

Investigation of Surface Treatments Undertaken Prior to Resin Composite Repair in Clinical Practice

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Abstract

In dentistry the repair of a failed resin composite (RC) restoration is a recognized alternative to its replacement as it is less invasive and destructive to tooth-structure. There is minimal evidence available in support of a definitive repair protocol, limitation in understanding the mechanisms behind effectiveness of repair protocols and as such there is no clear guidance for clinical practice.

Therefore a questionnaire-based study was undertaken to explore how dentists manage fractured RC restorations in primary care dental practice in the UK. This information, along with the relevant literature surrounding the repair protocols was investigated. This then, informed the design of *in vitro* investigations to better understand potential repair protocols for RC as described by the outcome measures of shear bond strength (SBS), roughness, surface morphology and water sorption.

The questionnaire-based study suggested that 70% of dentists would repair RC restorations and they used a varied combination of pretreatments in the process of making a repair. The most prevalent repair methods were surface modification by the use of a bur, application of phosphoric acid or a bonding agent. The *in vitro* investigations undertaken as part of the study demonstrated differences in SBS level between repair protocols. The application of a bur followed by acid etch and a bonding agent led to the highest SBS. Differences in roughness and surface morphology appeared to affect SBS level. In contrast, water sorption and aging showed minimal impact on SBS level.

Conclusion: Dentists are currently repairing fractured RCs using different protocols. The *in vitro* investigations revealed surface treatments have different contribution to the SBS level and these differences in SBS may be explained by the accompanied change in roughness and surface morphology of RCs. The highest SBS level was achieved by the sequential use of bur, phosphoric acid and bonding agent.

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Dedication

I dedicate this thesis to:

My country

My Sponsor (Kurdistan Regional Government)

My Family

Author`s Declaration

I declare the work presented in this thesis is original, has been carried out by the author and has never been presented in part or in full to any university in support for any application for any degree.

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List of abbreviations

BAC	Bearing area curve
BisEMA	Bisphenol-A hexaethoxylated dimethacrylate
BisGMA	Bisphenol-A diglycidyl ether dimethacrylate
CP/BA	Composite/priming bonding agent
CQ	Camphorquinone
D3MA	1, 10-decanediol dimethacrylate
DA	Dimer acid monomers.
DC	Degree of conversion
DDCDMA	Dimer dicarbamate dimethacrylate
DW	Distilled water
IQR	Inter quartile range
mg	Milligram
mm	Millimeter
Mr ₁	Material ratio of peaks
Mr ₂	Material ratio of valleys
μm	Micrometer
Ν	Number
Ra	Roughness average
RBS	Repair bond strength
RC	Resin composite

Rk	Core roughness
Rpk	Reduced peak height
Rvk	Reduced valley height
SBS	Shear bond strength
SD	Standard deviation
SEM	Scanning Electron Microscopy
SiC	Silicon carbide paper
TBS	Tensile bond strength
TEGDMA	Triethylene glycol-dimethacrylate.
UDMA	Urethane dimethacrylate
WS (%)	Water sorption percent

Chapter 1. Introduction

The developments achieved in RC technology have enhanced both the mechanical and physical properties of these dental materials. This in turn has led to them being widely used to restore both anterior and posterior teeth. However, in clinical service they are still susceptible to failure, particularly in the form of fracture and secondary caries. Repairing of these restorations has been identified to be one of the treatment options to manage failed RC restorations which possess a number of advantages over replacement. For example, this approach showed to increase longevity of repaired restorations, and to be cost-effective and time-saving (Fernández *et al.*, 2015; Casagrande *et al.*, 2017).

RC restoration repair involves a number of systematic treatments applied to the substrate materials, for example the application of adhesive promoters. However, at present there is lack of clear evidence in support of an effective repair protocol to guide dentists in their clinical practice. Furthermore, it is not clear whether all dentists do repair restorations in clinical practice and what repair protocols they use when undertaking a repair. Having knowledge on the treatment protocols is proposed to allow an insight in to the type of treatment that is delivered to patients in every day clinical practice. Therefore, a questionnaire study aimed to investigate how primary care dentistry in the UK used the practice of RC restoration repair and then to investigate different elements of the repair protocols used by general dentists for their individual and combined contribution to final RBS. This might then allow determination of the required steps for a repair protocol that could optimise RBS.

In vitro studies were designed to use both quantitative and qualitative measurements to better explain the findings around RBS and surface characteristics. *In vitro* measurement of RBS was considered to be fundamental for making comparisons between the protocols in these investigations, for that purpose SBS was evaluated between surface treatments and RCs. Bearing area curves, along with Ra measurements were used to further describe the microundercuts that the repair protocols created on the RC surfaces. Surface morphology was also investigated through the use of Scanning Electron Microscopy to provide further description of

surface morphology of the RC samples before and after some of the surface treatments.

A further challenge that RCs face in the oral environment is the potential effects of being immersed in oral fluids. Investigators have shown that fluids can negatively affect the RBS between RCs. Therefore, SBS of repaired RC samples were evaluated over different immersion time periods. Furthermore, the water sorption properties of RCs were investigated in an effort to understand saturation time of all RCs. It is believed after saturation RCs highly likely start to dissolve in solvents such as DW.

Chapter 2. Aims and objectives

Aims

To increase understanding of the repair of different RCs in relation to:

- Better understand how dentists repair fractured RC restorations.
- Better understand the different methods of repairing RCs *in vitro* and how they impact on the success of a repair.

Objectives

To achieve these aims, the following objectives were set:

- To explore whether or how dentists prepare surfaces prior to the repair of fractured RC restorations
- To develop an *in vitro* method to evaluate the impact of different repair methods upon RBS level.
- To investigate the impact of simulated aging upon the RBS level in vitro

Hypotheses

- The method of repairing RC restorations has impact upon the level of bond strength achieved *in vitro*.
- Simulated aging has impact upon the level of bond strength in vitro.



Figure 2.1 A flow diagram outlining the three phases of work plan in this thesis.

Chapter 3. Literature review

3.1 RC restorative materials

RC materials can be described as a solid that contains two or more distinct essential materials or phases. In dentistry, the term RC refers to a restorative material and commonly denotes a reinforced polymer system used to restore tooth tissues such as enamel and dentine (Sakaguchi and Powers, 2012b). The main contents of dental RCs are resin matrix, fillers, filler/matrix interface and initiators and activators (McCabe and Walls, 2008).

3.2 A summary from the historical perspective

RCs are a key group of restorative materials that are widely in use in dentistry today. There has been ongoing improvements of the constituent fillers, bonding and curing technologies within these materials. This development has given current RCs the properties which can restore physical function at the same time as mimicking the aesthetic appearance of human biological tissues, specifically teeth. Prior to the introduction of RCs, several other materials were in use to restore damaged teeth, such as amalgam, glass-ionomer cement, silicates, and acrylic resins. However, none of these alternatives are able to restore both the physical function and the appearance of teeth to the same extent that RCs are currently able (Peutzfeldt, 1997; Randolph *et al.*, 2018).

The development of RC restorations began with Bowen's discovery of Bis-GMA in the early 1960s (Bayne, 2005). This monomer proved to be durable owing to its high molecular weight, formation of a cross-linked matrix, and ability to link to fillers via a coupling agent (Vasudeva, 2009). This technology is still in use today within some of the current RCs, but there have been many further improvements and developments since its introduction.

Van Noort and Barbour, in their textbook, outline a number of improvements that have occurred in the area of filler components. For example, filler particles have

become smaller, mainly in the interest of improving aesthetics and polishability. Glass and barium inorganic fillers have been developed to transmit radiopacity, compensate for curing shrinkage, enhance manipulation and handling, and promote mechanical properties. In the 1970s, macrofill RC was introduced which possessed good mechanical properties, improved water sorption, and better thermal expansion than unfilled acrylic. The main disadvantages of these materials were staining and excessive wear. Further improvement in RCs to reduce the disadvantages of macrofill materials has been achieved, with enhanced surface smoothness and better physical and mechanical properties. Small filler particle size RCs have also been developed in which the filler size ranges from 0.5 to 3 microns. For example, microfilled and hybrid RCs contain a different range of filler sizes, but are smaller than macrofill materials (Van Noort and Barbour, 2013).

A number of methods have been used to cure or set RC materials. Initially, RCs were cured by a chemical reaction alone which required mixing two phases of a material as the base and activator. The main disadvantage of this was air entrapment during mixing, which led to weakening and staining. Another disadvantage of a chemical cure system is uncontrolled working time during placement, because the setting reaction depends on the amount of incorporated activator during mixing. Further, advancements in curing units yielded the production of light curing systems; the main advantages of a light curing system are that the RC materials are supplied in a single tube with no need for mixing. This consequently improved the mechanical properties with a decrease in staining, as well as improved working time of materials, as after placement the dentists activates the polymerisation reaction by the application of the curing light (Marghalani, 2016).

The key landmarks in the development of RCs described by (Bayne, 2005) are outlined in Figure 3.1. It is worthy of note that the terminology around midifill, midihybrid and mini-hybrid is not in contemporary use. The following sections will consider RC composition in greater detail.



Figure 3.1. A timeline to show the historical development of dental RC materials with respect to the content of monomer, filler, bonding and curing technologies. Macrofill, microfill, midifill, midi-hybrid and mini-hybrid are RC category; where midi-hybrid has larger filler size range than mini-hybrid composites. 3c, 2c, 1c are three, two, one components (or steps) of bonding agent; UV is ultra violet. VLC is visible light curing, QTH is quartz tungstn halogene. PAC is plasma arc curing, LED is light emitted diode. Modified from (Bayne, 2005), figure 5, page 278.

3.2.1 Resin matrix

Resin matrix is the organic part of RC materials and is made of various polymerised monomers. The first type of monomer used in direct filling materials resin was methyl methacrylate. It suffered from significant problems in use such as polymerisation shrinkage, discolouration, and a coefficient of thermal expansion that differed from teeth significantly, which led to gap formation and consequently a high incidence of secondary caries and pulp damage. These limitations motivated the development of epoxy resin. However, slow hardening made these materials unsuitable as well. Further modification of epoxy resin introduced the use of dimethacrylate monomers (Peutzfeldt, 1997; Vasudeva, 2009), which nowadays are widely in use in commercial RCs, albeit of a different type and structure.

The structure of the resin matrix is dependent on the way the monomers combine. When a resin matrix sets, the monomers bond together to form polymer chains, and then the chains combine to form either linear or cross-linking polymers. Cross-linking polymers are denser in structure than linear polymers (McCabe and Walls, 2008). In most contemporary RCs, the organic resin matrix is formed as a combination of a cross-linking polymer. These are used because they can enhance RC properties, for example Bis-GMA, as a cross-linking polymer has better mechanical properties and is resistant to water solubility compared to TEGDMA linear polymers (Cramer *et al.*, 2011).

The following paragraphs focus on some of the monomer systems with chemical structures illustrated in Figure 3.2.



Figure 3.2 Chemical structure of some monomer systems. Scanned from (Sakaguchi and Powers, 2012b pp 164.; Anusavice *et al.*, 2013, pp 302, fig 13-24.)

Literature review

The resin matrix of a large percentage of commercially available RCs is based on Bis-GMA, a type of dimethacrylate monomer. Bis-GMA has a comparatively high molecular weight (512 g/mol) and a stiff, partially aromatic molecular structure. This makes it an excellent dimethacrylate because strength and polymerisation shrinkage are significantly better than the majority of epoxy resins (Moszner *et al.*, 2008a). However, it has a number of undesirable properties, such as high viscosity (600– 1000 Pas at 23 °C) and increased stickiness, compromising the ease of handling of the resulting RC materials during placement. High viscosity limits the incorporation of high filler loading, and to manage the high viscosity, the Bis-GMA is mixed with a monomer such as triethylene glycol dimethacrylate (TEGDMA), which has a lower molecular weight (286 g/mol) and viscosity (0.05 Pas at 23 °C) (Ferracane, 1995; Vasudeva, 2009), allowing higher filler loading which can consequently enhance the mechanical properties (Kim *et al.*, 2002; Hu *et al.*, 2003; Moraes *et al.*, 2009).

A further undesirable property of Bis-GMA is its low degree of carbon double-bond conversion at room temperature, which results in a higher amount of residual unreacted monomer that can leak into oral fluids. The consequence of this phenomenon may be the replacement of the spaces with water, which reacts with the polymer chains, causing chain scission from the main polymer chain (Cramer *et al.*, 2011); in turn, this may decrease the strength of the polymer structure.

TEGDMA is an alternative monomer, and has increased carbon double-bond conversion to polymer because it possesses more functional groups than Bis-GMA (Chen, 2010). The main disadvantage of low molecular weight and functionality in TEGDMA is higher polymerisation shrinkage compared to Bis-GMA (Vasudeva, 2009).

Other monomers have been described in the literature which are thought to demonstrate enhanced properties compared with Bis-GMA. An example is the partially aromatic urethane dimethacrylates (Moszner *et al.*, 1999; Cramer *et al.*, 2011). The most widely used dental monomer of this type is 1,6-bis-(2-methacryloyloxyethoxycarbonylamino)-2,2,4-trimethylhexane (UDMA) with a molecular weight of (470 g/mol) and viscosity (8–10 Pas at 23 °C). It has significantly lower viscosity than Bis-GMA, and this allows the incorporation of fillers to be higher

than Bis-GMA. When volumetric shrinkage is compared, Bis-GMA shrinks 6.1% compared to a 6.5% volume change for UDMA. This is considered by investigators to be a small difference in volumetric shrinkage (Moszner *et al.*, 2008a). UDMA has been used in resin matrix with Bis-GMA or as a substitute for it (Khatri *et al.*, 2003; Floyd and Dickens, 2006).

A number of improvements in UDMA have led to improvement in resin matrix properties. For example, when UDMA contains both an aromatic group (hard segment) and an aliphatic group (soft segment) in the same molecule, the resultant polymer is tougher than when they contain only one individually (Moszner *et al.*, 2008a). Further improvement in the structure of UDMA included an altered urethane dimethacrylate resin, created to reduce shrinkage because it possesses a high molecular weight compared with Bis-GMA and original UDMA (895 g/mol vs. 512 g/mol vs. 470 g/mol, respectively), such as DX511 monomer, which is present in Kalore[®] RC (Ferracane, 2011).

An ethoxylated bisphenol-A-dimethacrylate (Bis-EMA) is a dimethacrylate monomer. The higher molecular weights of Bis-EMA at 540 g/mol and fewer double bonds per unit of weight have resulted in less shrinkage compared to TEGDMA. In addition, it has lower viscosity than Bis-GMA, allowing for incorporation of high filler loading into the resin matrix. Therefore, to reduce shrinkage in RCs materials, TEGDMA has been substituted with UDMA and Bis-EMA, and has lowered the negative effect of ageing and environmental factors, such as changes in RC properties due to moisture, and acid attack (Yap *et al.*, 2000).

Further developments in the resin matrix beyond Bis-GMA, TEGDMA, UDMA and Bis-EMA dimethacrylates as the main constituent in a RC have been made; however, many still include dimethacrylate monomers as the copolymer. Two examples of this are Kalore[®] and N'Durance[®], which are described below.

Anusavice and colleagues described a RC that contains an innovative monomer called Kalore, also identified as "DX-511". Kalore contains urethane, a monomer with a high molecular weight and a long, rigid central unit with flexible methacrylate end groups. The central unit helps to reduce polymerisation shrinkage, while the flexible end groups promote a higher degree of monomer conversion to polymer (Anusavice *et al.*, 2013).

The innovative monomer contained within N'Durance is Dimer Dicarbamate Dimethacrylate (DDCDMA), which has a high molecular weight and contains a bulky central group. This group phase separates dicarbamate between side groups, composed of Ethoxylate Dimethacrylate (EBPADMA) and Urethane Dimethacrylate (UDMA) monomers. The central chain separation is very long (molecular weight 843), which allows a higher degree of conversion and reduced polymerisation stress before the gel formation stage of matrix because the rigid central group does not shrink; this reduces polymerisation shrinkage and stress (Anusavice *et al.*, 2013). A further advantage of this resin matrix is that both the central group and side dimethacrylate groups are hydrophobic, which restricts water sorption and solubility.

Variation in ratio and types of monomers in resin matrix significantly affect RC properties, such as the degree of conversion, water sorption and polymerisation shrinkage (Goņalves *et al.*, 2009; Gajewski *et al.*, 2012; Cornelio *et al.*, 2014; Gonçalves *et al.*, 2015). A great variety in RC products (Ferracane, 2011) is possibly one reason for diversity in co-polymers between resin matrices, and this has likely resulted in the need for further investigation into the newly developed RCs in comparison to those that stand superior in properties.

3.2.2 Fillers

Fillers are inorganic particles added to the resin matrix of RC materials and act as a reinforcing phase to enhance their mechanical/physical properties. The composition of the most widely used fillers used in RCs are, glass and silica (quartz). Silica is radiolucent but adding radiopaque alkaline oxides, such as strontium oxide, barium oxide, or ytterbium triflouride, can overcome this problem (Ferracane, 1995; McCabe and Walls, 2008). Historically, changes to fillers have been led by how RC has been classified; Figure 3.3 shows developments in filler types and how filler size has changed over time.



Figure 3.3. Chronological development and changes in size of fillers in RC materials. Scanned and adapted from Curtis *et al*, 2008, pp. 187, Fig 1.

Quartz was the first type of filler used by Bowen in a macrofilled RC (Rueggeberg, 2002; Vasudeva, 2009). Availability and excellent optical match to the polymer resin were the main advantages of using quartz. These fillers were produced by milling/grinding large quartz blocks. The original quartz filler in macrofilled RC had a large particle size $(1-100 \ \mu m)$. These characteristics are on one hand advantageous because they enhance the strength of RC material to a great extent. On the other hand, they have a number of drawbacks. First, the hardness of quartz fillers made materials that were abrasive to tooth tissue. Second, the polymerised restoration's surface looked considerably rougher compared to the enamel it was intended to replace/restore. This was due to chemical and mechanical factors that would abrade the soft resin matrix earlier than quartz fillers, leave the fillers raised and exposed from the surface or, after dislodgement, leave large craters. Third, these RCs were more susceptible to debris collection and then discolouration. Fourth, the original quartz filler was difficult to detect on radiographs because it is radiolucent. Fifth,

because of excessive wear, there was a risk of the large filler becoming dislodged from the resin matrix during mastication and inhaled, causing lung pollution and making this class of RC hazardous to health (Ferracane, 1995; Combe and Burke, 2000). The development of RCs has seen considerable and ongoing changes in filler size as seen in Figure 3.3 from that of the original macrofill RCs (Ferracane, 2011; Ilie and Hickel, 2011).

One development in RCs was the introduction of microfilled RCs in the 1970s. This type of RC contained microfillers with an average particle size of 0.04µm. The fillers were composed of silicon dioxide or pyrogenic silica, and the particles are created from silicon after firing and sintering in an oxygenated atmosphere. The surface of microfill silica is highly energetic and can bond with resin matrix to form a thick paste. Silica is also softer than quartz. The resulting composition and size of silica microfill means that these RCs retain smooth surfaces better and have excellent aesthetics, as during polishing and mastication the fillers are torn away to the same extent as the resin matrix. However, like quartz these fillers are not radiopaque (Ferracane, 1995). Although these materials are more aesthetically pleasing, low filler loading resulted in the microfilled composites having poorer mechanical properties, and in particular they would fracture in areas of stress concentration, and had a higher coefficient of thermal expansion than macrofilled RCs (Ferracane, 1995; Ferracane, 2011). The limited filler loading capacity of microfiller particles was due to the fact that they have a tremendously high surface area. For this reason, there have been attempts to increase filler loading capacity in microfill by adding prepolymerised fillers (Marghalani, 2016).

Prepolymerised fillers are organic and inorganic fillers which were developed in the early 1980s by grinding cured RCs into particles that contain nano and/or micronsizes glass fillers. The size of these prepolymerised fillers is between 1–200 µm (Ferracane, 2011). This filler was introduced with the aim of minimising RC polymerisation shrinkage upon curing at the same time as keeping the advantageous properties of the enhancement of filler loading. First, it is incorporated into microfilled RC. Adding this type of filler to microfilled RC enhanced strength more than traditional microfilled RCs because prepolymerised fillers allow higher filler loading (Habib *et al.*, 2015). Some studies have demonstrated that microfilled RC containing prepolymerised fillers showed a reduction in stain (Imamura *et al.*, 2008), lower

polymerisation shrinkage (Yukitani *et al.*, 1997), increased fracture toughness (Kim *et al.*, 2000) and enhancement of mechanical properties (Suzuki *et al.*, 2005). In contrast, other investigators showed inferior properties compared to hybrid RCs, particularly in yield strength (Blackham *et al.*, 2009). The main disadvantage of prepolymerised fillers was their susceptibility to wear in the load bearing area if not surface treated, as they struggle to bond to resin matrix via the covalent bonds on their surface (Ferracane, 1995). In general, these fillers are regarded to be a useful means of reinforcing RCs, but there is limited understanding of the effect of these fillers on the properties of RCs (Waknine, 1985; Okada and Omura, 1993; Angeletakis *et al.*, 2005).

In the early 1980s, hybrid composites were introduced which contained macrofillers $(1-50 \ \mu\text{m})$ and microfillers $(0.04 \ \mu\text{m})$. They contained quartz, strontium and barium silicate, and lithium and aluminium silicate with amorphous silica microfillers. A further development in filler size was a reduction in hybrid filler sizes to midifill hybrids $(1-5 \ \mu\text{m})$ and minifill hybrids $(0.6-1 \ \mu\text{m})$ (Ferracane, 1995). A further change in hybrid RCs in the 1980s was the replacement of quartz filler with Ba-glass and ytterbium/yttrium trifluoride fillers, because of the disadvantages of quartz fillers as described earlier in this section (Itota *et al.*, 2004). Hybrid RCs were thought to combine the advantages of small and large fillers such as filler loading and different sizes. These two phenomena were shown to enhance physical and mechanical properties at the same time, such as strength and aesthetics, and so such hybrid composites were thought to be suitable for both anterior and posterior teeth (Ferracane, 2011).

A further reduction in the filler size of hybrid RCs was introduced in the early 1990s. The mean filler size for glass zirconia fillers is $0.4-1 \mu m$ and $0.04 \mu m$ for amorphous silica fillers. These RCs have been suggested to be used on both anterior and posterior teeth as their mechanical properties, wear resistance, and polishability have been enhanced. This type of hybrid RC is known as a microhybrid (Marghalani, 2016).

A more recent development in RC fillers is due to the introduction of nanofillers in the 2000s. These are composed of silica or zirconia, either in the form of discrete/ nonagglomerated particles (5–20 nm) or fused agglomerated nanoclusters (0.6–10

 μ m). The size range of these fillers was smaller than microfillers because of the solgel technology used in their creation. Nanoclusters were developed to obtain the best properties of the large and small filler sizes, for example large fillers allow high filler loading, while small filler particles produce superior wear resistance (Pontes *et al.*, 2013). The use of nanoclusters has allowed high filler loading to exceed 80 wt% and has enhanced the strength and aesthetics of the resultant RCs.

Filtek Supreme Universal Restoration was the first RC that contained nanofill and nanoclusters and was introduced in 2003. The manufacturer suggested this material had enhanced aesthetic properties similar to microfilled RC, and maintained physical and mechanical properties comparable to hybrid RC (Mitra *et al.*, 2003); as such, it promoted its use for anterior and posterior restorations. Curtis and colleagues assessed the mechanical properties of nanofill RC in comparison to microhybrid, microfill and nanohybrid RCs. Their results showed that nanofill RC had superior properties in fracture toughness and strength than the other RCs, and they suggested this is because of the presence of nanoclusters containing spherical particles which associated with reduced stress concentration compared with the other RCs contained irregular filler particles in shape (Curtis *et al.*, 2009b; Curtis *et al.*, 2009a).

Different proportions of nanofillers, nanoclusters and prepolymerised fillers have been incorporated into hybrid RCs, known as a nanohybrids. Studies have shown that nanofill and nanohybrid materials have similar mechanical properties, wear resistance, and reduced polymerisation shrinkage, and may improve resistance to fracture, retaining anatomical contour and decrease in water sorption, solubility and microleakage (Ilie and Hickel, 2009a; Sideridou *et al.*, 2011; Jun *et al.*, 2013; Pontes *et al.*, 2013; Takahashi *et al.*, 2013; Jin *et al.*, 2014).

Advancements in filler technologies have now led to a great variety of filler size, shape and concentration. RCs contain different compositions and combinations of fillers, in different loading ratios, from 60% to 90% by weight (McCabe and Walls, 2008). This has improved the strength and modulus of elasticity of the subsequent restorations, which has also reduced polymerisation shrinkage, improved the coefficient of thermal expansion, and reduced water absorption (Ferracane, 2011).

In the reviewed literature, a number of studies have investigated the effect of filler characteristics on RC properties. Studies have shown that in order to increase the mechanical properties of RC restorative materials, fillers must be optimally loaded and dispersed in the resin matrix (Kim *et al.*, 2002; Hu *et al.*, 2003). Inorganic filler particles act as a reinforcing phase, increasing the elastic modulus of the RC, providing resistance to wear, and improving fracture toughness; also, prior to curing, it influences handling properties for shaping and sculpting by the practitioner. When a number of commercial RCs were investigated for the effect of filler loading on properties of RCs, the results showed RCs with higher inorganic filler contents (>75 wt %) were associated with the highest mechanical properties, such as flexural strength and modulus of elasticity, with lowest solvent sorption (Randolph *et al.*, 2016a).

As described earlier, small filer size enhances the polishability of RC materials, and filler size may also have an impact on the roughness of the RC surface. Marghalani and co-workers showed that roughness parameters such as Ra increased as the mean filler size increased, yielding a linear relation (Marghalani, 2010). When nanofill and microhybrid RCs were assessed, nanofilled RC was found to be less rough than microhybrid RC (Oliveira *et al.*, 2012).

3.2.3 Filler/matrix interface

For fillers to be able to reinforce resin matrix, they need to be linked to it through a sufficiently strong bond. The interaction between fillers and resin matrix influences the mechanical properties of RCs because the stress distribution between the two phases is a crucial aspect in the strength of RC and durability of the material and subsequent restorations (Matinlinna *et al.*, 2018). There is a large difference in the elastic modulus between the fillers (>10MPa) and resin matrix (1–3MPa). Therefore, when stress is applied, the resin matrix deforms earlier than the filler, potentially leading to the formation of a gap between the two phases (Miletic, 2018). The lack of a bond and good wetting between fillers and resin matrix is thought to accelerate void formation and then lead to early RC failure (Santerre et al., 2001). Therefore, fillers are frequently chemically bonded to the adjacent resin matrix to promote a stable interaction between the two phases (Ferracane, 2011).

To achieve this bond, fillers are normally silane treated with a 0.025%-2% aqueous solution. A silane coupling agent is a difunctional molecule which contains two different functional end groups, alkoxy and C=C. The alkoxy group bonds to the inorganic fillers, in which the reaction is activated by acid, and the C=C bonds to the resin matrix monomer C=C, in which the reaction is brought about by the activation of free radicals, which is initiated by photo-activation in the resin matrix (Santerre *et al.*, 2001; Matinlinna *et al.*, 2018).

The main advantage of silica and other fillers surface treated with silane agents is that it has been shown to enhance the strength of RCs (Marovic *et al.*, 2014; Lung *et al.*, 2016) by increasing their toughness compared to those that have had no surface treatment (Söderholm, 1984; Matinlinna *et al.*, 2007; Matinlinna *et al.*, 2018). In contrast, silane structures may increase water sorption and negatively impact on stability of the bond between filler and matrix, and negatively affect the durability of RC. To manage this, silane systems have been developed with a level of hydrophobicity. The evidence suggests that when two different kinds of silane structures have been investigated, water sorption of the RC, which used γ -MPS silane, had less water sorption than UDMS silane (Karabela and Sideridou, 2008). Nihei has shown that hydrophilicity of silane affects the durability of RCs; that is, the more hydrophobic silane is, the stronger the bond and the less water sorption. This is because hydrolysis of the functional group of silane bonds to inorganic fillers are destroyed by the reaction with water (Karabela and Sideridou, 2008; Nihei, 2016).

3.2.4 Setting of RC materials

RC in the form of a paste assists its application to teeth, but it then needs to become set to form a hard material in the mouth. The process of setting RC to a hard material is by a polymerisation reaction. Polymerisation is the chemical action of converting shorter molecule monomers into a long polymer chain. In response to polymerisation reaction, there is a dimensional alteration and chemical reduction called polymerisation shrinkage. This is caused by transforming interatomic positioning between molecules by which double bonds are altered to single covalent ones. This conversion of monomers to polymer results in combining monomers and lessening of the intermolecular space between monomer units. Consequently, this conversion results in a shrinkage of the polymerised resin matrix. The reaction can
also release energy and, with an accompanying thermal contraction, generate internal stresses (McCabe and Walls, 2008).

There are two key methods to form a polymer: addition, and condensation polymerisation. During the addition reaction, no by-product is formed as the chain grows. Addition polymerisation is alternatively called free radical polymerisation because a molecule is supplemented to the polymer chain as the reaction proceeds. The majority of the RCs used in dentistry polymerise by an addition reaction. Polymerisation has three phases. The first phase is activation and initiation, which requires external energy such as heat, chemicals, or light to form an extremely reactive single electron called free radical from initiators. A free radical attacks the functional or reactive group, resulting in the creation of a single bond with additional carbon and an unpaired electron. The monomer linked to a free radical becomes a new free radical. In the second propagation phase, the fresh free radical can bond with another double bond which, in turn, attacks an alternative double bond, and so on, and thus chain enlargement is formed. Chain propagation continues until the viscosity of the resin is so high that there would be no enough migration of monomers to form a polymer chain, leading to the termination phase (Van Noort and Barbour, 2013).

Polymerisation of RC materials has been achieved using a number of methods, including heat, chemicals, and light activation. These are explained further below.

Heat activated

Heat activation is initiated by benzoyl peroxide when the resin material is heated above 65°C to form benzoyl free radicals. This method is mostly used for polymerising of denture base materials and has little application in the direct restoration of teeth (Marghalani, 2016).

Chemically activate

These RCs are usually provided as two-paste or powder/liquid systems; one is comprised of an aromatic tertiary amine, such as n, n-dimethyl-p-toluidine, used as an activator as an alternative to heat application. The other is a benzoyl peroxide initiator, which responds to activators to yield a (benzoyl) free radical. The RCs polymerised by this system are less stiff as the degree of conversion of cold-cured resins is lower than that of heat-cured resins (McCabe and Walls, 2008).

Light activated

RC photopolymerisation can be initiated by a molecule or system. According to the reviewed literature, a number of photoinitiators have been described, the most widely used of which is the champhorquinone system, combined with a co-initiator, frequently a tertiary amine. The free radical polymerisation process initiates when these molecules absorb reciprocal photons. CQ absorbs above the 400–500 nm range, with peak of absorption at 470 nm (Neumann *et al.*, 2005; Price and Felix, 2009).

Several alternative photoinitiators have been produced as curing systems for RCs, either to develop better aesthetic qualities by decreasing CQ concentration and its yellowing effect, or to improve polymerisation efficiency. Examples of alternative photoinitiators include phenylpropanedione (PPD), mono- or bis-acylphosphine oxides (MAPO and BAPO, in that order), benzoyl germanium or benzyl (Neumann *et al.*, 2005; Neumann *et al.*, 2006; Ogunyinka *et al.*, 2007; Moszner *et al.*, 2008b; Arikawa *et al.*, 2009; Asmussen and Vallo, 2009; Leprince *et al.*, 2011).

Photoinitiators are different in colour: champhorquinone is yellow, while phenylbis (2, 4, 6-trimethylbenzoyl) phosphine (TPO) is white, and Ivocerin^M, a germanium-based photoinitiator, is in between these colours. Photoinotiator systems may affect the stability of the RC pigmentation, which is supposed to be related to photoinitiator structure; moreover, when the photoinotiator is composed of more cross-linked polymer, better colour stability can be achieved (Palin *et al.*, 2014; Randolph *et al.*, 2016b). For example, phosphine oxide-containing RC has been shown to have better colour stability experimentally than champhorquinone products (Albuquerque *et al.*, 2013; Manojlovic *et al.*, 2016).

An initiator's efficiency relies on several features, for example the absorption yield efficiency (ϵ , in L/mol.cm) (Neumann *et al.*, 2006), which defines how many photons need to be absorbed per initiator molecule at a given wavelength. When comparing CQ and TPO in relation to absorption, TPO has greater efficiency at lower concentrations (10x lower). The aromatic rings or chromophores of TPO are thought

to be related to greater absorption maxima. Furthermore, the TPO molecule, upon excitation, yields two active radicals which can enhance a higher degree of conversion in the resin matrix (Jockusch *et al.*, 1997), while a type II photoinitiator system, such as CQ/amine, yields one (Cook, 1992). For example, monoacylphosphine oxide (MAPO) is considered to have more efficiency than CQ. When MAPO was compared to CQ, the degree of conversion, strength and modulus of elasticity were significantly enhanced in RC containing MAPO (Palin *et al.*, 2014).

For the process of setting RCs with light, different curing units have been developed. The first light curing system was ultraviolet but was replaced by a visible light curing system because of the following disadvantages:

- Incremental build-ups were essential which were no greater than 1mm (Rueggeberg *et al.*, 2017);
- Demand to expose every increment for 20 to 60 seconds per increment led to longer treatment time and slow adoption into clinical practice (Rueggeberg *et al.*, 2017);
- Concerns were expressed about the destructive potential of short wavelength energy on unprotected human eyes, leading to cataract formation and corneal burns, as well as possible changes in the oral microflora of the patient (Mangat *et al.*, 2014);
- Lamps to produce these wavelengths were expensive (Rueggeberg *et al.*, 2017).

The alternative technology that produced visible light also has limited depth of cure but is overall less hazardous. The first visible light curing system introduced in 1970s was Quartz Tungsten Halogen. This light is blue and has a wavelength of 410–500 nm of visible light, and there was a need for light exposure of materials of between 20–60 seconds to cure RC. The devices based on this technology had a number of disadvantages:

• This light generated heat during operation, thus requiring fan cooling, which was noisy (Uhl *et al.*, 2003);

- The challenge with these lamps was that only 9% of the energy consumed produced light, with the other 91% produced heat. This led to a short life of the halogen bulb of nearly 30 to 50 hours, before they started to reduce in efficiency and needed replacement (Mangat *et al.*, 2014);
- The filter within these devices can gather dust, crack, or peel, and may require cleaning if contaminated. This can alter the wavelengths of light transmitted, allowing harmful UV rays to be emitted (Mangat *et al.*, 2014).

A Plasma Arc unit is another curing method characterised by high light intensity in 470 nm wavelengths. The recommended time to set RC materials was significantly lowered to three seconds, and then nine seconds was suggested as the efficient exposure time.

Light Emitting Diodes were also introduced, and the main drawback of earlier generation models was low light intensity, but in later generations this was improved by enhancing multiple emission areas or two or more diode frequencies to be able to activate different types of photoinitiators. For example, G-Light width was developed by the GC Company, and is characterised by a high intensity, narrow spectrum of light. This model can be used for all types of light curing RCs, is rechargeable, and can be used over 400 times in ten seconds with no reduction in light intensity. This property gives the device a long lifetime.

Laser technology is another method of curing RCs. The wavelength of an argon laser is between 450–500 nm. The size of the device's tip is small and this is advantageous for interproximal curing of RCs in the class two cavity; however, the heat generation, visual damage, weight and portability of these devices are the key disadvantages of this method of curing (Rueggeberg, 2011; Mangat *et al.*, 2014; Rueggeberg *et al.*, 2017).

3.2.5 Classification of RC materials

Dental composites have generally been classified according to their filler, resin matrix, activation method, handling characteristics and application, such as to anterior and posterior teeth. Also, other classes of RCs have been labelled by the manufacturer as low-shrinkage and bulk-fill (McCabe and Walls, 2008; Anusavice *et*

al., 2013; Van Noort and Barbour, 2013; Marghalani, 2016). The most widely used classification system is that based on filler size distribution. Several authors have broadly classified RCs into the following groups associated with the size of the filler particles (Willems *et al.*, 1992; Ferracane, 2011):

1. Macrofill (conventional) composites have a particle size of $1-50 \mu m$ and typically contain 60–80% filler by weight. The main advantage of these materials is that they are very strong, but they are aesthetically unacceptable because it is difficult to polish them and impossible to maintain surface smoothness;

2. Microfill composites have a particle size of 0.01–0.1µm with a common average diameter of 0.04 µm. Filler loading is 30–60% by weight. Small particle sizes produce a massive increase in surface area for a given volume of filler that needs more resin matrix for wetting. Accordingly, individual microfill fillers cannot be incorporated at a high level but pre-polymerised filler can be added to the matrix, to which additional filler is joined. This RC can retain a smooth surface over time but is weak due to the low level of filler (McCabe and Walls, 2008; Ferracane, 2011);

3. Hybrid composites are produced with the aim of improving strength, polishability, and aesthetics. For this reason, there has been a decrease in filler size to produce what is called a small particle hybrid or midifills, for which the mean particle size is larger than 1 μ m, in a mixture of conventional glass or quartz fillers, but also some submicron silica particles. Further grinding of the fillers can result in RC with particles of average diameter between 0.4–1 μ m; these are named minifills or microhybrids. Filler loadings are about 75% conventional size and 8% submicron size, so a total filler content of 83% or more may be attained (McCabe and Walls, 2008);

4. Nanocomposites have a filler particle size of between 0.1–100 nm, and the fillers are in the form of individual or nanoclusters (Beun *et al.*, 2007; Ferracane, 2011). There has also been incorporation of nanofills in the form of individual nanofill and agglomerates in hybrid composites (Ferracane, 2011). This type of hybrid RC is called nanohybrid, but some authors consider it to be a class of nanocomposites (Senawongse and Pongprueksa, 2007). This class of material has satisfactory strength and improved polishability and aesthetics that are not easily distinguished from microhybrids (Ferracane, 2011).

An interesting point to consider is that microfilled composites, as with an average filler size of 0.04 μ m, could also be seen as the first nanocomposites, as 0.04 μ m is also included within the range of the nanofiller size. Due to the absence of identification of the concept of "nano" at the time of their development, they were not identified as nanofilled (Ferracane, 2011).

Filler is considered a major factor affecting the physical and mechanical properties of RC (Venhoven *et al.*, 1996; Turssi *et al.*, 2005). Accordingly, RC classification based on filler size distribution seems to be widely used as the RC classification system. Most manufacturers use this type of classification system to describe their commercial products, for example microfilled or microhybrid. Researchers have also adopted this type of classification system to help describe the difference in performance of different categories of RC products.

3.2.6 RC bonding systems

RCs bond to tooth structure through a bonding system, of which there are different types, they have been classified based on the steps of application in the clinic or strategies of use or generations (Van Noort and Barbour, 2013; Miletic, 2018). This section focus on the strategies of bonding systems, the number of steps are being mentioned without explaining them. Bonding system strategies include: etch and rinse, self-etch, and universal adhesives. In the etch and rinse strategy, the primer and bond are applied to the etched surface, while, in the self-etch strategy the acid monomer replaces the phosphoric acid. Because acidic monomer is less effective on enamel than phosphoric acid within self-etch system, the third and newest strategy developed and includes etching enamel or enamel and dentine with phosphoric acid followed by the application of a self-etch bonding system. The steps of application for all strategies that are currently available with the possible steps.



Figure 3.4. Current adhesive system strategies with the possible steps of application. A, P, B, SE indicate acid, primer, bonding resin and self-etch respectively. Scanned from (Miletic, 2018), p. 200)

3.3 Effect of environmental factors on RC properties over time

RCs are vulnerable to degradation in the oral environment, for example being constantly subjected to forces from mastication, sometimes in the form of parafunctional habits, at very high levels. This can produce degradation over time and cause failures such as wear or bulk fracture. In addition, chemical substances from food, bacterial by-products, and salivary enzymes can lead to RC degradation such as softening, swelling, and surface roughness (Asmussen, 1984; Ferracane and Marker, 1992; Ferracane and Berge, 1995; de Gee *et al.*, 1996; Santos *et al.*, 2017; Xu *et al.*, 2017).

As a heterogeneous material, every component of RC, including the polymer matrix, filler and filler/matrix interface, can be involved in the process of degradation. Santerre *et al.* (2001) described the mechanisms of RC degradation to include material loss and uptake (extraction and sorption) and physical changes such as stress cracking, fatigue fracture, and wear (Santerre *et al.*, 2001). Also, Bamford and Grassie (as cited in Göpferich, 1996) identified factors that may affect polymer degradation, such as chemical fluids, mechanical loading, polymerisation shrinkage, and thermal degradation.

In a clinical situation, these types of degradation are likely to be interrelated and have the potential to build upon one another and accelerate the rate of degradation more than the effect of mechanical or chemical factors individually. For example, chemical degradation can increase the mechanical degradation rate by softening a resin matrix (Curtis *et al.*, 2008b). When knowledge of the magnitude effect of each factor in the degradation process is required, clinical data may be difficult to interpret because these multiple factors combine. Therefore, to understand the mechanism of action of each factor, *in vitro* studies can simulate clinical factors individually to help determine the magnitude effect of each degradation type upon the properties of RCs. Further explanation of the common types of degradation is given below, according to whether the impact is chemical, mechanical, or photopolymerisation reaction.

3.3.1 Chemical impacts

The main sources of chemical fluid interaction can be either from food and drinks or saliva (Delaviz *et al.*, 2014). These sources have been simulated with different chemical fluids during *in vitro* studies, for example distilled water or phosphate buffered saline or acidic fluids (Al-Mulla *et al.*, 1989; Finer and Santerre, 2004b). The chemistry of the fluid that surrounds the RC is one factor that influences the degree of interaction and then degradation (Göpferich, 1996; Santos *et al.*, 2017). Hydrolysis is one mechanism by which RC can degrade (Ferracane, 2006), and every element within RC can be affected by chemical impacts.

Chemical degradation in resin matrix

The chemical degradation of resin matrix can occur due to the solubility and erosion of the polymer matrix. Solubility is a measure of the degree of dissolution of a material in a fluid, while erosion is a measure of the dissolution of the surface of a material (McCabe and Walls, 2008). Basically, solvents enter polymer spaces by a process known as sorption, and this triggers the chemical degradation of the polymer chain due to chain scission. Following this, monomer and oligomer leave the polymer matrix. These reactions lead to changes in resin matrix properties, such as a decrease in strength, hardness, and wear resistance, as well as increasing roughness on the surface of the RC, which can affect appearance (Ferracane, 2006).

A number of factors are thought to play a role in polymer degradation, such as hydrophilicity of the polymer between the polymer and the solvent and cross-linking density, the porosity of the network, and the pH of solvents (Göpferich, 1996; Ferracane, 2006; Cramer *et al.*, 2011). Investigators have demonstrated that the condensation type bonds within the resin matrix can be susceptible to hydrolysis (Santerre *et al.*, 2001) such as esters, urethanes and amides in the presence of acids or bases or enzymes (Williams and Zhong, 1994). It therefore seems there will be differences in chemical degradation between different polymer types. Investigations have reported that TEGDMA absorbed more water than Bis-GMA, which in turn absorbed more water than UDMA. This variance was due to the presence of hydrophilic ether linkages, hydroxyl groups, and urethane linkages in TEGDMA, Bis-GMA, and UDMA, respectively. Ester groups are another reason for water uptake in all three types of polymer network, and researchers have established the lowest

water sorption for ethoxylated Bis-GMA (Bis-EMA) due to the lack of hydroxyl groups and urethane linkages of Bis-GMA or UDMA (Sideridou *et al.*, 2003; Finer and Santerre, 2004a; Curtis *et al.*, 2008b; Pan and Kopecek, 2008; Shah *et al.*, 2008; Kerby *et al.*, 2009; Gajewski *et al.*, 2012).

The features of the polymerised network also play an essential role in defining the degree to which water uptake and swelling occur when a polymer is placed in a solvent such as water, acid, acetone or alcohol (Kao, 1989). Water moves into the polymer network via porosities and intermolecular spaces, but the extent and rate of water uptake is reliant on the density of the polymer network. Arima and colleagues have suggested a strong association between high cross-linking polymers and a decrease in solubility of polyethylmethacrylate polymers. However, they found that when the cross-linker contained hydrophilic ether linkages, water sorption essentially increased with cross-linker concentration, indicating that the chemical nature of the cross-linking agent may overtake the effect of complex molecular density (Arima et al., 1996). A high degree of porosity or microvoids has also been shown to assist fluid passage into and out of the polymer by acting as locations for molecules to separate, leading to enhanced solvent uptake and elution (Krongauz et al., 2016). Other factors that may influence water sorption include: hydrogen bonding and polar interactions of the polymer chain; the degree of conversion of the polymer; and, the quantity of pendant molecules existing within the network (Ferracane, 2006).

Another condition within the mouth which is likely to affect RC is pH. The pH of oral fluids may range from pH 4–8.5, depicting an environment from the mildly acidic to mildly alkaline. Soft drinks and chalk-containing toothpastes broaden this range between pH 2–12. It is possible for RC to be stable at near neutral pH values but to erode rapidly at extremes of either acidity or alkalinity. This partially explains why certain materials perform adequately with some patients but not with others. One of these studies has shown that RC can withstand hydrolysis well in neutral and acid rather than alkaline environments (Cilli *et al.*, 2012). It is important to note the slightly lower pH mediums that have been chosen within *in vitro* studies to simulate degradation in clinical situations to RCs, and how these pH act slowly on polymer degradation (Prakki *et al.*, 2005). According to the reviewed literature, there is no direct study on the effect of low pH solvents such as phosphoric acid etching on the surface of a RC. However, a number of attempts can be found which illustrate the

effect of phosphoric acid etching on the surface characteristics and interfacial bond strength between RCs. They suggest its action is possibly limited by the cleaning of the resin debris layer. However, nano-scale measurements showed some degree of reduction in the resin matrix level (Fawzy *et al.*, 2008; Loomans *et al.*, 2011a; Celik *et al.*, 2014).

