Concentrating Food Materials Using Electrokinetically Enhanced Filtration (EKEF):

Impact on Filtration Efficiency, Quality Compounds and Power Consumption

Thesis submitted for the Degree of Doctor of Philosophy

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Abstract

Countries around the world are suffering from many problems caused by the misuse of their resources either by using non-renewable ones or destroying the valuable features of the resources that are available.

Some of the most important challenges facing humanity are related to food and energy security. Food security includes many aspects besides providing people with an adequate amount of safe and healthy food; it also includes improving food processing to produce acceptable, high quality food products with reduced losses of nutrients, carbon footprint and using fewer resources in the production/packaging processes.

Dried and concentrated foods have become an important category in the food products marketplace. Many of the usual traditional methods require very high energy inputs. These methods may also have significant negative impacts on the foodstuffs, especially their chemical properties, such as loss of essential nutrients, as well as their appearance, aromas and flavour.

This project aimed to combine the electrokinetic phenomenon of electro-osmosis with the traditional functions of filtration to form a process of electrokinetically enhanced filtration (EKEF). This process is based on using electrokinetic effects to enhance conventional filtration technologies to concentrate foodstuffs at low temperature which saves energy, time, and product constituents associated with product quality, especially the temperature-sensitive components such as Vitamin C, fruit/vegetable colours and key aromas.

This study is concerned with factors affecting the efficiency of the EKEF process after its application to concentrate orange juice (14% dry matter content, pH=3.6) and malt extract (20% dry matter content, pH=4.55), and monitoring the changes that occur in indicators of food quality, such as the change in pH value, vitamin C content, colour, dry matter as well as the energy inputs to facilitate the EKEF.

The process of electrokinetics was combined with micro-filtration (MF) to speed up the process and save time and energy. This is carried out in an EKEF-rig which was designed to meet the project needs with an ability to change all the dependent parameters to find the optimum conditions to run this application.

Applying EKEF improved the filtration process and achieved net dewatering efficiencies up to 7.43% and 4.86% for orange juice and 10.68% and 6.26% for malt extract after 5.5hrs of processing under 30V and 15V respectively; and increased dry matter content from 14% to

33.09% and 29.52% for orange juice and from 20% to 34.2% and 32.14% for malt extract after 5.5hrs under 30V and 15V respectively. The maximum dry matter content was 28.75% and 29.63% under the control condition after the same time for orange juice and malt extract respectively.

Results showed that the performance of the EKEF process was dependent upon several parameters such as the sample's conductivity, applied voltage used, applied pressure, initial sample's thickness (the distance between electrodes) and filters pore size; all of these factors had a positive impact on improving the process efficiency except of the initial sample's thickness which had a negative impact.

Regarding the impact on the heat-sensitive food compounds, here represented by vitamin C; applying EKEF for 5.5hours reduced vitamin C retention in the concentrated juice to 41% under 15V and 7% under 30V. The retention of the total filtrate was also influenced by the EKEF process, where it fell to 48% under 15V and 35% under 30V.

Vitamin C retention was better under the EKEF process compared with its value under conventional thermal methods, where it needed about 10-15min at 50-75ºC and (3min) at 90ºC to drop to 50% or less *(Vikram et al., 2005)*. EKEF also had a negative impact on the juice colour and pH value.

The direction of water flow and the profile of fluid flow between the two electrodes were thoroughly investigated to identify the main filtration outlet and its position; the power consumption of the application was also calculated and compared with power consumption of conventional thermal methods.

The EKEF process resulted in a large power consumption addition compared with applying the pressure only; however, the required additional energy needed to achieve the same increase in dewatering efficiency by increasing the pressure only without EK was relatively small. On the other hand, EKEF showed a good efficiency in energy saving compared with the power consumption required in more conventional thermal methods used to evaporate an equal amount of water. This saving was up to 18.35 times and 47.34 times in OJ and ME dewatering respectively.

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Thanks all I really appreciate everything you gave to me..

Hasan…

List of Abbreviations

A/C Ratio	Anolyte to Catholyte Ratio
DE	Dewatering Efficiency
EK	Electrokinetic
EKEF	Electrokinetically Enhanced Filtration
EKG	Electrokinetic Geosynthetics
FFR	Filtrate Flow Rate
IEP	Iso-electric Point
LCP	The Lowest Conductivity Point
ME	Malt Extract
NDE	Net Dewatering Efficiency
OJ	Orange Juice
pH/IEP Ratio	pH to Iso-electric Point Ratio
WPD	Water Profile Direction
VCP.	Voltage Crossing Point.

Glossary

Anode: is the positively charged electrode, where the negative ions (anions) move to under a voltage application.

Cathode: is the negatively charged electrode where, the positive ions (cations) move to under a voltage application.

Anolyte: is the electro-osmotic flow corresponding to the respective movements toward the anode *(Yang et al., 2005)*.

Catholyte: is the electro-osmotic flow corresponding to the respective movements toward the cathode *(Yang et al., 2005)*.

Total Filtrate: is the total discharged filtrate exiting from both outlets, (anolyte and catholyte combined together)

Flow rate: is the discharged filtrate weight per minute (g/min).

Dewatering efficiency: is the ratio of the total filtrate weight (the anolyte and the catholyte weight) relative to the initial water weight in the sample.

Net dewatering efficiency: is the total dewatering efficiency minus the efficiency of the control (without EKEF).

Dewatering Process Outcome: is the weight (g) of the total filtrate streams exiting from the outlets through filters all over the dewatering process time.