Chemical degradation of fillers

Chemical degradation can occur directly on the exposed filler surface of RCs through hydrolysis and etching with acids, or the reaction may take place through the diffusion of water through the resin matrix to subsurface fillers at filler-matrix interfaces. Fillers can leak ions into the surrounding storage media (Söderholm, 1981; Drummond, 2008). The leaked ions can combine with water due to their polar nature and this leads to a change in a small hydrogen ion in the filler network (Michalske and Freiman, 1982; Michalske and Freiman, 1983). Tarumi et al (1995) reported that each filler type has a different susceptibility to hydrolysis. Glass filler was found to be more susceptible to degradation in water than guartz because glass contains ions that can leak into water. Barium containing glass fillers in RCs have been shown to have more water absorption than quartz-filled RCs, and surface degradation through hydrolysis (Söderholm, 1981; Tarumi et al., 1995). Also, glass modifiers leak more silicon ions than quartz and have more susceptibility to degradation by water and solvents (Söderholm et al., 1996). Water has a pH that is regarded as a weak acid, and thus it has the potential to degrade some types of filler such as silica and zirconia (Milleding et al., 2003). The degradation of glass filler can be reduced by isolating it from the surrounding environment using silane. There is also evidence that etching with weak acid, such as with 37% phosphoric acid for short time (20 seconds) results in no dissolution of filler or silane, but a strong acid such as hydrofluoric acid for 20 seconds dissolves the silane layer and glass filler quicker than silica fillers (Loomans et al., 2011a). Therefore, filler type and acidic challenge concentration and time has an impact on the outcomes such as roughness and surface morphology.

Filler size can accelerate the chemical degradation of RCs. The surface smoothness of RCs has been shown to affect the chemical degradation of these materials. RCs with small fillers have the potential to be smoother than other RCs containing larger

sized fillers because the space between the fillers is less. De Gee and colleagues reported less degradation of microfilled composites but significant degradation on microhybrid RCs when subjected to acidic challenge (de Gee *et al.*, 1996). One possible explanation for such findings is that small-sized particle fillers are less susceptibility to fluid accumulation on the surface of microfilled RC, due to the outer surface roughness of RCs. Therefore, differences in filler size may be regarded as one of the factors leading to the dissimilar surface degradation of RC materials in relation to hydrolysis.

Chemical factors affecting filler/matrix interface

Silanes are the most widely used filler/matrix coupling agents and are susceptible to chemical degradation. Arikawa and colleagues demonstrated that increasing the number of silanated fillers was associated with increased deterioration in RC properties. Arikawa and co-workers examined high and low silanated filler in RCs. They demonstrated that the composite with higher silanated filler degraded more by acidic media than the RC with low silanated fillers (Arikawa et al., 1995). Cilli et al. tested different types of silane treated fillers for degradation under acidic, neutral and alkaline media. It was shown that degradation began with silanated fillers, and also that differences in silane structure contributed to differences in the extent of the degradation of the RCs. Unfilled resin was compared to silanate-filled RCs. The latter had more surface roughness due to degradation than non-filled resin, and this was considered to be due to the greater susceptibility of silanized fillers to chemical degradation than resin matrix (Cilli et al., 2012). Therefore, one development with silane has been to enhance its resistance to chemical challenges by decreasing hydrophilic monomers and increasing cross-linking to lower the susceptibility to fluid absorption (Cramer et al., 2011; Marghalani, 2016).

3.3.2 Mechanical impacts

Mechanical degradation of resin matrix

The mechanical degradation of resin matrix is best described by changes that occur under occlusal loading and masticatory function in relation to how they are used to restore teeth. Mechanical loading produces stress in the polymer matrix and this

may lead to rupture in one or a number of chains, which appear as cracks and microcracks (Shah et al., 2009b). With further loading, stress around the crack tip may concentrate and cause crack propagation, increasing the length of the crack, and fusion between cracks can then lead to failure or a defect in the restoration (Lohbauer *et al.*, 2013; Van Noort and Barbour, 2013). The failure in RCs due to mechanical loading over time often produces changes in the form of wear and fractures (Baran *et al.*, 2001).

The rigidity of the monomer systems in the resin matrix can have an influence on increasing resistance to crack initiation and propagation. EI-Safity and colleagues showed that a RC containing Bis-GMA was more resistant to fracture under mechanical loading than other RCs which contained more flexible monomer systems in resin matrix, such as TEGDMA, Bis-EMA and UDMA (EI-Safty *et al.*, 2012). Also, all the factors that contribute to resin matrix degradation may increase and hasten the mechanical degradation rate, for example aged resin matrix fracture resistance is lower than unaged resin matrix (Lohbauer *et al.*, 2013).

Mechanical degradation of fillers

In the literature, fillers have been shown to increase resistance to mechanical degradation and enhance strength in RCs, through having a higher percentage of inorganic fillers (Ferracane *et al.*, 1987; Pontes *et al.*, 2013). Also, the composition of fillers reacts differently to mechanical degradation. Regarding glass fillers, although studies have not shown that these break down under stress and loading (De Souza *et al.*, 2011), pre-polymerised fillers have been recorded via surface topography to fracture under mechanical loading (Kim *et al.*, 2000). However, fiber-reinforced RCs seem more resistant to crack growth than particulate RCs (Keulemans *et al.*, 2009; Badakar *et al.*, 2011). Thus, filler morphology is another likely factor in mechanical degradation. Indeed, filler size is likely to decrease mechanical degradation, as Souza *et al.* (2011) found when they investigated the crack growth rate of two RCs of different filler sizes but equal ratios of filler. In the same study, RC with fine particles seemed to resist crack growth better and enhance the fracture resistance (De Souza *et al.*, 2011). It therefore appears that filler size and morphology influence fracture resistance differently, but this needs further investigation as commercial RC

manufacturers are continually changing their products in terms of filler size and concentration.

Mechanical degradation at the filler/matrix interface

Interfacial bond strength between the fillers and the resin matrix is likely to play an important role in the susceptibility of RCs to degradation under mechanical loading. Investigators have shown that when the interface bond is stronger than the applied stress between matrix and filler, the stress can be transmitted from the resin matrix to the filler via the interface, without destroying the interfacial bond, and stops void formation between filler and the matrix (Wang et al., 2008; Talreja and Singh, 2012). In addition, the thickness of the interfacial layer is likely to influence resistance to rupture under loading. Wang and colleagues showed that, when the same amount of stress was applied to two types of RCs of different interfacial bond thickness, the thicker bond could withstand higher stress before debonding than the thinner interfacial bond (Wang et al., 2008). Further, a strong interfacial bond between filler and matrix may deflect crack propagation through the interface to the resin matrix, reducing stress on the RC via energy dissipation. In contrast, when a void forms at the interface between fillers and resin matrix, it behaves similarly to a crack. The consequence would then be stress concentration at the crack tip and propagation at the interface, which is also likely to accompany filler extrusion. Therefore, the efficacy of bonding across the interface is a key dependent factor in the fracture resistance mechanism of RCs (Kim and Mai, 1991; Lohbauer et al., 2013).

3.3.3 Polymerisation reaction impacts

The amount of converted monomer to polymer, or the degree of conversion (DC) and the amount of unreacted monomers, are two factors likely to have an impact on the properties of RCs (Miletic, 2018). One of the most important aspects that may affect RC polymerisation is the effectiveness of the activating system of the polymerisation reaction, such as a heat or light cure. This is because it has the potential to affect the DC, of polymers in RCs (McCabe and Walls, 2008), and consequently the DC influences the mechanical and physical properties of RC. For example, Lohbauer *et al.* demonstrated the effect of three light curing devices on the DC and mechanical properties of RCs. The results suggested that the higher the DC, the better the

mechanical properties (Lohbauer *et al.* 2005). In addition, other investigators have found that the DC affects RC properties, such as water sorption, resistance to wear, and flexural strength (Ferracane *et al.*, 1998; Gajewski *et al.*, 2012; Mangat *et al.*, 2014).

3.4 RC restoration clinical failures

Contemporary RC materials are regarded to have adequate properties that allow them to be predictably used in anterior and posterior teeth (Ferracane, 2011). However, RC restorations are subject to different challenges, such as patient (heavy occlusal loading) and dentist (light exposure time) factors. In addition, the oral environment is harsh towards restorative materials, potentially leading to their degradation. These factors, individually or in combination, might degrade RC restorations and result in weakening of the structural integrity of these restorative materials and consequent of early failure. The range of failure of RC restorations, however, has been suggested to be within the recommendation of the ADA (American Dental Association), at no more than 2.5% annually. A number of studies with different observational periods ranging from 6–22 years have estimated annual failure rate at between 1.5%–2.2%, respectively (Gaengler *et al.*, 2001; Qvist, 2003; da Rosa Rodolpho *et al.*, 2006; Da Rosa Rodolpho *et al.*, 2011), which is below the range of failure suggested by ADA. Therefore, current RC could be regarded as a suitable restorative material.

The failure modes of RCs, particularly in relation to load bearing area, were found by Ferracane (2013) to be multifactorial, although the most prominent identified reasons for failure were caries, wear and fracture. Secondary caries is related to bacterial infection that occurs adjacent to a RC restoration, while fracture and wear are only regarded to be of concern in cases of a heavy occlusal pattern and/or large cavities (Ferracane, 2013). Accordingly, there is a preference for repairing remaining parts of failed RC restorations if they are still in good intact with the tooth structure (Tyas *et al.*, 2000; White and Eakle, 2000; Moncada *et al.*, 2008; Featherstone and Doméjean, 2012; Lynch *et al.*, 2013).

Clinical studies have not shown important differences in performance between RC materials when investigated for annual failure rates and the more apparent risk factors for clinical failure. For up to five years, hybrid RCs showed lower failure rates

than nanofill RCs, but the two materials did not show major clinical differences in performance for up to five years (Palaniappan et al., 2009; Sadeghi et al., 2010; Qin et al., 2013). Nanohybrid and microhybrid RCs showed differences in clinical performance for direct class two restorations over two years, where microhybrid RC showed statistically significantly lower annual failure rates compared with nanohybrid RC (Laske et al., 2019). Laske and colleagues argued that the time of the study was too short to be able to make a strong conclusion about the performance of microhyrid and nanohybrid RCs; over ten years of investigation nanohybrid and microhybrid RCs showed statistically non-significant difference in annual failure rates for class two restorations (van Dijken and Pallesen, 2014). For microfill RCs, a study showed that their performance was similar to fine and coarse filled hybrid RCs for class one and class two posterior restorations over eight years (Collins et al., 1998), but in patients with tooth wear microfill RC showed worse clinical performance than those microfill RCs in patients with no evidence of tooth wear (Bartlett and Sundaram, 2006). Indeed, in a more recent study, tooth wear was indicated as one of the factors of RC restoration failure that related to patients because tooth wear is indicative of destructive mouth habits of these patients (Demarco et al., 2012).

3.5 RC restoration repair

RC materials are advocated as materials of choice for both anterior and posterior restorations owing to the development of their physical properties and ability to match tooth colour (Lynch *et al.*, 2014). However, they are susceptible to localised defects under clinical function, most of which develop gradually, with the exception of fracture. Repair has been considered a better treatment decision (Casagrande *et al.*, 2017) than replacement, which is suggested as the last treatment of choice (Blum and Özcan, 2018). While repair may be defined as a conservative intervention in order to maintain dental health (Ericson, 2007), the replacement of RC restorations requires the removal of the fractured restoration. The most common rationale for repairing restorations is minimising unnecessary treatment to the sound part of the restoration and avoiding potential damage to sound tooth structure, even though there is significant potential for iatrogenic damage to the tooth during preparation (Lynch *et al.*, 2012; Brunton *et al.*, 2017). Other potential advantages of repairing restorations include the reduced time and effort of operation, the lower cost of

treatment for the patient, and the decrease in the use of local anaesthesia. These advantages are especially important for patients with complex medical histories, as suggested by Blum and co-workers (Blum *et al.*, 2012b).

Within new RC restorations, there is usually an amount of unreacted monomer that has the potential to react chemically with the monomers of the newly applied RC, providing a strong bond between them. This becomes a challenge when unreacted monomers are unavailable within a previously polymerised or old restoration, which may not provide the viable bond strength associated with this mechanism (Göpferich, 1996; Santerre *et al.*, 2001).

The most important consideration for a repaired RC is thought to be the interface between old and new RC materials (Eliasson *et al.*, 2014). Various *in vitro* study methods have been proposed to ensure adequate bond strength is generated between the original composite and the newly applied material (Turner, 1993; Brosh *et al.*, 1997; Denehy *et al.*, 1998; Shahdad and Kennedy, 1998; Lucena-Martín *et al.*, 2001; Frankenberger *et al.*, 2003; Oztas *et al.*, 2003; Tezvergil *et al.*, 2003; Bonstein *et al.*, 2005). There have been a number of different types of surface treatment investigated, and they are summarised as follows:

- Rotary cutting instruments carbide and diamond burs
- Rotary polishing instruments polishing burs and discs
- Microabrasion sandblasting and CoJet (silaination) systems
- Acid etching phosphoric or hydrofluoric acid
- Adhesion promotors dentine bonding system and/or silane.

However, there is still limited understanding and identification of an effective repair protocol for clinical use (Hickel et al., 2013; Özcan and Koc-Dundar, 2014; Valente et al., 2016).

3.6 RC restoration repair-current evidence

3.6.1 RC restoration repair within primary care

Dental researchers have taken into account the experience and opinions of dental practitioners, in order to be aware of their clinical preferences and practice. This is owed to the practical experience gained as a general dental practitioner, through daily interaction with materials, enabling practitioners to regularly highlight issues that are not easily apparent or identified by researchers in an academic environment (Schleyer *et al.*, 2013). Information on clinical practice has been used to provide a more generalisable line of investigation, by focusing upon what works in the practitioners' hands, and which thus might more effectively support changes in clinical practice (Field *et al.*, 2009). The information has also been used to identify the types of treatment delivered to patients, which might vary in decision making in the same or similar clinical situations (Gordan *et al.*, 2009; Gilbert *et al.*, 2010). These clinical protocols can, therefore, contrast with the available evidence (Klosa *et al.*, 2015).

In relation to RC repair, a number of studies have surveyed behavior of dentists in clinical practice for different purposes. When dentists were surveyed in the dental Practice-Based Research Networks in the United States and Scandinavia on whether they repair or replace defective restorations, including direct RC restorations, the investigators reported that less than one third of the defective restorations were repaired (Gordan *et al.*, 2012). The study was more concerned with dentists' decisions and their specific reasons for deciding whether to repair or replace defective restorations. The authors reported that date of qualification and practice type had some relation to the decision to repair restorations, but they did not report on the techniques and materials used for repairs (Gordan *et al.*, 2012).

In a more recent survey within the dental public service in Norway, repair was more common than replacement for RCs, particularly with a smaller defect size. The study was less concerned with treatment methods compared to the decision to repair or replacement. However, dentists were only asked about types of bonding agent strategy use when placing RC, or performing repair or replacement, and no record was made of the types of mechanical and/or chemical surface treatments other than the bonding agent (Staxrud *et al.*, 2016). Similarly, two studies surveyed dentists in

general dental practice for their attitude, practice, and experience regarding restoration repair, including RCs. The results showed 93.4% and 98.5% of RC restorations were repaired by dentists in Germany, and in Switzerland respectively (Kanzow *et al.*, 2017a; Kanzow *et al.*, 2017b). The participants asked about the use of phosphoric acid etching, bonding agent, and silane, but they were only asked about performing roughening of the fractured surface, and not the types of instruments used for roughening. In contrast, another study asked more detailed questions about the types of mechanical and chemical preparations during RC repair in Greece, and these included the use of a diamond bur and air-abrasion in addition to phosphoric acid etching, bonding agent, and silane (Maria *et al.*, 2017).

In a cohort study, Blum *et al.* undertook an investigation of the changes in newly graduated students' views on repairing defective direct RC restorations in the UK. These data provided information on the materials and techniques used to repair direct RC restorations, giving an insight into the experience of vocational dental practitioners (Blum *et al.*, 2005). The authors reported that diverse techniques were used to repair RC restorations. These included a decrease in the number of practitioners using diamond finishing instruments, and an increased number performing no mechanical roughening of the RC surface. There was also an increase in the use of bonding agent without acid etching. However, the results from this study cannot be regarded as a direct insight into the clinical practice of RC restoration repair, as this study was performed to determine the quality and parallelism of the RC restoration repair methods of recently graduated dentists compared with what these practitioners had been taught in dental schools.

According to the reviewed studies, dentists do repair RCs but it is unclear which methods are being used, particularly in the UK, and this information is required to ensure the types of treatments patients receive which might affect the quality in the care provided to patients in clinical dental practice.

3.6.2 RC restoration repair within dental schools

Teaching the repair of failed restorations, particularly direct RC restorations, is reportedly taught in most European, Scandinavian, and American dental schools (Gordan, 2003).

The dental schools were investigated in terms of RC restoration repair at two different times, approximately a decade apart. They were surveyed to establish the prevalence of the repair of failed direct RC restorations, and the manner in which this was taught (Blum *et al.*, 2002; Blum *et al.*, 2003a; Blum *et al.*, 2003b; Gordan, 2003). Second, with the progress made in relation to the materials available and emergent evidence supporting new approaches in operative dentistry, further surveys were undertaken in 2010. It was hoped that any changes to teaching styles which adopt direct RC restoration repair would be demonstrated (Blum *et al.*, 2011abstract; Blum *et al.*, 2012a; Lynch *et al.*, 2012). The overall response rate from dental schools was approximately 80% in the UK and 100% in Ireland, and it was found that the level of repair was greater in the 2010 data compared with that of 2000. For example, the teaching of restoration repair increased from 14 American dental schools to 52 between the two time periods (Gordan, 2003; Lynch *et al.*, 2012).

In relation to the nature of teaching, dental schools taught repair techniques based on evidence from the literature or the clinical experience of the teacher. More recent studies on RC repair showed teaching programs were more dependent on evidence from the literature (Blum *et al.*, 2012a; Blum *et al.*, 2012b). Despite this, the clinical techniques of repair seem to have remained the same when comparing the two time points, which included both mechanical and chemical preparation in most of the dental schools. Mechanical preparation was conducted predominantly using diamond finishing instruments, and less frequently with finishing discs and sandblasting (Gordan, 2003; Brunton *et al.*, 2017).

Regarding chemical preparation, several types have been employed, including acid etching with phosphoric acid and hydrofluoric acid, the application of an adhesive only, and the use of a multi-stage adhesive system and silanes; they are used with different frequencies by dental schools (Blum *et al.*, 2003a; Lynch *et al.*, 2012). With the exception of dental schools in Germany where more bonding agent is applied without phosphoric acid etching, all others in the UK, USA and Scandinavia typically applied both phosphoric acid and then an adhesive system were applied at the same time during the repair of RC restorations (Gordan, 2003).

Indications for repair were also investigated in dental schools, and it was reported that both tooth- and material-related defects were accounted for during decision

making. Overall there was variation between indications in dental schools, with no clear set of criteria identified. For instance, when dental schools in the UK and Ireland were reviewed, more than 75% of schools had performed repairs for partial loss of restorations, but around 86% of schools had included the repair of bulk fractures in posterior restorations to their teaching program in Germany (Blum *et al.*, 2012a; Kanzow *et al.*, 2018). Also, approximately 50% of Scandinavian schools indicated repair of worn restorations (attrition/ abrasion/ erosion), while 75% of UK and Irish schools identified wear as an indication for repair (Blum *et al.*, 2012b; Blum *et al.*, 2012a).

According to more recent studies undertaken for dental schools in 2012 (Blum *et al.*, 2012a; Blum *et al.*, 2012b), the repair of direct RC restorations has been incorporated into taught programs, but variation in indications and clinical techniques remains to be a persistent issue. This inconsistency may be related to the fact that indications and methods of repair are not universally agreed; these variations can be due to the fact that the teaching is mostly based on clinical experience and the lack of robust clinical evidence.

3.6.3 RC repair-in vitro testing of different approaches

I. Mechanical preparations

<u>A. Bur</u>

The commercially available diamond burs used in dentistry have various grit sizes, and, generally, coarse, medium, fine and extra fine are available. In addition to the name of the manufacturer, diamond bur sizes can be identified by a colour coded label, in black, blue, red, yellow and white, from coarse to extra fine, respectively. However, the colour label may not be the same from one commercial manufacturer to another. These diamond burs are one of the instruments used to mechanically roughen RC surface during repair (Özcan and Koc-Dundar, 2014; Valente *et al.*, 2016), owing to the fact that they are simple, cost-effective, and require no extra equipment or chemical substances in comparison to a number of surface treatments. For example, hydrofluoric acid and sandblasting are less convenient compared to

surface roughening with diamond burs (Joulaei *et al.*, 2012). In addition, abrasive grit size affects the cutting efficacy of burs and can change the composite surface micromorphology potentially increasing the interlocking with the repair material (da Costa *et al.*, 2012; Valente *et al.*, 2015).

In vitro studies on mechanical roughening by bur have revealed varying results. In comparison to the negative control (no surface treatment added to the prepared samples), the diamond bur led to a significant increase in RBS (Yesilyurt et al., 2009; Valente et al., 2015; de Jesus Tavarez et al., 2017), a substantial decrease (Wendler et al., 2016), or made no difference when comparing the negative control and treatment with diamond bur (Cavalcanti et al., 2007; Loomans et al., 2011b). One of the limitations of these studies is that there is inconsistency in the surface treatment of negative control samples before roughening with diamond bur. For example, a number of investigators prepared the negative control sample surface with a Mylar strip (Cavalcanti et al., 2007; Valente et al., 2015), another prepared with a diamond finishing bur (Al-Asmar et al., 2017) or coarse diamond bur (Wendler et al., 2016), while others prepared RCs with SiC paper (Rathke et al., 2009; Loomans et al., 2011b). This variability with the negative control sample preparation likely affected the outcomes measured for the control. In addition, the diamond burs used had different grit sizes, and in some of the studies no grit size was specified. It is therefore unclear how these variables affected the final outcomes, and so drawing conclusions based on the available data is difficult.

Like the negative control samples, the results for the diamond bur in comparison to other surface treatments are contradictory. The RBS using a diamond bur has been shown to be higher (Federica Papacchini *et al.*, 2007; Nassoohi *et al.*, 2015) or lower (Rathke *et al.*, 2009) or similar (Baena *et al.*, 2015; Al-Asmar *et al.*, 2017) in RBS than microabrasion, at 50 to 25 μ m. In comparison to the cohesive strength of the RCs, a group of authors revealed that a coarse diamond bur could enhance RBS by about 86% (Loomans *et al.*, 2011b). Also, Cavalcanti *et al.* (2007) showed that coarse diamond bur was significantly lower in bond strength than the cohesive strength of the RC. The main limitation of these studies is that the final outcome was investigated without a thorough description of the reasons for the outcomes.

Investigators have considered a number of variables in explaining the effects of RBS or RCs after the application of a diamond bur. Examples of such variables are RC types, roughness, surface appearance and surface area. Most of the studies examined microhybrid RCs (Cavalcanti et al., 2007; Federica Papacchini et al., 2007; Bektas et al., 2012; Joulaei et al., 2012; Kiomarsi et al., 2017) and some other studies tested nanofill (Yesilyurt et al., 2009; Ahmadizenouz et al., 2016) or nanohybrid (Baena et al., 2015; Wendler et al., 2016), while a few examined more than one type of RC (Loomans et al., 2011b; Celik et al., 2014; Nassoohi et al., 2015; de Jesus Tavarez et al., 2017). Generally, the studies do not clarify how RC type influences the effectiveness of a diamond bur on RBS. However, Celik et al. (2014) showed that nanohybrid RC resulted in better RBS compared to microhybrid after using a diamond bur. Joulaei et al. (2012) found that when two microhybrid RCs were treated with a diamond bur, the resultant bond strength was significantly different between the two materials. The surface appearance viewed with SEM and the roughness measurement of RCs were substantially increased when treated with a diamond bur compared to their negative control samples (da Costa et al., 2012; Valente et al., 2015). However, this relationship is not clear in comparison to other surface treatments. For example, Valente et al. (2015) found that a fine grit bur resulted in the roughest appearance compared to the medium grit bur when viewed with SEM, while the Ra measurement showed less roughness for the fine grit bur than medium bur. In addition, da Costa et al. (2012) measured the surface area of fine 46 µm, medium 91 µm and coarse 151 µm diamond bur. The highest surface area was for the medium bur and the lowest for coarse, but this difference did not impact on the RBS with the burs producing a similar RBS.

In summary, the application of a diamond bur appears to be inconsistent in enhancing RBS, and there is a limitation in explaining the effect of additional covariables on bond strength. Therefore, studies designed to understand these additional variables are required to further describe their magnitude of effect of diamond bur on RBS.

B. Microabrasion

Airborne-particle abrasion can be delivered via chairside air abrasion devices for intraoral use with two to three bar pressure. Metal, ceramic, composite, or amalgam

substrate materials are usually abraded for around 10 s from a distance of about 10 mm with the intention to create a clean and roughened surface, although longer air abrasion may be required for zirconia (Özcan *et al.*, 2013b). The abrasion particles are comprised of 30–50 µm aluminum oxide particles coated with a silicon dioxide layer, known as "silicoating" or "tribochemical surface conditioning" (Edelhoff *et al.*, 2001). Alumina or silica particles cover the surface, and these then create covalent bonds through the siloxane layer contained within a silane coupling agent. The CoJet system has particles that are intended to penetrate and become embedded in the top layer of the substrate, leaving it partly covered with silica (Lung and Matinlinna, 2012). Potentially, the embedded particles become microretention locations for the new composite added to the surface, which explains the better bond strength. One weakness of air abrasion is the aerosol containing abrasive particles, and there is a need for an effective suction device to stop inhalation of these particles.

In the literature, in vitro reports have evaluated the impact of sandblasting/air abrasion against other surface treatment approaches and have found conflicting results. The repair of many RC brands only achieved bond strengths near the cohesive level after sandblasting with 50 µm alumina particles (Loomans et al., 2011b). Several reports have shown superior RBS values for 50 µm aluminium oxide sandblasting than the other studied surface treatments, such as applying a bur and hydrofluoric acid (Hannig et al., 2006; Junior et al., 2009; Costa et al., 2010; Lührs et al., 2011; da Costa et al., 2012; Cho et al., 2013; Palasuk et al., 2013). Celik et al. also noted that air abrasion led to the smallest microleakage values compared to the other bur treatment (Celik et al., 2015). In contrast, other research groups have presented lower or statistically similar RBS values achieved by air abrasion to those from diamond bur application (Brosh et al., 1997; Yesilyurt et al., 2009; Hasan, 2012; Ahmadizenouz et al., 2016). In addition, a review study concluded that sandblasting may not enhance RBS better than other surface treatments (Valente et al., 2016). However, da Costa et al. (2012) explained that the reason for the effectiveness of sandblasting was the higher surface area of sandblasted RC, compared to the bur and no treatment for microhybrid RC. Most of the studies do not go on to explain the mechanism behind their findings, and therefore it is not clear how great the magnitude effect of sandblasting is in comparison to no surface treatment, or other surface treatments on different RCs.

II. Chemical preparations

A. Phosphoric acid etch

Phosphoric acid has been investigated either as a type of surface treatment alone or as one of the steps in a bonding system. The reviewed literature revealed the emergence of several contrasting themes. As a surface treatment, Loomans and colleagues (2011) showed phosphoric acid positively enhanced RBS (Loomans *et al.*, 2011b), while other groups of researchers reported that phosphoric acid did not improve RBS in comparison to other surface treatments (Fawzy *et al.*, 2008; Rathke *et al.*, 2009; Eliasson *et al.*, 2014; Bahari *et al.*, 2018). The following sections discuss the findings from the above studies and their strengths and weaknesses.

Loomans and colleagues (2011) studied the effectiveness of 37% phosphoric acid in comparison to nine other surface treatments on five types of RC that were different in composition. Their results showed phosphoric acid improved RBS significantly for the microhybrid and hybrid RCs compared to no surface treatment. In addition, Loomans *et al.* (2011b) compared the bond strength of RCs treated with phosphoric acid to the cohesive strength, in other words internal strength, of RCs, and phosphoric acid enhanced RBS by 90% in comparison to cohesive strength. The authors did not propose an explanation for the mechanism of action for phosphoric acid in the repair of bond strength.

In contrast, four groups of researchers showed phosphoric acid did not enhance RBS significantly in comparison to other surface treatments. Phosphoric acid was used in combination with other surface treatments such as coarse bur and silicon carbide paper (Rathke *et al.*, 2009; Eliasson *et al.*, 2014; Ahmadizenouz *et al.*, 2016). However, the results showed lower improvement in the RBS of the repair methods including phosphoric acid than those without. The authors did not investigate RBS before and after the application of phosphoric acid to determine its cleaning effect on RBS. Fawzy *et al.* (2008) also demonstrated no significant effect of phosphoric acid in terms of RBS for micro-fine hybrid when excluding phosphoric acid in the repair protocols. Further, a study by Bahari *et al.* (2018) showed a negative impact of phosphoric acid on RBS compared to no surface treatment when investigating the Beautifil II giomer, which is an RC containing glass ionomer fillers.

It has been proposed that phosphoric acid may improve RBS via increasing microundercuts on the RC surface. One of the main approaches used to quantify microundercuts is measuring roughness before and after the application of phosphoric acid. Fawzy and colleagues (2008) showed a significant change in roughness values for non-treated and grinded RC after treatment with 37% phosphoric acid etch. Additionally, a number of investigators have shown increases in roughness for hybrid and nano-filled RCs after treatment with 37% phosphoric acid etch –significantly higher for hybrid RC compared to a non-etched hybrid RC surface (Loomans *et al.*, 2011a). The main weakness of Loomans *et al.* (2011a) is that they did not test RBS.

In contrast to the findings of Fawzy *et al.* (2008) and Loomans *et al.* (2011a), a study by Bahari *et al.* (2018) explored changes in surface roughness in relation to RBS. Application of 37% phosphoric acid resulted in a smoother surface than before etching (Bahari *et al.*, 2018). This study concluded that the decrease in roughness resulted in low RBS. As mentioned before, Bahari *et al.* used a RC that contains glass ionomer fillers which were not present in the conventional RCs used by the Fawzy and Loomans groups. Therefore, it is unclear how filler chemistry may have affected roughness and RBS

Another suggestion for the effectiveness of phosphoric acid is the cleaning of the RC surface from loose particles and resin debris. Fawzy and colleagues (2008) used SEM and AFM as an alternative to investigate the surface characteristics of RCs before and after the application of phosphoric acid. Their findings showed that RC treated with 37% phosphoric acid resulted in smaller irregularities in size compared to an untreated surface and the removal of smear grinding debris for the ground RC. However, these authors did not find relation from the cleaning action of phosphoric acid on RBS.

Phosphoric acid has been used in combination within bonding systems for RC repair, and a number of studies have assessed the efficacy of phosphoric acid etch for improving RBS by inclusion or exclusion of it within the bonding systems. Generally, bonding system types comprised of bonding agent alone, or bonding agent with primer (self-etch), and the addition of phosphoric acid to either bonding agent or selfetch adhesive is known as the etch and rinse system.

For RC repair, the impact of phosphoric acid on the bonding system varies across studies. The Celik group found no difference between the etch and rinse and selfetching systems on reducing microleakage or enhancing RBS for the microhybrid and nanohybrid RCs (Celik et al., 2014; Celik et al., 2015). They explained similar findings between self-etch and etch and rinse systems by suggesting that the acidic primer of the self-etching systems produced effective surface cleaning, as does phosphoric acid in an etch and rinse system. In contrast, the Cavalcanti group showed self-etch enhanced RBS better than etch and rinse systems on microhybrid RC. They also included phosphoric acid with self-etching system, but the results showed no difference with exclusive use of self-etch bonding in RBS (Cavalcanti et al., 2007). The superior effect of the self-etching system was explained by its lower viscosity than etch and rinse adhesive system. Eliasson and colleagues' findings agree with the Cavalcanti group in that the two step self-etch system significantly increased RBS compared to the etch and rinse adhesive system on nanohybrid RC. The findings indicate that the RCs behaved differently to the bonding systems containing phosphoric acid, and the main limitation of the studies is that they could not explain the findings thoroughly.

This point can be summed up by suggesting that the current literature on the effect of phosphoric acid has shown evidence on the effectiveness of phosphoric acid on its own on RBS and impact on surface roughness and appearance. Also, RCs reacted quite differently to phosphoric acid. However, the findings are limited, and have produced inconsistent findings. The methods of preparing the samples were different and the RC performance was not described systematically before and after application of phosphoric acid. Therefore, it is not clear what the mechanism of action of phosphoric acid was in most of the reviewed studies, and so a conclusion cannot be drawn.

B. Hydrofluoric acid

Hydrofluoric acid has been used to etch RC surface during repair. It can dissolve filler particles and resin matrix to a different extent depending on the composition and surface area available for contact with the acid, and the concentration of the acid (Swift Jr *et al.*, 1992; Loomans *et al.*, 2011a). Hydrofluoric acid has been evaluated for different RCs for its effectiveness on RBS, but the findings are contradictory. Investigators have shown that hydrofluoric acid is likely to enhance RBS better than

phosphoric acid etch and bur (Loomans *et al.*, 2011b; Al-Asmar *et al.*, 2017; Bahari *et al.*, 2018), while other researchers have shown a negative effect of hydrofluoric acid compared with bur, phosphoric acid, and microabrasion (Crumpler *et al.*, 1989; Özcan *et al.*, 2005; Junior *et al.*, 2009; Yesilyurt *et al.*, 2009). The main limitation of these studies is that every study used a different composition of RC, and the concentration of the tested hydrofluoric acid was also different. The studies' findings dependent on the RBS did not explain the reasons for the outcomes. However, the problem with the usage of hydrofluoric acid is linked to the intraoral use of hydrofluoric acid (Özcan, 2003). If it contacts skin or mucosa, it can cause deep tissue necrosis and therefore it has been considered unsuitable for intraoral repair because of the potentially dangerous effect (Hatzifotis *et al.*, 2004).

C. Dental bonding systems

In relation to RC repair, the primary aim of the bonding agent is to provide retention for the repair material with the substrate RC via chemical or micromechanical interlocking or a combination of both. For non-aged RC, dental bonding reacts with the unreacted monomers of the polymer matrix and accordingly may enhance RBS (Veiga de Melo *et al.*, 2011). For aged RCs, due to the absence of unreacted monomers, the primary mechanism for the bonding agent would be interlocking in the micromechanical retentions upon polymerisation (Van Meerbeek *et al.*, 2003), because dental bonding can penetrate the irregularities and wet the surface of RC better than RCs. The potential wetting and capillarity of the dental bonding is the key mechanism for the micromechanical interlocking. In addition, traditionally a clean and dry surface is necessary for the bonding agent to be able to wet the substrate.

Since the high viscosity and low wetting potential of RCs may not allow proper wetting of the substrate with the repair RC (Kim *et al.*, 2011), this may be one of the mechanisms that a number of studies show as optimising RBS between the aged RC and the repair materials, in comparison to the situation of excluding a bonding agent (Staxrud and Dahl, 2011; Özcan *et al.*, 2014). In addition to that, the improvement in the RBS could also be attributed to the micromechanical locking of the bonding agent after setting with the irregularities in the surface (Cavalcanti *et al.*, 2007; Onisor *et al.*, 2007; Kallio *et al.*, 2013).

Different types of bonding systems have been used in clinical practice and tested in in vitro studies, particularly the total-etch and self-etch bonding systems. Total etch can be two or three steps, and self-etch can be one or two steps. Three-step bonding systems are the preferred bonding technology when performing RC restorations (Van Meerbeek et al., 2003; Manuja et al., 2012). Within published works in the literature, it is not clear which type of bonding system has the potential to enhance RBS in comparison to other types of bonding system. Nonetheless, Eliasson et al. found more enhancement in mean tensile repair strength values after the use of a two-step self-etch than the use of both one step self-etch and three step etch and rinse (Eliasson et al., 2014). Cavalcanti et al. (2007) showed a similar finding as Eliasson et al., where the authors found a two-step self-etch had the highest RBS on RC than bonding agent alone, or two step etch and rinse. In contrast, a number of investigators showed no difference between bonding agent alone and self-etch bonding (Özcan et al., 2013a). However, the studies used different types of RCs and different types of mechanical roughening before the application of the dental bonding and none of the investigators offered a mechanism to explain the mechanism behind their findings.

Bonding system type may have a different effect on improvement in RBS, even when applied to the same type of mechanically prepared surface. A number of studies have shown that both bonding agent alone and a self-etch bonding system can significantly enhance RBS when applied on r) nanohybrid and nanofill RCs prepared with p1200 SiC (Özcan *et al.*, 2013a). However, the use of a bonding agent on hybrid RC prepared with P1200 SiC did not show significant improvement in RBS (Brendeke and Özcan, 2007). Both studies used water to clean the samples. Because the studies on it measured RBS it is not clear whether surface roughness of prepared RCs with P1200 SiC related to the difference in findings or was not the reason for the outcomes.

In summary, bonding agent has been used during RC repair and there is not general agreement on their effect on enhancing RBS with the main limitation of the reviewed studies did not clarify the mechanism behind the findings of the effect of dental bonding, which could be clean surface, difference in wetting ability and retention of the bonding agents. Therefore, insufficient current evidence would support the need for further investigations.

D. Silane

After mechanical surface treatment, chemical adhesion can also be enhanced with silane (Brendeke and Özcan, 2007; Matinlinna *et al.*, 2018). Silane may improve RBS by increasing the surface energy, which is a requirement for optimal wetting of the surface to ensure close contact between the RC materials during repair (Staxrud and Dahl, 2015).

Generally, the chemical structure of silane is stated by Z3SiX, where Z is the part which reacts to inorganic materials and X is the part which reacts with organic materials, such as the monomers in bonding resin and RC matrix (Nihei, 2016). The strength of the chemical reaction of silane Z group is dependent on the type of inorganic material, in the order silica > quartz > glass > aluminium etc., as described by Lung and Matinlinna (2012) (cited in Matinlinna *et al.*, 2018). The Z group of silane is susceptible to hydrolysis, and therefore within the development of silane there has been enhancement of the hydrophilicity of the Z group (Lung and Matinlinna, 2012). Silane has been applied to enhance coupling between fillers and resin matrix. In addition, silane has been widely used to repair ceramic crowns, and there have been some attempts to evaluate the effect of silane in RC repair (Matinlinna *et al.*, 2018).

A number of studies compared the effect of silane in comparison with the bonding agent alone but the results are conflicting. Nassoohi *et al.* found that silane use gave similar bond strength when applied after hydrofluoric acid etching on microhybrid, nanohybrid and nanofill RCs, aged for 24 hours, in comparison to bur and bonding agent (Nassoohi *et al.*, 2015). Lima *et al.* found comparable RBS values with no application of silanes with bonding agent on silorane RC, either roughened with bur or aluminum oxide sandblasting (Lima *et al.*, 2014). In contrast to the former authors, Barcellos *et al.* found that use of silane after either surface roughening by bur or hydrofluoric acid led to significantly lower RBS compared to bonding agent on nanofill RC. The repaired samples were aged for seven days in distilled water (Barcellos *et al.*, 2015). Further, Imbery *et al.* reported lower repair strength values when the aged RC surface was initially silanised and an bonding system was used compared with the use of bonding system alone on prepared nanofill RC, when the repaired samples were aged in distilled water for 24 hours (Imbery *et al.*, 2014). However,

Fawzy and colleagues' findings suggest that silane be included in the repair method as it could significantly enhance RBS in combination with a bonding system, rather than excluding silane on prepared micro-fine hybrid RC in which the substrate samples were aged for thirty days and the repaired samples aged for 24 hours in distilled water (Fawzy *et al.*, 2008).

There appears to be a need for clarification of the role of silane on RBS. The current studies do not identify a consistent magnitude effect of silane on RBS based on its mechanism of action with the fillers or unreacted monomers in resin matrix of either RC materials or the bonding agent. In addition, the effect of storage in fluid and roughness of the substrate on silane effectiveness have not been determined well. Therefore, the current situation of evidence would justify the need for further investigations.

3.6.4 Assessment of RC repair in vitro

Most RC samples are ground and prepared with SiC paper prior to measuring any parameter to assure perfect parallelism of the surface, and this considered to be negative control of the samples before any interventions. In terms of RC repair, studies have either not prepared the specimen surfaces (Cavalcanti *et al.*, 2007; Veiga de Melo *et al.*, 2011) or prepared RC samples with SiC paper (Junior *et al.*, 2009; Loomans *et al.*, 2011a; Loomans *et al.*, 2011b; Özcan *et al.*, 2013a). The range of particle grit size used was from P80–P4000 (201-6µm). One of the drawbacks of the current situation is that there is no agreement or standarisation on which grit size of SiC to use to prepare samples before the application of surface treatments. For example, Loomans *et al.* (2011b) used P600 grit, while other authors have used P500 grit (Oztas *et al.*, 2003) or P4000 grit, and this may result in a number of problems with the research studies.

A number of studies prepared samples with different SiC papers and did not applied further roughening as a simulation of fractured surface, and the studies likely assumed preparing RC with SiC paper will make the surfaces similar to each other. However, there is evidence that when RCs are treated with the same SiC paper grit size, the findings are different. When Eliasson *et al.* (2014) prepared nanohybrid RC with P320 grit SiC paper, the RBS was 41 MPa, while Hamano *et al.* (2011) showed that when nanohybrid RC was prepared with P320 grit SiC paper, the bond strength

was 29.9 MPa. In addition, when nanohybrid and nanofill were prepared with P1200 grit SiC paper, the RBSs were 32 and 23 MPa, respectively (Özcan *et al.*, 2013a). Brendeke and Özcan (2007) presented lower RBS for hybrid RC prepared with P1200 grit SiC paper, at approximately 11 MPa. The main limitation of these studies is thus that they do not clarify differences due to the interaction of the SiC paper grit size and RCs, or another factor.

When comparing different studies, it is not clear how preparing RC samples with these different grit sizes of SiC paper affects the findings. The study by Kallio *et al.* (2013) showed that a higher grit size of SiC paper resulted in higher roughness and RBS. The authors prepared microhybrid RCs with P320, P800, P1200 and P2400, and the P320 exhibited higher RBS and a rougher surface than the other grit sizes of SiC paper. Therefore, there is a possibility that grit size influences the final results and it is likely to be a cofounding factor. For this reason, studies should consider this issue when preparing samples and comparing results with other studies.

The main limitation of the reviewed studies is that they assume that when RCs are prepared with any grit size, the surface will have fewer imperfections and a reduced oxygen inhibited layer, without making consideration for the effect of SiC grit size on RC surface characteristics, and the resultant effect on RBS. In addition, the studies have not clarified the magnitude of the effect of the prepared RCs on RBS, and thus it is not clear whether preparing RCs with SiC paper grit size is one of the reasons for the difference or similarity in the findings.

3.6.5 Measurement of RC repair in vitro

In vitro studies on the repair of RC have used both quantitative and qualitative methods to understand the effect of different surface treatment protocols on RBS. Both quantitative and qualitative methods have limitations, but combining the two methods allows the weaknesses of each method to be reduced, and thus there is a likelihood of providing stronger evidence. In addition, using the two methods can enhance insights and understanding of the findings that may be otherwise overlooked. The following sections are a summary of the methods used to describe the surface characteristics of RCs in terms of repair.

A. Scanning Electron Microscopy

SEM has been widely used in dental research since 1962 for the observation and characterisation of materials from the nano- to micro-scale. It can produce 2D or 3D images which provide qualitative information about the shape of surface features of the samples examined, and also about the location of features relative to each other. In dentistry, it has been used for image analysis, to provide information on the surface quality of RCs before and after interventions. However, although the qualitative information from SEM is subjective, time consuming and less able to be generalised, it can be used to provide a complete and detailed description of the examined sample. Consequently, this qualitative information has the potential to help interpret and explain quantitative data. For example, more degradation of resin matrix and the extrusion of fillers in nanohybrid RC by ageing in water coincides with lower RBS, while nanofilled RC reveals more resistance to degradation and higher RBS (Özcan et al., 2013a). In relation to RC repair, the aims of using SEM can be summarised as follows:

- To identify fracture modes by categorising them into cohesive, adhesive and mixed types, and to produce information which may link these categories to RBS. However, a generally accepted link has not been identified (Brendeke and Özcan, 2007; Junior *et al.*, 2009; Eliasson *et al.*, 2014);
- 2) To examine the degradation effect of water sorption and phosphoric or hydrofluoric acid etching on fillers, the resin matrix, and the interfacial layer (Hamano *et al.*, 2011; Loomans *et al.*, 2011a; Hamano *et al.*, 2012; Özcan *et al.*, 2013a). These studies have provided a deeper understanding of the location of degradation based on RC components, as well as the aggressiveness of that degradation;
- To describe surface pattern and morphology before and after surface treatments (Fawzy *et al.*, 2008; Rinastiti *et al.*, 2010; Hamano *et al.*, 2011; Veiga de Melo *et al.*, 2011; Baena *et al.*, 2015; Ahmadizenouz *et al.*, 2016). Studies have aimed to determine the difference in the quality and how much the qualitative change could have impact on the changes in quantitative findings;

Therefore, SEM can be regarded as a useful tool for providing qualitative information on the surface morphology of the examined samples.