Control orientation: is a phrase used in this study to describe an experimental orientation when the EKEF is off and the voltage value is 0V (no voltage applied).

Normal orientation: is a phrase used in this study to describe an experimental orientation when the EKEF is on, anode (+) is the bottom electrode and cathode (-) is the top electrode.

Reversed orientation: is a phrase used in this study to describe an experimental orientation when the EKEF is on, the cathode (-) is the bottom electrode and anode (+) is the top electrode.

Water Profile Direction: The direction of the major volume of discharged filtrate (or water) towards one of the two electrode.

The main outlet: is the outlet where the water profile goes to (the outlet of the major discharged filtrate volume).

Anolyte to catholyte ratio (A/C ratio): the percentage (or the volume) of the anolyte relative to the percentage (or the volume) of the catholyte.

Iso-electric Point (IEP): is the pH at which a particle is electrically neutral and carries no net electrical charge.

The Lowest Conductivity Point (LCP): (used for this study) is the pH at which foodstuff has the lowest conductivity.

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Chapter 1. Introduction and Aims

1.1. Introduction

The UK food sector is one of the most important industrial sectors, and one of the richest areas for innovation. The need for better food quality as well as variety have increased in the last decade as the consumers have become better educated in terms of health issues, nutritional aspects and food quality. All these factors encourage food producers and companies to seek new methods and ways to offer better food products with higher quality and greater safety.

Within this new-found demand for innovation, non-thermal processing of food has appeared as a possible opportunity for the industrial sector to provide better wellness and health for consumers, and potential new products with excellent quality and high inherent safety.

These emerging technologies are surrounded by immense challenges. However, there is a long list of research groups interested in these research area as these non-thermal applications are developing and making a significant positive impact on energy and the food sector. It also provides an excellent balance between cost and high quality on one side, and safety and reduced processing on the other side.

Non-thermal methods could be understood as an effective alternative to conventional thermal methods. However, non-thermal processing can also be effectively combined with thermal processing to achieve very good results in different areas such as inclusion of antimicrobials and/or bacteriocins, pH, and water activity modifiers.

Very attractive prices and superb quality make the opportunities for these new products commercially attractive *(Zhang et al., 2011)*.

Within more recent food processing innovation, drying and concentrating are being re-examined as effective methods to preserve food by removing water, which reduces water activity and thereby inhibits or decreases the growth of microorganisms and hinders decay and subsequent loss of quality. Dried and concentrated materials are classified into several categories according to the method used to dry them. Different methods of dewatering are used to give different products of different quality, physical and chemical properties with different prices *(Russell and Gould, 2003)*.

There have been significant improvements when using these methods, as drying food (to prevent spoilage) using the sun and wind are very old practices which have been used consistently since ancient times. Water is usually removed by several different methods; some of them depend on evaporation (air, sun, smoking or wind drying) but, in the case of freeze-drying, water is removed by sublimation after freezing food. Drying using heat to evaporate water is used widely.

Each of these methods has several disadvantages and may have negative impacts on the quality of the resulting foodstuffs. They impact especially on the chemical properties such as loss of essential nutrients, as well as the appearance, the odour and the flavour. In addition, many of the previous methods require high energy consumption *(Fellow, 2000)*.

Nowadays, there are many non-thermal methods used to reduce water content such as industrial high-pressure or to minimise the microorganisms' activities such as pulsed electric field (PEF). Researchers are still working to improve these methods and innovate others, all these methods depend on controlling the activation, inactivation, or retention kinetics and mechanisms of microorganisms, viruses, allergens, nutrition and toxins subjected to non-thermal processes *(Knorr, 1993; Zhang et al., 2011)*.

One of the ways used successfully in the field of dried and concentrated material to avoid these problems is microfiltration, "*Membranes with a pore size of 0.1-10 µm. Microfiltration membranes remove all bacteria. Only part of the viral contamination is caught up in the process, even though viruses are smaller than the pores of a microfiltration membrane. This is because viruses can attach themselves to bacterial bio-film" (Lenntech, 2015)*.

Microfiltration can be implemented in many different water treatment processes when particles with a diameter greater than 0.1 µm need to be removed from a liquid; and ultrafiltration, "*a high-level filtration system for the treatment of water and other liquids; based on filtering colloids and molecules between 0.002 and 0.1 µm in size by forcing liquids through a membrane with extremely fine pores. This method is capable of removing solids, viruses and bacteria*" *(Chisti, 2007)*.

Most filters in use today play a passive role, e.g. geo-membrane barriers help to prevent the passage of liquids. In addition, reinforcement in its turn provides tensile resistance, but that happens only after an initial strain has occurred. The passage of water is provided by the drain but the drain does not itself cause water to flow. Trying to improve this process, new methods need to be used as a separate process or a supportive one by combining it with other applied processes such as filtration.

Applying an electric field to a charged liquid or semi-liquid material using two electrodes results in several processes starting at different components: charged liquid ions, charged liquid particles and the liquid itself. These processes, which are described as Electrokinetics, cause a movement of these different components towards one of the electrode according to the process and the ion number and types.

"*The movement of charged particles in an aqueous medium to an electrode of opposite polarity is known as electrophoresis*" *(Weber and Stahl, 2002; Lockhart, 1981)*. The second type of movement, electro-osmosis, is defined as the displacement of liquid relative to the medium under the influence of an electric field (Weber & Stahl, 2002; Lockhart, 1981). There is another type of movement under electrokinetics which is produced by the same mechanism as electrophoresis but at the liquid level and works in the opposite direction to the charged ions that moves to the opposite polarity electrode under electrophoresis (Aziz, Dixon, Usher & Scales, 2006).

Previous processes and their application have been a very rich interesting area for many researchers to understand the impact of applying an electric field to a liquid and control this impact to enhance the dewatering process by combining these electrical phenomena with the water motion.

For instance, in the civil engineering area, new applications for geosynthetics have been created to provide an active role, initiating physical, chemical or biological changes to the matrix in which it is installed, by combining the electrokinetic phenomena of electro-osmosis, electrophoresis and associated electrokinetic functions such as electrolysis with the traditional functions of geosynthetics of drainage, filtration, containment and reinforcement to form electrokinetic geosynthetics (EKG) *(Nettleton et al., 1998; Hamir et al., 2001)*. Electrokinetic geosynthetics (EKG) is an important technology which can be considered as a platform, one which combines a wide range of materials and processes to perform such various functions as dewatering, drying and concentrating for different materials such as food, soils, sludge and slurries *(Jones et al., 2010)*.

There is a relatively good number of published papers about using electrokinetics in the civil engineering area to dewater non-food materials such as soil, mine tailing, waste and others; and a few studies about using the pulsed electric field on food material to increase the shelf life of these materials during the storing period. However, the number of publications describing using electrokinetic phenomenon to improve filtration in the food process area is rare and mostly confined to one study was done by *Ng et al. (2011)* to investigate the impact of electrokinetics on dewatering food waste and to compare the power consumption of the application with the power consumption needed to dewater the same volume of water using conventional thermal methods.. Therefore, this study has been carried out to test the impact of the electro phenomena (specifically the electrokinetically enhance filtration application EKEF) on the filtration efficiency including the flow rate amount and direction, the chemical properties and the power consumption.

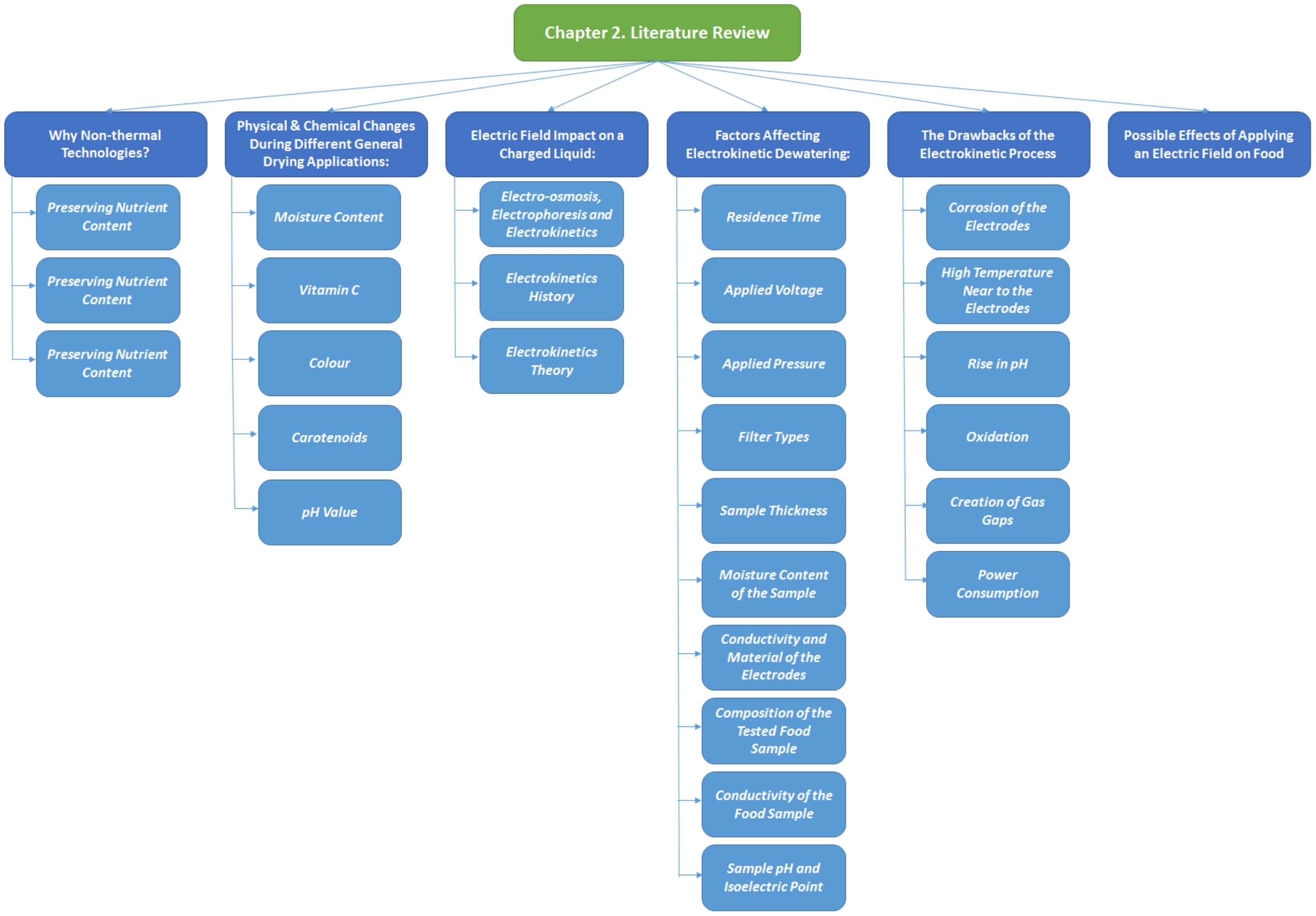
1.2. Aims and Objectives

This project aimed to investigate the efficiency of the application of EKEF to food concentration and the factors affecting it, and the potential physical and chemical changes in the appearance and quality of the product.