B. Roughness of RCs

In terms of RC repair, roughness has been quantified by the use of different methods and parameters within a number of *in vitro* studies. The aim of roughness measurement is to determine the impact of surface roughness on RBS. Average roughness (Ra) has been used to measure surface roughness using different methods. Firstly, Atomic Force Microscopy has been used to measure the roughness of RCs before and after surface treatments (Fawzy *et al.*, 2008; Rinastiti *et al.*, 2010). Secondarily, both non-contact (Loomans *et al.*, 2011a; Wendler *et al.*, 2016) and contact profilometers have been used to calculate average roughness (Junior *et al.*, 2009; da Costa *et al.*, 2012; Kallio *et al.*, 2013; Valente *et al.*, 2016). Another method of quantifying Ra is a white light 3D non-contact interferometer, which was used to quantify the average roughness of the treated surfaces of two RCs with different filler compositions (Loomans *et al.*, 2011a). In this study, the hybrid RC had a rougher surface than the nanofill RC before and after surface treatments, but this study did not examine RBS.

The findings from the studies in this area are conflicting, as some have shown that an increase in the Ra is likely to increase RBS. Rinastiti and co-workers showed significant increase in Ra of microhybrid, nanohybrid and nanofill RCs after silica coating compared to the Ra polymerised against Mylar strip, and this increase in Ra had an impact on the increase in RBS, except for one RC. Similarly da Costa et al. found an increase in the Ra of microhybrid RC when treated with fine/medium/coarse burs and sandblasting. However, the repair bond increased for all RCs but the highest bond strength was not associated with the highest roughness (da Costa et al., 2012). In addition, preparing microhybrid with P320 gave the highest Ra and RBS compared to smaller SiC grit papers of P800, P1200 and P2400. Nonetheless, this was not the case for P800 which showed higher Ra but similar RBS compared to P1200 and P2400 (Kallio et al., 2013). In contrast, it has also been found that an increase in Ra did not impact on an increase in RBS. Kallio et al. and Wendler et al. found higher roughness to be associated with a higher grit of diamond bur, while the highest bond strength was exhibited by the smaller SiC grit size (Kallio et al., 2013; Wendler et al., 2016). Unfortunately, these studies did not fully explain the reason for

the difference in Ra and RBS, and thus more studies are needed to explain the characteristics of surface treatments that might provide higher RBS, and how they differ from those which do not.

Average roughness is one of the most widely used parameters in studies investigating RC repair. It is defined as the arithmetic average height of roughnesscomponent irregularities (peak heights and valleys) from the mean line, measured within the sampling length (*Aerospace Engineering Guide*, 2008), as seen in Figure 3.5. The formula for Ra calculation is:

$$\mathsf{Ra} = \frac{1}{L} \int_0^L |Y(X)| d(X)$$

Where Y is the vertical deviation from the mean line, d is the number of deviations included in a sample length (L), and X is the numerical number of deviations.



Figure 3.5 Roughness average (Ra) of a surface finish. Scanned from (Aerospace Engineering Guide, 2008)

Literature review

When there is difference in the average roughness measurement between two profiles, this means there is a difference in the roughness quality, and such a difference may affect performance in real world application. Average roughness has been criticised for its limitation when used alone in qualifying roughness(Field *et al.*, 2010). Also, it is likely that important information may be overlooked when it is the only description of roughness. The limitations of roughness average are illustrated in Figure 3.6; the two profiles have different surface quality but, when profiled, have the same Ra value (Field *et al.*, 2010). The surface in Figure 3.6 (A) has wide peaks and narrow valleys, while in Figure 3.6 (B) the surface has narrow peaks and wide valleys; despite this, the calculation of Ra is the same. Furthermore, the difference does not only include peaks and valleys, as two profiles can have similar shapes but be different in spacing, and this might lead to variability in performance. For this reason, there is a need to distinguish surfaces that differ in shape and spacing, and these factors should be determined along with measuring the heights and depths of peaks and valleys.

A number of other calculated roughness parameters besides Ra have been used to describe roughness quality (Löberg et al., 2010; Loomans et al., 2011a; da Costa et al., 2012; Field et al., 2013). A bearing area curve (BAC) summarises shape information with a number of parameters consisting of three sections: the small peaks which are the upper part of roughness above the main plateau; the plateaux or the core part in the middle of the roughness; and, the deep valleys which are the lowest part of roughness between plateaux. Figure 3.7 illustrates how BAC parameters can categorise a rough surface in terms of shape and spacing into the three parts described (peak, core, valley). Within a BAC, a rough surface is measured for height and width –the height is by micron (μ m) and the width by percent. The parameters that represent heights are Rpk, Rk, Rvk, which are the heights of peaks, cores and valleys, respectively, and the parameters that represent the width of the roughness are Mr₁ and Mr₂. Mr₁ represents the material ratio of the peak part and Mr₂ represents the material ratio of the core. Table 3-1 describes the parameters used with a BAC, and Figure 3.8 shows two roughness examples of different quality. Both surfaces have similar Ra but differ in BAC parameters, and therefore the figure illustrates how BAC parameters characterise two roughness examples with the same Ra.


Figure 3.6. Two profiles with the same Ra value having different surface characteristics (the red line indicates the ideal form of the surface). The profiles have the same Ra value because they deviate from the ideal form by the same magnitude. Scanned from Field et al. (a) (2010, pp.185)



Figure 3.7. How the bearing curve of a rough surface and the parameters (Rpk, Rk, Rvk, MR1, and MR2) are used to categorise the rough surface distribution into peaks, cores and valleys. Scanned from Field et al. (a) (2010, pp. 186)

BAC parameters	Description	
Core Roughness Depth (Rk)	Correlates with the depth of the working part that carries load, and contacts the matting surface.	
Reduced Peak Height (Rpk)	The small peaks above the plateau of a surface worn off during the run-in period of a surface.	
Reduced Valley Height (Rvk)	An estimate of the depth of valleys that retain lubricants in the working part of a surface.	
Material Ratio of Peaks The fraction of the surface consisting of sm (Mr_1) peaks above the main plateau.		
Material Ratio of Valleys (Mr ₂)	The fraction of the surface that will bear load during the practical lifetime of the part.	

Table 3-1 The parameters used to describe BAC modified from (Field et al., 2010).



Figure 3.8. Comparison of two different surfaces with similar Ra using the bearing curve. Surface A has significantly more, and deeper, troughs (higher Rvk and higher Mr2), and therefore has a much greater pooling/lubricative potential. Surface B has significantly more, and higher, peaks (higher Rpk and higher Mr1) and is therefore likely to suffer significantly more early surface loss in the future. Scanned from Field et al. (a) (2010, pp. 187)

C. Surface area

Surface area measurements have been used to investigate surfaces qualitatively for different types of treatment protocols on microhybrid RC. SEM images at 1200 magnification, analysed with image analysis software, were taken to measure a surface area via a 3D scan of the treated surfaces (da Costa *et al.*, 2012). Da Costa and colleagues measured surface area for no surface treatment, three different grit sizes of abrasion bur, and sandblasting. Sandblasting and a coarse diamond bur reported the highest increase in surface area but only sandblasting showed the highest increase in RBS; and burs had medium values in surface area and RBS. When surface roughness patterns were examined by SEM, sandblasting showed more peaks, valleys, and microundercuts than burs. The authors considered the quality of the surface area was an important factor for RBS. Fawzy and co-workers measured surface area for micro-fine hybrid RC before and after treatment with polishing bur and/ or acid etching, and reported no impact of a higher value in surface area on changes in RBS. They found the highest surface area value for no surface treatment, which showed lowest RBS compared to other surface treatments

(Fawzy *et al.*, 2008). SEM examination showed less prominent microundercuts on no surface treatment than the samples treated with abrasive stone bur, but the increase in surface area contributed to the presence of wide shallow depression areas on no treated RC. These differences in outcome indicate that, although surface area measurement seems to provide a measurement of a sample, it could not describe the quality of the surface area.

D. Measurement of RBS

RBS has been evaluated widely using *in vitro* tensile or shear stress testing as a measure of repair effectiveness (Fawzy *et al.*, 2008; Junior *et al.*, 2009; Loomans *et al.*, 2011b; Özcan *et al.*, 2013a). The two test methodologies differ in the direction of the load application to the test specimens.

In tensile stress testing, a load is applied perpendicular to the interface where the repair has been undertaken, while in shear stress the load applied is parallel to it (Anusavice *et al.*, 2013). Both tests can be performed using a universal testing machine such as an Instron machine. In the shear test, the interfacial bond is challenged using a single-edged chisel, a flat-end rod, or a wire loop to dislodge the repair material from the substrate. There are two scales for measuring the outcomes of bond strength: macro- and micro-scale for both tensile and shear test methods. For example, there is both macro-shear and macro-tensile which is usually named shear or tensile only, and micro-shear or micro-tensile. The size of the sample used for shear or tensile tests are dependent on the scale of measurement of the outcomes. The size of the sample for macro-scale is between 2-28 mm² and 1 mm² for micro tests.

These two methods have some limitations which reduce their ability to predict clinical performance, summarised as follows (Sakaguchi and Powers, 2012a):

- Stresses at the interface are not uniformly distributed: flaw size and crack propagation affect interface debonding stress and mode;
- A high incidence of mixed and cohesive failure: deviation of crack propagation from the interface is likely to prevent the assessment of interfacial bond strength;

- Results of different studies are not comparable: this is due to differences in testing conditions, such as bonding substrate, sample preparation, storage condition and loading method;
- Bond strength tests lack clinical significance: this is the case with these two tests, because the variability in stress concentration relevant to clinical performance cannot be determined for bond strength.

The dental literature has not shown agreement on any of the test method to be used for evaluating bond strength. However, a number of studies commented on the test methods. shear bond strength testing has been considered by some investigators as more associated with affecting the substrate compared to the interface itself (Della Bona and Van Noort, 1995; Versluis et al., 1997). The reasoning behind this is thought to be due to the non-uniform stress distribution generated at the interface, as maximum tensile strength arises adjacent to the area of load application in shear bond testing, while in tensile bond strength tests, stress is likely to be distributed at the interface. Further, micro-tensile testing is regarded by some authors as a more reliable laboratory test method than macro-tensile and macro-shear testing due to better correlation of micro-tensile testing with clinical data of cervical restorations (Heintze, 2013). When the stress distribution pattern of the macro and micro of both shear and tensile test methods was evaluated for stress distribution patterns, the findings showed that non-uniform stress distribution occurred in all the test methods (Scherrer et al., 2010). Micro-shear testing has been used to evaluate the bond strength between dentine and RC, and is suggested as an alternative to micro-tensile testing because the findings are comparable with other micro-tensile testing (McDonough et al., 2002; Foong et al., 2006).

For RC restoration repair, tensile, micro-tensile, shear and micro-shear bond strength testing methods have been used to determine the RBS of RCs (Fawzy *et al.*, 2008; Rinastiti *et al.*, 2010; Giachetti *et al.*, 2012; Baena *et al.*, 2015). Similar to dental literature, there has not been agreement on the use of a particular test method in preference to other test methods. Generally, few studies commented on test methods. It has been suggested that macro-shear testing may be preferred, similarly to the micro-tensile test method, because the findings from studies which used the micro-tensile test method were comparable (Brendeke and Özcan, 2007). When the stress distribution between shear and micro-tensile test modes were evaluated for

RC repair, both test modes showed RBS was dependent more on the adhesive interface rather than on the composite (Rinastiti *et al.*, 2011b).

Investigators have shown a number of advantages of using either shear or microshear over tensile test methods as follow:

- 1) Both sample preparation and testing procedure are simpler than tensile and micro-tensile testing methods.
- 2) There is no need for a cutting procedure, which may affect the number of pretest failures and give false low strength results due to the inclusion of defects at the periphery of the bonded interface during cutting (Giachetti *et al.*, 2012).
- 3) Shear testing can assess very weak bond strengths. When bond strength is lower than 5-7 MPa, this would be considered as a pre-test failure in a tensile test because of the effect of an aggressive cutting procedure transmitting vibration to the samples; this acts as additional stress on the samples other than the tensile test (Pashley *et al.*, 1995).
- 4) Shear testing results are also likely to be dependent on the modulus of elasticity of a tested material, indicating that a shear test might be specifically used to test fracture resistance, depending on the material deformation rather than formation of crack and flow at the interface, as in a tensile test. This may be due to the direction of the force at the interface, since, in a shear test, the force applied is intended to displace one of the materials from the other, while in a tensile test it tries to separate the materials. This information may be crucial when the effectiveness of certain geometry is to be evaluated due to applied force at the interface (Leloup *et al.*, 2001).
- There is preference for shear over micro-tensile tests because, clinically, repaired restorations are mainly exposed to shear forces (Rinastiti *et al.*, 2011b).

One of the disadvantages of using shear testing is that the flexural modulus of RCs may influence the performance of the material during testing, and this may become more apparent if both the substrate and repair material are not the same material

(Heintze, 2013). This disadvantage have not been identified to be contributed to the effectiveness of the test method with any of the RC repair studies. Further, there is no evidence on the relevance of either testing method being used with clinical data for RC repair (Eliasson *et al.*, 2014).

E. Interpretation of RBS

Besides quantitative measures of RBS, other methods have been used to provide a better explanation and assessment of RBS in *in vitro* studies. The findings from RBS have been interpreted based on fracture modes, material fracture strength, and minimum required bond strength in clinical settings. However, there is no acceptance of any of the three methods because none have been well related to RBS.

Determination of fracture modes has been used by a number of authors as a method of interpreting RBS. Fracture modes can be categorised as: cohesive failure in substrate and repair material that appears as small indents on the surfaces of both materials; adhesive failure at the interface, leading to a completely smooth surface; and, a mixed type of failure involving both cohesive and adhesive failure, characterised by both a smooth and indented surface. After shear or tensile test of repaired samples investigators have looked to the fractured surface of the samples to determine the fracture mode. This type of analysis has been used to verify a repair failure propagated through the adhesive zone or out of it, where cohesive failure may indicate more stable RBS than the adhesive or mixed failure types because it is assumed fracture occur within the weakest part of the repaired samples. Thus, cohesive fracture indicates the interface between the substrate and the repair material is stronger than the material itself. Indeed, one study found that adhesive breaks are typical when RBS is lowest (Baur and Ilie, 2013). However, this finding have not been confirmed by other studies. Stereomicroscopy or SEM have been used to determine the fracture mode (Loomans et al., 2011b; Kiomarsi et al., 2017; Altinci *et al.*, 2018).

RCs showed inconsistent results between studies in terms of fracture mode. For example, nanohybrid RCs showed variable behavior in relation to fracture mode. In the study by Özcan et al., both nanohybrid materials were reported to have less than 50% cohesive failure (Özcan et al., 2013a), but a study by Loomans *et al.* found

approximately 90% of nanohybrid RC had cohesive failure (Loomans *et al.*, 2011b). A number of investigators showed that RCs with higher surface roughness and RBS values contributed to higher cohesive fracture mode (Baur and Ilie, 2013). In contrast, there was no difference in fracture mode between higher and lower values of nanofill and nanohybrid RCs, respectively, as both of the materials showed around 50% cohesive fracture mode (Özcan *et al.*, 2013a). Junior and colleagues' findings also agree with Baur and Ilie's results in that higher bond strength is associated with more cohesive fractures; however, with lower RBS, most of the fracture mode was mixed type (Junior *et al.*, 2009). This may indicate that differences in microstructure and resin matrix contribute to differences in results regarding fracture mode more than RC type and RBS value. The reason for this may be the fact that crack propagation impacts the type of fracture mode, and in itself crack propagation is greatly influenced by the filler content, characteristics and interfacial bond of fillers and resin matrices (Shah *et al.*, 2009a; De Souza *et al.*, 2011; Ornaghi *et al.*, 2014).

Another approach used in *in vitro* studies to interpret RBS is a comparison of repair bond to the material fracture strengths (cohesive strength) without repair or ageing (Loomans *et al.*, 2011b; Baena *et al.*, 2015; Ahmadizenouz *et al.*, 2016). One of the limitation of this approach is that it may not simulate RC performance in clinical conditions, where ageing is the most influential failure mechanism of a bulk restoration placed for the first time in a cavity, and may be the same for repaired restorations. In addition to the former limitation, the fatigue resistance of RCs is lower than their fracture strength (Keulemans *et al.*, 2009), therefore making a comparison of two properties realistic when one of these never occurs in the oral environment. Another limitation of this approach is that there are no recommendations on what RBS should be in comparison to cohesive strength in successful clinical practice.

Another way to consider RBS is to compare it with the minimum value required for bond strength for restorative materials in clinical practice. Although there has not yet been a determination of minimum RBS that can survive under occlusal function (Lucena-Martín *et al.*, 2001), one study has suggested that a range between 18–20 MPa can be regarded as the minimal clinical standard required for RBS (Yesilyurt *et al.*, 2009). Such an approach may be more relevant to the clinical conditions.

3.6.6 Impact of RC materials on RBS

RCs have been considered as an important factor influencing RBS, and both their properties and composition play a role in this. In relation to properties, Baur and Ilie showed that RC with higher wear resistance have higher RBS (Baur and Ilie, 2013). Also, nanofill RC with less degradation from ageing in water had higher RBS than the nanohybrid with more degradation from ageing in water (Özcan *et al.*, 2013a). RC properties is affected by their compositions. Composition of RCs is key aspects which impact RC properties include the mobility and hydrolysis of constituent monomers (Sideridou *et al.*, 2003; Ferracane, 2006), and filler characteristics in terms of composition, filler loading, surface area, and quality of the interfacial boundary (Kalachandra, 1989; Ferracane, 2006).

In addition to properties, investigators have shown that the different compositions of RCs are likely to have an impact on RBS. Loomans and colleagues investigated RBS on five classes of commercial RCs that differed in composition, and most importantly filler size. The authors concluded that RC composition greatly influenced the reaction of RCs to surface treatments, and so impacted upon RBS, to the extent that a universal repair protocol could not be suggested (Loomans *et al.*, 2011b). Further, a number of investigators have demonstrated the difference between two RCs belonging to the same classification, nanohybrid. The RCs showed a significant difference in RBS even though both were subjected to the same treatment; the RCs with higher filler concentration exhibited higher RBS than those with lower filler concentration (Rinastiti *et al.*, 2010).

RCs are different in their composition and this likely impacts their repairability, it is therefore important that studies base their design and then consider conclusions on the wide classification of RC types. Most of the studies on RC repair have not tested more than one to three RC types and, based on a review of the published work, there was only one which has studied five RCs (Loomans *et al.*, 2011b).

3.6.7 The effectiveness of RC restoration repair in vivo

Robust, well-designed clinical investigations in dentistry should ideally underpin the evidence base in supporting any intervention a clinician undertakes. A multitude of

corroborative evidence in clinical trials in dentistry is a rarity, and significant emphasis is still placed upon the controlled environment of a laboratory. In contrast to the laboratory environment, which is designed overall to be uni-environmental, clinical experimentation leads to interventions being tested in a multi-layered and complex environment. This, in turn, leads to challenges in controlling the covariant of any investigation and subsequent interpretation of the findings. The resulting problem is therefore that much of the evidence on the repair of direct RC restorations is not based on robust clinical studies, which might support the wider implementation of this more minimal intervention (Sharif et al., 2010b; Gordan, 2013; Hickel et al., 2013). Sharif et al. reviewed the literature in 2009 to search for evidence on the effectiveness of the repair versus replacement of direct RC restorations in permanent premolar and molar teeth. None of the reviewed clinical studies were included under the criteria of randomised clinical trials or controlled clinical trials. However, they concluded that the identified studies might indicate the effectiveness of alternative treatments, including repair, which facilitates increased longevity of the restorations and teeth with minimal intervention after two years of follow up (Sharif et al., 2010a).

Undertaking randomised clinical trials may not be feasible for many areas of investigation, and, in relation to longevity, a RC repair appears to be one such area. Key issues supporting this stance relate to the randomisation of treatment groups, with respect to the variety of defect types which could be indicated for repair, for example a marginal gap or bulk fracture. In this scenario, it is likely that the one treatment procedure may not be effective in all situations. Further, defect sizes can be difficult to control during randomisation or stratification, which could lead to bias. Adequate sample size, meaningful outcome measures, and loss to follow-up are further problems generally related to randomised, controlled trials (Sharif *et al.*, 2010b). Ethical issue likely to be another barrier for identifying treatment groups because some patients may refuse repair even the restoration condition indicated for repair. However, a number of studies evaluated the effectiveness of RC restoration repair.

Two clinical cohort studies have reported on the effectiveness of repair in comparison to replacement over a longer follow-up of up to seven years (Gordan, 2009) and ten years (Fernández *et al.*, 2015) in the University of Florida College of Dentistry and Dental School of the University of Chile individually. Fernández *et al* repaired class I

and class II RC restorations using self-priming bonding agent in comparison to the same classes but treated with replacement. The longevity and success of repaired restorations was similar to the replaced restorations for marginal adaptation, secondary caries, colour and anatomic form. Filtek Supreme was used as the repair material. Gordan repaired class II, III, IV and V using phosphoric acid etch and bonding agent and the results showed that repair is a viable treatment as it can increase the longevity of the restoration. Over the follow up time, the failure rate of repaired restorations was zero, and 21% for replaced restorations. Filtek Z250 was used as repair material. The studies did not use any mechanical roughening, except for a cutting bur to remove any defects around and within the RC restorations. Further investigations have included the repair of large class two preparations with three to five surfaces, where only phosphoric acid etching and the application of an adhesive system were used, without any further mechanical roughening. The results after four years suggested that the performance of repaired restorations could enhance the longevity of the RC restorations and they also found that the repair of secondary caries survived longer than those repaired due to fracture. The annual failure rate for repaired RC restorations was 5.7 %, while for amalgam it was higher at 9.3 %. Two types of hybrid RCs were used as repair material for RC restorations which were Clearfill photo posterior, Clearfill AP-X (Opdam et al., 2012).

Overall, from the reviewed literature, we might conclude that the repair of RC restorations has been a successful treatment in comparison to replacement, while there is variety in many aspects relating to the repair of RC restorations. These include defect size, type, and method of repair, and indeed many have not been investigated *in vivo* in either a controlled or combined way. A further question that also arises is whether a randomised controlled clinical trial can be designed and delivered to cover this highly variable topic of restoration repair.

3.7 RC repair: current recommendations

Based on the limitations identified in this chapter, the following recommendations should be considered by future studies on RC repair.

 Identification of the surface treatments that are used by dentists in clinical practice. RC repair has been adopted by clinicians but there is limited information on the types of surface treatments they undertake during RC restoration repair. This information will allow investigation of their effectiveness, and these findings have the potential to be used as a guide for RC repair in clinical practice.

- 2. Identification of whether dentists repair fractured RC restorations. This is for two reasons. Firstly, the repair of fractured RC restorations has been taught in dental schools, but there is evidence that dentists are less likely to repair them compared to secondary caries. Secondly, fractured RC restorations are likely to leave larger cavities than other defects such as a marginal gap and wear. Therefore, dentists should be asked about the repair of fractured RC restorations as these fractures may challenge dentists more than other defects.
- 3. Standardisation of samples and thorough description of them before and after surface treatments. A drawback within current *in vitro* studies on RC repair is their heterogeneity; one of the main factors is that most of these studies did not describe their samples before and after surface treatment, to enable a meaningful comparison between the findings. Additionally, another factor in the heterogeneity is that different SiC paper grades were used to prepare the samples before surface treatment, and again there has generally been no description of the samples before the application of surface treatments. Thus, SiC grades may act as a confounding factor for the RBS.
- 4. Increased understanding of the effectiveness of surface treatments. There is limited understanding of the individual, cumulative, and synergistic effect of surface treatments on the level of RBS *in vitro*. Once there is adequate information, then the design for a clinical study may be easier.
- 5. Inclusion of the different types and categories of RCs used in clinical practice, or those which have the potential to be used in future in their study design. Of the published *in vitro* works, most examined one RC type, and a few examined two to three RCs. In addition, RCs have varied compositions, even within one category. Previous works have demonstrated that the composition of RCs is a significant factor affecting RBS. This approach has two potential advantages: the results from such studies will probably be more generalisable to the clinical situation, where different types of RCs are in use; and this approach may clarify the effectiveness of the categorisation of RCs for implication in the design of future studies and clinical practice.

3.8 Summary of the literature review

The repair of RCs has been investigated both *in vivo* and *in vitro*. All the *in vivo* studies focused on evaluating the longevity of repaired RC restorations, but did not evaluate the effectiveness of surface treatments. Investigators have shown the surface treatments undertaken when dental schools teach RC repair, but there is limited information on the surface treatments undertaken by dentists during RC repair in clinical practice.

In vitro studies have evaluated surface treatments by ranking them for their effectiveness in increasing the level of RBS or understanding the mechanism of action of surface treatments. A number of approaches have been used for increasing understanding of the mechanism of action of surface treatments, with the most widely used approaches being the calculation of roughness and qualitatively assessment of RC samples for surface morphology. Because *in vitro* investigations have been performed using a wide variety methods, particularly for sample preparation and ageing, there is heterogeneity in the findings. As such, a clear conclusion has not been drawn on the effectiveness or the mechanism of action of surface treatments. Another limitation of *in vitro* studies is that most included one type of RC in the study design. Because there is a wide variety of RCs, the studies' findings are limited and less relevant to the clinical situation, where different types and categories are used.

Chapter 4. Investigating the clinical practice of general dental practitioners in repairing RC restorations

4.1 Introduction

Direct RC restorations, like other restorative materials, are susceptible to defects and failure (Ferracane, 2013). In most cases, these defects start gradually either adjacent, near or within the restoration. At this stage, when the extent of defect is not large, an alternative repair treatment instead of replacement is usually advocated. This is because repair can elongate the lifetime of the restoration (Casagrande et al., 2017), reduce treatment time and cost for the patient (Kanzow et al., 2016), and lower the risk of pulp and tooth-structure damage (Hickel et al., 2013). These points may mean that patients also strongly prefer repair (Kanzow et al., 2017a; Kanzow et al., 2017b). The evidence in the literature is that dentists do repair in clinical practice in different countries. For example, Gordan et al. (2012) found that fewer than one third of dentists perform repairs in the United States and Staxrud et al. (2016) found more than half of dentists do repair in Norway. However, the lack of robust evidence or an accepted protocol for repair on an effective repair protocol may be challenging for a clinician when choosing to repair or replace (Sharif et al., 2010b). Furthermore, the experience and thoughts of dentists who do repair has not been investigated fully. Therefore, a cross-sectional study was performed to collect data in the North East of England to describe current practice on RC repair at a single time point.

In the present study the objective was to develop a self-administered questionnaire to investigate the potential repair methods used by dentists in the clinical practice. Postal questionnaires are a reproducible method of collecting information on the practice of dentists. A number of authors have used postal questionnaire to collect information on dentists practice or attitude on aspects of dentistry other than RC repair (Iqbal and Glenny, 2002; Leggate and Russell, 2002; Field *et al.*, 2009). In addition, self - completed postal questionnaires have been shown to have a higher possibility of disclosing sensitive information compared to interview and electronic modes (Bowling, 2005). An alternative approach is electronically sending

questionnaires. This method has been used to collect information about RC repair at dental schools (Blum *et al.*, 2012a; Blum *et al.*, 2012b; Kanzow *et al.*, 2018). The main advantages of electronic questionnaire may be that they are cheaper and a larger number of participants could be engaged compared to the postal one, but it requires a sampling frame which might not be feasible with every study. Further, it has been suggested that electronically sending a questionnaire may not increase the response rate in comparison to postal questionnaire (Leece *et al.*, 2004).

In the present study a case-based scenario was included on managing a fractured RC restoration in the anterior tooth. The questionnaire contained questions about demographic characteristics and RC restoration repair based on the scenario. The first question on repair was about whether they do repair or not. The participants who do repair were asked to answer three additional questions, two of them were on approaches for RC repair, mechanical and chemical preparations.

Objectives

- To identify whether respondents are performing repairs to fractured RC restorations.
- To identify whether dentists use mechanical and/or chemical preparations during RC repair.
- To determine the factors that are important for respondents when considering whether to repair or replace fractured restorations.

Hypotheses

- Dentists are performing repair of fractured RC restorations.
- Dentists are using multiple methods to undertake repair of restorations.
- Dentist are motivated by a number of factors to repair restorations.

4.2 Materials and Methods

4.2.1 Questionnaire design

A questionnaire was designed to collect information from dentists about their practice for RC repair. To support the design the relevant literature was reviewed for types of questionnaires used in studies related to RC repair. The studies identified that used questionnaire to collect information on the practice of RC repair were performed on dental schools and a cohort study within a vocational dental program at the time of this study (Blum et al., 2005; Blum et al., 2012a; Blum et al., 2012b). The limitations of the questions were that within dental school studies the questions were thought to be too long to be asked in the dental practice, and the cohort study included very limited answer options for the questions asking about methods of repair. For example in the cohort study (Blum et al., 2005) the answer options for mechanical surface preparation were mostly related to finishing instruments while in clinical practice there are a variety of cutting instruments available along with polishing and finishing instruments. In addition, there were no questions on the demographic characteristics of the participants giving background of respondents, and there was a lack of questions related to patient factors that may affect repair decisions. Furthermore, the questionnaires used in the former studies were designed to specific aims of the studies.

Therefore, a new self-completed questionnaire was designed for current study. The basic requirements, instructions and reducing bias for a questionnaire were considered during the design using three published works (McColl *et al.*, 2001; Fink, 2003; Choi and Pak, 2004). The question topics and answer options were drafted with the guidance of the expertise of the supervisory team, to ensure that the questions were modelled to fall within the dentists' conceptual framework in the UK and particularly in the North East of England. The questionnaire consists of five topics and thirty-four answer options. The questions and answer options are presented in Table 4-1.

Topic 1 (demographic characteristics): Question 1: Please tick all responses that apply to your current practice. o NHS practice. o Private practice. o NHS and Private practice. o Multiple working dentists. o Single handed dentist. Question 2: Do you have any postgraduate qualifications? o None. o MSc. o MFDS. o Other. Topic 2 (treatment decision): Question 1: What would be your most likely treatment decision (based on the provided scenario)? o Full replacement. o Smoothing the fractured surface. o Repairing the fractured surface. Topic 3 (mechanical preparation): Question 1: Do you routinely mechanically prepare the fractured surface? o No. o High speed coarse diamond preparation. o High speed fine diamond polishing. o Slow speed disc polishing. o Microabrasion/sandblasting. o Other. Topic 4 (chemical preparation): Question 1: Do you routinely chemically prepare the fractured surface? o No. o Rinse with water. o Solvent (acetone, ethanol or chloroform). o Phosphoric acid etch. o Hydrofluoric acid etch. o Composite priming/bonding agent. o Silane coupling agent. o Flowable composite. o Other. Topic 5 (factors important to do repair): Question 1: What factors are important to you when deciding how to repair a fractured composite restoration? o How long ago the restoration was placed. o If I knew the restoration was sound underneath. o The size of the restoration. o The size of the fractured piece. o If natural tooth tissue was also involved in the fracture. o Heavy occlusion or parafunctional habits. o Presence of tooth surface loss or gastric reflux/vomiting. o Presence of bruxism habits. o Other.

Table 4-1. Key topics of the questionnaire and their answer options.

The proportion of dentists was measured by asking the participants whether they perform repair or not. Potential repair protocols were identified by asking the types of mechanical and chemical preparations during repair. Similarly, they were asked about factors that are important to them when repairing. The wording of the questionnaire sought to provide clear instructions, so that it would be accurately and consistently interpreted by the participants. Each question asked about only one topic, and question sequences were also considered, to enhance participation and reduce the potential of missing answers. In the questionnaire, the most important questions were put first, and the least at the end. Demographic characteristics were asked before the questions on RC repair in order to determine if the respondents were similar to the representative sample. The questionnaire's appearance was made simple to allow the readers to follow the questions easily. Blue, black and white colours were selected to identify the questions, and answer option boxes were provided for responses. Questions were closed-ended, with a list of answer options, except for three questions which included the option of comment. This option allowed participants to write down other options that they considered legitimate which had not already been identified within the questionnaire. A small section of the questionnaire was identified for office use, where the question and answer options were coded, and these were also separated from the respondent part by a white line, above which was written "Office use only". The questionnaire was designed to be completed within five minutes, because the area where dentists work is usually busy and we wanted to reduce questionnaire fatigue. The first draft of the questionnaire is shown in Appendix A.

4.2.2 Refining and piloting of the questionnaire

The question and answer options were revised by an experienced researcher, independent of the research team, who reviewed the first draft and gave his opinion on how accurate the questions were in relation to the aim of the study. Feedback was used to amend the wording of the questions, provide additional response options for demographic characteristics, and add more detail to the case-base scenario on the symptoms of the case. Also advice was given for numbering of the questions on RC repair which were repair decision, mechanical and chemical preparations, and factors important to do repair. These comments were used to modify the questionnaire before piloting it.

To check the ease of understanding of the questions and that the questions were properly worded and could be interpreted in relation to the aim of the study by participants, two local dentists with similar characteristics to the proposed target population were invited to complete the questionnaire. These dentists were clinical teachers in the School of Dental Sciences at Newcastle University, UK, but worked for the majority of the week in primary care. The two dentists provided comments via a list of questions shown in Appendix B. One participant commented that the picture did not clarify the reason for the fracture, nor indicate the size of the restoration. The second participant preferred to be able to identify the size of the restoration from the picture, as size of the fractured RC was important in her decision to repair or replace. Then, this feedback was used to modify the questionnaire further, and based on their comments the picture was removed from the guestionnaire as it appeared to confuse rather assist them with the questions. In addition, within the pilot study the time spent on the questionnaire by participants was monitored, and it was supposed to be no more than five minutes. The final draft of the questionnaire was created based on the all comments, as illustrated in Appendix C.

4.2.3 Study location and research team

The questionnaire was sent out to the clinical practices of dentists based in the North East of England, UK. The area is geographically diverse in relation to the provision of primary care dental services.

4.2.4 Sample population

Dentists working in primary care dentistry in the North East of England, UK, were invited to take part in this study. The GDC (UK) website was used to obtain names and contact addresses of dentists and generate a master list of 1,059 registrants. North East England was then defined in five geographical areas, as the distribution of dentists appearing to vary between areas. A stratified random sample of 100 names (approximately 10% of the number of dentists in the north east) was generated. This was based on the number of dentists in relation to postcode areas in the North East of England (postal codes beginning NE, DH, SR, TS and DL). A stratified sample approach was undertaken to ensure that the sample was representative of the population in relation to their geographic spread in the region. The approximate

distribution of dentists between each postcode were as follows: NE 70%, DH 10%, DL 5%, TS 5% and SR 10%. Within each postcode area, random sampling was undertaken by taking every tenth name in each area, with the exception of TS, where every third name was used. This approach to sampling was chosen to ensure an adequate number of dentists were included within the sample in terms of their proportion in the TS area.

4.2.5 Questionnaire administration

Before sending the questionnaire packs out to potential participants, an initial introductory letter was sent to all selected dental practices on October 30th 2014, see Appendix D. This letter aimed to prime practices about the study and to ask them to prime dentists working in clinical practice about the questionnaire when they received it. It was hoped that by raising the profile of the study, the questionnaire pack that was to follow was not just discarded on receipt at the practice. Initial packs were sent out to the identified participants on March 23rd 2015. There was a delay in sending the questionnaire pack out to the dentists from the time of sending the prenotification introductory letter. This delay was due to further editing of the questionnaire and printing the questionnaire pack over the Christmas holiday.

Participants approached in this study received a questionnaire pack, containing the questionnaire itself (see Appendix C), an introductory letter (see Appendix E), an information sheet (see Appendix F), and a consent for future contact form (see Appendix G). A stamped self-addressed envelope was included for returning the completed questionnaire and other documentation.

Eight weeks after posting of the questionnaire pack, a reminder letter was sent to any non-respondents, see Appendix H, to try to increase the response rate. The non-responders were also asked to contact the investigators further if they were willing to participate in this study and had lost the questionnaire. The contact details of the non-respondents were confirmed against the GDC website to check for the possibility of a change in contact addresses during the follow up stage. The data collection phase of this study was closed on 15th June 2015.

4.2.6 Inclusion criteria

The following inclusion criteria were applied in this study:

- Current registrant dentists were identified via the UK General Dental Council (GDC) list;
- Dentist were identified with a professional address in the North East of England, UK.

4.2.7 Exclusion criteria

The following exclusion criteria were applied in this study:

- Professional addresses outside of, or not working in, the North East of England, UK
- Dentists who had retired
- Dentists who were no longer working in clinical practice
- Dentists who would be not undertaking any restorative dentistry for an extended period of time.

4.2.8 Data management and confidentiality

An initial spreadsheet was generated in Excel based on the questionnaire contents, such as question number, answer options, and coding for data analysis. The detail of this spreadsheet (codebook) is described in the following sections.

The spreadsheet contained an identifier number for each participant; for the first participant in our sample, 1001 was the identifier number, and for the second, 1002, and so on until the last participant in our sample was given 1100 as their identifier number.

Each question and its corresponding answer options were given a label, name and coding. For example, one question on the demographic characteristics in the questionnaire, 'Do you have any postgraduate qualifications?' was named

postgraduate qualification and coded number 3 in the spreadsheet. Answer options for each question were given alphabetic coding, for example the coding for the answer options of question 3 were A, B, C, D, E, F. Accordingly, the spreadsheet included identifier numbers, question names, question labels, and coding for the answer options.

After closing the data collection process, the final spreadsheet was developed to be fit to undertake the data analysis. For each questionnaire, returned data were coded for each answer option mark (either yes (coded as 1), no (coded as 0) or missing (coded as 99).

Data entry was verified to ensure the accuracy of data transcription. Data were initially entered by hand and then all entries were reviewed a second time against the paper questionnaire. This was then followed by a further screening of 10% of the questionnaires for transcription errors, incomplete data or incorrectly formatted data. Finally, responses from those participants who did not fit our inclusion and exclusion criteria were removed. Based on this process, five participants were excluded as they did not meet the inclusion criteria, the respondents were student, retired, having another specialty that they do not do repair such as surgeon and orthodontics and the final respondent written not working as dentists currently. Therefore, from 52 respondents, 47 questionnaires were used in the final statistical analysis.

For confidentiality purposes, each questionnaire had an identifier number as described before. The identifier number was used during the process of data management. The participant's name and address were kept in a separate spreadsheet and Excel file retained on a password protected computer-based data system (Newcastle University servers), and were not used for the reporting or dissemination of the research findings. During the follow up process, this file, to which only the research team had access, was reopened to identify the address of non-respondents.

4.2.9 Statistical analysis

Descriptive statistics were undertaken to give frequency and relative frequency as proportions for each of the responses from the questionnaire and presented in graphical and table format using Excel software.

4.2.10 Ethical review

This study was reviewed and was given a favorable opinion through the Newcastle University Ethical review Combined Ethical Approval Form Version 1.3 (03/09/2014) code number (753137).

4.3 Results

4.3.1 Demographic characteristics

From the 47 respondents to the questionnaire, all of them (47) answered this question. The demographic characteristics according to clinical practice type, number of dentists within practice, and type of postgraduate qualifications held of the forty-seven respondents are shown in Table 4-2. The distribution of the type of practice in which the responded dentists worked appeared to be split almost equally between NHS practice only and mixed NHS and private practice, while few dentists worked in private practice. In regard to the number of dentists within practices, half of the respondents stated that their practice was of the multiple working dentists, while a minority reported that they worked as a single-handed dentist. Nearly half of the respondents did not answer this question. Analysis of postgraduate qualifications showed nearly half of the respondents reported no postgraduate qualifications, while the remainder held different types of postgraduate qualification. Those respondents who held postgraduate qualification, most of them held one type of postgraduate qualification, as shown in Figure 4.1

Demographic characteristics		Frequency	Relative frequency %	
Practice type of respondents				
	NHS practice	20	43	
	Private practice	2	4	
	NHS and Private practice	25	53	
Number of dentists within practice				
	Multiple working dentists	24	51	
	Single handed dentist	4	9	
	Missing response	19	40	
Postgraduate qualification				
	None	24	51	
	MFDS	12	26	
	Other	11	23	
	MSc	3	6	

Table 4-2. Demographic characteristics of the respondents based on type of practice, number of dentists within practice and postgraduate qualification.

Frequency and relative frequency as proportion of the respondents to each answer option in every question was calculated based on the number of respondents to this question (47). Total frequency of respondents to every question (type of practice, number of dentists within practices and postgraduate qualification) on demographic characteristics was 47 but answer options in postgraduate qualification is more than 47 respondents because some of the respondents showed to have more than one type of postgraduate qualifications.



Figure 4.1. Number of postgraduate qualifications for respondents held postgraduate qualification.

The relative frequency as proportion and frequency, within brackets, of respondents for number of postgraduate qualifications they hold was calculated using descriptive statistics.

4.3.2 Treatment decisions

From the 47 respondents to the questionnaire, all respondents gave a response to this question and are summarized as Figure 4.2. The distribution of the responses indicates that almost two-thirds of the respondents would repair a fractured restoration, just over one quarter would replace the whole restoration, but none of them would simply smooth a restoration.



Figure 4.2. Treatment decisions for fractured RC restoration based on repairing, replacement and smoothening.

The analysis based on responses to question one in the questionnaire (In this scenario what would be your most likely treatment decision?). The relative frequency as proportion and frequency, within brackets, of respondents for every answer option to treatment decisions was calculated based on the number of respondents to this question (47).

4.3.3 Surface treatment of repaired fractured direct RC restorations

The next section in the questionnaire was designed to ask specific questions about methods used by dentists to repair RC restorations. As 34 respondents indicated they repair fractured RC restorations data are presented from these respondents only (Figure 4.2).

Mechanical preparation

All 34 respondents answered this question on mechanical preparation of fractured RC restorations. From those who would perform mechanical preparation, more than three quarters reported that they would use one method for mechanical roughening, while around one quarter indicated that they would use two or three methods for mechanical roughening in their clinical practice, as shown in Figure 4.3.

The responses to the answer options of the mechanical preparation in the questionnaire, as shown in Figure 4.4, less than one-tenth do not perform mechanical preparation before the application of a repair material. However, the majority of the respondents do perform mechanical roughening using a variety of methods. The use of a coarse diamond bur was the most popular preparation used by three-quarter of respondents. Less than quarter repair fractured RC restoration using polishing discs or polishing diamond bur and less than one-tenth use microabrasion.



Figure 4.3 Number of methods used by respondents during mechanical preparation, prior to repairing a fractured RC restoration.

The relative frequency as proportion and frequency, within brackets, of respondents was calculated based on the number of respondents to this question (34).



Figure 4.4. Mechanical preparation methods used by respondents who repair fractured RC restorations.

The data analysis based on responses to question two in the questionnaire (Do you routinely mechanically prepare the fractured surface?). The relative frequency as proportion and frequency, within brackets, of respondents was calculated based on the number of respondents to this question (34).

Chemical preparation

Thirty-three from the 34 respondents that would repair fractured RC restoration answered this question. Three types of data analyse was performed on the answer options from this question in the questionnaire. The first analysis concerned the number of methods that respondents use during repair. Figure 4.5 shows that one, two or three methods of chemical preparation were used by dentists. This generally split the respondents into three equal groups.

The second analysis was performed to determine the responses to specific chemical preparation methods given in the questionnaire. Based on the findings from Figure 4.6, more than three-quarters of the respondents reported the application of a composite priming/ bonding agent, and more than half applied a phosphoric acid etch. No respondents reported the use of a solvent or a silane coupling agent prior to RC repair. The other chemical preparation methods such as water, flowable composite and hydrofluoric acid were used less often.

Third data analysis presented the clinical processes of chemical preparation used during RC repair are summarised in Figure4.7. Nearly one-third of respondents reported that their clinical processes included acid etch with composite prime/bonding agent. Approximately one-quarter reported only using composite prime/bonding agents, whereas, less than one-tenth of respondents used phosphoric acid etch without composite priming/bonding agent. None of the respondents reported they rinsed with water alone, as this was only used in combination with either phosphoric acid etch and/or composite priming/bonding agents. Hydrofluoric acid etch was used by a minority of the respondents followed by application of composite priming/bonding agents.



Figure 4.5 Number of methods used by respondents during chemical preparation, prior to repairing a fractured RC restoration.

The relative frequency as proportion and frequency, within brackets, of respondents

was calculated based on the number of respondents to this question (33).



Figure 4.6. Chemical preparation methods undertaken by respondents prior to repair of fractured RC restoration.

The analysis based on responses to question three in the questionnaire (Do you routinely chemically prepare the fractured surface?). The relative frequency as proportion and frequency, within brackets, of respondents was calculated based on the number of respondents to this question (33).



Figure 4.7. Processes for chemical preparation methods prior to repairing fractured RC restoration.

The relative frequency as proportion and frequency, within brackets, of respondents was calculated based on the number of respondents to this question (33).

4.3.4 Response to the scenario

All 34 respondents undertaking restoration repair answered the question which had been designed to improve understanding of the background factors related to the patient or restoration that might be considered important for the respondents to consider prior to the repair of a fractured RC restoration.

According to the findings in Figure 4.8, most of the factors were nearly equally identified by respondents as being important in the decision about whether to do repair. A sound restoration underneath was the most frequently chosen factor. Fractured restorations due to bruxism habits and gastric reflux were of less often considerated in repair or replacement compared to other factors.



Figure 4.8. Factors that respondents considering during repair of fractured RC restoration.