Several experiments were carried out with the following objectives:

- Designing an experimental rig to help to control the effective factors that have an impact on the EKEF process such as conductivity, applied voltage, applied pressure, filter pore size, initial sample thickness (the distance between the two electrodes).
- To investigate the efficiency of the application of EKEF in concentrating different food materials (orange juice and malt extract); and the impacts of the main factors which control the EKEF process progress and efficiency (the ones mentioned above), as well as the chemical properties of pH and LCP (the lowest conductivity point) values.
- To investigate the potential changes that could have resulted from applying EKEF to foodstuffs by analysing some quality compounds, such as pH, vitamin C (ascorbic acid) content, colour, dry matter and moisture content, and compare these results of this application with the results of published conventional methods.
- To measure the power consumption of this application under different running conditions.
- To suggest ways to control the conditions that affect the EKEF process to increase the outcome (total filtrate), decrease the negative impacts on foodstuff quality compounds and reduce the power consumption.
- Finally, to set up theoretical guidelines depending on the relationship between the chemical properties of samples and the direction of the water profile after applying an electric field which helps in the choice of the optimal application specification and to predict the process progress according to the food material properties.

Chapter 2. Literature Review



Flowchart 1. Chapter 2 (The Literature Review) Content

2.1. Why Non-thermal Technologies?

The drying and concentrating of food materials utilised in several industrial applications to reduce the volume of foodstuff which in turn reduces transportation cost and storing space; Drying and concentrating are some of the oldest methods used to preserve food by reducing the water content to inactive microorganisms and increase the food products shelf life.

The most common processes used for food preservation are by thermal pasteurisation and thermal sterilisation. These two methods depend on heat to deliver the microbial inactivation and enzyme activity reduction in the food products which results in greater safety and an extended storage life compared with the raw equivalent.

Thermal processes aim mainly to inactivate the pathogens and the microbial spores to produce food which are microbiologically inactive and therefore safer. However, thermal processes result in a number of changes which can affect the quality of the final product, for instance, colour, flavour, texture and the general appearance of the final product.

Due to the recent increase in the consumers' needs and demands for quality food products as well as their concern about the safety of their food, consumers start looking for more fresh food characteristics in their purchases, along with high nutrient content and sensory quality.

Consumers have also become more aware of food content and processing technologies *(Zhang et al., 2011; Evans and Cox, 2006)*. Thus, the need for an alternative method that preserves food's fresh characteristics, which achieves microbial inactivation, produces environmentally-friendly products, and at reasonable cost, has become very crucial and considered as a significant present challenge of food producers, scientists, and technologists all over the world.

In the last few years, non-thermal processes and applications have been explored widely as it represents a novel area of food processing. It is also very appropriate to combine these novel technologies either among themselves or with traditional ones.

There are many factors which have drawn attention to the need for new methods to dry and concentrate foodstuffs such as preserving the food, reducing power consumption, the loss of nutrients and the cost at the same time.

2.1.1. Preserving Nutrient Content

The need to inactivate microorganisms is a main condition to preserve food and extend the shelf life, but in most cases the processes that achieve this detrimentally affects the nutrient content of some food products due to the thermal sensitivity of some nutrients; for instance, juice, meat, eggs, fish, milk, and other important sources of protein which are affected greatly once they are subjected to thermal treatment such as pasteurisation or sterilisation.

Thus, there is a big need for a process that can maintain original nutrient content and does not affect the functionality or the structure of ingredients.

Non-thermal technologies are good ways to reach this goal , for example high hydrostatic pressure (HHP) has shown to have a negligible effect on food nutrient content; this was very clear with the anthocyanin content in fruits and vegetables after processing *(Tiwari et al., 2009; Zhang et al., 2011)*.

2.1.2. Sensorial Quality of Food

It is very common to observe changes in the sensory characteristics of food after using thermal processing. Temperature works as catalyst for some chemical reactions between fats, amino acids, proteins, vitamins, mineral salts, pigments and other chemical species in food, resulting in a number of physical changes.

Protein denaturation, coagulation or precipitation, browning, gelation, loss of colour and flavour, starch retrogradation, oxidation changes in microstructure and final texture loss of functionality, and other related chemical reactions occur in foodstuffs during thermal treatment.

Some studies of using pressure in juice processing showed insignificant changes between the quality of fresh juice and the pressurised orange juice during storage for about 3 months at 5°C *(Knorr et al., 2002)*. In consumer tests, consumers preferred the pressurised version when they were asked to compare it with fresh-squeezed and thermal pasteurized juices *(Evans and Cox, 2006)*.

2.1.3. Power Shortage and Environmental Issues

Food production is a multi-billion pound industry that converts animal and plant products into intermediate or final products for human consumption (or animal consumption as animal feed). This industry is dominated by large-scale, capital-intensive firms. The food-processing sector typically consumes significant high amount of energy and considered as one of the largest manufacturing energy consuming sector; in USA for instance, the food processing sector has the fifth-highest energy consumption of the sectors considered in the analysis included in a report for *U.S. Environmental Protection Agency (2007)* prepared by ICF International in 2007.

Energy is an important input cost for the food industry, and comes in as the third most significant portion of final cost along with raw materials and labour.