The analysis presented responses to question four in the questionnaire (What factors are important to you when deciding how to repair a fractured composite restoration?). The relative frequency as proportion and frequency, within brackets, of respondents was calculated based on the number of respondents to this question (34).
Summary of the findings

1- Demographic characteristics showed most of the respondents worked in the NHS or NHS and private practice, while the minority worked in private clinics. Half said they were multiple working dentists and they had no postgraduate qualifications.

2- More than three-quarters of the respondents performed repair, and less than onethird performed replacement for fractured RC restorations.

3- Most of the respondents who did mechanical preparation used one mechanical preparation method, and the selection of the method differed between the respondents, although the most widely used ones was a high speed coarse bur.

4- All the respondents who repaired RC used various chemical preparation methods, within which they used one or two or three methods. The most common methods were composite priming/bonding agent and phosphoric acid.

5- Respondents stated that a number of factors were important to them when they repair RC restoration, and the most commonly identified factor was sound restoration under the restoration, while bruxism habits and gastric reflux were less often considered by respondents.

4.4 Discussion

In this chapter, response rate, demographic characteristics, treatment decision, surface treatments and factors affecting treatment decision of dentists is discussed when they undertake RC restoration repair.

In the current study the response rate was relatively low at 52% after one reminder. A number of studies surveyed dentists for RC repair in the clinical practice and showed approximately similar or lower response rates. Staxrud and colleagues' study reported an approximately similar rate of response (55.8%) after three reminders. These authors sent questionnaires electronically to dentists in the public service in Norway and asked about RC repair (Staxrud et al., 2016). Another study concerned with RC repair by dentists in clinical practice in Greece used a mail questionnaire, and had a 40% response rate after two reminders, which is lower than those in the current study (Maria et al., 2017). According to the reviewed literature, similar studies that were performed in the clinical practice on topics other than RC repair showed higher response rates. A study by Field et al. (2009) in the North East of England in the UK, in the area of study similar to the current study, used a postal questionnaire; when they asked dentists about provisional implants, the response rate was 74%. In addition; another study by Leggate and Russell, interested in understanding general dental practitioners and evidence-based dentistry, used a postal questionnaire in Scotland in the UK, and the rate of response was higher (70%) than the current study (Leggate and Russell, 2002). The higher response rate of the former two studies in comparison with the current study may be related to the subject being more interesting to the participants than RC repair. Generally, it is accepted that there is no clear required response rate in the literature; 25% and higher response rates are recommended as long as there is no simultaneously high percentage of missing data (Fink, 1995). However, it has been identified that a higher response rate is more precise and reduces the risk of bias from low response (McColl *et al.*, 2001). Accordingly, in the current study a number of approaches were used to enhance the response rate, such as: using the Newcastle University logo on the questionnaire; pre-notifying dentists with a letter that included the signature of the research team; employing a short questionnaire requiring just five minutes to complete; sending a second copy of the questionnaire as a follow up; using an

anonymous questionnaire; assuring confidentiality; and using coloured ink and high quality paper with no staples. These strategies were used in another study that collected clinical practice information in the same area of current study (Field *et al.*, 2009). This would suggest that in comparison a good rate of return was achieved in the current study from a sample population, which was difficult to engage with a postal questionnaire.

Based on the demographic characteristics of the participants, the identified sample are representative of the population of the dentists in primary care in the UK. The findings showed that most of the responded dentists worked in primary care in the NHS (43%), or in both the NHS and private sector (53%) or in private sector only (4%), either in the form of multiple working dentists or single handed dentists. In addition, 51% did not have any postgraduate qualifications but the other half holding different types of postgraduate gualifications. The findings for the practice types approximately match another study's findings that was performed in the North West of England in which nearly half of the dentists works in primary care and less than one-tenth work in the private sector and the remained part worked in both primary and private care (Iqbal and Glenny, 2002). Furthermore, a study that was performed in the Scottish dental practitioners showed a lower proportion on postgraduate qualifications compared to the present study, where one-sixth of dentists showed further postgraduate qualifications (Leggate and Russell, 2002). The difference in the area and time may have affected the results. This finding suggests the sample is representative of the population in the clinical practice. Not to forget, there were descriptive terms from the NHS (National Health Service) on the demographic characteristics of dentists in the UK, but this information was not available at the time of this study. If there was information on demographic characteristics of dentists within NHS, I would compare the demographic characteristics of my sample to those in the NHS or I would able to get demographic characteristic of my sample from NHS without asking about it in the questionnaire.

According to a case-base scenario in the questionnaire, it was found repairing RCs as a concept is an acceptable and practiced technique for a large number of general dentists in the North East of England. A large proportion of the sample indicated that they repaired fractured RC restorations, while around one third did not include it in their current practice Figure 4.2. This finding is in agreement with more recent studies in the literature. Staxrud et al showed dentists in the public dental service in

Norway preferred to repair (89.6–54.1%) rather than replace a fractured restoration (Staxrud et al., 2016). Although Staxrud et al study's results on dentists' attitudes to repair are comparable with current study, they are not of the same background and the way of questioning was different from that in the current study. Similarly, another study by Maria et al. (2017) showed 86.8% of respondents repaired restorations that included RCs. In contrast, the current study had a higher rate in the population of repairing RCs compared to that reported by Gordan and colleagues, who found that one third of dentists in their practice-based research network undertook repair (Gordan et al., 2012). A possible reason for the difference between Gordan et al. and the present study may be that the current study was performed 3–5 years later; moreover, its findings may have been affected by the number of recently graduated dentists in practice who had been taught repair in dental school (Blum et al., 2012a), and vocational dental practitioners' training programs (Blum et al., 2005) in the UK. Those respondents who did not repair probably did not accept it as an effective alternative to replacement at the time of the current study. However, the current sample size was relatively small that a proper proportion of dentists that do repair in the North East of England could not be determined. The finding indicates that large number of the responded dentists do repair that should give insight in to the potential methods that they use during repair.

To determine the potential methods that dentists use during RC repair, the dentists were asked about the types of mechanical and chemical preparations, the respondents showed they use a variety of instruments and materials for that purpose Figure 4.4 and Figure 4.6. This finding is consistent with that of Maria *et al.* (2017) and Staxrud *et al.* (2016), who showed the respondents use both mechanical and chemical preparations. However, Staxrud et al did not ask on repair methods based on any case-based scenario, and did ask about the strategies and number of steps for enamel-dentine adhesive system without asking about mechanical preparation except preparation of extra retention in adjacent restoration. In addition, Maria *et al* asked about the mechanical and chemical preparation when to undertake repair of marginal defect of RC restoration and RC veneer only.

For mechanical preparation Figure 4.4, it appeared there was inconsistency between respondents on the mechanical preparation methods used. More than three-quarters of the respondents who do repair use one method of mechanical preparation while less than one-quater use either two or three methods Figure 4.3. Then according to

the findings in Figure 4.4, high speed coarse diamond preparation used by more than half of the respondents then followed by polishing instruments either polishing disc or fine diamond polishing bur. Whereas least number of respondents use microabrasion and less than one-tenth do not do any mechanical preparation. This finding indicates most responded dentists use a method to roughening the fractured surface by a coarse diamond bur or smoothening the fractured surface using polishing instruments and minority do not do any type of smoothening or roughening. Accordingly, it can be suggested more than half of the respondents that use one method to roughen the surface then from the remained percent most of them smoothen the surface while the least percent do microabrasion. The least use of microabrasion by our respondents is an unexpected finding, not only because most of the modern clinics in the NHS and private sector use microabrasion, but half of the responded dentists also stated they work in private and NHS sector (Wong and Winter, 2002; Ashfaq et al., 2019). It is believed the low proportion of respondents used microabrasion is not related to the lack of the device in their practices. The least use of microabrasion is likely related to their behaviour and attitude rather than availability of the device. These results align with what is reportedly taught in dental schools, in that the majority used mechanical roughening over microabrasion; however, most of the schools did not define the instrument types used in mechanical roughening (Blum et al., 2003b; Gordan, 2003; Blum et al., 2012b; Blum et al., 2012a). The use of rotary polishing instruments by our respondents is in alignment with the mechanical preparation reportedly used by vocational dental practitioners and dental schools in Germany (Blum et al., 2005; Kanzow et al., 2018). The use of roughening and polishing instruments is likely related to the taught programs in the dental schools and vocational training practice because more than half of the respondents showed they do not have any postgraduate gualifications. Another explanation might be related to peer review on treatment options. Furthermore, less than one-tenth showed no use of mechanical preparation. This finding contradicts the taught programs in the UK at dental schools (Blum et al., 2003b; Blum et al., 2012a) but its consistent with the vocational training program (Blum et al., 2005). Further, based on the reviewed published works within in vitro there is conflicting findings when the bond strengths achieved between roughening by microabrasion or bur compared to negatives of no preparation control (Brendeke and Özcan, 2007; Özcan et al., 2013a; Wendler et al., 2016). Therefore this may explain the use of different mechanical preparations being related to the lack of robust evidence.

Similarly, the chemical preparations used by respondents were diverse in the number, types and processes. More than one third of the respondents use two types of chemical preparation while less than one third performed one or three types of chemical preparation Figure 4.5. Based on the types of chemical preparation in Figure 4.6, the most widely used one is composite priming bonding agent then phosphoric acid, while none of the respondents indicated that they use silane or solvent. Further analysis for chemical preparation based on the processes that respondents used are shown in Figure 4.7. The highest frequency is seen for phosphoric acid etch with composite/priming bonding agent. With the exception of phosphoric acid surface treatments, the findings of adhesive system processes appear to be consistent with what is taught in the dental schools (Blum et al., 2003b; Gordan, 2003; Blum et al., 2012a; Blum et al., 2012b) and performed in the Norway dental practice (Staxrud et al., 2016). While, the use of phosphoric acid as a single surface treatment contrasts with the accepted processes of RC adhesive system (self-etch and etch and rinse). None of the respondents reported use of silane which is contradicting both dental schools and vocational dental program findings. The findings from the present study related to the use of phosphoric acid as a single surface treatment and no use of silane suggest a diversity in the approach of applying chemical preparation that has not been reported by any of the reviewed published work in the literature. Within the reviewed in vitro studies, generally there is conflicting findings for including phosphoric acid particularly within adhesive processes (bonding agent only, self-etch adhesive system) (Cavalcanti et al., 2007; Kashi et al., 2011; Loomans et al., 2011b; Irmak et al., 2017), and no published work investigated the effect of phosphoric acid as the only chemical surface treatment.

Another finding from this study was that the decision of our respondents to repair or replace a fractured restoration appeared to be based on multiple factors which they considered to be important Figure 4.8. Having sound tooth tissue under a fractured restoration appeared to be the most important factor reported. This contrasts with the findings of Blum, who suggested that teaching in dental schools in the UK and Ireland recommends repair for both fractured restorations and those with secondary caries (Blum *et al.*, 2012a). Opdam and colleagues found that motivating factors for RC repair, such as fracture and secondary caries, were not a significant factor in repair longevity (Opdam *et al.*, 2012). This may indicate less awareness among these respondents of the success of secondary caries for repair. The factor that the

respondents considered to be of least important for their treatment decision was the presence of a heavy bruxism habit, tooth surface loss, or gastric reflex. The latter factors have not been considered by other studies, accordingly, this is the first study to determine patient-tooth related factors for RC repair. However, the reason behind the selection of these factors within this study was not determined.

However, the current survey study performed in a small area (North East England) in the UK, based to the best of my knowledge this is the first study that collected information on the mechanical and chemical preparations for fractured RC restorations within dental practice. Also, this study provided evidence on the number and types of methods used in primary care and to show diversity in the mechanical preparation used in the clinical practice compared to the taught program in the dental school. Some of the selected methods and factors for performing repair by respondents are interesting and warrant further investigation because there is no clear conclusion around superiority of any of them, even within *in vitro* studies. Providing evidence in relation to the methods used in the clinical practice may be an important step for improving clinical practice in RC repair.

In conclusion

- Dentists do perform RC repair in the primary care in the North East of England in the UK.
- Most of the dentists use both mechanical and chemical preparation methods during RC repair.
- Dentists showed a diversity in the use of repair methods.
- Sound tooth under the restoration is the most motivating factor for repair

Chapter 5. Investigation of surface treatments undertaken prior to RC repair

5.1 Introduction

Mechanical or chemical surface treatments were performed on RC before the application of repair materials (Valente *et al.*, 2016). The published literature has investigated different types and combinations of surface treatment, but the findings are diverse and conflicting. In addition, there are no recommendations on a particular surface treatment method for clinical practice, and the participant dentists used a variety of treatments for chemical and mechanical preparations, as reported in the questionnaire chapter in this thesis. The selection of surface treatments investigated in this chapter was based on the findings from the questionnaire, and what was found conflicts with the results discussed in the literature review. This study compared chemical and mechanical preparations to identify whether there is a need to increase roughness during RC restoration repair based on *in vitro* investigations. This approach has been evaluated by a number of authors (Brendeke and Özcan, 2007; Özcan *et al.*, 2013a).

This study used the standard methods seen in the literature to measure the effectiveness of comparative surface treatments in increasing RBS. The most widely used method to measure the effectiveness of RBS is by quantitative measurement using a shear or TBS test. In this study, shear bond strength was used to measure RBS and was compared with other studies. To understand the action of surface treatments, a number of studies have measured average roughness, such as Ra (Kallio *et al.*, 2013; Lima *et al.*, 2014; Valente *et al.*, 2015; Wendler *et al.*, 2016; Bahari *et al.*, 2018). The studies using this approach did not identify the link between changes in roughness profile and RBS (da Costa *et al.*, 2012; Ayar *et al.*, 2018). BAC parameters have been identified to be a more informative set of measurements for roughness profile than Ra, and if Ra is used alone, substantial differences in roughness profile may be unnoticed (Field *et al.*, 2013). Therefore, in this study, BAC parameters were used along with Ra. In addition to roughness, surface morphology has been evaluated by a number of authors and provided valuable information for the analysis of the RBS of the surface treatment performed (da Costa

et al., 2012). Accordingly, in the present study, surface morphology was evaluated for some of the surface treatments using SEM.

RCs have different filler sizes, which is an important factor in their roughness characteristics. However, studies have shown that when RCs are prepared with coarse to fine grades, they decrease in roughness (Schmitt *et al.*, 2016). Within RC repair studies, a review study reported RC samples have been prepared with SiC papers in a number of studies before the application of surface treatments using different grades, on the assumption of creating similarity between RC samples (Özcan and Koc-Dundar, 2014). However, it is not clear how the use of different SiC grades impacts on the consistency of roughness and the RBS of RCs with different filler characteristics. In this chapter, different SiC paper grades were investigated for their impact on consistency in roughness and SBS on five different RCs.

Objectives

- To investigate SiC grades for RC samples preparation before surface treatments.
- To investigate the cumulative effect of chemical preparation on RBS.
- To investigate the effectiveness of mechanical preparation by bur individually, and comparing them to RC samples prepared with P600 SiC.
- To investigate the synergistic effect of combined mechanical and chemical preparations in comparison to mechanical preparation alone.
- To investigate how the differences in surface treatments impact on the changes in roughness and SBS.
- To investigate the impact of roughness changes on the SBS.
- To investigate the usefulness of BAC parameters compared to Ra parameter.
- To investigate the performance of different RCs when treated with different surface treatments.

Hypotheses

- All SiC grades produce consistency in roughness, and this roughness does not impact on SBS.
- Phosphoric acid impact on roughness and result in statistically non-significant increase in SBS.

- Increase in abrasive particle sizes of surface treatments, increases roughness profile particularly increasing peaks proportions compared to valleys proportions.
- Increase in abrasive particle sizes, results in increase in SBS compared to the prepared RCs.
- Ra misrepresents roughness profile compared to BAC quantification.
- RCs perform differently when prepared with different surface treatments.

5.2 General method

In this section firstly, the RCs used in these experiments are shown in Table 5-1. The justification of the selection of these materials was that Filtek Supreme XTE, Heliomolar and Filtek Z250 contain commonly used monomer systems and Kalore and N'Durance contain newly developed monomer systems. Also the selected materials cover all groups of a classification system that would be based on filler size and distribution. It was thought that this choice included a diverse set of RC materials that might represent what is used in current clinical practice. Secondly, a number of methods described that apply for all surface treatments. Then the method of every type of surface treatment was described separated such as SiC, chemical preparation, mechanical preparation and combined chemical and mechanical preparations.

Resin composites	Classification	Monomer type	Filler size range	Filler% (WT/VOL)
FILTEK Z250				
universal	microhybrid	Bis-GMA, UDMA, Bis-EMA,	0.01-3.5 μm	82%/60%
A2		TEGDMA		
3M ESPE				
Filtek Supereme XTE				
universal	nanofill	Bis-GMA, UDMA, Bis-EMA,	0.004-10 <i>µm</i>	78.5% /63.3%
A3-Enamel		TEGDMA, PEGDMA		
3M ESPE				
GC Kalore				
universal	nanohybrid	UDMA, dimethacrylate ,	0.016 -17 μm	82 wt%
A2		DX-511 co-monomers		
Heliomolar				
A3	microfill	Bis-GMA, UDMA, D3MA	0.04-0.2 μm	66.7 %/46%
lvoclar vivadent				
N`Durance		ethoxylated Bis-GMA		
multipurpose	nanohybrid	UDMA,	0.01- 0.5 µm	80%/65%
A2		dicarbamate-		
Septodent		dimethacrylate dimer acid		

Table 5-1 RC materials used in this study: RC names, classes, matrix and filler type, size and concentration by volume and weight percentage.

5.2.1 Sample preparation

In this experiment, five RC materials were used, as shown in Table 5-1.

Standardised substrate samples of the materials were made using a ring mould of PTFE (polytetrafluoroethylene, diameter of 10 mm, 3mm high). RC was injected into the mould incrementally, which was slightly overfilled and covered with a translucent Mylar strip on the top and bottom surfaces (polyethylene terephthalate matrix strip (0.15 mm thick, Goodfellows, Cambridgeshire, UK). The RC was then compressed between two Perspex blocks (clear acrylic, Bay Plastics, North Shields, UK; 6 mm thick) by hand, to create a sample with minimal voids and a flat superficial layer. With the matrix strip *in situ*, the RC was then polymerised for 40 seconds on both the top and bottom surfaces using a light emitting diode light cure unit (Coltolux®LED, Coltene/ Whaledent INC, CE). The intensity of the light curing device was checked before use with a light intensity meter (Coltolux Light Meter, Mahwah, CE) and found to be between 580–600 mW/cm² for all samples, with a peak wavelength of 470 nm.

After polymerisation, the RC substrate samples were left overnight in air. Then, each sample of RC substrate was embedded in self-cure resin (Bonda clear casting resin, Bondglass-Voss Ltd, UK), and left to set for 24 hours inside the moulds. This process created cylinders (diameter 25 mm; height 20 mm) in which the RC samples were located in the center on the top surface of the cylinder base (Figure 5.1), so that the samples were easier to handle during preparing with SiC and mounting in the shear bond testing jig.

Fifteen Samples were prepared for each of the five materials. Accordingly, the samples were in five groups with n=15 in each group.



Figure 5.1 The top view (A) and lateral view (B) of example test samples, consisting of a RC substrate sample embedded in an epoxy resin block; the samples were identified using both letter and numeric code. The letter represents material type and the number represents the grit size of surface treatment.

5.2.2 Sample repair protocol

Repair composite was built up on the substrate surface with the same RC used as the substrate for each group. The repair material was applied by means of a transparent gelatin capsule size 4 (Agar Scientific LTD, Essex, UK), which was used as a mould to apply the repair materials to the substrates. This ensured standardisation of the size of the repair material. Capsules consisted of two pieces and were therefore separated into two halves, with the smaller half used only as a mould (5 mm in diameter and 6 mm high).

First, the mould (capsule half) was slightly overfilled with RC with the aid of a plastic spatula and then placed onto the central part of the substrate by hand to ensure the adaptation of the repair material. Excess material around the mould was removed

using a clean plastic spatula. The repair composite was subsequently light cured on four sides (at 90° to each other) for 40 seconds per side to ensure the curing light reached the whole depth of the repair material, using the same light unit described in 5.2.1 (see Figure 5.2). All samples were then stored in a sealed container (500 ml microwavable food container), each material being held in a separate container, and covered with distilled water and held at 37°C in a temperature controlled oven for 24 hours to allow dissolution of gelatin capsules before testing RBS.



Figure 5.2 An example of a RC sample, where the substrate material embedded in epoxy resin, after setting the RC substrate surface treated and then repaired with the same material as the substrate.

5.2.3 Measurement of shear bond strength

Once a sample had been removed from the oven, the shear bond strength of the repair was tested using a compressive load with a universal testing machine (Instron model 5567, Berks, UK). A blade was applied at the interface of the substrate and repair RC (Figure 5.3), after the sample had been mounted on the load frame.

The test was performed with a 1.0 kN load cell and a cross head speed of 1.0 mm/min. The force was applied until the bond between the substrate and the repair material failed and the sample broke.

The blade was then cleaned and visually examined after each sample had been tested, to ensure that any excess material was removed prior to testing the next sample, and to evaluate the integrity of the blade throughout testing.

The shear bond strength (SBS) was calculated using equation (1) shown below:

 $SBS = Load \div \pi r^2$ (1)

SBS was calculated then recorded in MPa. Load was recorded in Newton. The value of π used was = 3.14. r represents the radius of the samples and was recorded in millimeters.



Figure 5.3 An example of a test sample inside the Instron machine jig to test SBS at the interface of substrate and repair material.

5.2.4 Statistical analysis

After collecting the raw data for each measurement they were arranged in a Microsoft Excel (2016) spread sheet including raw data, variables name (resin composites and surface treatments) and sample numbers. Then normality distribution was evaluated using the Shapiro-Wilk test, to choose appropriate statistical test (parametric or nonparametric). These tests (two-way analysis of variance, or Kruskal-Wallis test where normality failed) was performed to look for differences within and between resin composites after surface treatments and differences between surface treatments; either a Tukey test or Dunn's test was used to form all pairwise multiple comparisons, with a significance level of 0.05 using Sigma Plot (version 13) statistical software. The bar graphs for median or mean and IQR or SD were made showing any statistically significant differences, using Graph PadPrism (versions 5 and 8).

5.3 SiC method

In this experiment, four SiC grades used were P600, P800, P1000 and P1200, for preparing five RCs, the grades investigated for roughness and SBS. Roughness was measured using a contact profilometer with a tip size of five microns, and Ra and BAC parameters were constructed. In addition, the surface morphology of the RCs prepared with P600 was examined under SEM. In this experiment, the RC samples were repaired with the application of repair materials only after preparing with SiC without application of intermediate materials.

5.3.1 Preparing RCs with SiC

A surface treatment of each RC substrate sample that was embedded in the resin block was undertaken by preparing with either P600, P800, P1000, and P1200 SiC(Tri-M-ite Wet or Dry paper, 3M, St Paul USA) on a lapping machine (MetaServ[™] 250 Single & Twin Grinder Polishers, Minhang, Shanghai, China). The grades of the grit respectively were 25.8, 21.8, 18.3, 15.3 µm, and applied at 200rpm for 60 seconds, using running water as a lubricant. To remove any residual debris from the surface, prepared samples with SiC were thoroughly rinsed with water, and finally dried with compressed air.

Each RC substrate sample was surface treated first with P600, and after the repair and shear bond test for P600, the samples were reused for surface treatment with P800, the remnant or holes on RC from the P600 SBS test were removed using P600 till the surface became flat again. After the SBS testing for P800, the sample was reused for a third and fourth time for surface treatment with P1000 and P1200, in the same way as for P800. To be clear, after flattening the used sample with P600, the grade which was required to be investigated was applied directly after preparing with P600 SiC. For example, when P1200 investigated, after completing SBS on RC samples prepared with P1000, the same RC samples (substrate material embedded in resin block) prepared with P600 to make the samples flat again then P1200 grade used to polish RC samples. So, there was not sequential use of the SiC paper grades when every grade was investigated. The reuse of the substrate samples was not performed when the SBS testing produced holes in the substrate material due to debonding of the repair material.

5.3.2 Measuring roughness

After treatment with each SiC grade and before the application of the repair RC material, the surface of RC substrate samples that was embedded in resin blocks was profiled using a contact stylus profilometer (Mitutoyo Surftest SV-2000 and Surfpak-SV Mitutoyo Corp V1.600). Samples were mounted in the profilometer perpendicular to the stylus tip, using a spirit level.

A diamond stylus (radius= 5 μ m) was placed in contact with a RC substrate sample and then moved laterally across the sample for 4 mm under 4 mN contact force, with a speed 0.5 mm/s and a height range of 800 μ m. Five successive measurements, perpendicular to the grinding direction, were recorded on each RC substrate sample.

Roughness average (Ra), reduced peak height (Rpk), core roughness depth (Rk), reduced valley height (Rvk), material ratio of peaks (Mr1), and material ratio of valleys (Mr2) were measured for each RC sample.



Figure 5.4 An example of stylus profilometry undertaken on a RC sample.

5.3.3 Photomicrographs of prepared RCs

A scanning electron microscope (SEM, Stereoscan 240, Cambridge Instruments, Cambridge, U.K.) was used to evaluate the morphological surface features on the tested RCs. For this purpose, one sample from each RC substrate was embedded in resin block was prepared. Therefore five samples for this investigation were prepared (n=1/group) with P600 silicon carbide paper (Tri-M-ite Wet or Dry paper, 3M, St Paul USA) on a lapping machine (MetaServ[™] 250 Single & Twin Grinder Polishers, Minhang, Shanghai, China), using the method described in the sample surface treatment for P600 silicon carbide paper in Section 5.3.1. Each sample was then sputter-coated with gold and was then ready for SEM investigation.

5.4 SiC results

In this experiment the effect of SiC grades was evaluated for consistency in roughness, SBS and relation between roughness and SBS of prepared RCs.

Roughness

Typical roughness profile height for Filtek Supreme XTE, Heliomolar, Filtek Z250, Kalore and N'Durance are shown in Figure 5.5, Figure 5.6, Figure 5.7 and Figure 5.8. The figures illustrate peak heights and valley depths, which are roughly between 2.5 and -2.5 μ m. The profiles are representing roughness profile of prepared RCs with either P600 or P800 or P1000 or P1200. The peak heights and valley depths decreased with a decrease in SiC grit size.

Similarly, there was a difference between RCs when prepared with P600, but this difference became less obvious in profile height for the samples prepared with lower SiC grades.

In addition, typical BACs for Filtek Supreme XTE, Heliomolar, Filtek Z250, Kalore and N'Durance are shown in Figure 5.9, Figure 5.10, Figure 5.11 and Figure 5.12, respectively. BAC describes the distribution of material ratio within the profile height,

and is described in the literature review. The shape of the curve and material ratio are used to describe RC roughness in relation to peak, core and valley. All RCs showed a small Mr_1 and a large Mr_2 with relatively deep valleys, and this feature appeared to be consistent over the four SiC grades.



Figure 5.5 Typical stylus profilometry profile for roughness on RC samples treated with P600, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.6 Typical stylus profilometry profile for roughness on RC samples treated with P800, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.7 Typical stylus profilometry profile for roughness on RC samples treated with P1000, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.8 Typical stylus profilometry profile for roughness on RC samples treated with P1200, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.9 Typical bearing area curve for roughness on RC samples treated with P600, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.10 Typical bearing area curve for roughness on RC samples treated with P800, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.11 Typical bearing area curve for roughness on RC samples treated with P1000, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.12 Typical bearing area curve for roughness on RC samples treated with P1200, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.

<u>Ra</u>

Ra is the average of a set of individual measurements of a surface's peaks and valleys (*Ra & RMS: Calculating Surface Roughness*). Ra measurement was calculated for the samples for each SiC grade and RC. Figure 5.13 compares Ra measurement between the SiC grades for every RC, while Figure 5.14 compares Ra between RCs for every SiC grade.

As the Figure 5.13 shows, in general there were a significant decrease in Ra when samples were prepared with P600 compared to other grades (P<0.01), although no significant difference was found between P600 and P800 treatment for Kalore and Filtek Supreme XTE. For all RCs, there was a significant decrease in Ra when P800 treated samples are compared with P1000 and P1200 treated samples (P<0.01). No significant change in Ra was found for P1000 and P1200 treated samples.

For the same SiC grade group, Figure 5.14 shows that, in a comparison of the roughness of the five RCs, generally all RCs had lower Ra compared with Kalore. Filtek Z250 showed a marked decrease in roughness compared to Kalore, and there was a significant difference between them except for the samples treated with P600 and P1200. For Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar, there was a steady decrease in the order of roughness but no significant difference was observed, except for the samples treated with P800 and P1200.

Furthermore, it is clear that Kalore had the greatest roughness as opposed to Heliomolar, which had the least roughness, with the exception of P1200. This difference was significant for all SiC grades (P<0.01). The roughness of Filtek Supreme XTE was significantly lower than that of Kalore, with the exception of the P600 and P800. Also, there was no significant difference between Kalore and N'Durance, other than for the samples treated with P800 and P1200 (P<0.01).

What can be clearly seen in Figure 5.14 is that the lowest number significant difference between RCs was shown with P600, where only one was demonstrated between Kalore and Heliomolar.



Figure 5.13 Effect of SiC grades on Ra level for each RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Ra (n=15); * represents significance difference between SiC paper, and horizontal bars connect the RCs with a statistical significance of ** point to P<0.01.



Figure 5.14 comparing Ra level between RCs for every SiC grade.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Ra (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with a statistical significance of * points to P≤0.05 and ** P<0.01.

<u>Rpk</u>

This parameter is used to calculate reduced peak height of the BAC curve. The Rpk measurement was calculated for the samples for each SiC grade and RC. Figure 5.15 compares the Rpk measurement between the SiC grades for every RC. Figure 5.16 compares the Rpk of RCs for every SiC grade.

As can be seen in Figure 5.15, the Rpk data overall were similar to the Ra data for RCs treated with SiC. For all RCs, P600 and P800 treated samples were significantly higher in Rpk compared with P1000 and P1200 treated samples (P<0.01). None of the differences between P600 and P800 treated samples were significant except for Filtek Z250 and N'Durance (P<0.01); P1000 and P1200 treated samples showed no significant difference.

As can be seen in Figure 5.16, the results of Rpk were similar to Ra findings, with a few exceptions in that there was a decrease in Rpk for all RCs compared to Kalore, and this decrease in Rpk was significant for Heliomolar for the samples in the same SiC grade groups. Also, N'Durance showed significantly lower Rpk compared with Kalore, except for the P600 group (P<0.01). However, Filtek Z250 exhibited no significant change in Rpk compared with Kalore with the exception of the samples treated with P800 (P<0.01).



Figure 5.15 Effect of SiC grades on Rpk level for every RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Rpk (n=15); * represents significance difference between SiC paper, and horizontal bars connect the RCs with a statistical significance of * mark P≤0.05, ** P<0.01 and *** P<0.001.



Figure 5.16 Comparing Rpk level between RCs for every SiC grade.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Rpk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with a statistical significance of * point to P≤0.05, ** P<0.01 and *** P<0.001.

<u>Rk</u>

This parameter is used to calculate the core roughness of the BAC curve. The part which carries load is most closely in contact with the repair material. Rk measurement was calculated for the samples for each SiC grade and RC. Figure 5.17 compares Rk measurement between the SiC grades for every RC, while Figure 5.18 compares Rk between RCs for every SiC grade.

As illustrated in Figure 5.17 the Rk data overall were similar to the Ra data for RCs treated with SiC, with a slight difference. In general, there was a significant decrease in Rk with samples prepared with P600 compared to other grades (P<0.01), although no significant difference was found between P600 and P800 treatment for Kalore and Filtek Supreme XTE. For all groups of RCs, there was a significant decrease in Rk when P800 treated samples were compared with P1000 and P1200 treated samples (P<0.01) with the exception of Kalore. No significant change in Rk was found between P1000 and P1200 treated samples.

It can be seen in Figure 5.18, that Rk data were similar to the Ra data in that there was a decrease in Rk for all RCs compared to Kalore. This decrease in Rk was significant for Heliomolar (P<0.01) for the samples in the same SiC grade group. Also, the Rk for Heliomolar was significantly lower than Filtek Z250 and N'Durance for the samples treated with P800 and P1000. Further, Heliomolar was significantly lower than Filtek Z250 in Rk for samples prepared with P1200. There was a significant decrease in Rk when N'Durance and Filtek Z250 samples were compared to Kalore samples treated with P800. N'Durance samples were significantly lower in Rk compared with Kalore samples (P<0.01) treated with P1200. For the samples prepared with P1000, Filtek Supreme XTE was significantly lower in Rk compared with Kalore samples (P<0.01).


Figure 5.17 Effect of SiC grades on Rk level for every RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Rk (n=15); * represents significance difference between SiC paper, and horizontal bars connect the RCs with a statistical significance of * indicate P≤0.05, ** P<0.01 and *** P<0.001.



Figure 5.18 Comparing Rk level between RCs for every SiC grade.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Rk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with a statistical significance of * point to P≤0.05, ** P<0.01 and *** P<0.001.

<u>Rvk</u>

This parameter is reduced valley depth of the BAC curve that accumulate fluid. The Rvk measurement was calculated for the samples for each SiC grade and RC. Figure 5.19 compares the Rvk measurement between the SiC grades for every RC, while Figure 5.20 compares Rvk between RCs for every SiC grade.

As can be seen in Figure 5.19, the findings in the Rvk data were similar to the results for the Ra measurement, in which there was a significant decrease in Rvk when the samples prepared with P600 were compared to other grades (P<0.01); however, no significant difference was found between P600 and P800 treatments for Kalore and Filtek Supreme XTE. For all RCs, there was a significant decrease in Rvk when P800 treated samples were compared with P1000 and P1200 treated samples (P<0.01). No significant change in Rvk was found between P1000 and P1200 treated samples.

The Rvk for Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar RCs for various grades of SiC is displayed in Figure 5.20. The results of Rvk were similar to the Ra findings, with the exception of the P1200, but the significant difference was only between Kalore and N'Durance (P<0.01).



Figure 5.19 Effect of SiC grades on Rvk level for every RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Rvk (n=15); * represents significance difference between SiC paper, and horizontal bars connect the RCs with a statistical significance of ** mark to P<0.01.



Figure 5.20 Comparing Rvk level between RCs for every SiC grade.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Rvk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with a statistical significance of ** point to P<0.01 and *** P<0.001.

<u>Mr1</u>

This represents the proportion of peaks. The effect of SiC grades and RC types on Mr₁ is illustrated in Figure 5.22 and Figure 5.21.

The effect of P600, P800, P1000 and P1200 SiC grades on the Mr₁ of a RC is shown in Figure 5.21. Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar RCs showed the trend that Mr₁ in the P600 and P800 was lower than those in the P1000 and P1200. This difference was significant for the Filtek Z250. For Kalore, Mr₁ was significantly lower for the samples treated with P800 compared to those treated with P600, P1000 and P1200 (P<0.01). For N'Durance and Heliomolar, the Mr₁ for the samples treated with P600 was significantly lower than that treated with P1200 (P<0.01). For Filtek Supreme XTE, the Mr₁ for the samples treated with P600 was significantly lower than those treated with P1000 (P<0.01). No significant change in Mr₁ was found for the samples treated with P600 and P800, except for Kalore.

Figure 5.22 compares the Mr₁ between Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar RCs on a grade of SiC. Here, a clear trend in roughness could not be recognised. For the P600 prepared group, the Mr₁ of Kalore was significantly higher than those of the Filtek Z250, N'Durance, and Heliomolar. For the RCs treated with P800, the Mr₁ of the Filtek Supreme XTE was significantly greater compared with Filtek Z250 and Kalore. For the samples treated with P1000, the Mr₁ of the Heliomolar and N'Durance was significantly lower compared to Filtek Supreme XTE and Filtek Z250. No significant difference was found for the samples treated with P1200.



Figure 5.21 Effect of SiC grades on Mr_1 level for every RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Mr_1 (n=15); * represents significance difference between SiC paper, and horizontal bars connect the RCs with a statistical significance of ** indicate P<0.01.



Figure 5.22 Comparing Mr₁ level between RCs for every SiC grade.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Mr₁ (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with a statistical significance of * point to P≤0.05 and ** P<0.01.

<u>Mr2</u>

This represents the proportion of valleys. The Mr₂ measurement was calculated for the samples for each SiC grade and RC. Figure 5.23 compares the Mr₂ measurements of the SiC grades for every RC, and Figure 5.24 compares the Mr₂ between RCs for every SiC grade.

It can be seen in Figure 5.23, that no significant change in Mr_2 was found for the RCs treated with P600, P800, P1000 and P1200, with two exceptions. For Kalore, the Mr_2 for the samples treated with P800 was significantly lower compared with the samples treated with other grades (P<0.01). For N'Durance, the Mr_2 for the samples treated with P600 was significantly lower than those treated with P1200 (P<0.01).

Figure 5.24 compares the Mr_2 between Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar RCs treated with a grade of SiC. Heliomolar exhibited the least roughness in each SiC grade group. Heliomolar samples were significantly lower than other RCs (P<0.01) when treated with P1200, and also significantly lower than Filtek Z250, Filtek Supreme XTE and Kalore (P<0.01) when treated with P1000. Heliomolar samples were significantly lower compared with Filtek Z250 and Filtek Supreme XTE when treated with P800. Heliomolar samples were significantly lower than Kalore when treated with P600. In addition, the Mr_2 of N'Durance samples was significantly lower than those of Filtek Z250 and Filtek Supreme XTE when treated with P600. The Mr_2 of Filtek Supreme XTE when treated with P600 and P1000. The Mr_2 of Filtek Z250 and Kalore were similar except for the samples treated with P800 (P<0.01).



Figure 5.23 Effect of SiC grades on Mr₂ level for every RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Mr₂ (n=15); * represents significance difference between SiC paper, and horizontal bars connect the RCs with a statistical significance of ** indicate P<0.01.



Figure 5.24 Comparing Mr₂ level between RCs for every SiC grade.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of Mr₂ (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with a statistical significance of * point to P≤0.05 and ** P<0.01. * point to P≤0.05 and ** P<0.01.

<u>SBS</u>

Typical load-extension curves for Filtek Supreme XTE, Heliomolar, Filtek Z250, Kalore and N'Durance are illustrated in Figure 5.25 to Figure 5.28. As a whole, the samples demonstrated similar behaviour. The primary area of the extension curve shows a low line which is the area where no load is placed on the sample, but when the guillotine blade is heading towards the sample to make contact. Next, there is a small growth in load, which shows the moment when the guillotine blade begins to touch the sample. The load then starts to go up sharply in a straight line as the guillotine shears the sample. This is followed by a steep decline in the load as the interfacial bond between substrate and repair material fails.



Figure 5.25 Typical load extension-curve for a shear bond strength on RC samples treated with P600, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.26 Typical load extension-curve for a shear bond strength on RC samples treated with P800, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.27 Typical load extension-curve for a shear bond strength on RC samples treated with P1000, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.28 Typical load extension-curve for a shear bond strength on RC samples treated with P1200, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.

In this experiment, there were a number of pre-test failures for Filtek Supreme XTE for the P600 grit (n=6), P800 grit (n=1), P1000 grit (n=9), and P1200 grit (n=6) pretest failures.

Generally, the various SiC grades resulted in only a minimal difference in the SBS of the RCs. Figure 5.29 illustrates the SBS of four SiC grades for every RC. Figure 5.30 compares RCs for every SiC grade. As shown in Figure 5.29, statistically non-significant difference in the SBS was found between RCs when treated with P600, P800, P1000, and P1200.

In Figure 5.30 we can see that, for the same SiC grade group, and the SBS comparison for the five RCs, Filtek Z250 and Kalore showed a markedly higher SBS compared to Filtek Supreme XTE and Heliomolar, with a significant difference between them for the P600 prepared group (P<0.01). However, for the P800 and P1200 groups, only Filtek Z250 was significantly higher in SBS than Filtek Supreme XTE (P<0.1).



Figure 5.29 Effect of SiC grades on SBS level for every RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of SBS (n=15).



Figure 5.30 Comparing SBS level between RCs for every SiC grade.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P800, P1000, P1200 represent different SiC grades. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with a statistical significance of * point to P≤0.05, ** P<0.01 and *** P<0.001.

Photomicrographs

Figure 5.31 shows typical SEM images for every RC. The RC sample was surface treated with P600 silicon carbide paper. All the RCs exhibited surface features that were essentially dependent on the SiC grit size, and thus the surfaces were indicative of the resin matrix imparted by the abrasive, or in other words, striation, as seen in the photomicrographs at 500X magnification. This striation was less prominent for Heliomolar and Filtek Supreme XTE, and therefore both RCs appear to look smoother than the others. At 5000X magnification, the same striation can be noticed at 500X magnification. There were, however, some differences in pores and resin matrix loss, as described below.

Kalore exhibited a rougher surface than Heliomolar and N'Durance, characterized by small craters due to the extrusion of fillers. Also, some fillers were merely loosely retained and other particles were covered with resin matrix on the surface. The Filtek Supreme XTE surface showed minor holes (pores or craters) apparently created due to the plucked-out fillers, leaving a small number of loose particles on the surface. The surface feature of N'Durance looked smoother than Kalore which possibly due to less often in filler debonding from the surrounding matrix. The surfaces of the Filtek Z250 were similar to that of Kalore but with less loss of resin matrix, and most of the fillers were still covered with resin matrix. In contrast, Heliomolar and N'Durance showed much less damage to resin matrix with very few obvious pores.

Surface treatment	P600	
Resin composites	500X	5000X
Kalore	Î	
FiltekZ250	Î	
N`Durance	Î	$\overrightarrow{}$
Filtek Supreme XTE	Î	
Heliomolar	Î	↑ ↑ ↑ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓ ↓

Figure 5.31. Scanning electron photomicrographs at 500X and 5000X magnification of RCs after preparing with P600.

The grooves ($\hat{1}$), caused by the SiC abrasive particles, debonded fillers (\Rightarrow) and pores (\Leftarrow) are clearly seen.

Summary of findings:

- 1- The different SiC grades affects RC roughness where P600 resulted in a significantly higher roughness compared with P800, P1000, and P1200;
- 2- P600 resulted in the lowest number of significant differences in roughness between RCs compared with the other SiC grades;
- 3- The RC type affects roughness where Kalore contributed to the highest and Heliomolar to the lowest roughness for every SiC grade;
- 4- The SiC grades did not have an impact on SBS because there was no significant difference between RCs as a result of different SiC grades;
- 5- The RCs influenced SBS, where Kalore and Filtek Z250 showed highest value and Filtek Supreme XTE and Heliomolar presented the lowest values in SBS for every SiC grade;
- 6- RCs were illustrated in photomicrographs to show differences, where Kalore and Filtek Z250 had a rougher surface appearance than Filtek Supreme XTE and Heliomolar.

5.5 Chemical preparation method

In this section the chemical preparation includes sequential application of phosphoric acid, bonding agent and silane were investigated. The effectiveness of the surface treatments were investigated by measuring both SBS and associated roughness and surface morphology when applicable.

5.5.1 Phosphoric acid, bonding agent and silane pre-treatments

The samples were prepared with P600 as described in Section 5.3.1. Then the first surface treatment was applied to RC samples which consisted of 36% phosphoric acid gel (DETREY[®]Conditioner 36, DENSPLY, Germany). The phosphoric acid gel applied on the RC samples, using the manufacturer's instructions, for 15 seconds. Next, the phosphoric acid was washed with water and air for 15 seconds and dried with air for 15 seconds using air water spray. The second surface treatment involved etching and bonding. After preparing the samples with P600 and then acid etching with phosphoric acid, Optibond[™] Solo Plus bonding agent (Single component totaletch dental adhesive, Kerr, USA) was applied with a cotton applicator tip (Soft-Tip Applicator, DE, USA), using the manufacturer's instructions. The bonding agent was applied on RC samples for fifteen seconds using a light brushing motion, dried for three seconds, and light cured for twenty seconds using the light curing device described previously. The third chemical preparation was adding silane to the former surface treatment. Silane was added to RC samples after treatment with phosphoric acid, silane primer (ESPE[™] Sil, 3M ESPE, Germany) was applied, using the manufacturer's instructions for intraoral repair. The silane was applied with a cotton applicator (Soft-Tip Applicator, DE, USA) for ten seconds, and then left to dry for 30 seconds, followed by the application of bonding agent.

5.5.2 Measurement of RC roughness

The surface of the RCs was profiled as described in Section5.3.2. In this investigation, the measurement of roughness was performed after preparing with P600 and acid etching the samples.

5.5.3 Photomicrographs of acid etched RCs

A SEM examination was performed for the RC samples (n=1/group). The method was similar to that described in Section 5.3.3. The only deference was in the surface treatment of the samples, where the samples treated with both P600 and acid etching.

5.6 Chemical preparation results

In this experiment the cumulative effect of 37% of phosphoric acid, CP/BA and silane were evaluated for roughness and SBS. Firstly, a 37% of phosphoric acid was applied to RCs prepared with P600 to simulate when dentists do not perform any mechanical preparation, and only use phosphoric acid as chemical preparation; they then apply the repair RC directly without the use of any other intermediate materials. Secondly, CP/BA was applied to RC samples treated with P600 plus phosphoric acid as noted in the first treatment. Thirdly, silane added to the last chemical surface treatment after etching with phosphoric acid.

5.6.1 Phosphoric acid pretreatment

Roughness

Typical roughness profiles for the five RCs when treated with P600 plus phosphoric acid etch are illustrated in Figure 5.32. The vertical axis of the figures displays the peak heights and valley depths of the RCs, which were between 2.5 and -2.5 μ m. Filtek Z250 and Kalore appear to be higher in roughness profile than other RCs, particularly in comparison with Heliomolar. However, all RCs were similar to the corresponding profile roughness as described for RCs prepared with P600, discussed in Section 5.4.

In Figure 5.33, the typical BAC for the RCs treated with polish plus acid etch is illustrated. The triangular area above and below the red line represents peaks and valleys, respectively, and both occupy a small portion of the curve. The area between the peak and valley represents the core part of the roughness. The slope angle of the red line was similar in steepness for all RCs. The two vertical lines represent the

material ratio of roughness. The yellow line on the right side is the material ratio of a peak, and the area between the two vertical yellow lines represents the material ratio of the valleys. The material ratio of the peak is rather small in comparison to the valley. The first and second material ratios were quite similar between the RCs. The BAC curve for all the RCs in this experiment were broadly similar to those prepared with P600, seen in the previous experiment.



Figure 5.32 Typical stylus profilometry for roughness on RC samples treated with P600 plus acid etch, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.



Figure 5.33 Typical bearing-area curve for roughness on RC samples treated with P600 plus acid etch, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.

<u>Ra</u>

The Ra data in the polish plus acid etch. Kalore showed the highest roughness and Heliomolar exhibited the lowest. Kalore and Filtek Z250 showed significantly higher roughness than N'Durance, Filtek Supreme XTE, and Heliomolar (P<0.001). Further, Heliomolar showed significantly lower roughness compared with N'Durance and Filtek Supreme XTE. In contrast, none of the differences between Kalore and Filtek Z250, N'Durance, and Filtek Supreme XTE were significant (P>0.05, Kruskal Wallis test, see Figure 5.34). Additional analysis of data revealed no significant differences between the roughness of samples prepared with either P600 or P600 plus acid etch (P>0.05), and they followed the same trend as preparation with P600 in all RCs, as shown in Figure 5.35.

In summary, two trends could be found from the Ra data; first, there is the significant difference of Kalore and Filtek Z250 from the other RCs; second, there was no significant difference between the roughness`s of the samples treated with either P600, or P600 plus acid etch.



Figure 5.34 Ra level of RCs treated with P600 plus phosphoric acid.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Ra (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * mark P \leq 0.05 and *** P<0.001.



Figure 5.35 Ra level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 and P600 plus phosphoric acid represent the two treatments. Bar charts represent median and the vertical lines IQR of Ra (n=15).

<u>Rpk</u>

Analysis of the data illustrated in Figure 5.36 and Figure 5.37 revealed that the Rpk measurement for the RCs was analogous to the Ra measurement, with a few differences in significant value. Another difference in the Rpk is that Kalore was significantly higher in Rpk than Filtek Z250 (P<0.1).



Figure 5.36 Rpk level of RCs treated with P600 plus phosphoric acid.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Rpk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * mark P≤0.05 and *** P<0.001.



Figure 5.37 Rpk level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 and P600 plus phosphoric acid represent the two treatments. Bar charts represent median and the vertical lines IQR of Rpk (n=15).

<u>Rk</u>

The data of acid etched samples exhibited in Figure 5.38 and Figure 5.39. Comparable difference between RCs were found between Rk and Ra with few differences in the significant values.



Figure 5.38 Rk level of RCs treated with P600 plus phosphoric acid.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Rk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of *** indicate P<0.001.



Figure 5.39 Rk level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 and P600 plus phosphoric acid represent the two treatments. Bar charts represent median and the vertical lines IQR of Rk (n=15).

<u>Rvk</u>

The finding from Figure 5.40 and Figure 5.41, was similar to the two trends illustrated within Ra data analysis. In addition to the first trend, N'Durance showed significantly higher Rvk compared with those of Filtek Supreme XTE and Heliomolar. One exception from the second trend was that significant difference was found between the Rvk measured for Filtek Z250 samples treated with either P600 or P600 plus acid etch (P<0.1).



Figure 5.40 Rvk level of RCs treated with P600 plus phosphoric acid.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Rvk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * point to P≤0.05, ** P<0.01 and *** P<0.001.


Figure 5.41 Rvk level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 and P600 plus phosphoric acid represent the two treatments. Bar charts represent median and the vertical lines IQR of Rvk (n=15); * represents significance between RCs, and horizontal bar connect the treatments with statistical significance of * point to P≤0.05.

<u>Mr1</u>

Significant differences were found between the Mr_1 measured for samples treated with P600 plus acid etch. Filtek Supreme XTE showed the highest Mr1 and was significantly different compared to N'Durance, Filtek Supreme XTE, Filtek Z250 and Heliomolar, see Figure 5.42. In Figure 5.43, the data for P600 plus acid etch follows the same trend as P600 treatment for RCs, and non-significant difference was found between the Mr₁ for the RCs (P>0.05, Kruskal Wallis test).



Figure 5.42 Mr_1 level of RCs treated with P600 plus phosphoric acid.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Mr₁ (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of ** indicate P<0.01 and *** P<0.001.



Figure 5.43 Mr_1 level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 and P600 plus phosphoric acid represent the two treatments. Bar charts represent median and the vertical lines IQR of Mr₁ (n=15).

<u>Mr2</u>

Significant difference was found between the Mr₂ measured for samples treated with P600 plus acid etch. Filtek Supreme XTE showed the highest Mr₂ compared to Kalore, Filtek Z250, N'Durance and Heliomolar, see Figure 5.44. The data for P600 plus acid etch following the same drift as P600 treatment for all RCs, where non-significant difference was found between the Mr₂ for the RCs (P>0.05, Kruskal Wallis test), as can be seen in Figure 5.45.



Figure 5.44 Mr₂ level of RCs treated with P600 plus phosphoric acid.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Mr₂ (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of ** indicate P<0.01 and *** P<0.001.



Figure 5.45 Mr₂ level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 and P600 plus phosphoric acid represent the two treatments. Bar charts represent median and the vertical lines IQR of Mr₂ (n=15).

<u>SBS</u>

Typical load-extension curves for the five RCs are illustrated in Figure 5.46. Overall, the samples all exhibited a similar behaviour, and the performances are similar to those described in Section5.4. The first area of the extension curve shows a horizontal line which is the area of no load practiced to the sample when the guillotine blade touches the sample prior to making contact with it. Next, there is a small increase in load, which signifies the moment the guillotine blade begins to touch the sample. The load then starts to rapidly rise in a straight line as the guillotine shears the sample. This is followed by an abrupt drop in the load as the interfacial bond between substrate and repair material decline.



Figure 5.46 Typical load-extension curve for a roughness on RC samples treated with P600plusacid etch, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.

The SBS of the RCs increased within P600 plus acid etch particularly for the Filtek Supreme XTE and Heliomolar. Significant differences were found between materials treated with P600 plus acid etch, as shown in Figure 5.47. Kalore and Filtek Z250 showed significantly higher bond strength compared with Filtek Supreme XTE and N'Durance. Heliomolar was significantly higher in bond strength than those of N'Durance. The other differences between materials in SBS were non-significant (P>0.05). Compared to the P600 treatment, there was an increase in the SBS for the RCs treated with P600 plus acid etch. However, the higher value expressed by SBS were not significant with the exception of Filtek Supreme XTE and Heliomolar, see Figure 5.48.



Figure 5.47 SBS level of RCs treated with P600 plus phosphoric acid.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * point to P≤0.05, ** P<0.01 and *** P<0.001.



Figure 5.48 SBS level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 and P600 plus phosphoric acid represent the two treatments. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the treatments with statistical significance of * point to P≤0.05 and *** P<0.001.

Photomicrographs

Figure 5.49 shows typical SEM images of the five RC samples' surfaces after P600 plus phosphoric acid surface treatment. The surface texture was similar to RCs prepared with P600 but with a few differences.

At low magnification, Filtek Supreme XTE and Heliomolar appear similar in striation to other RCs. This indicates RC were similarly affected by P600 plus phosphoric acid surface treatment.

At high magnification, Filtek Supreme XTE showed pores, de-bonded fillers and morphology of deeper scratches. The surface texture of Heliomolar was very smooth than other RCs and exhibited narrow and deep scratches. Kalore and Filtek Z250 and N'Durance characterized by a surface with deep grooves and very few loose fillers.

Generally, etched RCs showed less loose fillers on surface and deeper grooves compared to prepared RC samples with P600 particularly for Filtek Supreme XTE and Heliomolar, see Figure 5.50 and Figure 5.51.

Surface treatment	P600+acid etch	
Resin composites	500X	5000X
Kalore	Î	
FiltekZ250	Î.	
N`Durance	Î	
Filtek Supreme XTE	Î	→
Heliomolar	Î	$ \begin{array}{c} \uparrow \\ \hline \\ \rightarrow \end{array} $

Figure 5.49 Scanning electron photomicrographs at 500X and 5000X magnification of RCs after P600 plus phosphoric acid surface treatment.

The grooves (\uparrow) caused by the SiC abrasive particles, debonded fillers (\Rightarrow) and pores (\leftarrow) are clearly appear.

Surface treatment	P600	P600+acid etch
Resin composites	500X	500X
Kalore		
FiltekZ250		
N`Durance		
Filtek Supreme XTE		
Heliomolar		

Figure 5.50 Scanning electron photomicrographs at 500X magnification of RCs afterP600 preparation and P600 plus phosphoric acid surface treatment.

Surface treatment	P600	P600+acid etch
Resin composites	5000X	5000X
Kalore		
FiltekZ250		
N`Durance		
Filtek Supreme XTE		
Heliomolar		

Figure 5.51 Scanning electron photomicrographs at 5000X magnification of RCs after P600 preparation and P600 plus phosphoric acid surface treatment.

5.6.2 Phosphoric acid plus bonding agent pretreatment with or without silane

<u>SBS</u>

Typical load-extension curves for Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar are illustrated in Figure 5.52. In most cases, the samples all showed identical behaviour, and the performance was similar to those described in Section5.4. The initial area of the extension curve shows a level line which is the area of no load practiced to the sample when the guillotine blade was moving to the sample prior to making contact with it. Next, there is minor increase in load, which signify the moment when the guillotine blade begins to touch the sample. The load then starts to rapidly rise in a straight line as the guillotine shears the sample. This is followed by an abrupt drop in the load as the interfacial bond between substrate and repair material falls.



Figure 5.52 Typical load - displacement curve for a shear bond test on repaired RC samples treated with P600, phosphoric acid etch and bonding agent, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE, (E) is Heliomolar.

As can be seen Figure 5.53, the RCs treated with P600 plus acid etch plus bonding showed difference in SBS values, the Filtek Z250 and Kalore and Heliomolar presented higher SBS compared with Filtek Supreme XTE and N'Durance. This difference is only significant for Filtek Supreme XTE in comparison to Filtek Z250 and Kalore.

Comparing the surface treatments for each RC, all materials showed an increase in SBS as the number of chemical preparations increased successively, as illustrated in Figure 5.54. For example, Filtek Supreme XTE showed the lowest SBS for the P600 treatment and highest value for P600 plus acid etch plus bonding agent treatment, while medium value for P600 plus acid etch treatment. Significant differences were found between the SBS measured for the RC samples treated with the P600 plus acid etch plus bonding sequentially than those prepared with P600 with the exception for the Filtek Z250. In contrast, none of the differences between the SBS measured for samples treated with the P600 plus acid etch plus bonding and P600 plus acid etch acid were significant with the exception for the N'Durance (P<0.001).



Figure 5.53 SBS level of RCs treated with P600 plus phosphoric acid plus bonding agent.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * indicate P≤0.05 and *** P<0.001.



Figure 5.54. SBS level of RCs with three treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P600 plus phosphoric acid and P600 plus phosphoric acid plus bonding agent represent the three treatments. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the treatments with statistical significance of ** indicate P<0.01 and *** P<0.001.

Silane treatment

Typical load-extension curves for Filtek Z250 are illustrated in Figure 5.55. In the main, the samples all exhibited similar behavior, and their performance is similar to those described in Section 5.4. The first area of the extension curve shows a horizontal line which is the area of no load practiced to the sample when the guillotine blade was establish to the sample prior to making contact with it. Next, there is small increase in load, which signify the moment when the guillotine blade begins to touch the sample. The load then starts to rapidly rise in a straight line as the guillotine shears the sample. This is followed by an abrupt drop in the load as the interfacial bond between substrate and repair material decline.



Figure 5.55 Typical load - displacement curve for a shear bond test on repaired RC samples treated with P600 plus acid etch plus silane plus bonding agent.

Significant difference was found between the SBS of the samples for the Filtek Z250 when silane was included in the treatment than excluding silane (P<0.001), see Figure 5.56.

Within this experiment, unusual performance from the tested samples was noticed. Firstly, there was extrusion of the RC samples from the resin blocks before shear testing the interfacial bond between the substrate and the repair materials. To solve this problem, the sample position was changed to be about 5 mm away from the outer border, the situation of extruding RC samples did not occur again.

Secondly, another problem arose which was a change in the shape of the acrylic block during the SBS test from round to oval, see Figure 5.57; and the interfacial debonding between the substrate and repair materials during the SBS test was taking longer time around 10-20 minutes for each sample. Previous SBS tests were around 2-3 minutes. To handle this problem, the acrylic block material was changed to

polyester casting resin, but the same problem was exhibited even though it was decreased slightly. Among the five samples, three suffered this unusual change in the shape of the block. Also, the setting time of the polyester casting resin increased from 24 to 48 hours, but remained within the manufacturer's instructions the setting time was from 24–48 hours, but the same situation recurred.

The effect of silane on the resin block is believed to cause this unusual behaviour of resin blocks because for the previous experiments, in which silane was not used, the samples under SBS testing behaved as usual with no change in shape in the resin bocks for both the acrylic and polyester clear resin, as illustrated in Figure 5.57. Also, the load extension curve for the silane samples showed a convex line graph, while load extension curve for other samples in the all other experiments showed a linear graph line, see Figure 5.58. The deformation in the load extension curve was belonging to the acrylic or polyester blocks rather than to the RC samples. It is thought the block deformation was acting as a confounding factor on these results. Hence, this line of experimentation was not repeated again and the results from deformed acrylic blocks under testing have not been included further in this thesis.



Figure 5.56 SBS level of RCs with four treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600, P600 plus phosphoric acid, P600 plus phosphoric acid plus bonding agent and P600 plus phosphoric acid plus silane plus bonding agent represent the four treatments. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bar connect the treatments with statistical significance of *** indicate P<0.001.



Figure 5.57 Same silane treated sample before (A) and after (B) shear test.

RC treated with P600 plus acid etch plus silane plus bonding agent. The red line in Sections A and B of the figure indicates the radius of the same sample before and after shear testing which were 56 and 65 mm respectively. It is apparent that the radius of the tested sample increased by about 9 mm which indicates the change in the radius of the resin block under shear testing.



Figure 5.58 Load-extension curve of the silane treated samples without change in the shape of the samples (A) and with change in shape of the tested samples.

Summary of findings

- 1. No significant change in roughness was found for each RC treated with P600 plus phosphoric acid compared to those prepared with P600 alone.
- 2. Surface morphology of RC treated with P600 plus phosphoric acid showed similar striation between RCs less loss filler particles. When these findings compared with those of RCs that prepared with P600, the changes could be noticed clearly particularly for Filtek Supreme XTE and Heliomolar.
- 3. RC treated with P600 plus phosphoric acid enhanced SBS for all RC which was statistically significant for Filtek supreme XTE and Heliomolar.
- 4. P600 plus acid etch plus bonding agent decreased the number of statistically significant differences between RCs because it enhanced SBS for all RC but in different ranges. The range of improvement for N'Durance was significantly higher in comparison with P600 plus phosphoric acid treatment.
- P600 plus acid etch plus silane plus bonding agent improved SBS at twice for Filtek Z250 in comparison for the exclusion of silane in the chemical preparation.

5.7 SiC and chemical preparation discussion

This section discusses the findings from SiC grades used to prepare RC samples, and this is followed the surface treatments in the form of chemical preparation on prepared RCs.

SiC paper is made from sheets of paper in which abrasive material, silicon carbide, is glued to one side. SiC paper is created from a variety of grit sizes; generally, it is used to remove materials from surfaces, to make them smoother or rougher (*Sandpaper*). For RC repair, SiC paper has been used to remove the outer surface layer of RCs rich with resin matrix, to produce a similar roughness between samples or, in other words, consistency between samples. In the literature, the studies have either not prepared at all, or prepared samples before the application of surface treatments. Those studies which did prepare RC samples used a variety of different grit sizes. Özcan and Koc-Dundar (2014) reviewed a number of studies and identified the range of grit size of the SiC paper used to prepare the RC samples as 60–1200 grits. The authors raised concerns about the use of different grit sizes for preparing RC samples between studies, stating that the use of different grit sizes has resulted in a difference in starting surface roughness, and this difference is likely to have an impact on the results.

In restorative dentistry, instruments used for cutting, contouring, finishing and polishing have used grit sizes from 3–150 μ m (Ferracane, 2001). With regard to abrasive particle hardness, the main feature of cutting instruments is that the abrasive grit size is usually larger than those of individual contouring, finishing and polishing instruments (Sakaguchi and Powers, 2012a; Van Noort and Barbour, 2013). Abrasive particle size has an impact on the roughness of a material, where the greater the abrasive particle size, the rougher the material surface due to greater abrasion (Barbosa *et al.*, 2005). Finishing and polishing instrument grit sizes are approximately 3–40 μ m (Jefferies, 2007). In this experiment, four grades of SiC were carefully chosen, such that their abrasive particle sizes were 15.3, 21.8, 18.3 and 25.8 μ m for P1200, P1000, P800 and P600, correspondingly. These grades were selected because it is the size located between coarse finishing and final polishing instrument grit sizes which is supposed to remove a layer of the RC surface without

excessively changing roughness so that most of the roughness is the same level. Accordingly, it is assumed that preparing RC samples with SiC would allow better consistency between RCs.

The consistency of the treated RC samples was measured by determining roughness within and between RCs treated with different SiC grades, and further measurement of consistency included determining the impact of roughness of prepared RC samples with SiC on SBS. No data were found that had previously investigated SiC grades for better consistency. Therefore, the main question was whether there was significant variance between the four SiC papers upon roughness and whether this impacted upon SBS.

Assessing the roughness of RCs after surface treatments is a valuable measurement for determining surface topography. A number of studies have measured average roughness (Ra) using a contact profilometer (Junior *et al.*, 2009; Costa *et al.*, 2010; Rinastiti *et al.*, 2010; Kallio *et al.*, 2013). In this experiment, a line scan profilometry that used a physical probe (contact stylus) was used to ascertain RC roughness after surface treatments by measuring parameters such as Ra and BAC.

These data showed that SiC grades (P600, P800, P1000 and P1200) impacted roughness. Based on the results, as the SiC grade decreases, the RC Ra profile also decreases; moreover, the five materials behaved in the same way during SiC preparation. Generally, the samples treated with P600 and P800 were statistically significantly rougher than those treated with P1000 and P1200 for Ra measurement (Figure 5.13).

This result is consistent with those of Kallio *et al.* (2013), who also found preparing microhybrid RC prepared with P800 SiC paper contributed to more measured roughness than those of P1200 grit. In agreement with my findings, Endo *et al.* prepared three nanohybrids and one nanofill with SiC paper using grades of P600 ($20 \mu m$), P800 ($18 \mu m$) and P1200 ($15 \mu m$). Their findings showed that as the grade size decreased, the Ra of the three RCs also decreased (Endo *et al.*, 2010). Similar to both Kallio *et al.* and Endo *et al.*, Lee *et al.* (2002)showed the RCs prepared with P600 SiC has greater Ra values compared to the RCs prepared with P1000 and P1500 for five types of RCs. The earlier studies used a contact stylus profilometer, and the tip diameter of the stylus was five microns in Kallio *et al.* and Endo *et al.*

studies which is the same size as the one used in the present study, but Lee et al did not report the whether the profilometer was contact stylus or not.

The contact profilometer was able to detect differences in roughness profile between SiC grades, but one limitation is that the findings should be interpreted based on the sensitivity of this method, which is based on a stylus tip diameter of 5 microns. Another limitation of the contact profilometer is that it only provides information on roughness in only one dimension. However, alignment of the findings with published studies in the literature (Lee *et al.*, 2002; Endo *et al.*, 2010; Kallio *et al.*, 2013) most probably indicates that the profilometer used is an appropriate method for detecting difference in roughness between RCs, at least for the Ra and BAC parameters.

Because Ra has a limitation in describing the quality of roughness, it is not clear if the decrease in Ra values with the decrease in SiC grades was due to the removal of peaks. It has been assumed the smaller grade of SiC will remove the upper most layer of roughness profile which is peak. Peaks is the weakest part of roughness profile particularly in comparison to core part when a force applied to roughness profile, which wear off earlier than the core parts (Field *et al.*, 2013). Therefore, it is believed evaluating RCs after preparation with SiC grades for BAC distribution in relation to peaks, cores, valleys and peaks and valleys proportion might explain the findings from SBS.

The findings from the BAC parameters showed that as the SiC grade decreased, the Rpk decreased in value, but this was also true for Rk and Rvk (Figure 5.15, Figure 5.17, Figure 5.19). Similar to Ra, the RC samples treated with P600 and P800 were statistically significantly rougher than those treated with P1000 and P1200. This finding indicates that a decrease in SiC grade simultaneously affected the profile height of peaks, core, and valleys, rather than the peaks only. For the Mr¹, generally P600 contributed to lower peaks proportion for most of the RCs except for Kalore, some of the differences were statistically significant. For the Mr², most of the RCs showed statistically non-significant difference between SiC grades number of (e.g. P1200) of SiC (e.g. P600) (Figure 5.21, Figure 5.23). Based on the limitations of this study these findings suggest that preparing RCs with smaller SiC grade (e.g. P1200) did not result in statistically significantly less peak proportions (Mr1). Thus, the assumption that a smaller SiC grade would effectively remove peaks cannot be accepted.

There are a number of explanations for this finding. First, SiC particles have a hardness similar to diamond particles (Jefferies, 2007), and the harder the abrasive particle, the more effective it is in abrading the surface of RC (Sarrett et al., 1991). This likely produces excessive abrasion from the previous roughness profile than only the peak, in such a way that the roughness profile was representing the smaller SiC grade rather than the previous roughness profile. Second, the samples in this thesis were prepared for 60 seconds, and this could be considered sufficient time for the hard particles of SiC to remove not only the peaks but also the entire previous roughness profile. Finally, RCs consist of two phases, filler and resin matrix, so it is a non-homogenous material. Consequently, even small grades of SiC may produce more changes in roughness profile than the grade of the SiC particles due to the extrusion of fillers during preparation. However, within the current experiment it could not be explained why there was minimal statistically significant change in Mr₂ if the smaller grades of SiC (e.g. P1200) removed the roughness profile of the greater SiC grades such as P600. As the abrasive particle size and shape of SiC grades had not been investigated, it is difficult to make further explanation.

An interesting finding from investigating SiC grades was that P600 contributed to the smallest number of statistically significant differences between the RCs in comparison to all other grades for Ra, Rpk, Rk, Rvk (Figure 5.14, Figure 5.16, Figure 5.18, Figure 5.20). For the Mr₁ and Mr₂, P600 showed least number statistically non-significant difference between RCs compared to P1000 only. Therefore, P600 showed better consistency in roughness profile between RCs in comparison to the other grades, possibly due to that P600 abraded RCs in a more similar way than the other grades. In contrast to the present study, Lee *et al.* showed the smallest SiC grade (P1500) contributed to fewer statistically significant differences between the RCs. Lee *et al.* (2002) used five RCs but the commercial names of their RCs were different from those used in the present study. The authors reported they prepared RC samples for 50 strokes of 15-cm length, which was different from the preparing method and time of RCs with SiC in the current study.

The inclusion of BAC parameters in addition to Ra for measuring roughness was advantageous because via the use of BAC the distribution of roughness profile in relation to peak, core and valleys and peaks and valleys proportion was evaluated, which was not possible by the use of Ra. Therefore, the use of the BAC parameter provides more detailed information on the roughness profile than the Ra parameter

alone. This conclusion is in agreement with Field *et al.* (2010), who showed that using BAC with Ra will allow an additional and more valuable description of the surface quality. Knowing these differences in roughness profile has the potential to better reveal how differences in roughness can effect changes in SBS.

The roughness data showed a number of outliers unspecific to a particular material or SiC grade, as they occurred randomly. It is believed the outliers are related to voids or foreign body entrapment inside the RC, either during manufacturing or the placement of the layers of the materials inside the mould. Owing to this, median and interquartile ranges were used for data analysis, as the findings were expected to be less affected by the outliers in comparison to mean calculation.

While considering the effect of SiC grades on SBS, SBS increased as the SiC grades decrease, but none of these differences between P600, P800, P1000 and P1200 for the five RCs (Figure 5.29) were shown to be statistically significant. In the present experiment, the substrate samples were not aged in any solution and no intermediate material was applied, but the findings accord with two earlier studies even though they used intermediate materials and different surface treatments. The first study found statistically non-significant difference in SBS on the microhybrid RC samples treated with either P800 or P1200 SiC grades (Kallio et al., 2013). Özcan et al. compared prepared RCs with P1200 SiC paper with abraded RCs with 30 µm using the CoJet system. The authors showed statistically non-significant difference in microTBS between P1200 (15.3 µm) or the CoJet system (30 µm) for nanohybrid, nanofill, and microhybrid RCs (Özcan et al., 2013a). However, the CoJet system differs from SiC paper and the abrasive particle size is very close to that of P600 25.8 µm. In contrast, Brendeke and Özcan (2007) showed the CoJet system (30 µm) had greater statistical significance of SBS compared with those of P1200 (15.3 µm) for conventional hybrid RC, and it is possible that the RC ageing of these samples affected the findings. In Özcan et al. and Kallio et al, the substrate samples were aged for one week but Brendeke and Özcan aged them for two months in DW. The one-week ageing condition of the Kallio et al. study appears to be closer to the testing conditions in the present study. Ageing time is a factor that affects the performance of RCs (Schmidt and Ilie, 2013; Eliasson et al., 2014), in that the ageing could result in leakage of monomers including unreacted monomers from RCs (Cokic et al., 2018; Khalid et al., 2018). Unreacted monomer is one factor that can enhance bond strength (Veiga de Melo et al., 2011), and this may be the reason why the

findings are comparable. However, Özcan *et al.* aged the samples for one week but they did thermocycling after one week storage in DW. Furthermore, the statistically non-significant difference between SiC grades in SBS was not in a line with the statistically significant findings from Ra and BAC parameters (discussed in the former paragraphs) Therefore, within limitation of this study it can be suggested roughness of RCs prepared with different SiC grades did not impact on SBS.

Another finding was that SiC grades impacted on the performance of RCs in SBS. The statistically significant differences in SBS between RCs were changed from one SiC grade to another (Figure 5.30). The RC samples prepared with P1200 showed statistically non-significant difference in SBS, while RCs prepared with P600, P800 and P1000 showed statistically significant difference between them in SBS. This finding is comparable with another study that showed no significant difference between nanofill and microhybrid RCs in SBS at lowest roughness due to polymerisation against Mylar strip (Ra < 50 μ m),), but the difference between the two materials became statistically significant when the roughness increased due to silica coating (Ra> 300 µm), the authors did not use bonding agent and did not aged the samples (Rinastiti et al., 2010). This finding on SBS does not seem to be influenced by roughness values. If roughness impacted SBS, the samples treated with P1200 would have returned a statistically significant difference between three to four RCs. Also, RCs treated with P600 would have showed only one statistically significant difference in SBS. Therefore, within limitations of this study it would be possible to make suggestion that statistically significant difference in roughness of SiC did not directly influence SBS. This finding is important, particularly as prepared RC samples with SiC were used as the control group. To the best of my knowledge, this is the first study to demonstrate the importance of the selected SiC grade to prepare samples in providing a roughness profile that is better in consistency and does not act as a confounding factor in the SBS results. Within limitations of this study it can be suggested that P600 should be used to prepare the studied RCs during sample preparation for RC repair at least for the RC samples in this thesis.

The first chemical preparation on RCs prepared with P600 was surface treatment with 36% phosphoric acid for 15 seconds. Based on the results, phosphoric acid produced a change in Ra measurement compared to those which were only prepared with P600; however, the changes were statistically non-significant for all RCs (Figure 5.35). One study is in agreement with the Ra measurement because

the authors showed statistically non-significant change in Sa measurement for nanofill RC (Filtek Supreme XT[®]) when prepared with 4000 grit SiC and after adding 37% phosphoric acid for 20 seconds (Loomans et al., 2011a). In contrast, a number of authors have shown statistically significant change in RMS measurement for a micro-fine hybrid RC (Gradia anterior[®]) when polymerised against a Mylar strip or ground with a stone bur, compared to both treatments when 37% phosphoric acid was added for 15 seconds (Fawzy et al., 2008). As noted, the parameters of the earlier studies were different from those used in the current study: RMS is guite similar to Ra but more affected by extreme peaks and valleys, while Sa is a 3D measure of overall surface texture and they are similar to Ra in that they are not differentiate peaks and valleys (Surface roughness). Another finding from phosphoric acid experiment is that phosphoric acid maintained the main features of roughness of RC samples prepared with P600. The roughness between RCs when treated with phosphoric acid showed the main trends for highest and lowest roughness for the nanohybrid (Kalore) and microfill (Heliomolar), respectively, similar to the situation for the RCs treated with the P600 grit for Ra and three of the BAC parameters (Rpk, Rk, Rvk). This aligns with the findings from Loomans et al. (2011a), who used a 3D non-contact optical interferometer to measure Sa and found that hybrid RC showed the greatest roughness, while nanofill RC had the least value both before and after adding 37% phosphoric acid for 20 seconds on RCs.

All BAC parameters, Rpk, Rk, Rvk, Mr1 and Mr2, showed changes in their values after the application of phosphoric acid compared to those prepared with P600. The changes in values were not statistically significant, except for Rvk (Figure 5.37, Figure 5.39, Figure 5.41, Figure 5.43, Figure 5.45). This finding confirms that phosphoric acid gel impacted upon all roughness elements of the roughness profile rather than on a particular element such as peaks or valleys alone. Further, the extent of the effect of phosphoric acid was that the profile and BAC graphs of the acid etched RCs showed similarity with those only prepared with P600 (Figure 5.5, Figure 5.9, Figure 5.32, Figure 5.33). Based on my best knowledge, this is the first study to evaluate changes in roughness using BAC on RCs treated with phosphoric acid. The present study only used the gel form of phosphoric acid, and therefore the phosphoric acid applied in the liquid form may not produce similar findings to this study because there may be difference between the flow of the gel and liquid forms.

Furthermore, the Ra and BAC parameter results showed a number of outliers which were unspecific to any parameter or RC type. One explanation for these outliers could be due to the voids and foreign bodies integrated within the materials during manufacturing or placement in mold, similar to those described within RCs prepared with SiC.

Based on the results, the application of 36% phosphoric acid for 15 seconds on prepared RCs with P600 resulted in a statistically non-significant increase in SBS for some of the RCs compared with those samples treated with P600 alone except for Filtek Supreme XTE and Heliomolar (Figure 5.54).

Filtek Z250 demonstrated a statistically non-significant increase in SBS. Based on the published studies, although this material has not been tested for the impact of phosphoric acid on RBS, Filtek Z250 is categorised as a microhybrid RC. Fawzy *et al.* (2008) tested a microhybrid RC and showed a statistically non-significant increase in microTBS after the application of 37% phosphoric acid, and their finding is in agreement with that of the current study for microhybrid RC. Moreover, Fawzy *et al.* aged the substrate before the application of surface treatments for 30 days in DW, which was not the case in the current study.

Similarly, Kalore and N'Durance showed a statistically non-significant increase in SBS when treated with 36% phosphoric acid compared with those treated with P600. As illustrated in Table 5-1, the two materials are nanohybrid in category. Kalore and N'Durance have not been tested for RBS after the application of phosphoric acid, but two studies did investigate different products of nanohybrid RCs. In their findings, Yesilyurt et al. (2009) showed a statistically non-significant increase in the shear bond strength of nanohybrid RC either before or after 35% phosphoric acid application for 60 seconds, polymerised against a Mylar strip. The difference between the current study and that of Yesilyurt et al. is that the latter used an aggressive ageing method before the application of surface treatments. This involved 300 hours of ageing by ultraviolet light and visible light, at a temperature of 43.3°C, with a programmed cycle of 18 minutes of DW spray within each 2-hour period. In contrast, Wendler et al. (2016) showed that the application of 35% phosphoric acid for 15 seconds on nanohybrid treated with diamond bur (grain size 27–76 µm) enhanced TBS (statistically significant) compared to bur treatment alone (P<0.05). The authors used Grandio SO[®] type RC, the composition of which

includes filler (89 wt %), 0.5–3 µm glass ceramic particles 0–40 nm SiO2 nanoparticles, and matrix –Bis-GMA, Bis-EMA, TEGDMA. Actually, the composition and filler size range are not similar to those of the nanohybrid materials used in the present study, in which the findings for the two nanohybrids (Kalore and N'Durance) do not agree with Wendler *et al.* Wendler *et al* aged Grandio SO[®] as substrate material for 30 days in DW before the application of surface treatments, but this was not performed for the substrate samples in the present study.

Based on the reviewed literature, neither Filtek Supreme XTE nor Heliomolar, respectively nanofill and microfill in categories, has been investigated for RBS after application of phosphoric acid. Therefore, this is the first study to show a statistically significant increase in SBS for the two materials after P600 plus phosphoric acid surface treatment.

Analysis of the SEM examination did show changes in RC photomicrographs due to phosphoric acid application. The RCs showed better appearance of the striation lines of the grooves, particularly for Heliomolar and Filtek Supreme XTE, and a decrease in the number of loose particles on the RC surfaces (Figure 5.49). These findings are in agreement with an earlier study by Fawzy et al. (2008), who showed clearance of microhybrid RC from resin debris and loose particles when the micrographs of prepared RCs with stone bur compared to those phosphoric acid added to the prepared surface. However, a number of other authors investigated prepared microhybrid RC with P400 before and after phosphoric acid etching with SEM but they did not comment on the photomicrographs (Ayar et al., 2018). Another study showed prepared nanofill RC with 4000 SiC grade, before and after application of phosphoric acid under SEM (X5000) but the authors cleaned the samples by ultrasonic device after preparing with SiC (Loomans et al., 2011a), the findings therefore is not comparable to those in the present study. Thus, it can be suggested that phosphoric acid can produce changes in surface morphology of RCs along with changes in roughness.

Based on the Ra, BAC, photomicrographic and SBS findings, the statistically significant changes in SBS were more compatible with the changes in the photomicrographs than those of roughness. This is owing to the obvious changes in striation lines for Filtek Supreme XTE and Heliomolar, which align to the statistically significant increase in SBS for the two RCs. Although this finding has not been

shown by other earlier studies in the literature, it indicates that changes in both roughness and surface morphology due to phosphoric acid application may impact on RBS *in vitro*. This finding also raises concern that, when RC samples are prepared and acid etched before any surface treatment, there is the possibility of a difference in RBS which is due to both roughness and surface morphology. Therefore, investigators should be aware of the differences in RBS between the samples that are prepared with SiC alone or prepared with SiC and then acid etched in *in vitro* because a number of studies used phosphoric acid after preparing RCs with SiC before application of surface treatments (Hannig *et al.*, 2006; Eliasson *et al.*, 2014).

The second chemical preparation was bonding agent applied to RC samples after they were treated with P600 plus phosphoric acid. The function of a bonding system is to assemble the substrate and the adhered RCs via different mechanisms, such as by chemical unification and micromechanical interlocking (Van Noort and Barbour, 2013). In the current experiment, SBS was investigated for the RCs treated with P600 plus phosphoric acid plus CP/BA, in comparison with the exclusion of either CP/BA or phosphoric acid plus CP/BA. Also, this investigation replicated the clinical use of CP/BA by dentists during RC restoration repair, as described within the questionnaire study in this thesis.

The adhesive systems available on the market are varied, but in clinical situations the ease of use based on minimal steps and lower risk of cross-infection may be preferred to more complicated multi-step products. Accordingly, in this study, acid etch was supplemented with a single step priming/bonding agent as this provided a simple and clinically relevant combination of materials available to a dentist.

CP/BA was in the form of a uni-dose instead of a bottle, to ensure that each uni-dose of bonding agent would be used for one patient. This uni-dose may ensure a reduction in the loss of some materials, such as ethanol, which may evaporate from the adhesive system if the cap of the bottle is left open too long. A composite priming bonding agent is likely to be preferred for RC repair to other bonding systems that do not contain a primer. However, there is evidence that the use of a priming bonding agent does produce a statistically non-significant higher RBS than other bonding agents, although the practice of using a primer might be beneficial for clinical situations with exposed dentine at the repair site (Rathke *et al.*, 2009). For the
current experiment, a CP/BA was selected because it is considered more relevant to the clinical situations in which RC and adjacent tissue may be involved in the repair process.

Based on the results, all RCs showed an increase in the level of SBS after adding CP/BA, and in particular N'Durance showed a statistically significant increase in SBS (nearly two folds) compared with excluding bonding agent (Figure 5.54). The relevant literature could not be found, however, a study compared RBS of prepared microhybrid and nanohybrid RCs with Soflex discs to those self-etch bonding agent added to them, those RCs self-etch bonding system added showed statistically significantly greater microTBS compared to those did not include bonding system (Lima *et al.*, 2016).

The second important finding was that all RCs showed statistically significant improvement in the level of SBS compared to the excluding of both phosphoric acid and CP/BA (Figure 5.54). Relevant evidence in the literature could not be found. Thus, this is the first study to use this approach to evaluate the effect of a single bonding system on multiple RC types. It is therefore likely that such an improvement exists in the SBS of repaired RCs in the clinical situation. However, there is no evidence of the possibility of having clinical differences in relation to *in vitro* significance values. More importantly (within limitations of this study), when dentists need to increase the level of bond of the repaired RCs in clinical practice, depending on chemical preparation only as a conservative approach, they could benefit from the use of both selective etching of RC and application of a CP/BA.

The results showed enhancement in SBS led to some of the RC SBS values reaching the recommended minimal required bond strength for clinical situations, which is between 18–20 MPa (Yesilyurt *et al.*, 2009). In this work, the SBS values for Kalore and Filtek Z250 treated with phosphoric acid plus CP/BA were 20.05 and 18.74 MPa, respectively. Also, Heliomolar bond strength value was very close to Filtek Z250, 17.74 MPa. This means that adding the bonding agent resulted in the repair SBS of most of the RCs being at the minimum level required for bond strength in clinical situations. However, the SBS values of N'Durance and Filtek Supreme XTE did not increase to this level. This indicates that the potential advantage of the bonding system is that it may enhance RBS on a number of RCs, so that it is strong

enough for the clinical situation. However, the shear test cannot represent all the forces that a repaired RC restoration faces in the mouth, and this is difficult to simulate *in vitro*.

Based on the reviewed literature, most of the studied RCs have not been investigated for RBS, except Filtek Supreme XTE and Filtek Z250. The categories of the studied RCs are illustrated in Table 5-1, and most studies investigated different products of RCs with categories similar to those used in the present study. The following section focuses on the categorical name of the RCs within published studies, bearing in mind that the RCs in the current study are identified by both categories and commercial names for clarity when compared to the published literature.

The SBS value for microhybrid (Filtek Z250) is in agreement with previous research, which showed 18 and 18.8 MPa RBS for microhybrid RC treated with SiC and bonding system (Kallio *et al.*, 2013; Lima *et al.*, 2014; Kaneko *et al.*, 2015). In contrast, a number of investigators found different values of RBS for microhybrid RC treated with a polishing system or a SiC or Mylar strip and bonding system from 11.93–17.53 or 22.7, or 31 MPa (Hannig *et al.*, 2006; Cavalcanti *et al.*, 2007; Veiga de Melo *et al.*, 2011; Özcan *et al.*, 2014; Ghavam *et al.*, 2018).

For the two nanohybrids (Kalore and N'Durance), SBS values were between 15–20 MPa. This is in line with (Rinastiti *et al.*, 2010), who found a similar range (15.8 to 19.9 MPa) of immediate RBS after the application of a bonding system on two nanohybrid RCs polymerised against Mylar strip. Similarly, El-Asirarya *et al.* (2012) showed 18 MPa bond strength on nanohybrid RC prepared with 320 SiC and treated with three-step etch and rinse adhesives. In contrast, a number of researchers have found different RBS values for nanohybrid than those in this study 28.6 and 42 MPa for Eliasson *et al.* and Baena *et al.*, respectively (Eliasson *et al.*, 2014; Baena *et al.*, 2015).

The SBS for nanofill (Filtek Supreme XTE) was 13.29 MPa in this study, but the published data showed RBS for nanofill RCs to be different – 14.62 and10.92 MPa by Rinastiti *et al.* and Ghavam *et al.*, respectively (Rinastiti *et al.*, 2010; Ghavam *et al.*, 2018).

Similarly, for microfill (Heliomolar) treated with phosphoric acid and prime and bond by Lucena-Martín *et al.* (2001), RBS showed a lower value of 5.96 MPa than the SBS value in this experiment. The differences in RC type, ageing, and method might have affected the results in the studies which contradict my findings, and this illustrates how methods of other studies influenced their values compared to those in the current study.

This study suggests the importance of using phosphoric acid plus bonding agent, not only to enhance the statistically significant level of SBS, but also potentially to improve the level of required bond strength in clinical practice. However, the RC RBS value in clinical practice suggested by a number authors (Yesilyurt *et al.*, 2009) was not based on any investigation.

The third chemical preparation investigated silane by adding it to phosphoric acid and bonding agent as a surface treatment. The results showed silane increased SBS for Filtek Z250 was (29.3 MPa), the SBS value was two-folds and statistically significant greater when compared to the exclusion of silane (9.9 MPa) (Figure 5.56). The reason for this increase in SBS was not investigated in this study, but numerous investigators have suggested possible explanations for the mechanism of the action of silane. Applying silane agent increases close contact between the substrate and repair RC due to the chemical bond between the glass or silica fillers being exposed on the substrate and the resin matrix of the repair material (Maneenut et al., 2011; Joulaei et al., 2012; Imbery et al., 2014; Matinlinna et al., 2018). In the current study, only Filtek Z250 was included in the analysis because of the problems encountered with producing and then testing samples, as described in Section 5.6.2. Filtek Z250 is a microhybrid RC with a filler type composed of silica and Zirconia. Fornazari et al. (2017) used a nanofill RC, prepared with SiC P600 grit, containing silica and zirconia fillers, and there was a statistically significant increase in RBS with the use of silane and a bonding agent which did not contain materials that promote chemical reaction with fillers. The study of Fornazari et al. reported that RBS changed from 8.4 to 15 MPa when compared to the application of bonding agent alone, which is nearly twice that of the bonding agent alone. However, the values in the current and Fornazari et al. studies are dissimilar, as the results showed an approximately similar range of increase in RBS; that is, it was nearly twice greater than the RBS without silane. The difference in SBS values between this study and those of Fornazari et al. is likely due

to the difference in the material categories. The current study used microhybrid RC, which contains filler size in the range of $0.01-3.5 \mu m$, larger than those of nanofill RCs that are usually between $0.004-10 \mu m$. However, Fornazari *et al.* did not mention the filler size of the nanofill RC they used. In contrast, Kaneko *et al.* (2015) showed statistically non-significant change in TBS when silane was added to bonding agent on microhybrid RC prepared with P600 SiC. The authors did not provide information on the composition of the resin composite. From these findings, it is suggested that the current study did not confirm that silane surface treatment to RCs has the potential to improve the level of bond strength.

For the two problems happened within the silane experiment (noted in the results of silane experiment), a solution could not be found. Although more than one month was spent trying to identify and solve this problem, no solution was found and so this line of experimentation was abandoned in preference for investigating other surface treatments. It was not believed the problem of changing the shape of acrylic blocks to be due to the acrylic material used around RC samples. This was owed to two reasons. Firstly, previous studies have used acrylic resin for the block around the RC samples for the shear test, but none which included silane in the treatment reported any of the aforementioned problems in their experiments (Brendeke and Özcan, 2007; Rathke et al., 2009; Veiga de Melo et al., 2011; Ahmadizenouz et al., 2016; Fornazari et al., 2017). Secondly, the problem of the change in block shape under shear test remained even when the blocks were changed to polyester, because polyester has more resistance to softening than acrylic resin. Furthermore, this problem was not clear to be due to the composition of silane material because the manufacturer did not provide the chemistry of the silane. However, Brendeke and Özcan (2007) used silane with a commercial name similar to that in this study (ESPE-SiL®), and they applied it for five seconds and followed it with the application of a bonding agent. The method is guite similar to the method used in the current silane experiment. The second problem of extruding RC samples from acrylic blocks under shear test, before failure in bond, between substrate and repair RCs happen, it was not believed to be due to an increase in RBS, where the mean and median SBS were 29.3 and 30.89 MPa, respectively. The samples treated with SiC plus phosphoric acid and bonding agent showed similar SBS mean 29.4 MPa, where even the samples located in the periphery of the resin blocks showed no extrusion; thus, the interaction of silane with resin blocks may be the reason.