For food manufacturing, the most important fuels are natural gas, purchased electricity, and coal. Thermal applications applied in food industry consume the major portion of the power and increase gas emissions. All data and research in this area mention the urgent need for developing new methods which reduce power consumption and gas emission *(U.S. Environmental Protection Agency, 2007)*.

2.2. Physical & Chemical Changes During Different General Drying Applications

Several changes occur in food materials during the drying process; these changes affect the chemical and physical quality of the final products negatively. Some parameters have been tested to observe these changes. These parameters help to evaluate the effectiveness of the different methods. The following are the parameters and their changes under different methods:

2.2.1. Moisture Content

The main aim of the dewatering process is to decrease the water content of the food material which in turn decreases the volume of the final product and the microbiological activities and increase the shelf life of the product.

2.2.2. Vitamin C (Ascorbic Acid)

Vitamin C (ascorbic acid) is highly sensitive to degradation and has very low stability in aqueous solution. Vitamin C is oxidized readily in light, air and when heated. Because it is water soluble, heating in water (like thermal treatment) causes the vitamin to be oxidized and also to leach out of the food into the water *(Tannenbaum and Walstra, 1985; Lee et al., 2004)*.

Orange juice is a highly valued product representing a significant source of vitamin C in the diet. Different methods are used to concentrate orange juice especially thermal methods, *(Polydera et al., 2003).*

Heat treatment during food processing would seem to be a real threat because of the high heat used. However, this high heat destroys the enzyme ascorbic acid oxidase, found in fruits and vegetables, before much vitamin C is oxidized, so paradoxically some heat treatment may be beneficial to the final vitamin C content of the foodstuff.

On the other hand, freezing also has a negative effect on vitamin C if the tissues of the product have been broken and exposed to air *(Sizer et al., 2009)*.

This suggests that all traditional dewatering methods would affect vitamin C negatively, either because of the high temperature, such as used in thermal treatment, or because of the oxidization in air after breaking the tissues such as in freezing, or as a loss with the output "effluent discharge" since vitamin C is water soluble.

Vitamin C degradation in OJ at various temperatures heated by different methods was investigated previously by *Vikram et al. (2005)*; manually squeezed orange samples were processed by different methods (conventional, ohmic, infrared and microwave) under different time and temperature combinations as shown in *Table 2.1*:

Table 2.1. Thermal treatment conditions for OJ heating (Vikram et al., 2005)

Conventional, Ohmic, Infrared Temperature (°C)	Microwave		Time (min)
	Power Level (W)	Approximate temp. attained (°C)	
50	245	100	1, 5, 8, 10, 15
60	315	105	1, 2, 3, 5, 10
75	385	110	1, 5, 8, 10
90	455	125	0.5, 1, 1.5, 2, 3

The results indicated that the heating methods had an impact on the retention of vitamin C. Under each heating method, the degradation was more rapid at higher temperatures. The retention of vitamin C under these methods of heating is indicated in *Figure 2.1*. Ohmic heating caused the most rapid degradation followed by microwave, and infrared heating. The conventional method had a longer lag period and the approximate vitamin C retentions at different temperature were as shown in *Table 2.2*, which shows that almost half of the vitamin C content was lost after 10min of the conventional heating method at 50°C.