The following paragraphs focus on the performance of RCs during different surface treatments. Firstly, the performance of RCs is discussed when prepared with P600. RCs showed statistically significant difference in Ra and BAC parameter values even when prepared with the same SiC grade (P600) (Figure 5.14). Kalore showed a statistically significant higher value of Ra compared with Heliomolar. All other materials showed a statistically non-significant difference in Ra. Lee et al. (2002) investigated Ra for five RCs after preparing with SiC P600. The authors showed statistically a significant difference between most of the RCs. The studied RCs were different from those used in the present study, and the authors did not provide information on filler size and categorical names. Generally, RC roughness was affected by both the polishing systems or SiC grades and the RC itself (Erdemir et al., 2013; Goncalves, 2018), particularly filler characteristics (McCabe and Walls, 2008). There are a number of explanations to this finding, however, one of the explanations was investigated in this thesis. According to the manufacturer's information on filler characteristics revealed in Table 5-1, Kalore includes larger and irregular filler particles with a high filler loading by weight, while Heliomolar contains smaller filler particles with the lowest filler loading by weight. A number of studies have shown that large filler size is a factor that results in greater roughness, such as Ra (da Costa et al., 2010), because this filler size may protrude from the surface of the resin matrix more than smaller sized filler; large filler particles also enhance roughness even during extrusion because they leave large craters compared with those of small filler particles (Kaplan et al., 1996; Barbosa et al., 2005; Rodrigues-Junior et al., 2015).

Another explanation is filler shape and interparticle spacing. Irregular fillers contribute to greater roughness while spherical ones lead to lower Ra (Marghalani, 2010). Interparticle spacing is another factor in roughness, as irregular particles result in more interparticle spacing than spherical particles (Marghalani, 2010). As the interparticle spacing increases, the resin matrix between the particles is more easily challenged (Yilmaz *et al.*, 2017) and this further increases interparticle spacing and roughness (Oliveira *et al.*, 2012). However, interparticle spacing is decreased in RCs by incorporating small filler particles between large ones (Jaarda *et al.*, 1997). Except for Heliomolar, the studied RCs contain a wide range of filler sizes based on the manufacturer's information (Table 5-1). For example, when comparing Ra values between Filtek Supreme XTE with Filtek Z250, it is interesting to note that they

showed statistically non-significant difference in Ra even though they are obviously different in filler particle size. The presence of small nano-particle size filler particles is likely to offset the influence of large sized nanoclusters (10 μ m). Similarly, there was a statistically non-significant difference in Ra between Kalore and N'Durance, which both contain irregular filler particles but with different filler size range. The presence of nanoparticles (0.1 μ m) and smaller glass fillers (0.7 μ m) likely equalises the impact of large prepolymerised filler particles (17 μ m) in Kalore.

The difference in roughness profile between RCs were not due to peaks or valleys individually. The use of BAC parameters confirmed the difference between RCs in roughness were due to difference in peaks, cores and valleys and proportion of peaks and valleys together. For example, the higher Ra of Kalore compared to Heliomolar was not due to the presence of higher Rpk only, but also higher Rk, Rvk Mr₁ and Mr₂ (Figure 5.16, Figure 5.18, Figure 5.20, Figure 5.22, Figure 5.24). This finding also explains why the finding from Ra and BAC parameters were in align with each other because Ra measurement dependent on core with valleys and core with peaks heights. Therefore, quantification of BAC parameters could be considered to be a useful for ensuring how the difference between RCs in roughness profile is because if Ra used alone the information on the peaks, valleys and cores would not be identified.

The statistically significant differences detected in Ra and BAC parameter values between RCs did not relate to the categories of microhybrid, nanohybrid, nanofill and microfill of the studied RCs (Table 5-1). For example, microhybrid had a statistically non-significant difference compared to all other RCs in Ra, Rpk, Rk and Rvk. These results corroborate the ideas of Costa *et al.* (2007) and Sadeghi *et al.* (2016), who presented statistically non-significant differences in Ra between microhybrid, microfill and nanofill. Further, the two nanohybrids, Kalore and N'Durance did not behave similarly in the present study. Kalore did exhibit a statistically significant difference in Ra compared to microfill (Heliomolar), while N'Durance showedNS difference in Ra to those of microfill. Similarly, the polymer systems did not affect the difference between RCs in roughness (Table 5-1), Kalore is based on UDMA and Filtek Z250 on Bis-EMA, but RCs showed statistically non-significant difference in roughness between RCs due to the monomer system before ageing.

Based on the SBS findings, RCs behaved differently even when they had the same surface treatment. Both Filtek Z250 and Kalore were statistically significantly stronger in SBS compared to Heliomolar and Filtek Supreme XTE, but N'Durance revealed statistically non-significant difference with all RCs. The SBS findings cannot be directly linked to roughness parameters as the statistically significant differences between RCs in Ra and BAC do not match with the statistically significant difference difference in Ra and BAC was between Kalore and Heliomolar. While in SBS Filtek Z250 also showed statistically significant difference with Heliomolar. Another example is that N'Durance presented statistically significantly lower Mr₂ value weaker compared to most of the other RCs, but in SBS N'Durance showed statistically non-significant difference with other RCs.

The surface morphology showed to some extent a link to SBS. It is interesting to note that the smoother surfaced morphology of the RCs was statistically significantly lower for SBS than those with a rougher surface. At low magnification (500X) of SEM examination, both Heliomolar and Filtek Supreme XTE showed a smoother surface than the other RCs. Both RCs showed statistically significantly lower SBS than Filtek Z250 and Kalore, but at high magnification N'Durance showed smoothness close to those of Filtek Supreme XTE, although N'Durance had a statistically non-significant difference with all RCs for SBS.

From SBS results the performance of RC in relation to category (microhybrid, nanohybrid, microfill, and nanofill) (Figure 5.30) was identified. Note to forget no evidence in the published works was found that compared RCs without chemical treatment, or prepared with P600, which used either a tensile or shear bond strength test. However, a number of studies have compared RC category in *vitro* using different surface treatments.

Microhybrid was statistically significantly stronger for SBS than nanofill. Microhybrid had statistically significantly stronger TBS than nanofill RC (Junior *et al.*, 2009), while Rinastiti *et al.* (2010) showed statistically significantly greater SBS for nanofill than microhybrid; the authors did not age the samples but did treat the RCs with silica coating (30 μ m). In Rinastiti *et al.*, with Mylar and bonding agent surface treatments, there was a statistically non-significant difference between the materials. Therefore,

the statistically significantly higher SBS of microhybrid over nanofill is not supported by published work.

Microhybrid showed statistically significantly and two-fold greater SBS compared to microfill. Two studies showed higher microTBS of microhybrid compared with microfill; nonetheless, the authors did not consider comparing the two materials for statistically significant difference (Loomans *et al.*, 2011b; Maneenut *et al.*, 2011), but microhybrid showed less than half-fold greater in SBS compared with microfill.

The two nanohybrid RCs presented a statistically non-significant difference between them, but one of the nanohybrids (N'Durance) showed a statistically non-significant difference in SBS with all RCs. The findings from the literature do not agree with this study finding. Rinastiti *et al.* (2010) showed a statistically significant difference between the two nanohybrids.

There was a statistically non-significant difference in SBS between microhybrid and the two nanohybrids for SBS. In contrast, Altinci *et al.* (2018) showed nearly two fold higher bond strength of microhybrid than nanohybrid.

One impact of prepared RC types with SiC on SBS was pre-test failure. Of the prepared RCs with SiC, Filtek Supreme XTE had a number of pre-test failures for the four SiC grades. The load extension graphs were checked in terms of whether there was a bond between the RC and RC. Therefore, the samples that failed before or even under the shear test but did not show adhesion were not considered as zero MPa. There are two explanations for this finding. One is that resin debris had accumulated on the surface and rinsing with water did not remove it because: first, the smaller interparticle space due to the high filler loading; and, second, the spherical particles might have meant that the rinsing water had less access to the resin debris and abrasive particles. In addition, uncontrolled loading during preparation with SiC may be another factor, as the samples with heavier loading during preparation with SiC generated heat and this affected the further DC in the resin matrix (Ferracane and Condon, 1992; Lucena-Martín et al., 2001), which minimised the advantage of the chemical interaction for these samples in comparison with other RCs. One study reported the exclusion of pre-test failure in the analysis, but this was not considered strong enough RBS (Ozcan et al., 2013a). However, Özcan et al. used a tensile test in which some pressure might have been produced on the samples during the cutting of the block material, and they did not evaluate with LEC graph to clarify if there was any bond, even for very weak values. The findings demonstrated that there is no general acceptance of how much bond strength in tensile or shear testing should be considered as a pre-test failure, or which procedure should be followed to identify pre-test failure. The pre-test failed samples were not included in the data analysis in this experiment due to failure in adhesion rather than low adhesion, and the load extension graph was checked for this purpose.

Secondly, the following paragraphs discuss how RCs performed when phosphoric acid added to RCs prepared with P600 and how much it was differ from those prepared with P600.

The number of statistically significant differences in Ra and BAC between RCs increased compared to those RC samples prepared with P600. (Figure 5.34, Figure 5.36, Figure 5.38, Figure 5.40). Furthermore, the statistically significant higher Mr1 and Mr₂ changed from those RCs prepared with P600 (Figure 5.42 and Figure 5.44). Filtek Supreme XTE was statistically significantly higher in Mr1 and Mr2 compared to Filtek Z250 and Kalore, while for RCs prepared with P600 this was not the case. This is because some of the RCs showed a decrease while others increased in Ra and BAC parameters` values; occasionally, a RC did not show any change in Ra and BAC parameters' values (Figure 5.35, Figure 5.37, Figure 5.39, Figure 5.41, Figure 5.43, Figure 5.45). Moreover, none of the RCs always showed a decrease or increase over Ra and all BAC parameters. When the roughness of prepared RC samples with P600 was compared to those treated with P600 plus phosphoric acid, Filtek Z250 showed an increase in Rvk but a decrease in Mr₁. The photomicrographic examination in both magnifications did not reveal changes in roughness or exposed filler particles (noted within phosphoric acid surface treatment). The limitations of the previous studies is not only in that they used no more than two types of RCs, and they prepared the substrate in different ways. It is not clear what the consequence of this is on the outcomes. Loomans et al. (2011a) compared two RCs, but they used one roughness parameter to quantify roughness profile which was representing average measurement. Thus, their findings could not be compared with BAC parameters. Generally, the findings showed changes in roughness and photomicrographs indicating phosphoric acid minimally eroded the resin matrix without affecting the filler particles or their interface with resin matrix. It's important to show if these changes in roughness and surface morphology how impact on SBS.

The difference in performance in roughness between RCs treated with P600 plus phosphoric acid did not link to categories of RCs. All the RCs based on microhybrid, nanohybid, nanofill and microfill showed increase in roughness over all the parameters due to the application of phosphoric acid, and this change was not specific to any category (example given within previous paragraph). In the same way, the difference in performance in roughness between RCs treated with P600 plus phosphoric acid did not relate to monomer systems. Filtek Supreme XTE and Filtek Z250 are based on Bis-GMA monomer, but they were statistically significantly difference in peak proportions (explained within previous paragraph).

Based on the SBS findings, RCs performed differently in SBS (Figure 5.48). Two of the RCs Filtek Supreme XTE (P<0.1) and Heliomolar (P<0.001) presented statistically significant increase in SBS, the other increases were statistically nonsignificant. The behaviour of N'Durance was not expected because it has nearly as smooth a surface as Heliomolar, and the surface was clear of loose debris in the same way as Kalore, but showed no change in SBS. N'Durance has recently been introduced to the market, and, to the best of my knowledge, there is limitation in knowledge on N'Durance in relation to RBS and surface treatments during repair. Furthermore, a published study could not be found in the literature that compared more than one product of RC after phosphoric acid surface treatment. One possible explanation of increase in SBS may be due to better microretention between the substrate and the repair material. However, this possibility has not been tested either in this study or other published literature. Another possible explanation for the statistically significant increase in SBS is unreacted monomers, as suggested by a number of authors, although they did not examine it (Veiga de Melo et al., 2011). In contrast, it has been found that the DC in resin matrix does not impact SBS (Ozcan et al., 2013a). The implication from phosphoric acid finding is that statistically significant increase in SBS may indicate it is highly likely that phosphoric acid increases SBS in the clinical situation, at least for some of the RCs.

Based on the changes that occurred in SBS, the statistically significant differences between the RCs were higher in number in P600 plus phosphoric acid treatment than those RCs prepared with P600. Within P600 plus phosphoric acid treatment, Heliomolar also showed greater statistically significant SBS than N'Durance, which was not the case in the prepared samples with P600 (Figure 5.47). This happened because some of the RCs showed more increase in SBS compared with the others.

Thirdly, RCs performed differently in SBS by adding bonding agent to RCs treated with P600 plus phosphoric acid. Furthermore, the statistically significant difference in SBS between RCs decreased compared to the exclusion of bonding agent (Figure 5.47) and Figure 5.53). The only differences were those found between Filtek Supreme XTE showed statistically significantly lower SBS compared with Kalore and Filtek Z250 (P<0.001 and P<0.1), respectively, and this two statistically significant difference were lower in number than those between RCs treated with P600 plus phosphoric acid. One factor of decreasing statistically significant differences in the number in SBS between RCs was that RCs reacted differently to the bonding agent (Figure 5.54). For example, N'Durance showed a statistically significant increase in SBS but Filtek Z250 was statistically non-significant and had a minimal increase in SBS. Similar findings in the performance between RCs were also reported by Rinastiti et al. (2010), who showed that due to the application of bonding agent to Quadrant Anterior Shine (microhybrid), Grandio (nanohybrid), Tetric Evo Ceram (nanohybrid) and Filtek Supreme XT (nanofill), only Filtek Supreme XT had a statistically significant increase in SBS compared to the other RCs. However, the number of statistically significant difference increased than before application of bonding agent. Rinastiti et al applied bonding agent to RCs polymerised against Mylar strip.

However, earlier RC repair studies have not identified the effect of bonding agent based on RC roughness. In the present study, it is believed that the effectiveness of bonding agent is likely related to the RC substrate roughness, where fluids accumulate within the irregularities of RC roughness (Field *et al.*, 2010), and would provide microretention after setting. One of the roughness measurements evaluated in this thesis was Rvk, which is the parameter which calculates the height of the roughness profile which holds fluid, so it can be used to evaluate the bonding agent's thickness (with disregard to the effect of flow and contact angle of the CP/BA within RCs). Therefore, Rvk values were used to investigate the impact of CP/BA on improvement in SBS based on roughness. In this experiment, the roughness of RCs was not measured because CP/BA was applied to the surface of the materials and polymerised by light curing. It was believed that the roughness measure for the RCs in this situation would not represent the real roughness of RCs. Further, no benefit for measuring the RCs in this situation was indicated in relation to RC repair, and it is not a reproducible method. Therefore, the roughness of P600 plus acid etch was

used to represent the surface roughness of RCs. Based on the findings, the differences in roughness between RCs did not impact on SBS. The Rvk of Heliomolar was lowest and Kalore the highest in Rvk values (Figure 5.40), but the SBS was enhanced in nearly the same way for both materials (Figure 5.53). In the P600 plus acid etch surface treatment, the Rvk of Filtek Z250 became more statistically significant compared to P600 (Figure 5.41), although this increase in roughness was accompanied by a statistically non-significant increase in SBS (Figure 5.54). Indeed, surface treatments for N'Durance led to a statistically non-significant change in Rvk, and there was a statistically significant increase in SBS when CP/BA was applied. Thus, it can be concluded that bonding agent effectiveness was not directly related to RC roughness.

In this experiment, the evaluation of the valleys of the RCs, in which fluids accumulate, is one of the advantages of using BAC calculation. As for the Rvk, it was possible to evaluate the impact of RC roughness on the effectiveness of bonding agent.

Another possible explanation of the difference in performance between RCs in terms of increase in SBS may be due to improvement in the level of micromechanical interlocking between the substrate and the repair material, which have been assumed to be one of the mechanism of action of the bonding agent. One study showed that the application of bonding system did not improve RBS, and the authors explained the findings by the presence of good micromechanical interlocking between the substrate and the repair materials, even before the application of adhesive promoters (Hamano et al., 2011). In the current experiment, the level of SBS did not increase statistically significantly for Filtek Z250 when treated with P600 plus acid etch plus bonding agent in comparison to those treated with P600 plus acid etch. For this finding, one possible explanation is that micromechanical interlocking between the substrate and the repaired Filtek Z250 material was superior to such an extent that the bonding agent did not contribute to a statistically significant increase in the SBS level. For the statistically significance improvement in SBS of N'Durance after the application of CP/BA, again, one possible explanation is that the bonding agent likely produced better micromechanical interlocking between the two surfaces compared to those between before adding CP/BA. Further explanation is that the chemical reaction compatibility between CP/BA with RCs monomer system were different in an extent influenced SBS level. It is likely that reaction with N'Durance

was superior, but lowest with Filtek Supreme XTE. The chemical bond between RC and bonding agent monomers has not been tested by any repair studies but have been assumed by a number of authors to be one of the mechanism of action (Eliasson *et al.*, 2014; Irmak *et al.*, 2017).

The statistically significant increase in SBS suggests the bonding agent has the potential to improve bond strength in a clinical situation, at least for some of the RCs. However, these factors need to be tested in clinical practice before final recommendations can be made. The findings from CP/BA investigation showed how differently RCs` SBS change when CP/BA added. Within limitation of this experiment, it showed to be advantageous for the SBS level because all RCs` SBS increased even some of was statistically non-significant.

Without consideration of monomer compatibility in chemical reaction between them, the covalent bond between the unreacted monomer on substrate with the adherent monomer was identified as an explanation of the statistically significant higher RBS on non-aged RCs (Veiga de Melo *et al.*, 2011). If true, it does not appear that unreacted monomers in the resin matrix of the RCs impacted on the SBS level. If the statistically significant increase in SBS is affected by unreacted monomer within the substrate, N'Durance would be one of the materials minimally affected by bonding agent, but this was not the case in this study. According to the manufacturer's information, the monomer system for N'Durance provides a high degree of double bond conversion, which was identified to be higher than Filtek Z250 RC (Boaro *et al.*, 2013). Nevertheless, the bonding system statistically significantly enhanced SBS for N'Durance.

The profilometer was able to detect differences in roughness profile between surface treatments and resin composites. This is owed to the following points:

 For the SiC grades, there was not only a difference between SiC grades in roughness profile, but also the difference in roughness profile between RCs changed from one grade to another one. P600 showed the least statistically significant difference between RCs than the other grades. These difference in roughness profile for both SiC and RCs was comparable with a number of earlier studies (Lee et al., 2002; Endo et al., 2010; Kallio et al., 2013). 2. When phosphoric acid added to RCs prepared with P600 the roughness profile changed from those prepared with P600, even the change was statistically non-significant. Every RCs showed difference amount of change in roughness profile, this reflected on that the statistically significant difference between RCs increased in number compared to those prepared with P600. Some of these findings are comparable with earlier studies even they used optical methods for measuring roughness profile (Fawzy et al., 2008; Loomans et al., 2011a), as explained with previous paragraphs.

Similar to profilometer, the shear test was able to detect differences in SBS between surface treatments and resin composites. This is explained in the following points:

- SiC grades resulted in difference in SBS. RCs showed statistically significant difference between them and this difference was greater in number for samples prepared with P600 and became less in number with the other grades. Some of these findings are comparable with an earlier study (Kallio et al., 2013) that used SBS.
- Adding phosphoric acid to prepared RCs with P600, led to increase in SBS. Every RC behaved differently and this mirrored in increasing the statistically significant difference between RCs compared to RCs prepared with P600. Some of these changed have been shown by an earlier study Fawzy et al. (2008). The detail of that study explained in former paragraphs.
- 3. Adding CP/BA to RCs treated with P600 plus phosphoric acid RCs increased SBS further than P600 plus phosphoric acid surface treatment, some of the increase in SBS was statistically significant. Every RCs showed increase in SBS, but they were difference in amount and this reflected on decreasing the statistically significance difference between RCs in number compared to those prepared and etched. A number of these findings are comparable with earlier studies that used similar or other types of RBS tests. For example, Kallio et al. (2013) and Rinastiti et al. (2010) used a SBS test, and Lima et al. and Kaneko et al. used micro-tensile or tensile tests, respectively.

Generally, the performance of RCs within different surface treatments was not directly related to their categories and monomer systems.

For the prepared RCs with SiC no direct relation between RC category and roughness profile could be identify. This is owed to that the only statistically significant change was between one nanohybrid and microfill when prepared with P600, but this difference increased between RCs over other SiC grades. If category was the only factor influencing roughness, the profile roughness between RCs would have remained the same over all other SiC grades. Similarly for SBS, if the category was the only factor the statistically significant difference between RCs would have remained the same over all SiC grades. For the monomer system, again if it was the main factor the difference between one nanohybrid which is based on UDMA monomer and the microfill which is based on Bis-GMA would have not changed. Moreover, the other RCs that are differed in monomer system would had showed statistically significant difference and Filtek Z250.

For the RCs treated with P600 plus phosphoric acid, a direct link between categories with roughness profile and SBS could not be found. The statistically significant difference in roughness profile between RCs increased when phosphoric acid added to RCs prepared with P600. If RC category was the main factor, this change would not have occurred. Also for SBS, if category was the main factor all RCs that have different category would have showed statistically significant difference between them. In contrast, the two nanohybrid materials performed differently because one of the nanohybrids (N'Durance) had statistically non-significant difference in SBS than other RCs. However, the second nanohybrid material (Kalore) revealed greater statistically significant SBS than nanofill and microfill. Moreover, microhybrid and the two nanohybrids showed a statistically non-significant difference in SBS, and the RC performance was not linked to monomer type in SBS. Both Heliomolar and Filtek Z250 contain Bis-GMA but the increase in the level of SBS was statistically significant different between the two materials.

For RCs treated with P600 plus phosphoric acid plus bonding agent, the difference in performance between RCs in SBS, were not directly related to category. N'Durance was one type of nanohybrid used but only this one showed a statistically significant increase in the level of SBS, and Kalore, the other nanohybrid, did not show a statistically significant change in the level of SBS. None of the other categories of RCs showed statistically significant increase in the SBS due to the use of bonding agent. Similarly, the monomer system did not show any direct relation to the

statistically significant increase of the level of SBS when treated with bonding agent. Both Kalore and N'Durance contain UDMA monomer but only one of them showed statistically significant increase in SBS by adding CP/BA.

In summary

Some of the SiC grades and surface treatments as a form of chemical preparation for RC repair investigated in this section resulted in statistically significant changes in roughness and SBS, and prominent change in surface morphology for the studied RCs. Furthermore, RCs performed differently over SiC and surface treatments.

First, SiC grades impacted on Ra, BAC and SBS, where a SiC paper with larger grade contributed to a rougher surface and a SiC paper with a smaller grade contributed to a smoother surface in Ra. Generally, RCs prepared with P600 and P800 showed statistically significantly more roughness (Ra) compared to those of P1000 and P1200. BAC parameters confirmed the Ra findings, because BAC distribution showed a decrease in roughness profile height when RCs were prepared with a smaller SiC grade. However, BAC parameters showed a statistically significant increase in peak proportion for some of the RCs, but this increase was equalised by a statistically significant increase in valleys too. Therefore, SiC grades affected both peaks and valleys.

SiC grade contributed to a statistically non-significant difference between RCs in SBS. The statistically significant changes in SBS were not aligned with the statistically significant changes in Ra and BAC parameters. Further, RCs treated with P600 showed more statistically non-significant differences between them in Ra and BAC findings compared to the other grades. Therefore, P600 was thought to produce a level of consistency in roughness and therefore selected to prepare RC specimens than the other studied grades when better consistency is considered between RC specimens.

Second, phosphoric acid increased SBS level for all RCs which was statistically nonsignificant compared to RCs prepared with P600. Both Filtek Supreme XTE and Heliomolar showed a statistically significant increase in SBS compared to P600 preparation. Both materials also showed a prominent change in surface morphology due to phosphoric acid application, such as change of blurred striation lines to clear ones. Thus, the increase in SBS appeared to be more aligned with the prominent changes in surface morphology than the changes in roughness, because all RCs showed a statistically non-significant change in Ra, and most BAC parameters, when phosphoric acid is applied to RCs prepared with P600. Similar to SiC grades, phosphoric acid affected peaks of roughness profile, and also core and valleys.

Third, P600 plus phosphoric acid plus bonding agent enhanced SBS level for all RCs which was statistically significant for N'Durance compared to P600 plus phosphoric acid treatment. Furthermore, this surface treatment resulted in a statistically significant increase in SBS for all RCs compared to the RCs prepared with P600. It is interesting to note that phosphoric acid plus bonding agent improved RC SBS without the need for modifying the substrate material by cutting at micro and macro levels; in other words, minimal intervention is required.

Finally, when silane was added to the above surface treatments, the finding from one RC presented, Filtek Z250, which showed a statistically significant increase in SBS compared to the exclusion of silane.

RCs performed differently due to the application of surface treatments. Preparing RCs with P600 resulted in statistically significant differences between them in Ra, BAC and SBS and obvious difference in surface morphology. Kalore showed statistically significantly rougher surface compared with Heliomolar. While only Filtek Supreme XTE and Heliomolar showed smoother surface morphology at X500 due to blurred striation lines. Both Filtek Supreme XTE and Heliomolar had statistically significantly lower SBS compared to Filtek Z250 and Kalore. Filtek Supreme XTE and Heliomolar showed a much smoother surface morphology than Filtek Z250 and Kalore. The statistically significant differences in SBS were more related to surface morphology smoothness than smoothness from Ra and BAC parameters. The use of Ra and BAC parameters and photomicrographs allowed having more detailed information on the roughness of prepared RCs with P600 before surface treatment could promote more precise interpretation of the SBS results.

Phosphoric acid resulted in clear striation lines and the removal of loose particles on RCs prepared with P600, particularly for Filtek Supreme XTE and Heliomolar. These changes were in a line with the changes in the level of SBS. Both Filtek Supreme XTE and Heliomolar showed a statistically significant increase in SBS, but the other RCs showed statistically non-significant increase in SBS level. While, the changes in roughness was not directly related to the statistically significant changes in SBS

When bonding agent was added to RCs treated with P600 plus phosphoric acid, the level of SBS increased which was statistically significant for N'Durance only. This increase in SBS was not directly related to Rvk of RCs

In conclusion:

- Different SiC grades (P600, P800, P1000, and P1200) contributed to the differences in roughness within RCs. RCs prepared with P600 and P800 showed statistically significant greater roughness than those treated with P1000, P1200.
- SiC grades did not contribute to the statistically significant difference in SBS.
- SiC grades affected the consistency in roughness between RCs. RCs treated with P600 showed better consistency in roughness compared to those treated with other grades.
- Phosphoric acid treatment generally increased SBS, and this was statistically significant for two of the RCs, Filtek Supreme XTE and Heliomolar. This increase in SBS aligned to the change in surface morphology.
- The addition of a bonding agent statistically significantly improved SBS for N'Durance only. The statistically significant increase in SBS was not in alignment with the statistically significant changes in roughness
- The sequential use of P600 plus phosphoric acid plus bonding agent resulted in the overall greatest increase in SBS for all RCs compared to preceding surface treatments. These increases in SBS were statistically significant in comparison to P600 preparation for most of the RCs with the exception of Filtek Z250 which showed statistically non-significant increase in SBS for all treatments.

5.8 Mechanical without/with chemical preparations method

In this section SBS and roughness of five RCs were investigated after surface treatments with medium coarse diamond bur; bur plus acid etch plus bonding agent without or with silane.

5.8.1 Mechanical preparation method

In this section SBS and roughness of five RCs were investigated after surface treatments with medium coarse diamond bur.

1. Sample preparation

The samples were prepared using the method described in Section 5.2.1. However, in this experiment RC samples were placed closer to the border of resin blocks, as revealed in Figure 5.59. This allowed the cutting bur to reach the RC samples during surface treatment. Before pre-treatment with the bur, the samples were prepared with P600 SiC using the method described in Section 5.3.1. Fifteen samples were prepared for each of the RC types, as summarised in Table 5-1.



А

В

Figure 5.59 The top view (A) and lateral view (B) of the test sample consisting of a RC sample embedded in an acrylic block. The samples were identified using letter and numeric codes. Both RC and surface treatment types were coded with different letters.

2. Sample surface treatments

Surface treatment was performed with a medium coarse round ended cylindrical diamond bur (95-130 µm,ISO 806314 199524 01410.0, Switzerland) using a high speed air motor (40,000rpm) (KAVO Super Tourque, model 625, reference number A46999) for five seconds under water cooling. The RC samples were then washed with air/water spray for three seconds.

3. Measurement of RC roughness

The surface of the RCs was profiled as described in Section 5.3.2.

5.8.2 Combined mechanical and chemical preparation methods

1. Sample preparation

The samples were prepared using the method described in Section 5.8.1.

2. Sample surface treatments

Surface treatment was performed with a diamond bur using the method described in Section 5.8.1. Then phosphoric acid and bonding agent were applied using the same materials and methods described in Section 5.5.1.

5.9 Mechanical without/with chemical preparation results

In this experiment firstly, the individual effect of mechanical preparation by the use of diamond bur was evaluated by comparing bur treatment to the RCs prepared with P600 SiC. Secondly, the synergistic effect of mechanical and chemical preparations were used within previous experiments in this chapter were evaluated. For both experiments the outcome measures of roughness and SBS were used.

5.9.1 Mechanical preparation results

A typical roughness profile of each RCs treated with the bur is shown in Figure 5.60. The vertical axis of the figures displays the peak heights and valley depths of the RCs that are below 30 and above $-30 \mu m$. All RCs appear to be similar in roughness profile without any obvious difference in height.

In Figure 5.61, typical BAC for each material treated with the bur is presented. The triangular area formed from above the blue line and below the red line represent the peak and valley, respectively. Both these parts occupy a small portion of the curve, and the middle segment represent the core part of roughness. The sloping angle of the red line seems to be of a similar steepness for all RCs. The two yellow vertical lines represent the material ratio of roughness. The yellow line on the right side represents the material ratio at peak (Mr₁), and the area between the two vertical yellow lines represents the material ratio at valley (Mr₂). The material ratio at the peak is moderately smaller in comparison to the material ratio at the valley. The BAC curve for all RCs in this experiment was similar to those prepared with P600, as described in Section 5.4, with a few exceptions. The RCs treated with bur shows a similar width for both Mr₁ and Mr₂, and the horizontal red line in Figure 5.61is steeper in slope than those prepared with P600 only.



Figure 5.60 Typical stylus profilometry profile for roughness on RC samples treated with a bur, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.



Figure 5.61 Typical bearing area curve for a roughness on RC samples treated with a bur, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.

<u>Ra</u>

Analysis of data revealed that N'Durance and Kalore had statistically significant lower Ra compared with those of Filtek Supreme XTE, Filtek Z250 and Heliomolar, the P values are shown in Figure 5.62.

As can be seen in Figure 5.63, further analysis of data illustrated that Ra for RCs treated with the bur was statistically significant higher than those treated with P600 grit SiC paper, the (P<0.001) for all RCs.



Figure 5.62 Ra level of RCs treated with bur.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Ra (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * mark P \leq 0.05, ** P<0.01 and *** P<0.001.



Figure 5.63 Ra level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 or bur represent the two treatments. Bar charts represent median and the vertical lines IQR of Ra (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of *** mark P<0.001.

<u>Rpk</u>

Analysis for Rpk data revealed statistically significant lower and higher roughness were for N'Durance and Filtek Z250 individually. N'Durance typically showed statistically significant lower Rpk compared with Heliomolar and Filtek Z250. Also, Filtek Z250 was statistically significant higher in Rpk compared with Kalore and Filtek Supreme XTE. The P values are illustrated in Figure 5.64.

Similar to Ra, RCs treated with bur compared to those treated with P600. The bur treatment showed statistically significant higher Rpk compared with P600 for every RC (P<0.001), see Figure 5.65.



Figure 5.64 Rpk level of RCs treated with bur.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Rpk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * mark P \leq 0.05, ** P<0.01 and *** P<0.001.



Figure 5.65 Rpk level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 or bur represent the two treatments. Bar charts represent median and the vertical lines IQR of Rpk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of *** mark P<0.001.

<u>Rk</u>

The differences between RCs are similar to those revealed for Ra. N'Durance and Kalore were statistically significant lowest values than other RCs, for Pvalues see Figure 5.66. In addition, the Rk for the RCs treated with bur showed statistically significant higher values compared to those treated with P600 (P<0.001), as explained in Figure 5.67



Figure 5.66 Rk level of RCs treated with bur.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Rk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * mark P \leq 0.05, ** P<0.01 and *** P<0.001.



Figure 5.67 Rk level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 or bur represent the two treatments. Bar charts represent median and the vertical lines IQR of Rk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of *** mark P<0.001.

<u>Rvk</u>

Data analysis for Rvk showed that the statistically significance difference was only between three RCs, Kalore showed statistically significant higher Rvk than N'Durance and Filtek Supreme XTE, for P- values see Figure 5.68. The other RCs showed statistically no significant difference between them. Further, Rvk for the RCs treated with bur showed a statistically significant higher values than those treated with P600 P<0.001, as illustrated in Figure 5.69.



Figure 5.68 Rvk level of RCs treated with bur.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Rvk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of* mark P≤0.05 and ** P<0.01.



Figure 5.69 Rvk level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 or bur represent the two treatments. Bar charts represent median and the vertical lines IQR of Rvk (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of *** mark P<0.001.
<u>Mr</u>1

It is apparent from Figure 5.70 that very few materials showed difference between them, only Filtek Z250 was statistically significant higher in Mr₁ than Filtek Supreme XTE P≤0.05. The other differences between RCs were statistically non-significant. Further analysis showed when Filtek Z250, N'Durance, and Heliomolar RCs treated with bur showed statistically significant higher Mr₁ compared to those treated with P600 P<0.001. The other RCs showed statistically no significant difference between them P>0.05, as explained in Figure 5.71.



Figure 5.70 Mr₁ level of RCs treated with bur.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Mr_1 (n=15); * represents significance difference between RCs, and horizontal bar connect the RCs with statistical significance of * mark P≤0.05.



Figure 5.71 Mr₁ level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 or bur represent the two treatments. Bar charts represent median and the vertical lines IQR of Mr_1 (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of *** mark P<0.001.

<u>Mr</u>2

Data analysis showed Filtek Supreme XTE was statistically significant higher in Mr₂ than Heliomolar; Kalore was statistically significant lower in Mr₂ than other RCs with the exception of Heliomolar, the P values illustrated in Figure 5.72. Further, when bur and P600 treatments were compared for Mr₂, Filtek Z250, Heliomolar and N'Durance revealed statistically significant different between the two surface treatments which was higher in value for bur treatment. The other RCs showed statistically no significant difference between them. The P values shown in Figure 5.73.



Figure 5.72 Mr₂ level of RCs treated with bur.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of Mr_2 (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * mark P≤0.05, ** P<0.01 and *** P<0.001.



Figure 5.73 Mr_2 level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 or bur represent the two treatments. Bar charts represent median and the vertical lines IQR of Mr_2 (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of; * indicate P≤0.05 and *** P<0.001.

<u>SBS</u>

Typical load-extension curves for Filtek Supreme XTE, Heliomolar, Filtek Z250, Kalore and N'Durance are illustrated in Figure 5.74. Overall, the samples exhibited a similar behaviour, and the performances were similar to those described in Section 5.4 with two exceptions. First, the SBS appears to be higher in the RCs treated with a bur. Second, the SBS at maximum load appears to be similar in height between RCs.

What stands out in Figure 5.75 is RCs showed statistically no significance difference between them in SBS P>0.05. Further, RCs treated with bur showed statistically significant greater SBS than those treated with P600 P<0.001, as shown in Figure 5.76.



Figure 5.74 Typical load-displacement curve for a shear bond test on repaired RC samples treated with bur, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.



Figure 5.75 SBS level of RCs treated with bur.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of SBS (n=15).



Figure 5.76 SBS level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. P600 or bur represent the two treatments. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of *** mark P<0.001.

5.9.2 Combined mechanical and chemical preparations results

The results from the SBS of RCs treated with bur plus acid etch plus bonding agent are described and then compared to the findings from the RCs treated with bur.

<u>SBS</u>

Typical load extension curves for Filtek Supreme XTE, Heliomolar, Filtek Z250, Kalore and N'Durance are illustrated in Figure 5.77. Overall, the samples showed similar behavior, and the performance was similar to those described in Section 5.9.1.



Figure 5.77 Typical load displacement curve for a shear bond test on repaired RC samples treated with bur plus acid etch plus bonding agent, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.

In Figure 5.78 Filtek Z250 showed statistically significant higher SBS compared to N'Durance P<0.01 and Filtek Supreme XTE P<0.001. Whereas, the other RCs showed statistically no significant difference between them P>0.05. What is interesting about the data in Figure 5.79 is that only Filtek Z250, Kalore and Heliomolar showed statistically significantly greater SBS for the bur plus bonding system treatment compared to bur treatment only P≤0.05. The other RCs showed statistically no significant difference between them P>0.05.



Figure 5.78 SBS level of RCs treated with bur plus acid etch plus bonding agent. Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of ** P<0.01 and *** P<0.001.



Figure 5.79 SBS level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bur or bur plus bonding system represent the two treatments. Bar charts represent median and the vertical lines IQR of SBS (n=15); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * indicates $P \le 0.05$.

Silane treatment

Typical load-extension curves for Filtek Supreme XTE, Heliomolar, Filtek Z250, Kalore and N'Durance are illustrated in Figure 5.80. The samples exhibited similar behaviour, and the performance was similar to bur plus phosphoric acid plus CP/BA, with two exceptions. Firstly, both Filtek Supreme XTE and Heliomolar exhibited gradual increase in the load when the blade affected the interface between the substrate and the repair RC. Secondly, there is a small area of fluctuation for the Filtek Supreme XTE when the blade affected the interface. The small curve before the main curve for the Heliomolar, Filtek Z250, Kalore and N'Durance seems to be due to the resistance of a small piece of excess RC or bonding agent around the sample



Figure 5.80 Typical load displacement curve for a shear bond test on repaired RC samples treated with bur plus acid etch plus silane plus bonding agent, where (A) is Kalore, (B) is Filtex Z250, (C) is N'Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.

From Figure 5.81, it can be seen that Filtek Z250 resulted in statistically significant higher SBS than N'Durance P<0.05. The other RCs showed statistically non-significant difference between them. From the data in Figure 5.82, it is apparent that adding silane to bonding system resulted in statistically non-significant change in SBS than the excluding it P>0.05.



Figure 5.81 SBS level of RCs treated with bur plus acid etch plus bonding agent.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bar charts represent median and the vertical lines IQR of SBS (n=5); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * marks P \leq 0.05.



Figure 5.82 SBS level of RCs with two treatments.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar represent different RCs. Bur plus bonding agent without/ with silane represent the two treatments. Bar charts represent median and the vertical lines IQR of SBS (n=15 for group with excludes silane; n=5 for silane included group).

In summary

The RCs treated with a bur were different in roughness. Kalore and N'Durance showed lowest roughness for the Ra, Rpk, Rk and Mr₂ measurements, with the exception of Rvk and Mr₁.

The RCs treated with a bur were statistically significant higher in Ra, Rpk, Rk, and Rvk than those RCs treated with P600 grit only. This difference for Mr_1 and Mr_2 measurements was less within the RCs.

There was statistically non-significant difference between RCs when treated with a bur in terms of SBS measurement. The SBS within RCs treated with the bur were statistically significantly higher in strength compared with those treated with P600 grit alone.

The SBS between the RCs treated with bur plus acid etch plus bonding agent was statistically significantly different. Filtek Z250 was statistically significant higher in strength than N'Durance and Filtek Supreme XTE, whereas the other differences were statistically non-significant.

Bur plus acid etch plus bonding agent increased the level of SBS in comparison to those RCs treated with bur but was not statistically significant except for Heliomolar.

The difference in SBS between the RCs treated with bur plus acid etch plus bonding agent with silane was statistically significant. Filtek Z250 was statistically significantly higher in strength than N'Durance but the difference between other RCs was NS.

Silane treated samples showed statistically non-significant increase in SBS compared to those of excluding silane.

5.10 Mechanical without/with chemical preparations discussion

This section discussed the findings from mechanical without and with chemical preparations. These preparations included a number of surface treatments first one was mechanical preparation by the use of a standard medium coarse diamond bur; second surface treatments were bur plus phosphoric acid plus bonding agent without or with silane.

Based on the results, a medium coarse diamond bur was an effective method for improving SBS.

To further understand the mechanism by which bur treatment improved SBS, the roughness of the RCs treated with the bur was compared to the RCs treated with SiC. Bur treatment contributed to greater roughness compared to SiC. The grit size of the bur was135–95 μ m, which is much larger than those of P600 (25.8 μ m). The grit size of the instrument has previously been shown to be a factor that influences RC roughness (Erdemir et al., 2013). In addition, the bur treatment resulted in statistically significant higher values for Ra, for all RCs in comparison to P600 (Figure 5.63). As noted within the previous section in this chapter, the limitation in the Ra parameter in qualifying a roughness profile does not detail how the bur changed the quality of RC roughness profile. For this purpose, the BAC calculation showed bur treatment resulted in not only statistically significantly higher peaks, but also greater values of cores and valleys (Figure 5.65, Figure 5.67, Figure 5.69). Bur treatment showed not only an increase in Mr₁ and Mr₂ for all RCs, but that increase was statistically significant for three RCs in comparison to RCs treated with P600 only (Figure 5.70 and Figure 5.73). There is reported evidence that an increase in roughness height enhances RBS better than an increase in width dimension when observed under SEM (da Costa *et al.*, 2012). None of the earlier studies showed this range of increase in roughness or RBS, even with the use of a bur of similar abrasive particle size to the one in bur experiment. The present and earlier studies' methods and results are illustrated in Table 5-2. The earlier studies mentioned in this paragraph only used Ra, without providing further information on roughness, so a more detailed comparison is impossible. This finding indicating an increasing roughness profile of RCs, particularly by the use of the medium coarse diamond bur,

has the potential to statistically significantly increase SBS particularly when that the increase in roughness profile is in all its segments of distribution simultaneously. The importance of the current investigation is linked to addressing the effect of bur treatment for five RCs of different composition and category against a standard surface treatment, before ageing the specimens. However, in ideal situations, clinical studies should confirm these findings, which, while preliminary, suggest that when a clinician requires a method to increase RBS, the use of a median coarse diamond bur may be an effective method. Another important point with the current investigation is that it has provided evidence of the effectiveness of a method that all dentists use in everyday clinical practice.

Author	Materials	Sample preparation	Surface treatment	Roughness (Ra µm)	Test method	RBS (Mpa)
Valente et al, 2015	Microhybrid	Mylar	Diamond bur 126-91 µm abrasive particle size	1.38/4	Microtensile	24.2/28
Wendler et al, 2016	Nanohybrid	Diamond bur 76-27 µm abrasive particle size	Diamond bur 126-64 µm abrasive particle size	1.07/3.36	Tensile	4.86/7.15
da Costa et al, 2012	Microhybrid	Soft-Lex Pop On	Diamond bur 151 µm abrasive particle size	0.17/3.82	Tensile	35.2/47.5
Present study	Microhybrid, two nanohybrids, nanofill, microfill	P600 SiC paper	Diamond bur 135-95 µm abrasive particle size	0.23-0.33/ 6- 6.8	Shear	1-11/27-30

Table 5-2 Present and earlier studies investigated impact of change in Ra on RBS by the use of bur pre-treatment against sample preparation.

The alignment of the current study's findings with a number of other studies that used microTBS and TBS tests indicate the usefulness of SBS for measuring difference within and between RCs due to P600 preparation and bur treatment.

Further investigation within bur treatment involved applying a bonding system to RCs treated with bur. When SBS was compared to the bur and to the bur plus phosphoric acid plus CP/BA, the latter treatment increased the level of SBS for all RCs. The maximum increase in SBS was for Kalore, Filtek Z250 and Heliomolar and the

minimum for N'Durance. The overall trend was for phosphoric acid plus composite/priming bonding agent to further increase the SBS for the RCs. However, this enhancement was not significant in N'Durance and Filtek Supreme XTE, with Filtek Z250, Kalore and Heliomolar returning the only statistically significant result. These findings reflect those of Rathke et al. (2009) and Cavalcanti et al. (2007), who also found an increase in RBS when bur plus composite priming bonding agent on microhybrid RC (Spectrum TPH) compared to bur treatment only. Both Rathke et al. and Cavalcanti et al. aged the substrate for 24 hours, which was not the case in the present study. Just to remind (noted in the previous section in this chapter), the literature have explained the findings for adhesive system by that the increase in RBS assumed to contribute to micro-retentions of bonding agent between irregularities in RCs (Brosh et al., 1997; Tezvergil et al., 2003; Staxrud and Dahl, 2011), or via the formation of a covalent bond between unreacted monomers within bonding agent and substrate material on non-aged RC substrates (Brosh et al., 1997; Tezvergil et al., 2003). As mentioned within the previous section in this chapter, the studies that have been reviewed in the literature have not investigated the effect of unreacted monomers, with the exception of a number of investigators, but they did not find any impact of the difference in DCs between RCs on TBS (Özcan et al., 2013a). Özcan et al investigated different RCs but their category were nanofill and nanohybrid which are similar to the categories of some of the RCs included in this studiy. Not to forget, the compatibility of monomer systems between CP/BA and studied RCs have been assumed to be another factor on RBS because a number of authors showed adhesive agent type impacted on RBS (Yesilyurt et al., 2009; Wendler et al., 2016).

The SBS test used in this study appears to be a useful method for detecting the difference in SBS for bur treatment before and after phosphoric acid plus bonding system, particularly when the findings showed change in RBS similar to other studies, even the RCs and testing condition are different, such as those by (Rathke *et al.*, 2009) that used TBS. Also, the comparability of findings with those of Cavalcanti et al that used SBS, possibly confirms the usefulness of SBS for that purpose.