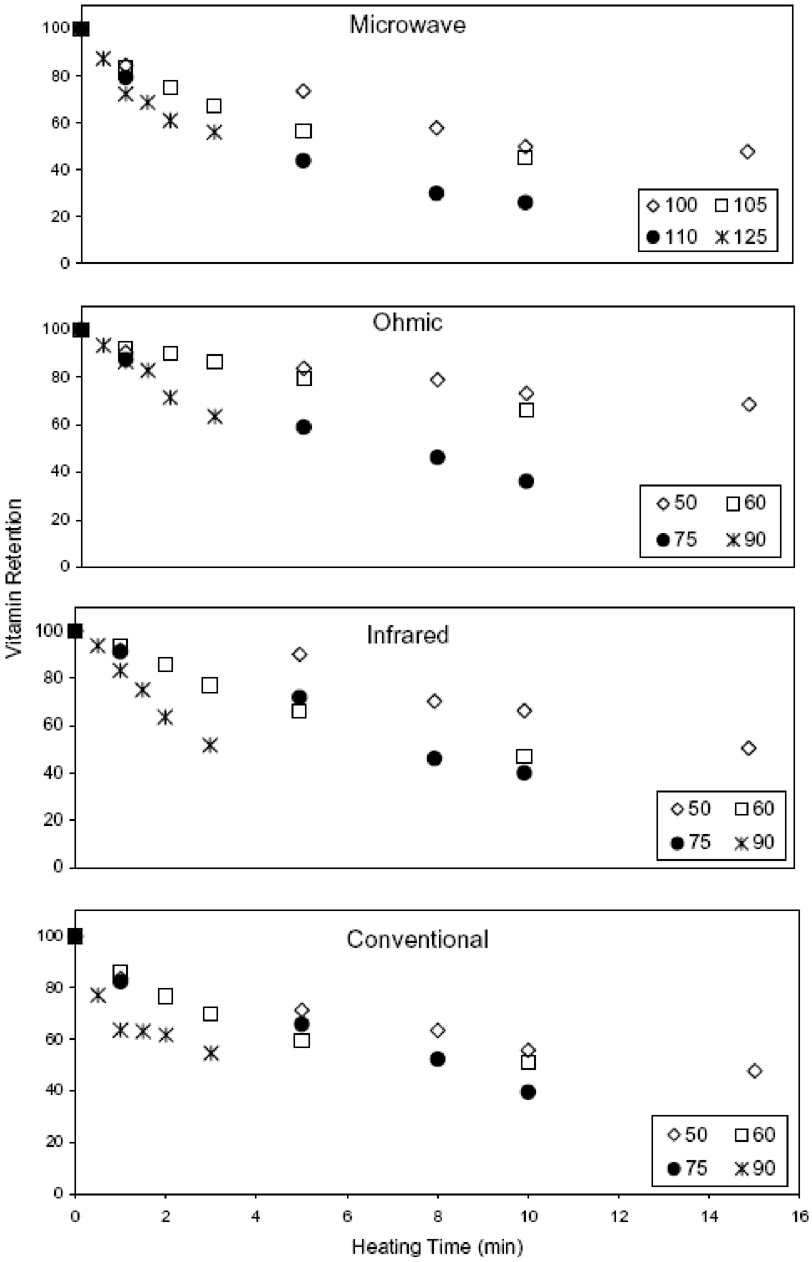


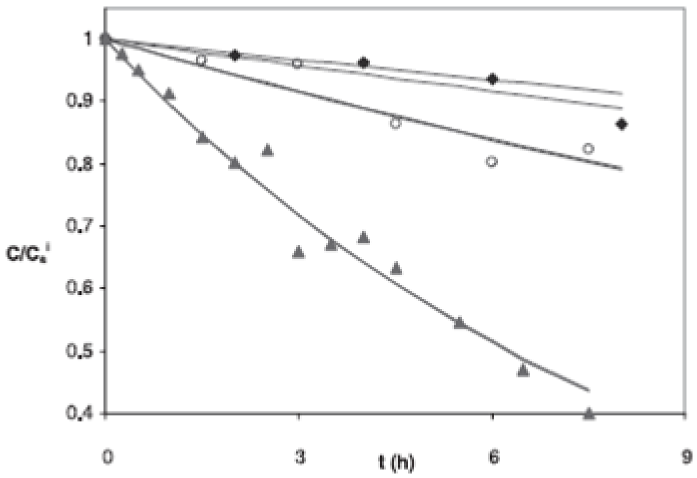
Figure 2.1. Change in vitamin C retention during heating by different methods at different temperatures (Vikram et al., 2005)

Table 2.2. Vitamin C retention during heating by conventional method at different temperatures (Vikram et al., 2005)

Conventional Temperature (ºC)	Time (min)	Vitamin C Retention %
50	10	58
60	10	50
75	10	40
90	3	57

Lower temperatures have also had a negative impact on vitamin C content, the thermal degradation of vitamin C in OJ was analysed over in a 20-45°C temperature range by *Manso et al. (2001)*.

Vitamin C retention change over 8 hours at 20, 30 and 45ºC was shown in *Figure 2.2*, which shows that vitamin C loss reached 5% at 20 ºC, 15% at 30 ºC, and 45% at 45 ºC after 5.5h.



First-order model fits to ascorbic acid data for times less than 8 hours at ◆ 20, ○ 30 and ▲ 45 °C. The dashed line indicates the individual fit and the solid line the global fit.

***Figure 2.2. Change in Vitamin C retention during heating at different low temperatures* (Manso et al., 2001)**

In addition to the heat impact vitamin C can be lost through other degradation reactions:

- ✓ *Ascorbic Acid Oxidation:* An enzymatic reaction by ascorbic acid oxidase in the presence of light and oxygen especially during any freezing treatment of foodstuffs *(Nursten, 2005)*.
- ✓ *Maillard degradation of vitamin C*: Vitamin C is a reducing carbohydrate and can react with amino acids, peptides, and proteins. These types of reactions between carbohydrates (sugars) and proteins belong to a class of reactions known as Maillard reactions. The Maillard reactions of vitamin C are involved in the browning of cut fruit and can cause changes in the flavour of foods. In addition, the Maillard degradation of vitamin C in the body may be involved in clouding the lenses of the eyes and in the age-related loss of elasticity in the skin and sinews *(Smuda & Glomb, 2013)*.

The process of vitamin C degradation has previously not been truly understood; *Smuda & Glomb, 2013* have comprehensively studied the amine-catalyzed degradation of vitamin C in a model system and clarified about 75 % of the Maillard-induced degradation reactions of vitamin C, the end products of these reactions are carbonyl and dicarbonyl compounds, carboxylic acids, and amides.

Among other compounds, the researchers identified N6-xylonyl lysine, N6-lyxonyl lysine, and N6-threonyl lysine as unique characteristic end-products of vitamin C Maillard systems.

2.2.3. Colour

Food product colour is considered to be one of the important sensory attributes of interest to customers and workers the food industry. It is also considered as a very important indicator of sensory evaluation *(Tepper, 1993)*.

Non-enzymatic browning of citrus beverages is a major quality deterioration factor. More than one type of mechanism may be involved in the formation of brown pigments: the reactions between amino acids and reducing sugars (Maillard reactions), the aerobic and anaerobic degradation of the ascorbic acid *(Azandouz and Puigserver, 1999; Johnson et al., 1995; Kacem et al., 1987 and Sakai et al., 1987)* and further reactions of the carbonyl compounds via aldol condensation or reactions with the amino acids to yield brown pigments *(Fustier et al., 2011)*.

Generally the browning reactions in foods could be divided into: an enzymatic reaction (phenolase browning) and non-enzymatic reactions (Maillard, caramelisation and ascorbic acid oxidation) *(Nursten, 2005)*.

- ✓ ***Phenolase browning*** is an enzymatic reaction by polyphenol oxidase in the presence of light and oxygen especially during any freezing treatment of foodstuffs.
- ✓ ***Maillard reaction*****:** Is a chemical reaction between free amino acids and a reducing sugar, usually requiring heat *(Madruga et al., 1995)*.

  A complex mixture of poorly-characterized molecules is formed when the sugar reactive carbonyl group reacts with the nucleophilic amino group of the amino acid; this complex mixture is responsible for a range of odours and flavours often characteristic of baked products. An alkaline environment accelerates this process, as the amino groups are de-proteinated and, hence, have an increased nucleophilic nature *(Belderok, 2000)*.

García et al. (2010) reported that it is hard to evaluate the pH influence of the Maillard reaction, because the reaction itself influences pH significantly. The ratio of products formed and the rate of colour formation are also influenced by the pH; colour can be reduced by decreasing the pH. However, pH has a less dramatic effect on aroma than temperature, time or water content. The required temperatures for the reaction depend on the pH value but normally it should be more than 90ºC. These conditions are achieved during thermal treatments more frequently than during non-thermal treatments.

- ✓ ***Caramelisation***: *Chen et al., (2008)* reported that caramelisation is the browning of sugar. As the process occurs, some compounds such as furanones are produced and volatile chemicals are released, producing the characteristic caramel colour and flavour.

  Like the Maillard reaction, caramelisation is a type of non-enzymatic browning. However, unlike the Maillard reaction, caramelisation is a pyrolysis reaction, as opposed to a reaction of the sugars with amino acids. In caramelisation, disaccharides are broken down into the monosaccharides fructose and glucose *(Ajandouz et al., 2007)*.

  Caramelisation is very difficult to induce in non-thermal applications as it needs a temperature of more than 120ºC.

- ✓ ***Ascorbic Acid Oxidation:*** A further mechanism appears to operate during the discoloration of dehydrated vegetables in which ascorbic acid is involved. The formation of dehydroascorbic acid and diketogluconic acids from ascorbic acid (mentioned in *Section 2.2.2*) is thought to occur during the final stages of the drying process and is capable of interacting with the free amino acids, non-enzymatically, producing the red-to-brown discoloration *(Eskin, et al, 1971)*.

Several factors can affect the formation of coloured complexes in food products. Among these are pH, temperature, moisture content, time, concentration and nature of reactants *(Lee, 1983)*.

- ➢ The rate of browning increases with rising temperature. Since these reactions have been shown to have a high temperature coefficient, lowering of the temperature during the storage of food products can help to minimize these processes *(Eskin, et al, 1971)*.

- These reactions being moisture dependent for optimum activity can be inhibited by reducing the moisture content through dehydrating procedures. In attempting to carry out these procedures one must ensure that the dehydrated product is suitable for sale in that form, and that the product is suitably packaged so as not to permit moisture uptake during storage.

- Since the Maillard reaction is generally favoured at the more alkaline conditions, if this type of browning is involved, lowering of the pH might provide a good method of control *(Eskin, et al, 1971)*.

- Gas packaging is extremely useful in excluding oxygen by using an inert gas. This reduces the possibility of lipid oxidation, which in turn could give rise to reducing substances capable of interacting with amino acids. While this reaction does not appear to influence the initial carbonyl-amino reaction, exclusion of oxygen is thought to effect other reactions involved in the browning process *(Eskin, et al, 1971)*.

- Chemical inhibitors have been used to advantage in limiting browning reactions during the production and storage of a variety of foods. Among those widely used are sulfites, bisulfites, thiols, and calcium salts *(Eskin, et al, 1971)*. Sulfites proved successful in controlling a variety of browning processes. Bisulfites inhibit the conversion of D-glucose to S-hydroxymethyl- furfural, as well as the conversion of ascorbic acid to furfural by complexing through the reducing group. Consequently the formation of furfurals is blocked, thus preventing the production of the coloured pigments. They can also block the carbonyl group of the reducing sugars involved in the carbonyl-amino reaction *(Fox, 1991)*.

- Calcium chloride was reported to be a possible inhibitor of browning. Its inhibitory effect is due to the chelation of calcium with the amino acids *(Eskin, et al, 1971)*.

2.2.4. *Carotenoids*

Carotenoids are some of the most important pigments in fruit, and they are present in moderate concentrations in orange juice (28µg Carotene/ 100g Orange juice). However, oranges also accumulate anthocyanin pigments such that their juices exhibit a characteristic reddish colour *(Arena et al., 2000; Kirca and Cemeroglu, 2003; Meléndez-Martínez, 2005)*.

The chromospheres of conjugated double bonds of carotenoids are responsible for several properties such as the colour and other functions of these pigments *(Britton, 1995; Meléndez-Martínez et al., 2007a,b)*. The instability of these compounds should be taken into account especially with respect to oxygen, light and heat. To avoid these factors affecting carotenoids, a series of precautions must be taken during their preparation and analysis *(Britton et al., 1992, 1996; Schiedt and Liaaen-Jensen, 1995; Rodriguez-Amaya, 1999,2001; Meléndez-Martínez et al., 2004)* such as separating them in a solution that help to increase their stability.

Thermal processing can often lead to detrimental changes to the sensory properties and colour of products *(Farnworth et al., 2001; Lee and Coates, 2003)*. For example, carotenoids are isomerised from 5, 6-epoxides to less intensely coloured 5, 8-epoxides, and anthocyanins are degraded to brown pigments *(Fellow, 2000)*.

Freezing treatment affects carotenoids in the same way as it affects vitamin C (oxidization with existence of air and light).