The final investigation of bur treatment included adding silane to the bonding system. Where the findings were compared to the same treatments minus silane, some of the RCs showed increased and others decreased SBS but these differences were

statistically non-significant. Not to forget, in the silane experiment, the problem of change in resin block samples` shape was similar to those samples prepared with P600, but with less frequency. As a result, it was decided to perform the experiment but with a smaller sample size (n=5). This relatively small sample size probably limits the interpretations from the findings.

Similar findings have been reported in other studies (Veiga de Melo *et al.*, 2011; El-Asirarya *et al.*, 2012; Lima *et al.*, 2014), which suggest that adding a silane treatment has a minimal impact on the reparability of RCs treated with a diamond bur plus acid etch plus bonding agent. Two of these three earlier studies investigated one type of RC, a microhybrid (Charisma[®]), and the other investigated a nanohybrid (Grandio Caps[®]), respectively. Within the literature, some explanations have been suggested for the ineffectiveness of silane. The ineffectiveness of silane has been explained by that the surface treatments did not properly uncover the fillers on the substrates, and the application of silane and an adhesive system in separate layers resulting in a weak, thick and multiphase interfacial layer, leading to the increased possibility of defects at every working step (Park and Jin, 2001).

Silane contributed to a reduction in the SBS for N'Durance and Kalore but this was statistically non-significant (Figure 5.82). N'Durance's SBS reduced from 24 to 21.6 MPa and for Kalore SBS from 27.1 to 26.1 MPa. Both RCs contain glass fillers as the main filler type rather than silica particles. This is in keeping with previous research by El-Asirarya *et al.* (2012), who reported a reduction in RBS after the inclusion of silane with a bonding system on 24 hour aged nanohybrid samples. El-Asirarya *et al.* used a nanohybrid that contains glass fillers as the main filler in addition to silica particles, and they roughened the surface with a diamond bur, although the authors did not mention its grit size. Other investigators have suggested that the minimal number of silica particles may have affected the results because silane reacts with silica particles better than other types of filler particles, such as glass (Matinlinna *et al.*, 2018). Therefore, within the limitations of this study, the findings suggest there may be no benefit to including a silane treatment step before an adhesive system for some of the RCs treated with a coarse diamond bur.

The effectiveness of silane cannot be attributed to the monomer system and categories of the RCs. The RCs that are different in monomer system such as Heliomolar and Kalore showed a statistically non-significant difference in SBS.

Similar to monomer system, RC category did not show direct relation to effectiveness of silane. Filtek Z250 is a microhybrid RC but showed a statistically non-significant difference with the two nanohybrid RCs (Kalore and N'Durance) similar to those found by (Yin *et al.*, 2001; Júnior *et al.*, 2013).

The shear test used in the present study could be considered an advantageous method for detecting difference in relation to silane application because the findings are in agreement with a number of other studies that used tensile tests, such as those by EI-Asirarya *et al.* (2012).

The following sections focus on the performance of RCs during roughening with bur as a form of mechanical preparation, and combined bur treatment with chemical preparation.

Firstly, the tested RCs showed differences in roughness when treated with bur alone. Generally, from the Ra and BAC findings, RCs showed a statistically significant difference between them, which was unique for each parameter (Figure 5.62, Figure 5.64, Figure 5.66, Figure 5.68, Figure 5.70, Figure 5.72). Based on the reviewed literature, the earlier studies used Ra measurement only. One study suggested that the statistically non-significant difference in Ra between the three types of nanohybrid RCs when treated with fine diamond finishing bur (Schmitt *et al.*, 2016). These findings are in agreement with the Ra results in the present study, which showed a statistically non-significant difference between Kalore and N'Durance. To the best of my knowledge, this is the first study to investigate the roughness of different RCs after treatment with a coarse diamond bur.

Another finding was that the roughness of the studied RC treated with bur was higher than those found in the literature. For example, the median value for Ra for the five RCs treated with a bur of 95–130 μ m abrasive grit size was approximately 6–6.8 μ m. A number of studies have shown that the Ra of microhybrid and nanohybrid RCs treated with diamond bur (grit sizes 91–151 μ m, 64–126 μ m and 91–126 μ m) had a Ra of 3.82 μ m, 3.36 μ m, and 4 μ m, respectively (da Costa *et al.*, 2012; Valente *et al.*, 2015; Wendler *et al.*, 2016). In the current investigation, each bur was used once to prepare one sample for 5 seconds. The earlier studies used different times or did not mention the time or how many samples were prepared per bur instrument. For example, da Costa *et al.* (2012) prepared the samples for ten seconds and Wendler *et al.* (2016) removed 0.3 mm from the samples. All the above-mentioned studies

also used hand pressure during sample preparation, which is a possible source of variation. Diamond burs show great variability in cutting efficiency and roughness after use due to the accumulation of debris, cutting time, and difference in wear of abrasive particles (Yin *et al.*, 2001; Júnior *et al.*, 2013). Therefore, these variables might have resulted in the difference between the roughness outcomes in these studies.

It is assumed similar RBS from a treatment method on a number of RCs would be better for clinical situations in which the clinician may be unaware of the type of RC restoration in the tooth. Having an effective repair method for increasing RBS on most of the RCs has been recommended and authors have investigated a number of surface treatments and RCs for such a purpose (Loomans et al., 2011b). A study investigated the RBS of five RCs. The results showed a statistically non-significant difference between the studied RCs when treated with bur (Figure 5.75). When the statistically significant difference in findings of roughness and SBS were compared. there was no alignment between the findings. For example, N'Durance and Kalore were statistically significantly lower in Rk compared to other RCs (Figure 5.66), but the materials did not show a statistically significant difference in SBS (Figure 5.75). Based on the reviewed studies that measured roughness Table 5-2, only one type of RC material had been investigated in every study. Therefore, within limitations of this study, it can be suggested the statistically significant differences in roughness between RCs did not directly impact on SBS. If this would be the case, bur treatment is likely to be an important factor for RBS that has potential to offset the impact of difference in roughness between RC materials.

The median values of SBS for the RCs treated with bur were approximately within a 18–25 MPa range, and these values are within a suggested minimum required level of RBS for clinical practice (Yesilyurt *et al.*, 2009). The following sentences show the RBS values of earlier studies that used similar surface treatment at least for diamond bur. The reviewed literature showed conflicting results. Cavalcanti *et al.* (2007) showed a higher value for microhybrid RC treated with the bur without the application of bonding agent, at 31.4 MPa. The authors used a microhybrid RC but they did not provide information on the composition; they aged the substrate sample for 24 hours and used a bur but did not provide its grit size. However, a number of authors used a bur to treat microhybrid and nanohybrid RCs and reported lower microTBS (4.4 and 4.5, respectively), where the substrate samples were aged for 24 hours in DW (Lima

et al., 2016) The authors in both studies did not provide information on the grit size of burs. Therefore, it appears the values of RBS is dependent on RC materials and the method. Therefore, it may be difficult to compare RBS values for surface treatments.

None of the monomer systems and RC categories impacted on the SBS findings of these RCs. This is owing to that all RCs differed in the monomer systems and category but this difference did not directly impact upon SBS findings. For example, Heliomolar and Kalore are based on Bis-GMA and UDMA, respectively, with categories of microfill and nanohybrid, also respectively. Considering all the former differences, there was statistically non-significant difference between them in SBS when treated with bur (Figure 5.75).

In this work, the BAC parameters not only showed statistically significant differences in values that were not aligned to the statistically significant difference in SBS, but they also showed a change in roughness profile similar to those found with Ra values. However, BAC calculates the difference in the roughness profile elements between RCs that are not provided by Ra calculations. For example, Ra did reveal a statistically non-significant difference between Filtek Z250 and Filtek Supreme XTE, but Mr1 showed the statistically significant difference between the two materials. This indicates that the SBS findings were explained by roughness profile distribution of RCs that is based on better information compared to the previous studies used Ra alone.

The phosphoric acid plus CP/BA treatment was carried out after mechanical preparation with a bur. Based on the findings, the statistically non-significant differences between RCs decreased compared to excluding chemical preparation (Figure 5.78). As noted for prepared RCs with P600, the element of a roughness profile that has the potential to accumulate fluid is Rvk (Field *et al.*, 2010). In this thesis, the differences in Rvk have been used to determine the effect of RC roughness on the effectiveness of bonding agent. Based on the findings, it appears there was no alignment between Rvk and SBS findings. RCs showed a statistically non-significant difference in Rvk, while there was statistically significant difference between RCs in SBS. These findings suggest no direct relation of RCs roughness treated with a medium coarse diamond bur on the effectiveness of the bonding system. This finding is important for clinical situations where there is a need for a method to have a similar effect on different RCs.

The SBS values for Filtek Z250, Kalore, Heliomolar, N'Durance and Filtek Supreme XTE treated with bur (135–95 µm) were 29.4, 27.1, 25.5, 24 and 22.7 MPa, respectively. These findings indicate that all the RCs showed SBS above 20 MPa. Hence, all the RCs showed SBS values above the suggested limit of the minimum required bond strength for clinical practice, of 18–20 MPa (Yesilyurt et al., 2009). The findings are approximately consistent with those of Valente et al. (2015), who showed 28 MPa microTBS of microhybrid (Opallis®, FGM) when treated with a bur grit size of abrasive particles ranged 126–91 µm (medium coarse grit), plus phosphoric acid and priming bonding agent. This finding indicates bur treatment with bonding agent has the potential to enhance bond strength above 20 MPa. However, the SBS values between the two studies are very close, although the Ra value was different for the RCs. Valente et al. (2015) showed lower Ra value for the microhybrid RC (4 µm) compared to those of the microhybrids in the present study (6.8 µm). This difference in roughness is explained by the difference in the range of bur grit size between the two studies. As noted for the bur treatment alone, it is difficult to identify a value of RBS for a surface treatment even usually more than one surface treatment apply and each one have some impact on RBS was not statistically significant.

Further analysis of the data showed the RC categories, as revealed in Table 5-1, were not linked to differences in SBS. The two nanohybrid materials (Kalore and N'Durance) performed differently. Kalore showed a statistically non-significant difference but N'Durance had statistically significantly lower SBS compared to Filtek Z250. Similarly, the monomer system was not linked to differences in SBS. For example, Heliomolar is Bis-GMA-based whereas N'Durance is UDMA-based, but there was no significant difference between the two materials.

Data analysis of the SBS for the RCs treated with bur plus acid etch plus silane plus bonding agent showed similarity of the materials, with Filtek Z250 having statistically significantly greater SBS than N'Durance. The statistically significant difference between N'Durance and Filtek Z250 is a continuation of the difference due to bonding agent, because before including silane the two materials had a statistically significant difference (P<0.01). It is possible that the changes produced by the action of silane did not lead to a loss of statistically significant difference between N'Durance and Filtek Z250 (p<0.05). However, silane treatment led to the loss of the statistically significant difference between Filtek Z250 and Filtek Supreme XTE

compared to those of not including silane treatment (Figure 5.78 and Figure 5.81). This is owing to the fact that silane treatment increased SBS for Filtek Supreme XTE but decreased SBS for N'Durance (Figure 5.82). Thus, previous statistically significant differences between materials in SBS were lost after the addition of silane to the surface treatment of some materials. This change increased the number of statistically non-significant differences between RCs.

Another finding was that the median value of SBS ranged between 21.6–32.2 MPa. The highest value was Filtek Z250 and the lowest N'Durance. The values of SBS in the current experiment did not match the RBS values in earlier studies which used silane with a bonding system for RCs treated with diamond bur. Loomans et al. (2011b) showed TBS to be higher than 40 MPa for microhybrid, nanohybrid and microfill RCs. Loomans et al. reported a value for the negative control samples near 40 MPa, and they ranked the values but without finding the statistically significant difference between surface treatments before and after adding silane. A number of authors have reported lower bond strength for silane with a diamond bur than those in the current study, which were around 10–16 MPa for microhybrid, nanohybrid and nanofill (Veiga de Melo et al., 2011; Ahmadizenouz et al., 2016; Kiomarsi et al., 2017). There are several explanations for this. Firstly, silane had a statistically nonsignificant increase on SBS and thus the SBS after silane treatment remained close to the value before the application of silane, while the SBS before silane application was lower than those of the present study. The lower SBS before silane treatment might be due to differences in RC material from those used in the current study and the type of bur and bonding system used before applying silane.

Silane treatment can increase statistically non-significant differences between RCs. However, they resulted in a statistically non-significant increase in SBS. Due to the limitations of this study and earlier studies a clear suggestion about silane use could not be drawn.

Summary

Bur treatment is an effective method for improving SBS. RCs treated with bur showed a statistically significant increase in SBS compared with those prepared with P600 alone. This increase in SBS was related to the increase in roughness. Adding a bonding system to the bur treatment did appear to improve SBS but was

statistically non-significant. Similarly, adding silane to the former treatments had a statistically non-significant increase in SBS.

RCs performed differently during surface treatments. With bur treatment alone, RCs showed a statistically non-significant difference between them. Adding the bonding system resulted in decreasing the statistically non-significant differences between RCs because three RCs showed a statistically significant difference between them, while silane treatment led to fewer statistically significant differences between the RCs. For all surface treatments, RCs showed SBS values to be above the suggested minimal required bond strength for clinical practice, 20 MPa.

In conclusion

- Bur pre-treatment increases both roughness and SBS in comparison to P600 prepared RCs. This increase in SBS was consistent between RCs.
- Adding a bonding system to bur treatment increased SBS but was statistically non-significant. This treatment resulted in increasing the number of statistically significant differences between RCs, which were unrelated to the differences in roughness profile of the RCs.
- The addition of silane to the bur and bonding system treatment resulted in a statistically non-significant increase in SBS. This treatment resulted in a loss of statistically significant differences between some of the RCs compared to exclusive use of silane.

Chapter 6. The impact of water sorption over time

6.1 Introduction

RCs in oral environments are likely to absorb fluids from saliva and foods such as water, acid, and alcohols. Fluids have the potential to release components from RCs into oral fluids. Both the absorption and desorption of a liquid is also thought to degrade the structure and mechanical properties of RCs (Boaro *et al.*, 2013; Alshali *et al.*, 2015), including RBS (Papacchini *et al.*, 2007; Eliasson *et al.*, 2014). These reductions in RBS have been attributed to the degradation of polymer matrix, fillers, and filler-matrix interface (Söderholm, 1983; Söderholm *et al.*, 1984; Kalachandra, 1989; Santerre *et al.*, 2001; Martin *et al.*, 2003; Ferracane, 2006). Therefore, a RC that can better resist the degrading impact of the oral environment is apparently of great interest. In addition, fluids may enter RCs via diffusion, which is time dependent and related to many factors such as content, degree of polymerisation, density, and the chemistry of polymers (Gajewski *et al.*, 2012; Albuquerque *et al.*, 2013; Cornelio *et al.*, 2014; Giannini *et al.*, 2014; Kumar and Sangi, 2014; Alshali *et al.*, 2015; Panahandeh *et al.*, 2017).

There is recognition of the importance of identifying the equilibrium uptake point of RCs when their properties are tested in relation to absorption (Musanje and Darvell, 2003), to ensure the loss of soluble and leachable materials which may cofound the findings. Accordingly, the aim of this study is to investigate the impact of water sorption of RCs on the changes in SBS of aged repaired RCs.

Objectives

- To evaluate the WS% of 5 RCs.
- To evaluate changes in SBS of 5 RCs as a result of aging.
- To evaluate the impact of water sorption of 5 RCs on SBS.

Hypotheses

- RCs different in WS%.
- Aging in DW will result in statistically significant decrease in SBS for all RCs.
- The ranking for WS% do not directly related to the ranking in decrease in SBS.

6.2 Method

6.2.1 Water sorption of RCs

In this study, the water sorption method was based on the BS EN ISO standard 4049:2000 with some modifications because the aim was not only to measure water sorption of the RCs but to increase understanding of the water sorption property of RCs over time. Circular discs (1 mm thick and 15 mm in diameter) of five RCs (Filtek Z250, Filtek Supreme XTE, Heliomolar, Kalore and N'Durance) were made using PTFE (polytetrafluoroethylene) circular ring moulds (n= 5/ RC) as illustrated in Figure 6.1. The method of preparing the samples was similar to that described in Section 5.2.1.

The circumference of each specimen of RCs was prepared to remove flash and irregularities. The specimens were prepared manually using P600 grit SiC paper and cleaned from debris with the compressed-air jet.

Before performing a water sorption test, the samples were dehydrated. For this purpose, the specimens were repeatedly weighed on an electrical analytical balance (Mettler AE 240, Leicester, UK). Every specimen was placed separately into a small vial (25*38 mm, polystyrene vial with polyethylene cap, Fisher Scientific), without the cap and transferred to a desiccator with silica gel in an incubator maintained at 37°C for 24 hours. After 24 hours, the desiccator was removed from the incubator, the specimens were weighed on the digital balance, placed back into the vial unit without the cap and transferred to a second desiccator with fresh silica gel in an incubator maintained at 37°C for another 24 hours. This cycle was repeated until a constant weight was achieved when the mass change of each sample did not exceed 0.1 mg in any 24-hour period. This weight was designated as m1. In addition to weight measurement, two measurements were made of the diameter at right angles to each

other using a digital Vernier Calliper (Mitutoyo, Japan) to calculate the mean diameter. In addition, the thickness of the samples at the circumference of four equally spaced points were measured to calculate mean thickness. The area was calculated, in square millimeters, from the mean diameter and then, the mean thickness was used to calculate the volume, V, in cubic millimeters.

Once a constant weight was obtained, the samples were prepared for the water sorption test. For this, the specimens were stored in 12 ml of DW in a sealed vial, see Figure 6.2, in a 37°C incubator with interval measurement of samples weight at 1, 2,3,4,7, 14, 21, 28, 56, 84, 112,140,168 days storage time periods in DW. After each storage time period, the specimens were removed from the storage bottle by tweezer and each side was allowed to drip on a tissue for two seconds and then waved in the air for 15 seconds to remove excess water prior to weighting them on the electrical analytical balance. Once a constant saturated weight was reached, it was designated as m2. Constant saturated weight was obtained when the second decimal number of the weight measurement record on the electrical analytical balance was not fluctuating. For every 5 samples the electrical digital balance was reset to zero.

The percentage amount of water sorption from weight change was calculated, WS %, using the following equation (Lagouvardos *et al.*, 2003; Sideridou and Karabela, 2011).

WS % = [(m1-m2)/m1]*100(2)

M1 is the mass of the sample, in micrograms, before immersion in water;

M2 is the mass of the sample, in micrograms, after immersion in water.



Figure 6.1. A sample of RC was prepared for water sorption test using a PTFE mould of 1 mm thick and 15 mm in diameter.



Figure 6.2. A sample of RC was stored in DW for water sorption test using a vial with its cap.

6.2.2 Ageing of repaired RCs

The sample preparation, surface treatments, repair and testing SBS were performed using a similar method as described in Section 5.8.2 but without a group testing treatment using silane. The only difference was in the sample size (n=7 for each RC). In this experiment, another step was added which was to investigate the effect on RC repair of ageing over time. The specimens were stored in DW for different periods of 7, 28, 84, and 168 days. Accordingly, for each time period five groups of specimens were prepared. Every group of RCs were kept in a plastic container that was sealed with a cap in DW in a 37°C incubator; the DW was changed every month for the groups whose immersion time exceeded more than one month to avoid the growth of bacteria in the DW. It was believed there might be contamination of the DW by bacteria from the surfaces of the containers and the RC samples that had not been sterile before use. Although, DW is pure H₂O that is free from other substances such as bacteria, bacteria could grow in DW if contaminated (Favero *et al.*, 1971; Walker *et al.*, 2000).

6.3 Results

In the first section of this chapter, the impact of immersion in DW over time of the RC samples for changes in weight and percentage of water sorption were evaluated. The second part of the experiment investigated the SBS of aged repaired RCs in DW over time.

6.3.1 Water sorption of RCs

Figure 6.3 illustrates the median value of the specimen weights of the RCs over immersion time points in DW. It can be seen that the RC's weight differed before immersion in DW, and in the order of heaviest to lightest these are: Filtek Z250, Kalore, N'Durance, Filtek Supreme XTE and Heliomolar. Nonetheless, the materials generally behaved in the same way. The samples showed very gradual increase in
weight within the first 14-days of immersion. After this immersion period, a consistent level of changes become more obvious.

The median values and IQRs water sorption (WS) of the RC samples over different immersion time points in DW are presented in Figure 6.4. Generally, all RCs behaved similarly and all showed WS values to be below 1.5%. The RCs showed a gradual increase in WS from the first day of immersion to approximately 28 days, after this period there was consistency of WS up to 168 days. So, from this aspect we might assume that 28 days can be considered a saturation time point for all the tested RCs.

N'Durance and Heliomolar showed the lowest and highest WS respectively. When statistical difference was investigated between the immersion time points for every RC, the materials behaved differently Table 6-1. For N'Durance and Kalore first day was the only time point that was the statistically significant lower WS value. While for Filtek Supreme XTE, Filtek Z250 and Heliomolar the second and fourth days were statistically significant the lower WS values. The rapid change in WS% values for RCs in the second and fourth days was not expected and may be related to some faults in the method. Similarly, the statistically significant higher values for WS was different between RCs. For Filtek Z250, N'Durance, Kalore Filtek Supreme XTE, and Heliomolar showed statistically significant higher WS values at 168th, 84th, 21th and 28th day respectively. The P values for immersion time points that showed P< 0.05 are illustrated in Table 6-1.

However, RCs performed similarly, they showed statistically significant differences between them over every immersion time point, as illustrated in Figure 6.5 to Figure 6.7. Within the first three days, the difference between the RCs were NS. In the 4^{th,} 7th and 14th day Filtek Z250 and N'Durance showed statistically significant lower WS than a number of RCs. In the 21th, 28th and 56th day N`Durance and Heliomolar showed statistically significant lower and higher WS values respectively. From the 84th to 168th day only N'Durance showed statistically significant lower WS values than the other RCs.



Figure 6.3. Weight change of RCs over different immersion times.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar depict different RCs. Each data point depicts median weight of the samples over a period of 168 days immersion in DW.



Figure 6.4 WS% change for RCs over 168 days.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar depict different RCs. Each data point depicts median WS% of the samples over a period of 168 days when immersed in DW (n=5).

Immersion time	Resin composite							
(days)	Kalore	Filtek Z250	N`Durance	Filtek Supreme XTE	Heliomolar			
	0.92	0.33	0.43 *•	0.49 ^{¤≠}	0.75			
1	(0.64-2.40)	(0.28-1.15)	(0.41.68)	(0.29-0.78)	(0.60-1.57)			
_	0.61 •••	0.40	0.67	0.75	0.69 ^{§§}			
2	(0.56-0.80)	(0.32-0.96)	(0.54-0.69)	(0.53-0.83)	(0.56-0.96)			
	1.15	1.11	1.17	1.11	1.45			
3	(1.02-1.96)	(0.44-1.39)	(0.54-1.68)	(0.67-1.64)	(1.07-1.62)			
	0.81	0.53*	0.65	0.97	0.81 [§]			
4	(0.79-1.05)	(0.44-0.57)	(0.57-0.77)	(0.81-1.15)	(0.77-0.93)			
	0.90	0.77	0.65	1.01	1.00			
7	(0.82-1.09)	(0.62-0.85)	(0.58-0.74)	(0.90-1.12)	(0.99-1.13)			
	1.10	0.00	0.00	4.40	4.05			
14	(1.01-1.33)	0.83 (0.78-1.04)	0.80 (0.75-0.86)	(1.05-1.35)	1.35 (1.21-1.54)			
		4.00	0.04	1.00				
21	1.14 (1.02-1.22)	1.08 (1.00-1.19)	0.94 (0.83-0.96)	1.32 (1.19-1.34)	1.44 (1.30-1.50)			
28	1.18 (1.08-1.31)	0.96 (0.93-1.31)	0.94 (0.82-1.08)	1.20 (1.14-1.37)	1.46 (1.39-1.57)			
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56	1.14 (1.07-1.30)	1.15 (1.12-1.26)	0.93 (0.74-0.96)	1.26 (1 15-1 48)	1.43 (1.40-1.53)			
	(1.07 1.00)	(1.12 1.20)	(0.1 + 0.00)	(1110-1110)	(1.10 1.00)			
84	1.33	1.20 (1.14, 1.33)	1.07 (0.80.1.10)	1.26	1.33			
	(1.27-1.30)	(1.14-1.55)	(0.09-1.19)	(1.13-1.31)	(1.51-1.40)			
112	1.23	1.18	0.91	1.24	1.33			
	(1.12-1.27)	(1.14-1.30)	(0.03-1.00)	(1.10-1.32)	(1.30-1.45)			
140	1.13	1.08	0.82	1.21	1.17			
	(1.07-1.17)	(1.14-1.20)	(0.78-0.98)	(1.1-1.32)	(1.12-1.25)			
168	1.25	1.26	0.93	1.11	1.23			
168	(1.16-1.28)	(1.20-1.36)	(0.88-1.09)	(1.08-1.34)	(1.21-1.4)			

Table 6-1 WS% of RCs over immersion time points.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar denote different RCs (n=5). * denotes statistically significant difference compared to 168th day of immersion in DW. • denotes statistically significant difference compared to 84th day of immersion in DW. ¤ denotes statistically significant difference compared to 56th day of immersion in DW. § denotes statistically significant difference compared to 28th day of immersion in DW. . \neq denotes statistically significant difference compared to 21st day of immersion in DW. One symbol P≤0.05; two symbols P<0.01; three symbols P<0.001.





Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar depict different RCs. Bar charts represent median and the vertical lines IQR or mean and SD of WS% (n=5); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * point to P≤0.05; ** P<0.01; *** P<0.001.





Heliomolar

Kalore





6.3.2 Ageing of repaired RCs

Typical load-extension curves for Filtek Supreme XTE, Heliomolar, Filtek Z250, Kalore and N`Durance after immersion in DW for 7, 28, 56 and 168 days are presented in Figure 6.8 to Figure 6.11. As a whole, the samples demonstrated similar behaviour, and the performances were identical to those described in Section 5.9.2. The small projection areas before the main straight line are possibly due to the presence of excess materials around the main sample border on the substrate.



Figure 6.8. Typical load extension-curve for a shear bond strength on RC samples treated with bur + acid etch + bonding agent and aged in DW for 7 days, where (A) is Kalore, (B) is Filtex Z250, (C) is N`Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.



Figure 6.9. Typical load extension-curve for a shear bond strength on RC samples treated with bur + acid etch + bonding agent and aged in DW for 28 days, where (A) is Kalore, (B) is Filtex Z250, (C) is N`Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.



Figure 6.10. Typical load extension-curve for a shear bond strength on RC samples treated with bur + acid etch + bonding agent and aged in DW for 84 days, where (A) is Kalore, (B) is Filtex Z250, (C) is N`Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.



Figure 6.11. Typical load extension-curve for a shear bond strength on RC samples treated with bur + acid etch + bonding agent and aged in DW for 168 days, where (A) is Kalore, (B) is Filtex Z250, (C) is N`Durance, (D) is Filtex Supreme XTE and (E) is Heliomolar.

This section presents SBS of RC specimens that were stored for different lengths of time, 1st, 7th, 28th, 84th and 168th days, in DW. The 1st day SBS data is the same data from the Section 5.9.2 but without silane treatment, where the SBS was evaluated for the RC specimens incubated for no more than 24 hours in DW at 37°C.

The changes in the SBS of the repaired RCs over immersion time points is illustrated in Figure 6.12. All RCs showed an overall decrease in SBS with an increase in immersion time except for the first 7 days where the highest SBS was recorded with the exception of N'Durance. Furthermore, every RC behaved differently over the immersion time, but the general pattern was that most of the RCs showed increase in SBS for the first 7 days and then a decrease to at or below the 1st day SBS level. N'Durance revealed a steady consistency in SBS values for the entire immersion time points, the lowest and lowest SBS values were on the 7th day and 1st days. All other RCs showed sudden increase in SBS values in the 7th day compared to the 1st day. From the 28th on to the 168th day, Filtek Z250 showed steady consistency SBS; Heliomolar revealed gradual decrease in SBS values; Filtek Supreme XTE and Kalore showed steeper decrease in SBS values compared to those of other RCs.

Table 6-2 shows statistically significant difference in SBS values between immersion time points for every RC. Kalore showed statistically significant lower SBS in the 168th day compared to those of the 1st, 7th and 28th day. Filtek Supreme XTE showed statistically significant lower SBS of the 168th day in comparison to the 7th and 28th day. Heliomolar revealed statistically significant lower SBS of the 168th day is of the 168th day compared with that of the 7th day. The other material differences were statistically non-significant.

Statistical analysis showed difference between RCs over every immersion time point, as illustrated in Figure 6.13. In the 1st day immersion time point the differences between Filtek Z250 against N'Durance and Filtek Supreme XTE; Kalore and Filtek Supreme XTE were statistically significant. Day 7 and day 28 N'Durance showed statistically significant lower SBS compared to a number of RCs. While over 84th and 168th days, Filtek Z250 revealed statistically significant higher SBS values than the other RCs.



Figure 6.12 SBS change for RCs over 168 days` immersion in DW.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar depict different RCs. Each data point depicts median SBS of the samples over 7, 28, 84,168 day immersion time in DW (n=7).

Immersion time (days)	Resin composites							
	Kalore	Filtek Z250	N`Durance	Filtek Supreme XTE	Heliomolar			
1	27.13 *	29.49	23.71	22.76	25.68			
	(3.94)	(3.98)	(22.16-25.89)	(4.34)	(20.83-28.81)			
7	32.50 ***	33.36	23.82	27.64 **	29.92 *			
	(4.61)	(4.03)	(18.13-26.25)	(4.28)	(26.35-38.14)			
28	29.95 **	29.22	23.13	26.01 *	25.79			
	(4.56)	(3.94)	(19.92-26.43)	(2.41)	(23.82-27.65)			
84	27.03	29.29	20.75	24.15	24.53			
	(3.61)	(3.21)	(20.36-21.59)	(2.99)	(20.39-26.94)			
168	21.76	28.70	22.39	19.59	22.10			
	(2.59)	(2.10)	(20.33-25.31)	(3.63)	(21.72-24.35)			

Table 6-2 Comparing SBS levels between aging time points for every RC.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar depict different RCs (n=7). 1, 7, 28, 84 and 168 day depict interval immersion time. * depicts statistically significant difference compared to 168^{th} day of aging period; one symbol indicates P≤0.05; two symbols P<0.01; three symbols point to P<0.001.





Figure 6.13 Comparing SBS levels between RCs for every aging time point.

Kalore, Filtek Z250, N'Durance, Filtek Supreme XTE and Heliomolar depict different RCs. 1, 7, 28, 84 and 168 day depict immersion time points in DW (aging); Bar charts represent mean and the vertical lines SD of SBS (n=7); * represents significance difference between RCs, and horizontal bars connect the RCs with statistical significance of * indicates P≤0.05; ** P<0.01; *** point to P<0.001.

Summary of findings

- The WS % of RC increased gradually but became more consistent after the 7th day until the 168th day of immersion in DW.
- 2. The maximum WS% between RCs was different. Filtek Z250, N'Durance and Kalore exhibited statistically significant maximum WS% after 56 days of immersion time, the 84th day for N'Durance and Kalore, and the 168th day for Filtek Z250. Heliomolar reached maximum water sorption on the 28th day and Filtek Supreme XTE on the 21st and 56th day.
- 3. Over every immersion time point in DW, the WS% between RCs was different. For the first three days, the difference was not significant, but from the 4th day until the 56th Filtek Z250 and N'Durance showed significant lower WS% than other RCs. However, from the 84th day only N'Durance was substantially lower in WS% compared to other RCs.
- 4. Every RC showed a reduction in SBS over the immersion times. SBS of RCs increased on the 7th day of immersion in DW compared to the first day, except for N'Durance. From the 28th day, the materials showed a gradual decrease in SBS, except for N'Durance.
- Kalore, Heliomolar and Filtek Supreme XTE showed a statistically significant reduction in SBS on the 168th day compared to the 7th day, while the difference for Filtek Z250 and N'Durance was not significant.
- 6. The SBS between RCs was different for every immersion time. Filtek Z250 and Kalore showed the highest SBS and N'Durance the lowest for the 1st, 7th, 28th and 84th days. Only on the 168th day did Filtek Z250 show a significant greater bond strength than other RCs; the other differences were statistically non-significant.

6.4 Discussion

In the oral environment, RCs absorb water from saliva and can release material elements to their surroundings. This can have a harmful effect on the structure and longevity of RCs, such as a reduction in physical and mechanical properties (Ferracane, 2006; Ilie and Hickel, 2009b; Giannini *et al.*, 2014). Studies have shown that the composition of RC is a crucial factor in the absorption of water (Curtis *et al.*, 2008b; Albuquerque *et al.*, 2013; Panahandeh *et al.*, 2017).

Most studies have simulated the effect of oral fluid by ageing their samples in different types of fluids such as DW, artificial saliva, and saline *and etc*. In the current study, DW was selected to simulate ageing by saliva. However, saliva does have different components, but 99% of it is water (Humphrey and Williamson, 2001). Furthermore, ageing in DW is a reproducible *in vitro* method in RC studies (Kumar and Sangi, 2014; Alshali *et al.*, 2015).

There are two different modes in the polymeric phase in which RCs absorb water. Firstly, the water is absorbed to the free volume between the polymer chains and nano-pores formed during polymerisation, and is called unbound water (Vanlandingham *et al.*, 1999). Secondly, the bound water is attached to the polymer chain by a hydrogen bond (Sideridou *et al.*, 2003). This process leads to an increase in weight of the RCs. It is worth highlighting that water uptake in the RCs is by diffusion, which is a time dependent process (Asaoka and Hirano, 2003; Costella *et al.*, 2010). Accordingly, in the current study, water sorption was measured at intervals at 1–4, 7, 14, 21, 28, 56, 84, 112, 140 and 168 days, these interval measurements were hoped to provide a more insightful description of the changes in RCs that occur during the immersion time which likely to affect the findings to be more accurate than taking fewer measurements.

The first finding was that water sorption percent (WS%) for all RCs increased gradually from the first day of immersion in DW until approximately 28 days, and thereafter WS% became more consistent (Figure 6.4). In agreement with the current study, Mustaza *et al.* (2014) showed that the equilibrium uptake day for methacrylate-based RCs was 26–33 days.

The maximum WS% of RCs over 168 days ranged from 1.07% to 1.49%. This value is comparable with the maximum values reported by a number of authors of less than 1.6%, even though the RCs between the studies are different. For example, Mustaza *et al.* (2014) reported WS% to be less than 1.5 and Ferracane (1997 as cited in Ferracane, 2006) showed to be less than 1.0 to 1.6%.

The WS% of the studied RCs was comparable to other findings in the literature. Kalore showed 1.4% at 150 days in deionised water (Wei et al., 2013), although in the present study WS% was measured for 140 and 168 in DW. The value of 150 days was even higher than the 168th day, on which WS% was 1.25% in the current study. The difference in storage media might have contributed to the difference in findings between the two studies. Another study showed higher water sorption for Filtek Supreme XTE (0.96%) than for Kalore 0.76% when the samples had been stored in DW for one week, and were 2 mm thick and 5 mm in diameter (Randolph et al., 2016a). The ranking for WS% in the current study for both materials is comparable with that of Randolph et al. (2016a), but the values in this experiment were higher for both Filtek Supreme XTE (1.01%) and Kalore (0.9%). The difference in values is likely due to the difference in sample dimensions between the two studies. Other studies have shown higher water uptake by Heliomolar (1.1%) than Tetric Ceram or Tetric Evo Ceram (a type of nanohybrid material) for 1, 7 and 30 days in DW (Musanje and Darvell, 2003; Sideridou et al., 2008). The ranking for the two materials is comparable to the findings of 7 and 28 days WS in the current study, while on day 1 the WS% of Kalore (nanohybrid) was higher than Heliomolar (Table 6-1).

Based on these results, RCs behaved differently for maximum WS% (Table 6-1). The maximum amount of WS of RCs from lowest to highest was N'Durance, Filtek Z250, Filtek Supreme XTE, Kalore, and Heliomolar. Monomer chemistry is considered one of the most important factors affecting water sorption (Gajewski *et al.*, 2012; Alshali *et al.*, 2015). The variance in water sorption of resin matrix is thought to be reliant on the hydrophilicity of monomer types; the order of the hydrophilicity of the monomers involved in the studied RCs, from lowest to highest, was DA (Dimer acid), D3MA (1, 10-decanediol dimethacrylate), Bis-EMA, UDMA, Bis-GMA, TEGDMA (Trujillo-Lemon *et al.*, 2006; Sideridou and Karabela, 2011; Bociong *et al.*, 2017). Moreover, the absorption ability of DX511 was higher than

those of Bis-EMA and UDMA combined (Sideridou *et al.*, 2015). For the monomer ratio and types, see Table 6-3 below:

Resin composites	Monomer ratio by %							
	Dimer acid	DX-511	Bis-EMA	UDMA	Bis-GMA	TEGDMA	PEGDMA	D3MA
N'Durance	2.5-10		10-25	2.50				
Kalore		1-5	1-5	10-20				
Heliomolar				10-20	10-25			2.5-10
Filtek Z250			1-10	1-10	1-6			
Filtek Supreme XTE			1-10	1-10	1-10	1	5	

Table 6-3 Monomer ratio and types of RCs.

Based on the results in Table 6-1 and Table 6-3, only N'Durance matched the ranking of the monomer hydrophilicity, as the other RCs do not match that ranking. For example, D3MA in Heliomolar ranked second for low hydrophilicity but the WS of Heliomolar was not the second lowest material for WS, as it had the highest WS% over the six months. Thus, it can be concluded that, based on one type of monomer, the WS of RC cannot not be determined. Every RC used in this study not only combines three to five different monomers, but the ratio of the monomers is revealed to be different between the RCs (Table 6-3). For example, both N'Durance and Kalore contain Bis-EMA and UDMA but N'Durance had a higher ratio of Bis-EMA and lower ratio of UDMA, while for Kalore the ratio was opposite. Similarly, Filtek Z250 and Heliomolar contain Bis-GMA and UDMA, and the ratio of the two monomers is much higher in Heliomolar than those of Filtek Z250. This difference in ratio is one of the factors which resulted in the difference in WS between the two materials.

Filler loading, size and bonding condition to resin matrix is also likely to have influenced WS% of RCs. The filler loading of the studied RCs is illustrated in Table 5-1. The amount of resin matrix is dependent on the filler loading, and as the filler loading increases, the amount of resin matrix decreases in RC. Although the resin matrix of Heliomolar contains D3MA, which is more hydrophobic than the monomers in Filtek Z250 and Filtek Supreme XTE, the low filler loading in Heliomolar is likely to

have resulted in a higher amount of resin matrix, which led to a larger amount of WS% in comparison to the former materials. Sideridou et al. (2008) and Janda et al. (2007) reported that high filler loading reduced the content of the resin matrix in RC, leading to lower water sorption. What is more, nano-particles mean a higher amount of silane between fillers and resin matrix, and the latter has the potential to absorb water within RC (Matinlinna et al., 2018). Accordingly, the higher WS% of Filtek Supreme XTE than Filtek Z250 is likely not only due to monomer chemistry but also the presence of nano-particles that have the potential to enhance water uptake. Nano-particles result in greater surface area to volume ratio compared to large particles and hence a larger area of hydrophilic silane is available for water sorption (Wilson et al., 2005). Furthermore, the absence of silane treatment for the particles in Filtek Z250 is another reason for the difference in WS between the two materials. However, this is not the case for N'Durance, which has a small filler size compared with Kalore. An explanation of these cases is likely due to the more hydrophobic resin matrix, DA, in N'Durance, which may protect the silane interface and the fillers from water uptake. From this, the WS findings of the RCs are more related to overall composition than the effect of monomer hydrophilicity alone.

For the impact of RC categories on WS, this study did not show a relation between categories and WS. The categories of the studied RCs are shown in Table 5-1. For example, the two nanohybrids (N'Durance and Kalore) showed statistically significant difference in WS on the 21st, 56th, 84th, 112th, 140th and 168th days. Indeed, the microhybrid performed differently to the two nanohybrids. It did not show statistically significant lower WS than Kalore, but was higher than N'Durance over a number of immersion time points (Figure 6.7). However, the statistically significant lower WS values of microhybrid than nanofill is in agreement with a number of earlier studies (Curtis et al., 2008a; Silva et al., 2008; da Silva et al., 2011). This difference is believed to be related to the presence of pores around aggregated filler particles in nanofill, due to poor impregnation of aggregated fillers with resin matrix rather than the difference in filler size. Furthermore, as discussed previously, the nonagglomerated nano-particles contribute to greater surface area than volume, and might have allowed more fluid accumulation at the filler/ matrix interface (Kalachandra and Wilson, 1992; Curtis et al., 2008a; Silva et al., 2008). Indeed, if filler size had an effect on WS values in the current experiment, N'Durance would be one of the RCs which would have shown high value WS, rather than microhybrid RC.

Another interesting finding was that N'Durance showed statistically significant lower WS% than the other RCs, particularly from the 112 to 168 days (Figure 6.5, Figure 6.6 and Figure 6.7). In contrast, Heliomolar showed statistically significant higher WS% than a number of RCs from days 7–84. These findings can be explained by the chemistry of RCs, which are linked to WS as discussed before. For example, N'Durance has a low resin matrix, the most hydrophobic monomer (dimer acid), and the ratio of Bis-EMA (10–25) is much higher than UDMA (2.5). In contrast, Heliomolar contains less hydrophobic monomer than N'Durance, as the resin matrix ratio is much higher than the other RCs. The ratios of Bis-GMA and UDMA are very high (10–25), and it does not contain Bis-EMA. The combination of the former factors lead to the two materials presenting statistically significant difference in WS over the longer term intervals. This finding is not in accordance with those of Boaro et al. (2013), which showed higher WS of Heliomolar than N'Durance in the 28 days in DW, but the difference was statistically non-significant. Boaro et al. used a quartz tungsten halogen light curing device intensity 900 mW/cm 2x20 seconds, while the present study used a light emitting diode, intensity 580 to 600 mW/cm 2×40 seconds. This difference in the light curing intensity and time might have affected the results. Light intensity has an impact on the DC of RCs (Lohbauer et al., 2005) and this is another factor that impacts WS (Gajewski et al., 2012). To the best of my knowledge, this is the first study to measure the WS% of these five RCs over a 168 day interval. The findings on the WS of RCs not only allow better understanding of the performance of RCs in WS, but have the potential to reveal the impact of WS on RC in relation to SBS.

Further, the data showed excessive and sudden increase in the WS values. This finding has two possible explanations. Firstly, the RCs absorbed large amount of DW more than the other intervals that led to increase in WS% compared with the 2nd and 4th days. While, on the 3rd and 5th days the RCs showed lower WS% than second and fourth days likely due to leakage of monomers from the RCs. Secondly, this finding is believed to be related to a difference in specimen dryness, such as the load of shaking the specimens in air being different over the 2nd and 4th day compared to the other days. Further, when the samples were dipped on tissue, it is likely they were less dried on the 2nd and 4th day than the samples over the other days, which might have left some small droplets of water on the sample that affected

the WS values. This was only obvious in the first four days when the researcher was new and less skilled at the experiment.

Water sorption by the RCs is likely to affect the mechanical properties via leakage of elements from the resin matrix, the silane interface, and fillers (Panahandeh et al., 2017) and plasticisation (Ferracane, 2006). In this study, the SBS of the RCs was investigated after ageing the repaired specimens in DW for different time intervals. Based on previous publications, the ageing processes which have been investigated include ageing of the substrate only, followed by ageing of the repair material or ageing of the substrate and repair materials together after repair. In this study, the substrate and repair materials were aged together after the repair. This type of ageing was selected because the study is interested in investigating the overall effect of absorbed DW on SBS, rather than simulating clinical situations. In the clinical situation, the remaining part of a fractured RC restoration might have been aged for a period of time due to the effect of oral fluid, before the application of a repair method and materials. It is believed this simulation better mimics the method of immersing RC specimens in DW to evaluate WS%, to allow better understanding of the impact of WS on RCs and the link to the level of SBS. In addition, repair studies used different immersion time points. In one review study, the authors identified the immersion times used by studies, and these ranged from 0.33 to 730 days (Özcan and Koc-Dundar, 2014). In the current study, the repaired RC samples were immersed in DW for 168 days and SBS was investigated on the 7th, 28th, 56th and 168th day intervals. This ageing method has been reproduced by other investigators in vitro through immersion in DW (Oztas et al., 2003; Celik et al., 2014; Eliasson et al., 2014; Özcan et al., 2014; Altinci et al., 2018) or other solutions (Rinastiti et al., 2011a; Baena et al., 2015; Comba L et al., 2015; Souza et al., 2017; Kouros et al., 2018). DW has been found to be similar to artificial saliva in terms of the effect on RCs (Sideridou et al., 2011). This therefore supports the use of this method for measuring difference in changes in SBS due to ageing in DW.

Also based on the findings, most of the RCs behaved similarly over the entire period aged in DW (Figure 6.12), as Kalore, Filtek Z250, Filtek Supreme XTE and Heliomolar showed an increase in SBS on the 7th day, followed by a decrease in SBS. However, N'Durance revealed a decrease in SBS on the 7th day and then a slight increase in SBS. From Table 6-2, overall it can be concluded that the ageing period resulted in statistically significant decrease in SBS for some of the RCs.

Kalore showed the greatest reduction in SBS over the ageing period, as the 168 day interval resulted in statistically significant lower SBS to three intervals (1st, 7th and 28th days) out of four intervals. Kalore was followed by Filtek Supreme XTE, as the 168 day interval resulted in statistically significant lower SBS to two intervals (7th and 28th days) out of four intervals. The lowest was for Heliomolar, and the 168th day interval resulted in statistically significant lower SBS on only the 7th day interval out of four intervals. In contrast, Filtek Z250 and N'Durance showed statistically nonsignificant change in SBS. This suggests some materials can maintain a consistent SBS over 168 days of ageing in DW. Based on best knowledge, this is the first study to test the five RCs for SBS after ageing for an extended duration in DW. In agreement with some of the statistically non-significant changes in the current study, earlier studies showed a statistically non-significant change in RC RBS after ageing in DW for six months (168 days). The authors used the same method to age the repaired samples, but the studied RCs were different. However, the category names are similar to two of the RCs in the current study. When the microTBS of Grandio[®] (a type of nanohybrid RC) was evaluated on the 1st day and after 6 months of ageing, a statistically non-significant change in bond strength was shown (Baena et al., 2015). Similarly, da Costa et al. (2012) showed a statistically non-significant change in TBS for Opallis composite® (microhybrid) compared to one month aged samples (Lovell et al., 2003). Further, the changes in SBS of RCs over storage periods was assumed to be impacted by their DC. However, the limitation of this study is that the DC of the studied RCs had not been measured. A number of authors showed that final curing of RCs occurred within 72 hours from polymerisation (Mair and Padipatvuthikul, 2010). Indeed, one study showed a higher DC for nanohybrid RCs after 30 days (70.04%) compared to 16 hours (58.7%) of storage in water (Wendler et al., 2016). In contrast, the consistency of SBS for N'Durance might be due to the primary high DC (Trujillo-Lemon et al., 2006), which is likely as there was not significant DC in the monomer matrix thereafter.

In the published literature, the monomer systems of RCs have been investigated for hydrophilicity and solubility properties. These properties have been identified to be one of the factors that influence mechanical properties (Kumar and Sangi, 2014). Accordingly, the changes in the SBS for repaired samples may be partially explained by the properties of their monomer systems. The reduction in SBS of Filtek Supreme XTE may be explained by the presence of TEGDMA, because this monomer is most

prone to degradation (Spahl *et al.*, 1998; Örtengren *et al.*, 2001). The statistically significant decrease in SBS of Kalore may be due to presence of DX-511 monomer, which showed higher hydrophilicity to DW than Bis-EMA and UDMA (Sideridou *et al.*, 2015) because it contains more hydrophilic polar groups such as urethane than the two other monomers (Wei *et al.*, 2013). The minimal reduction in SBS for Heliomolar may be explained by the highly cross-linking density of Bis-GMA and minimal mobility of D3MA, which likely resulted in less degradation of the resin matrix than the two former materials (Sideridou *et al.*, 2003; Goncalves *et al.*, 2008; Silva *et al.*, 2008). The statistically non-significant change in SBS for Filtek Z250 and N'Durance can be explained by the fact that the ageing process may not have degraded the RCs. This is because ageing in DW is likely to have leaked out the unreacted monomers without degrading the polymer chains to such a degree to cause an impact on the mechanical properties (Schneider *et al.*, 2009).

Another finding was that RCs behaved differently across all immersion time points (Figure 6.13). When comparing the intervals, Kalore and Filtek Z250 were statistically significantly stronger in SBS than a number of RCs, but on the 84th and 168th days, Filtek Z250 remained statistically significantly stronger in SBS compared to all other RCs. Similarly, N'Durance and Filtek Supreme XTE were statistically significantly lower in SBS compared with Kalore, but this difference became statistically non-significant on the 84th and 168th days of ageing. These results further support the idea that ageing the samples increased the variability between the RCs compared to before being aged (Rinastiti *et al.*, 2011a; Özcan *et al.*, 2013a). The finding suggests that when comparing the aged RCs, the ageing period should be considered as an important factor in the comparison and interpretation of the findings.

The changes in SBS cannot be directly linked to the amount of WS. For the RCs which showed a statistically significant decrease in SBS over immersion time, Heliomolar had the highest amount of WS on the 168th day, but showed the least statistically significant difference in SBS reduction compared to Kalore and Filtek Supreme XTE (discussed before in this section). In contrast, N'Durance showed the least amount of WS% and a statistically non-significant decrease in SBS over the ageing period. This work is in agreement with studies which showed that the amount of WS may not completely explain the changes in the mechanical properties of RC due to a period of ageing (Boaro *et al.*, 2013).

Another important finding was that even with the long duration ageing period, all RCs showed SBS values to be higher than 20 MPa. The SBS after the 168th day of ageing in DW ranged between 27.16–22.5 MPa. These values of SBS have the potential to withstand the loading of mastication because they are above 20MPa, which has been suggested as the minimal required bond strength in the clinical situation (Yesilyurt *et al.*, 2009). The earlier studies showed higher SBS values, 56.8 and 34.8 MPa, over the same ageing period in DW (da Costa *et al.*, 2012; Baena *et al.*, 2015). The RCs and surface treatments were different between the present and the former studies.

Based on the findings, the changes in SBS cannot be related to the categories of RC materials. For the two nanohybrids (Kalore and N'Durance), only Kalore showed a statistically significant decrease in SBS over the ageing period, while N'Durance showed a statistically non-significant change in SBS. If the changes in SBS due to ageing were related to categories, both Kalore and N'Durance had to show similar results. Furthermore, the changes in SBS appeared to be multifactorial rather than related to one factor (as noted before).

Overall, the five RCs had a similar behaviour for WS over the entire immersion time period. From the 1st to the 28th day, they showed a gradual increase in WS% and then became more consistent. Thus, the 28th day could be considered the equilibrium uptake point for these RCs. For the amount of WS, RC composition is a possible explanation of difference in WS% between RCs. The range of WS was similar to those reported in the literature. Most of the materials performed in a similar way in that they revealed an increase in SBS on the 7th day and then started to show a decrease in SBS over the ageing period, except for N'Durance. The decrease in SBS was statistically significant for some of the RCs, while others showed more stability in that the change was not significant. Over every ageing time point, RCs also behaved differently in that in the early stages of ageing only two of the RCs showed statistically significant greater SBS than other RCs, but at the later stage of ageing only one RC showed statistically significant greater SBS than the other RCs. Finally, the amount of WS could not be directly linked to a decrease in SBS. Some of the possible reasons for the findings on WS and decrease in SBS due to immersion in DW were explained by RC composition.

In conclusion:

- RCs performed similarly during WS, showing a gradual increase in weight change till the 28th day of immersion in DW, followed by a more consistent change in WS values. The 28th day is thus considered to be equilibrium uptake point for all RCs in this work.
- The maximum WS% for Kalore, Filtek Supreme XTE, and Heliomolar was higher in value than those of Filtek Z250 and N'Durance. This difference in WS is thought to be linked to material composition.
- The aged repaired RCs showed approximately the same performance in SBS after ageing in DW over different intervals of 168 days. They showed an initial increase in SBS and then a decrease in SBS, with the exception of N'Durance, which was more consistent over the ageing period.
- Some of the RCs were more consistent in their SBS value over the ageing period than others due to showing a statistically non-significant decrease in SBS such as Filtek Z250 and N`Durance. While, Kalore, Filtek Supreme XTE and Heliomolar showed statistically significant decrease in SBS over the aging period.
- The differences in SBS between RCs changed over the intervals of the ageing period. In the early stages, both Kalore and Filtek Z250 showed statistically significantly higher SBS than other RCs, but in the late stages of the ageing period, only Filtek Z250 showed statistically significantly higher SBS.
- The reduction in SBS could not be fully explained by WS values, as it appears the statistically significant decrease in SBS is not only related to WS%, but also other factors.

Chapter 7. General discussion

Repair has been identified as an alternative treatment for failed restorations (Hickel et al., 2013), as repair may increase the longevity of the restoration with minimal intervention (Setcos et al., 2004; Gordan et al., 2011; Fernández et al., 2015; Kanzow et al., 2016). RCs have become widely used restorations because of the aesthetic, strength, bonding and conservative cavity preparation, and has thus been adopted in practice by dentists. During literature review, it was identified the findings from the in vitro studies are diverse in which it is not clear which repair methods should be used by those dentists who undertake RC restoration repair. Nor is it clear how effective repair protocols are at increasing the level of RBS. Further, the available information on the surface treatments undertaken in the clinical practice was incomplete. Accordingly, the work in this thesis aimed to investigate the practice of RC restoration repair by dentists primarily for the potential methods of repair. This information, along with the identified limitations in the literature, was used to guide the design of the *in vitro* studies and allowed the experimental works in the laboratory to increase understanding for the effectiveness on a number of surface treatments that were undertaken by dentists in the clinical practice.

The first work undertaken in this thesis consisted of a cross-sectional study of management of fractures of RC restorations by dentists in the primary care. This study aimed to investigate the practice of RC restoration repair by dentists primarily for the potential surface treatments undertaken prior to repair and belonging to either mechanical or chemical preparations. Little information has been published in the literature about the methods of RC restoration repair particularly at the time of the questionnaire study which was between 2013 and 2015 and in the UK. The earlier studies that provided information on the surface treatments undertaken by dentists were published after 2015 or focused on adhesive system for surface treatment (Staxrud *et al.*, 2016) or repairing marginal defects and veneers in which the answer options were less than those in the present study and chemical and mechanical preparations was not identified (Maria *et al.*, 2017). Furthermore, the present study showed that a number of tooth and patient factors were important for dentists when deciding to undertake repair that have not been shown by earlier studies. For example, one of the important factors for undertaking repair is sound tooth under the

restoration. As commented in the questionnaire chapter, the factors need further investigations and accordingly dentists' needs to be motivated for undertaking repair. Consequently, it appears the present study was not only performed before the earlier studies, but also provided more thorough information on the situations around undertaking RC restoration repair by dentists. This might help in prioritizing the future investigations better for improving RC repair in clinical practice.

In the questionnaire study analysis of the data showed most of the dentists (72%) would repair fractured RC restorations. Most used (91%) both mechanical and chemical preparations, and only one tenth (9%) were dependent on chemical preparation alone. For those dentists who use mechanical roughening, high speed coarse diamond preparation (71%) was the most widely used ones. This sort of information was shown by this study only because the mechanical and chemical preparations asked separately from the participants. This finding became one of the themes for the *in vitro* investigations because it is believed providing evidence on the need for removing from the remained restoration in the aim to enhance roughness is important to be considered for the magnitude effect of the coarse bur in comparison to not using it as repaired based on minimal intervention. A study showed 84.7% of participated dentists roughen veneer surface when undertaking repair using diamond bur, but the authors did not identify the grade of the bur (Maria *et al.*, 2017).

For chemical preparation, the present study showed phosphoric acid was used alone (9%) or with CP/BA (32%) or use of CP/BA alone (24%). From this result it is concluded there is a need for understanding the effectiveness of phosphoric acid when undertaking repair. Therefore, phosphoric acid investigation was included in the *in vitro* experiments. However, previous study reviewed RC repair by dentists have shown some dentists did not use phosphoric acid with adhesive agent, they did not show a number of dentists undertake surface treatment with phosphoric acid without including adhesive system (Maria *et al.*, 2017).

It would be useful to know if the reason for the participants' choice of repair method is based on patient preference or dentist opinion, or other factors. This information would provide more insight into the thought processes of dentists more generally around the topic of resin composite repair. This point was not considered at the time of the study design because this study chose to focus on the repair methods first for the investigation of clinical practice for RC restoration repair, and asking about the

reason of due repair would be in need for further room of unstructured questions that need different analysis from the current questionnaire design.

The reviewed literature showed most of the earlier *in vitro* studies used RBS to evaluate the effectiveness of repair protocols. For the *in vitro* experiments in this thesis, shear test was used to determine the bond strength between the substrate and the repair materials. This method is an established method used by a number of studies in the literature. Based on the best of my knowledge, shear and alternative methods (tensile, microtensile, microshear) have not been related to the clinical situation. This test was used to rank the bond strength between RCs and surface treatments because shear force is one of the forces that occur in the mouth. Moreover, shear force is one of the forces that test how much the repaired part resist displacement from the opposite force which is assumed the repaired restoration face during chewing. Therefore, it is believed this test is more relevant to the real situation of repaired restoration.

However, there has been some attempt to understand the effectiveness of individual surface treatments. One of the approaches that has been used is that measuring roughness. In the present study, the roughness of RCs was measured to evaluate the irregularities and undercuts created by the action of SiCs and surface treatments quantitatively. The rough area was believed to be best for the mechanical interlocking of repair RCs and bonding agents. Thus, measuring roughness might give an insight in to that how the roughness that created by surface treatments impact upon the change in RBS. Average roughness measurements, including Ra, have been the most widely used roughness measurement within RC repair studies (Fawzy *et al.*, 2008; da Costa *et al.*, 2012; Kallio *et al.*, 2013; Bahari *et al.*, 2018). In the current study, BAC was calculated along with Ra measurement. It has been shown that BAC parameters provide useful information on roughness profile, as if Ra alone is measured it misrepresents the roughness profile (Field *et al.*, 2013).

For the measurement of roughness, a contact stylus profilometer was used to measure Ra and BAC parameters. The contact stylus tip is usually made from of diamond, steel, or tungsten carbide. A roughness profile is produced when the stylus tip scans the surface in radical or linear way. It does so because the stylus tip in the X, Y and Z planes is sampled at regular intervals. Although a contact stylus profilometer is slower than an optical one and deforms delicate samples, they have

been identified as more accurate in measurement than non-contact ones (Ireland *et al.*, 2008; Field *et al.*, 2010). A contact stylus profilometer has been compared to non-contact interferometer for measuring RC roughness after surface treatments, and it produced similar rankings of the surface treatments (da Costa *et al.*, 2012). In contrast, one study showed that a contact stylus resulted in different rankings for prepared RCs with Soflex discs in comparison to Atomic Force Microscopy (Kakaboura *et al.*, 2007). However, the latter study was not on RC repair but on smoothness of RCs after using dental polishing instrument, and this may have impacted the findings. In the present study, a diamond stylus profilometer was used to measure the roughness of the RCs, which were not deformed by the stylus tip because they were measured after setting, as in previous studies.

In addition to roughness measurement for increasing understanding the effectiveness of surface treatments, the surface morphology of the prepared RCs with P600 was examined using SEM. This line of investigation was undertaken because roughness measurement parameters have limitation in describing surface topography. This approach has been used by a number of studies (Comba L *et al.*, 2015; Ahmadizenouz *et al.*, 2016; Ayar *et al.*, 2018; Bahari *et al.*, 2018). In this thesis two magnifications were used, 500x and 5000x, which were on the scales of 100 and 10 μ m. These two magnifications enabled the surface morphology to be viewed better than the use of one scale. The studies of RC repair mostly used high magnification greater than 500x, which is usually used to evaluate the roughness pattern (noted in the literature review).

Another approach that was used to increase understanding of effectiveness of surface treatments has been by measurement of RBS of RC samples before undertaking surface treatments (Cavalcanti *et al.*, 2007; Lima *et al.*, 2014; Altinci *et al.*, 2018), but the limitation of most of the studies that used this approach they applied intermediate materials such as bonding agent (Loomans *et al.*, 2011b; Özcan *et al.*, 2013a; Ahmadizenouz *et al.*, 2016). Furthermore, those studies that included none treated samples they did not consider the impact of surface characteristics, such as roughness, of RC types on RBS. Therefore, the RBS of earlier studies likely affected by either intermediate materials or difference between RC samples.

The first experiment in this thesis evaluated four different grades of SiC for better consistency in roughness between RC samples and evaluating SBS without

application of intermediate materials, and how that roughness impacted upon SBS. The P600 grade resulted in better consistency in roughness between RC samples, but two materials showed statistically significant difference in roughness. This indicated RCs are complex materials and they behave differently under the same condition. Therefore, it is important to evaluate the roughness of RCs even when prepared with the same SiC grade before the application of surface treatments. This likely allows better identification for the magnitude effect of the surface treatments in increasing RBS.

Within *in vitro* experiments in the present study, the RC samples were not aged before the application of surface treatments, as it is believed that ageing the substrate samples would increase the variability between the RCs and obstruct understanding of the effectiveness of the surface treatments on the level of SBS. However, there may be concern about the impact of DC of RCs on the level of SBS achieved because of the presence of unreacted monomers. Özcan and co-workers showed that a difference in DC between non-aged RCs does not impact on TBS; the authors tested two RCs in which the two materials were given different measures for filler content. The nanohybrid RC was by weight and the nanofill RC by volume (Özcan *et al.*, 2013a).

Based on the *in vitro* findings, phosphoric acid statistically significantly increased the SBS of two RCs out of five RCs in comparison to those prepared with P600. Phosphoric acid was shown to clean the RC surfaces of loose particles and resin debris, which was believed to be one of the reasons that led to increased SBS. Nonetheless, phosphoric acid did not lead to statistically significant change in roughness profile. Therefore, the cleaning action and statistically significant increase in SBS indicates the importance of including phosphoric acid in surface treatments, particularly in clinical situations, where there is the possibility of pellicles and food debris covered RC surfaces. The use of an effective cleaning material that statistically non-significant impact on RC surface and positively on RBS is an important step for increasing RBS, particularly for some types of RCs. However, there are no data to give insight into the impact in clinical practice. Fawzy et al. showed phosphoric acid impact on surface morphology and statistically significant in roughness, but the authors did not investigate the impact of those changes created by phosphoric acid on RBS before adding bonding agent (Fawzy et al., 2008). However, a number of previous studies examined RBS of phosphoric acid but in

combination with adhesive system. The table below explains earlier studies that investigated phosphoric acid.

Authors	Materials	Sample preparation	Chemical preparation	Test method	RBS (MPa)
Hannig et al, 2006	Microhybrid	800	Etch and rinse 2 steps	Shear	22.7
Ghavam et al, 2018	Microhybrid Nanohybrid	Polish disk	35% phosphoric acid+self-etch	Microtensile	11.9 10.9
Baur and llie, 2013	Microhybrid, nanohybrid	400	Etch and rinse 2 steps	Shear	30.1, 21.3
Tavarez et al, 2017	Microhybrid, Nanohybrid	pumice and brush	37% phosphoric acid+ bonding agent	microshear	27, 30.7

Table 7-1 Earlier studies investigated phosphoric acid in combination with adhesive system.

Adding CP/BA to RCs treated with P600 and phosphoric acid resulted in a statistically significant increase in SBS for one RC only, but both phosphoric acid plus CP/BA in combination resulted in statistically significantly higher SBS than those without phosphoric acid plus CP/BA (P<0.001), except for Filtek Z250. The SBS of the last chemical surface treatment reached a level that has been suggested as a minimal required bond strength of RCs in clinical practice, which is 20 MPa (Yesilyurt *et al.*, 2009). Hence, it is likely that chemical preparation not only results in a statistically significant increase in SBS, but may have an impact in the clinical situation. Furthermore, phosphoric acid alone did not statistically significantly increase the SBS for most of the RCs. This suggests that using phosphoric acid alone would not be the same as combined phosphoric plus CP/BA. A number of earlier studies have examined bonding agent in comparison to excluding it but they did not included phosphoric acid step in the adhesive system, the results of these studies are summarised in the following table below.

Authors	Materials	Sample preparation	Chemical Aging preparation		Test method	RBS (MPa)	Statistical analysis
Lima et al, 2016	Microhybrid, nanohybrid	Soflex	Two-step self-etch adhesive (two types)	24 hours DW (substrate)	Microtensile	5 or 2.2 to 25.7	SS except 1 bondinhg agent on nanohybrid
Özcan et al, 2014	Two microhybrids	Mylar	Bonding agent	24 hours DW (substrate)	Tensile	(21.7 to 35.1) or (17.5 to 41.5)	SS
Kaneko et al, 2015	Microhybrid	600 SiC + ultrasonic cleaning	Bonding agent	1 month DW (repaired samples)	Tensile	3.84 to18	SS
Rinastiti et al, 2010	Nanohybrid	Mylar	Bonding agent	No	Shear	14 to 21	NS except one nanohybrid
Present study	Microhybrid, two nanohybrids, nanofill, microfill	P600 SiC	Etch and rinse 2 steps	Repair ≤ 24hr DW	Shear	1 to 11/12 to 20	SS

Table 7-2 Earlier and current studies investigated RBS before and after including bonding agent.

SS represents statistically significant difference.

Roughening RC samples with a medium coarse diamond bur resulted in statistically significant higher SBS compared to prepared RCs with P600 (P<0.001). The change in SBS by bur was accompanied by a statistically significant increase in roughness compared with prepared RCs with P600 (P<0.001). The earlier studies (discussed in the surface treatments chapter) used bonding agent when compared bur treated samples to samples not treated with a bur. The present study evaluated SBS between bur treatment and P600preparation of RCs without the use of adhesive system. Therefore, the present findings have potential to represent the real difference between the two surfaces that were differed in roughness than the earlier studies, where the impact of adhesive systems were not excluded. The table below shows the earlier and present studies compared SBS and Ra of RCs treated with bur.

Authors	Materials	Control group (surface treatments)	Experimental groups (surface treatments)	Roughness (Ra µm)	Test method	RBS (MPa)
Valente et al, 2015	Microhybrid	Mylar + adhesive system	Diamond bur (126- 91 µm) + adhesive system	1.38/4	Microtensile	24.2/28
da Costa et al, 2012	Microhybrid	Soft-Lex Pop On + adhesive system	Diamond bur (151 µm) + adhesive system	0.17/3.82	Tensile	35.2/47.5
Wendler et al, 2016	Nanohybrid	Diamond bur (76-27 µm)	Diamond bur (126- 64 µm)	1.07/3.36	Tensile	4.86/7.15
Lima et al, 2016	Microhybrid	Soflex	Diamond bur (regular grit)	Nil	Microtensile	2.2/4.4
						5/4.5
Present study	Microhybrid, two nanohybrids, nanofill, microfill	P600 SiC paper	Diamond bur (135- 95 μm)	0.23-0.33/ 6-6.8	Shear	1-11/27-30

Table 7-3 RBS and Ra before and after bur treatment.

The combined use of phosphoric acid and bonding agent showed statistically nonsignificant increase in SBS on bur treated N'Durance and Filtek Supreme XTE when compared to RCs roughened with bur only. This finding suggesting the effectiveness of adhesive system depend on the surface that applied to it. However, phosphoric acid plus CP/BA might be included with bur treatment for two reasons. Firstly, it is standard for RC application. Secondly, they increase SBS, and this increase results in SBS values of all RCs to be above 20 MPa, which is likely to have clinical impact (noted before).

Generally, the roughness parameters used in this study provided useful information for increasing understanding in the roughness profile of RCs with different surface treatments, and the alignment of the changes in roughness profile with the change in SBS. BAC provided more thorough information on the roughness profile in the present study, as the BAC parameters showed the distribution of profile height in relation to peaks, cores, and valleys, as well as the proportion of peaks and valleys for RCs with different surface treatments. However, Ra showed a deviation of profile height from the mean line only. Ra findings for bur treated RCs were in alignment with the BAC parameters in terms of showing a statistically significant increase in roughness. This is due to the statistically significant increase in roughness profile not being limited to peak and valley proportions, but also roughness profile height, and this height can be represented by Ra. Furthermore, the statistically significant change in Ra and BAC parameters was sometimes followed by statistically nonsignificant changes in SBS. For example, the statistically significant difference in Ra and BAC parameters for RCs prepared with SiC grades did not align with the statistically non-significant difference between RCs in SBS. Also, the statistically significant difference between RCs for peak and valley proportions when treated with diamond bur did not align with the statistically non-significant difference between RCs in SBS. In contrast, the statistically significant increase in profile height for Ra and Rpk, Rk, Rvk aligned with the statistically significant increase in SBS when P600 preparation and bur treatment of RCs were compared (discussed in the chapter on surface treatments).

The last in vitro experiments in this thesis focused on simulating aging of repaired samples and investigating the impact of water sorption property of RCs on decrease in SBS due to aging. Simulating aging has been performed by most of the earlier studies even they are different in the type of aging method and aging fluid. Most
widely aging methods are immersion in fluid or thermocycling. Aging by thermocycling has been considered by a number of authors to be a questionable method (Özcan *et al.*, 2013a). In this thesis, immersion in DW was chosen to simulate aging over different time points. Therefore, evaluation of water sorption property considered to be valuable for determining the immersion over time in DW on the change in SBS. Based on the best of my knowledge water sorption property of repaired RCs have not been evaluated. The findings showed all RCs performed nearly similar for saturation time, but were different for the amount of WS%. The ranking of WS% did not align with the ranking of decrease in SBS for the 168th day immersion in DW.

The composition of RCs has been identified as a significant factor for RBS and their composition is considered to affect their reaction to the surface treatments (Loomans et al., 2011b; Ghavam et al., 2018). In the present study, RCs were different in composition and consisted of five categories: nanohybrid (Kalore), microhybrid (Filtek Z250), nanohybrid (N'Durance), nanofill (Filtek Supreme XTE) and microfill (Heliomolar). The studied RCs represent a wide range of categories that dentists use in clinical practice and their composition includes those currently accepted as universal filling materials; Kalore and N'Durance have been developed to overcome the limitations of the other materials such as polymerisation shrinkage and enhanced hydrophobicity of the resin matrix (Sakaguchi and Powers, 2012b). Therefore, the studied RCs were of wide variety in composition. The selection of these RCs is believed to make this study more generalisable and relevant to the clinical situation. where a variety of RCs are in use. One of the main constituent parts of RC is filler. Fillers impact both the physical and mechanical properties of RCs (Masouras et al., 2008a; Masouras et al., 2008b; Ilie and Hickel, 2009b), and therefore the studied RC categories are based on filler size characteristics. Further, the resin matrix of the studied RCs is based on dimethacrylate monomers such as bis-GMA, UDMA and TEGDMA, and recently developed innovative monomers such as high-molecular weight urethane with a rigid central section and flexible groups (DX-511) and dimer dicarbamate dimethacrylate (DDCDMA). The two latter monomers are in two RC products that do not contain Bis-GMA Kalore and N'Durance, respectively. Kalore has enhanced reduced polymerisation shrinkage because DX-511 monomer contains a rigid central section. Similarly, N'Durance has enhanced reduced polymerisation shrinkage and hydrophilicity because DDCDMA contains a bulky rigid central group

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that is hydrophobic (Sakaguchi and Powers, 2012b). Dimethacrylate based RCs have been the most widely used commercial RC restorations in clinical practice (Vasudeva, 2009). Based on the findings in this study, Filtek Z250 and N'Durance showed statistically non-significant decrease in SBS after ageing in DW over 168 days. However, Filtek Z250 showed WS% to be higher than N'Durance. Indeed, N'Durance showed a minimal amount of WS%. This indicates that these two types of RCs were minimally affected by DW. If they resist other fluids in the mouth, they are likely to increase the longevity of repaired restorations. Furthermore, the studied RCs behaved differently for different surface treatments (noted in previous paragraphs), while the bur treatment resulted in similar performance between the RCs when they showed statistically non-significant in SBS.

One of the strengths of this thesis is that the cross-sectional study collected clinical data to assess the need for *in vitro* studies, and thus this study design can be considered a novel work in RC repair topic. Consequently, the evidence provided in this thesis is relevant to clinical practice. The level of evidence in this study is based on laboratory work, in which most of the variables were standardised, which is opposite to the oral environment where there are multiple variables. However, in vitro based studies can compare repair methods under a controlled environment, and this allowed a better understanding of the mechanism that repair methods enact to improve the level of SBS. This information has the potential to determine which mechanism is more important for improving the level of SBS. Moreover, the findings that were based on *in vitro* experiments showed a number of statistically significant increase in SBS such as when RCs treated with phosphoric acid plus CP/BA in comparison to P600 preparation only. Also, when a coarse bur was compared to RCs prepared with P600, the SBS between the former surface treatments was statistically significant, although it is not clear how much statistically significant difference in vitro has a practical effect. Previous in vitro studies generally assume the statistically significant higher RBS value for a surface treatment has potential to increase longevity of repaired restorations better than those with statistically significant lower RBS values. Further, the high statistically significant differences in SBS suggest that the findings can be used as a testing hypothesis for clinical studies (Janket et al., 2018). However, the clinical study design for RC repair has its own challenge because of standardising the variability between cases, identifying the reason for the failure of repaired restoration, whether it was from the repair part or

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not, and the loss of follow up (identified in the literature review). Therefore, performing a clinical study would not likely be easy and it is not clear what the problems will be and the reason for some outcomes may necessitate going back to the laboratory experiments.

In translational studies, epidemiological studies are an important step for facilitating the identification of problems, providing evidence and translating it to practice in the clinical situation (Chiappelli and Prolo, 2003; Afrashtehfar and Assery, 2017; Janket *et al.*, 2018). RC repair has been identified to be one of the areas that showed delay in the providing clinical evidence and translation of findings (Gordan, 2013). Therefore, the present study should be of paramount importance for establishing evidence and a hypothesis for clinical studies and subsequent translation to clinical practice. It appears that the use of clinical data to determine the need for further investigation has been performed within the dental literature. The following two paragraphs explain two examples of studies that used clinical data to decide on further investigations.

The first example is that Klosa *et al.* (2015) reviewed the practice of cementation of all ceramic restorations by dentists who participated in a conference over a period of eight years studied at three intervals using cross-sectional studies. The authors used a handout questionnaire asking about the methods used by participants during the cementation of all ceramic restorations. When the results were compared to the available evidence in the literature, there was diversity from what had been accepted for cementation of these restorations by the majority of the participating dentists. This study identified incorrect practice for all ceramic crowns. The authors also suggested the available evidence has not been translated to the clinical practice.

The second example is a study performed in Newcastle University. The study collected clinical data retrospectively to assess the success of fixed partial restorations dependent on cementation rather than retention (Jamous, 2008). The restorations were placed in the Dental Hospital of Newcastle in the School of Dental Sciences. The data suggested an unaccepted failure rate for these restorations. The main reason for the failure was de-bonding between the restoration and cementation, and therefore the authors suggested further investigation to improve bond strength between the restoration and cementation materials. This study used clinical data to identify the clinical problem and the need for further work, which was

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laboratory-based investigations. However, this study could not clinically test the success of the restorations within the laboratory findings. It could compare the developed methods to the available methods that followed for the restoration cementation in practice.

A number of authors have identified that there are some challenges in translating research findings to clinical practice. However, they concluded that training dentists is the first step because dentists need to acquire skill to ensure that they have the capability to apply the evidence when there is capacity (Fox *et al.*, 2014). One of the examples that can be used to train dentists at least in the UK is the use of phosphoric acid with bonding agent when doing RC repair, because the questionnaire study and an earlier study (Blum *et al.*, 2005) showed that dentists do not use phosphoric acid or only use phosphoric acid without a bonding agent. While, one of the main finding in this thesis assumed to has potential to be translated in to the clinical practice at least in the UK because the finding give strength to adopting a protocol of repair that includes the use of coarse diamond bur prior to the application of phosphoric acid and bonding agent.

7.1 Conclusion and suggestions

In spite of its limitations, the study certainly adds to our understanding of how dentists do RC repair in the clinical practices, and how surface treatments, whether chemical or mechanical, can impact on SBS of repaired RCs and how RCs performed. Furthermore, based on the findings in this thesis bur plus phosphoric acid and CP/BA has potential to be an effective surface treatment method for RC repair. However, there is need for further investigations and comparison of this method to other methods to provide more evidence on the effectiveness of the bur plus phosphoric acid and CP/BA method.

7.1.1 Future works

The following are recommendations for future investigations to add to the current understanding of resin composite restoration repair:

• To investigate the reasons for the selection of a particular surface treatment by dentists during RC repair in clinical practice.

- To test other methods for their effectiveness in comparison to bur such as sandblasting/microabrasion.
- To investigate the substrate materials when they are aged in fluids to evaluate the effectiveness of the repair method even when the RC material undergoes a degradation process. This is to simulate the clinical situation where the restoration has been in the mouth for a number of years before fracture.
- To test different RC combinations acting as a substrate and repair materials to simulate the clinical situation, where mostly the type and brand of the repaired RC restoration is not known, and there is a high possibility of repairing the RC restoration with a RC that would differ from the original.
- Undertaking well designed and in-depth cohort studies to evaluate the clinical effectiveness of RC restoration repairs

7.1.2 Implications of the findings in this thesis

A number of findings within this thesis are recommended to be used for further studies of RC repair either *in vitro* or *in vivo*.

Implications for in vitro studies

- P600 SiC should be used to prepare the baseline surface of samples rather than the other grit sizes examined in this study. P600 SiC grit could provide better consistency in roughness that did not impact on the findings of SBS.
- The use of bearing area curve (BAC) as a tool for qualifying roughness profile along with Ra, where the potential of missing out information about roughness profile would be reduced compared to the use of Ra alone.
- Also, it would be better if the surface morphology of resin composites is examined along with roughness, as roughness measurements have limitations in describing surface characteristics of resin composites.

Implications for clinical practice

- Phosphoric acid and bonding agent should be used in combination, whether the RC surface is roughened or not.
- Bur plus phosphoric acid plus CP/BA should be used in clinical practice as they have more potential to enhance RBS than the use of bur or phosphoric acid and adhesive individually.

Appendices

Appendix A. Initial questionnaire format of the crosssectional study



Thank you for taking the time to complete this questionnaire

Please read the clinical scenario overleaf and answer the questions in relation to

the practice to which this questionnaire was sent

	Office use only
Management of fractured resin composite restoration	
Imagine a patient attended to your clinic today has complaint about a restoration chip fracture at mesial corner placed by yourself three months ago on central incisor.	
U.S.C.	
Please answer the following questions. The questions are asking about your decisions in relation to the above clinical scenario in the practice to which this questionnaire was sent:	
Please state year of your qualification as a dentist Please state year of your professional qualification	
Q1- Would you restore this fracture? Yes No No If yes please continue.	
Q2- Would you Smooth the fractured corner with no further treatment? Yes No III	
Q3- Would you replace the whole restoration?	
Yes No	
If no please continue.	

Q4- If	repairing the fractured portion of the restoration is the preferred treatment, please	
tick or	ne or more of the given choices.	
Do you	u routinely <u>mechanically prepare</u> the surface?	
0		
0	High speed coarse preparation.	
0	High speed polisning.	
0	Slow speed polisning.	
0	Microabrasion/sandblasting.	
0	Pumice or prophy paste.	
0	Others	
Do yo	u routinely <u>chemically prepare</u> the surface?	
0	No	
0	Rinse with water.	
0	Solvent (acetone, ethanol or chloroform).	
0	Phosphoric Acid etch.	
0	Hydrofluoric acid etch.	
0	Composite priming/bonding agent.	
0	Silane coupling agent.	
0	Flowable composite.	
0	Others	
What restor	factors are important to you when deciding <u>how</u> to repair a fractured composite ration?	
0	How long ago the restoration was placed.	
0	If I knew the restoration was sound underneath.	
0	The size of the restoration.	
0	The size of the fractured piece.	
0	If natural tooth tissue was also fracture.	
0	Heavy occlusion or parafunctional habits.	
0	Presence of tooth surface loss or gastric reflux/vomiting.	
0	Presence of bruxism habits.	
0	Others	
	This is the end of the questionnaire. Thank you for your times in completing it.	

Appendix B. Pre-test questions of the cross-sectional study

Can you answer the following questions which will further help in developing this questionnaire?

1- Did you find any question difficult to answer?

- 🗆 No
- □ Yes (Please write your comments).....

.....

2- Are the items appropriate for you to choose?

- 🗆 No
- □ Yes

3- Are the instructions clear?

- 🗆 No
- □ Yes

4- Do you think the questionnaire format is flow?

- \square No
- □ yes

5- Does the title guide you to the questionnaire?

- 🗆 No
- □ yes

Thank you for your time and cooperation

Appendix C. The final questionnaire format of the crosssectional study



Management of Fractured Resin Composite Restorations by Dentists

Thank you for taking the time to complete this questionnaire

	Office use only Identifier Number 1001
Please tick all responses that apply to your current practice	
NHS practice	1A
Private practice	1B
NHS and Private practice	1C
Multiple working dentists	1D
Single handed dentist	1E
Do you have any postgraduate qualifications? Please tick all that apply.	
None	2A
MSc	2B
MFDS	2C
Other(please state)	2D

Please read the clinical scenario and answer the questions overleaf





This is the end of the questionnaire. Please return it in the stamped and addressed envelope provided. Thank you for your time for completing it.

Appendix D. Introductory letter to dental practice managers

Dear Practice Manager,

We are surveying dental clinical practices in the North East of England, to explore how dentists manage fractured resin composite restorations. These types of restorations are more prone to early failure especially when they are placed in posterior teeth. Repair is one of the treatment choices that are based on minimal intervention dentistry.

Therefore the School of Dental Science at Newcastle University wants to find out the way that dentists are managing fractured resin composite restorations. Currently there is little information on the clinical practices they follow for managing fractured resin composite restorations.

We would greatly appreciate if you would encourage the dentists who are working in your practice to complete a brief questionnaire that will be sent to your clinical practice in near future with the title "Management of Fractured Resin Composite Restorations by Dentists". We expect that it should take approximately five minutes for them to complete. The results from this study will help us to understand the best way of managing fractured resin composite restorations.

If you have any questions about this survey feel free to contact us by telephone or email using the information overleaf.

Thank you very much for taking time to read this letter.

Yours Sincerely,

Tara Al-Barazanchi PhD student Dr. Giles McCracken Clinical Senior Lecturer Consultant in Restorative Dentistry

Contact addresses:

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Dr. James Field PhD Supervisor	james.field@newcastle.ac.uk	+44 (0) 191 208 8515

Appendix E. Introductory letter to participants

Date: 2015 March

Dear Dr Donoghue

We would like your help to improve our understanding of resin composite restoration repair. Little is really known about how dentists repair fractured resin composite restorations in clinical practice. Therefore, School of Dental Science at Newcastle University is conducting a study on resin composite restoration repair. The results from this survey will help us understand current methods used to repair this type of restorative material.

You have been identified from a list of local dentists to take part in this survey. We have not asked all dentists in the North East of England to take part, so your experiences and thoughts on this subject are very important to us.

Enclosed is a copy of a questionnaire and further information about this study. Please take time to complete the questionnaire and return it in the self-addressed envelope as soon as you are able to do so.

If you have any questions please feel free to contact us as shown in the information sheet that we have enclosed.

Yours sincerely,

Tara Al-Barazanchi PhD student

Dr. Giles McCracken Clinical Senior Lecturer Consultant in Restorative Dentistry

Appendix F. Participant information sheet



Management of fractured resin composite restorations by dentists

Information for participants

You are invited to take part in this research study; it is very important that you understand why the research is being carried out and what will it involve before you decide if you would like to take part. Please take time to read the following information and if there is anything that is not clear or you would like more information then please feel free to ask us.

What is the purpose of the study?

The purpose of this study is to investigate how dentists manage and repair fractured resin composite restorations as currently there is lack of information in this field. You will be asked to complete a short questionnaire about this topic. This will tell us more about what dentists do when they encounter this clinical situation.

Why have I been asked?

You have been asked to take part in this study because you are a dentist and you might repair fractured resin composite restorations in your clinical practice.

Do you have to take part?

No, whether or not you take part is your choice. If you do not want to take part, you do not have to give a reason.

What will my participation involve?

This study will involve completing the included questionnaire once. It should take approximately five minutes to complete. Your participation in the study will be completed when you have returned the questionnaire to us and you will only be contacted further if you have indicated you are happy for us to do so.

What are the possible benefits of this study?

There are no direct benefits to you from participating in the study. By participating in the study you are helping in our research that might improve patient care.

Page 1 of 2

Version 1: 2014 July

Are there any risks for you if you take part?

We do not anticipate any risks to you taking part in this study.

Will my taking part in this project be kept confidential?

Yes, your questionnaire data will remain anonymous throughout the study. Each participant will be given a unique code and all the information collected will be stored in a locked cabinet which only members of research team will have access to. Your participation in the study will be strictly confidential during the research and dissemination of the research findings.

What will happen to the results of this study?

It is an intention to publish the results of the study and be used as part of a research degree (PhD).

Who is organizing the study?

Organization and funding of this study is by the Newcastle University. The funding covers only the costs of the necessary materials to run the study.

Who do I contact if I have any questions?

If you have any questions or would like further information then please contact:

NAME	EMAIL ADDRESS	TELEPHONE
Mrs. Tara Al-Barazanchi PhD Student	<u>t.r.h.al-barazanchi@ncl.ac.uk</u>	+44 (0) 191 208 6378
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Dr. James Field PhD Supervisor	james.field@newcastle.ac.uk	+44 (0) 191 208 8515

Version 1: 2014 July

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Appendix G. Consent for future contact form



Consent to future contact for research

Management of fractured resin composite restorations by dentists

Please provide the following information if you agree to be contacted for future research related to this PhD study:

Name:

Contact address:

Post code:

GDC number:

I consent to further contact in relation to this research.

Signature:

Date:

Appendix H. Follow up letter to non-respondents

Date: 2015 May

Dear Dr Gupta

It has been around 2 months we sent a questionnaire from (School of Dental Science-Newcastle University) asking for your participation in our study about the **Management of Fractured Resin Composite Restorations By dentists**.

To date we have not received your response from you and we would like to ask you to take part. The questionnaire we sent to you should only take five minutes to complete.

If you have disposed or lost the questionnaire we would be delighted to send you another, please feel free to contact us further.

The time and consideration you have given us is very much appreciated.

If you require additional information please contact, our contact details are included in this letter.

Yours sincerely,

Tara Al-Barazanchi PhD student Dr. Giles McCracken Clinical Senior Lecturer Consultant in Restorative Dentistry

NAME	EMAIL ADDRESS	TELEPHONE
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Dr. Matthew German PhD Supervisor	matthew.german@newcastle.ac.uk	+44 (0) 191 208 3928
Dr. James Field PhD Supervisor	james.field@newcastle.ac.uk	+44 (0) 191 208 8515

Appendix I. The presentations from the works in this thesis

26th Euro Dental and Oral Health Congress poster (Italy, 2018)

Al-Barazanchi T, German M & McCracken G (2018) Effect of mechanical and chemical surface treatments on resin composite repair. Journal of Oral Health and Dental Management, Vol. 17, pp 70.



Repairing fractured resin composite restorations has the potential to increase the longevity of the primary restoration of a tooth with minimal intervention compared to restoration replacement (Ericson, 2007). There remains, however, no conclusive evidence for a universally agreed method for surface pretreatment of the composite prior to repair (Valente et al., 2016). A previous questionnaire based study of general dental practitioners in the North East of England suggested that many used a high speed coarse diamond burr both with and without adhesive systems as the most widely used method of repair (Field et al., 2017).

Aim

The aim of this study was to investigate different surface treatment methods upon shear bond strength of repaired resin composite restorative materials. Four commercially available resin composite materials were used to build specimens to repair and test (diameter=10mm, thickness=3mm, n=60). Specimens were light polymerised for 40s on both faces with an LED light unit (Collolux®LED, Coltene/ Whaledent INC, CE) of intensity 580-600 mW/cm². Two groups of surface treatments were selected: I.High speed coarse diamond burrs were used for 5s with water spray; 2. As for treatment 1, phosphoric acid etched (DETREY@Conditioner 36, DENSPLY, Germany) for 15s washed with water spray for 15s, composite priming/bonding agent (OptiBond ™ Solo Plus, Kerr) applied for 15s and air dried for 3s then light cured for 20s.

For each group, fresh composite was applied to the treated surface (diameter 5mm and height 6mm) and polymerised for 40s seconds ther incubated at 37°C for 24 hours in distilled-water. Shear bond strength (SBS) was determined for each group using a universal test machine (crosshead speed 1 mm/min, model 5567, Instron, UK). Roughness of resin composites was measured after surface treatment with course diamond burr using a contact profilometer (Surflest SV-2000, Mitutoyo, UK).

Results

Using coarse diamond burr in isolation resulted in statistically significantly lower SBS (P=<0.05) compared to application of diamond burr with etching and bonding for most of resin composites (Figure 1). Average roughness was not significantly different between resin composites except for Kalore (P=<0.05)(Table 1).



Mean shear bond strength (MPa) and standard deviation of resin composites as function of different surface treatments. * signifies a significant difference (P<0.05).

Discussion

The combined mechanical and chemical surface treatments investigated in this study can statistically significantly increase repair bond strength compared to the use of mechanical preparation in isolation for all resin composites. This findings can be used to motivate dentists in the clinical practice to avoid the use of mechanical surface treatment in isolation with course diamond burr to be able to enhance mergin band strength.

be able to enhance repair bond strength. Based on roughness average, Kalore was significantly smoother than other resin composites, while Kalore was no significantly differ in SBS compared to most of the resin composites. This may suggest this difference in roughness average has no impact on repair bond strength.

Resin composites	Average roughness (µm) Median (IQR)	
Filtek Supreme XTE	6.7 (6.2-7.3) ^a	
Heliuomolar	6.6 (6-7.8) ^a	
Filtek Z250	6.8 (6-7.3) ^a	
Kalore	6.4 (5.5-6.9) ^b	

Table1 Surface profile=roughness average Median roughness average and inter quartile range of resin composites after surface treatment with burr. Median followed by same letter indicate statistically similar median.

Conclusions

Within the limits of this study, using course diamond burrs in isolation on resin composites prior to repair resulted in a lower SBS in comparison to addition of both etching and applying a dentine bonding agent. Further work is required to consider the compound effect of composite aging and repairing dissimilar composites together.

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IADR poster (San Francisco, 2017)

Field JC, Al-Barazanchi T, German M & McCracken G (2017) Management of fractured resin composites by dentists in North-East England. Journal of Dental Research, Vol. 96(Special Issue A) (pp 2806-2806)



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