2.2.5. pH Value

No significant change in pH of test samples during thermal treatment has been reported *(Friedman et al., 1990)*.Also in filtration and mechanical methods there is no change in the pH value.

2.3. Electric Field Impact on a Charged Liquid

Applying an electric field to a charged liquid or semi-liquid material using two electrodes results in several processes starting at different components: charged liquid ions, charged liquid particles and the liquid itself. These processes cause a movement of these different components towards one of the electrode according to the process and the ion number and types.

These processes are described as Electrokinetic phenomena.

Previous processes and their application have been a very rich interesting area for many researchers to understand the impact of applying electric field to a liquid and control this impact to enhance the dewatering process by combining these electrical phenomena with the water motion.

These phenomena are called electrokinesis in general but this electrokinesis has several types according to the relative movement of particles, surfaces and fluids *(Hunter, 1993; Aziz et al., 2006)*.

For example "*the movement of charged particles in an aqueous medium to an electrode of opposite polarity is known as electrophoresis (Weber and Stahl, 2002; Lockhart, 1981)*" which has been used in various applications mainly to determine the particles charge and to separate enzymes and proteins in general.

A second type of movement, which known as electro-osmosis is defined as "*the displacement of liquid relative to the medium under the influence of an electric field" (Weber and Stahl, 2002; Lockhart, 1981)*. These two types of processes are used to enhance solid–liquid separation.

A third type of movements which is produced by the same mechanism as electrophoresis but at the liquid level and works in the opposite direction to the charged ions that moves to the opposite polarity electrode under electrophoresis.

The benefits of electrokinetic type applications are not new as they have been reported in the mid-1970s by *Yukawa et al. (1976& 1978)*. Other researchers continued worked on the electro-osmotic and electrokinetic application in the area of dewatering such as removing heavy metal contaminants from soil by using an electric field in groundwater *(Grundl and Michalski, 1996; Shapiro and Probstein, 1993)*, enhancing the ultra-filtration process *(Zumbusch et al., 1998)*, and minimising the radioactive nucleotides level in radioactive materials *(Turner and Dell, 1984a& b)*.

The Operational parameters of these processes have been investigated such as the use of voltage and constant current conditions *(Yukawa et al.,1976& 1978; Yoshida et al., 1980)* as well as salt concentration, conductivity, ionic strength, suspension types, electrode material, intermittent current application and the effect of voltage *(Aziz et al., 2006)*.

Despite the fair amount of research on the potential use of an electrical field to improve dewatering of suspensions, the operational examples of their industrial use appear to be remarkably few and most of the research has been done in the soil, waste and mining areas.

In the food processing area, electrokinetic applications have been used mainly for analytical procedures such as protein and enzyme separation; but their use at an industrial scale application has been minimal and the amount of research which has investigated the potential benefits and application of the technology on foodstuffs has been limited. There is a small amount of research investigating the technology, but mainly in the food waste area where electrokinetics is used to improve the dewatering process to reduce the volume of the waste by removing the water component *(Rathore and Guttman, 2003)*.

2.3.1. Electro-osmosis, Electrophoresis and Electrokinetics

Electro-osmosis and electrophoresis are the main applications of the liquids and suspension transport processes used in a wide range of applications especially the bio-analytical ones such as isoelectric focusing and capillary electrophoresis, and capillary electro-chromatography *(Oddy and Santiago, 2004)*.

Both electro-osmosis and electrophoresis are caused by applying a low-intensity direct current between two electrodes on each side through a porous medium. Applying this current causes electro-osmosis of the aqueous phase as a migration of ions and electrophoresis of charged particles in the colloidal system to one of the electrodes according to the charge of ions and particles *(Oddy and Santiago, 2004)*.

Scientifically, electro-osmosis is defined as "*the movement of the capillary water under the electric field due to the existence of the electrical double layer at the interface of water and the solid surface*", while electrophoresis is "*the migration of charged particles or ions in a colloidal system towards the counter charged electrode*"*(Aziz et al., 2006)*.

The direction of movement of ions under the electrophoresis process is decided by the nature of the charges on the surface of solid particles towards the electrode of opposite polarity; a positively charged particle will travel towards the cathode while the negatively charged particles will travel towards the anode *(Aziz et al., 2006)*.

Depending on the charge of the ions liquids moves in the opposite direction to the ions; This means that the movement of the water is divided between the electrodes in proportion to the ionic proportions (i.e. if the majority of liquid charged ions have a negative charge, the main ion movement will be towards the anode via electrophoresis). The liquid then migrates toward the opposite polarity electrode which is the cathode *(Aziz et al., 2006)*; this movement of water in the opposite direction to ion particles is a result of the electrokinetic phenomena.

These evolving technologies showed an effective impact not only in dewatering applications and soluble ions removal, but also in the removal of insoluble organics in porous media *(Aziz et al., 2006)*. Research about the electrokinetic application have focused on the sludge dewatering, soil remediation from heavy metal, and organic contamination *(Gladman et al., 2005)* and waste dewatering; but research about electrokinetics use in the food area are still too few and insufficient to cover this application's benefits and parameters.

2.3.2. *Electrokinetics History*

As mentioned before electrokinetics (the movement of water profile in the opposite way to the direction of travel of ions under an electrical field application) were investigated before in several studies mainly in dewatering sludge, soil and mining.

Raats et al. (2002) reported that the solids content in drinking water sludge increased from 17% to 24% by combining electrokinetic dewatering with a belt press in a gravity-driven thickening belt combined with an additional energy consumption of 60 kWh/t of sludge. A high water content reduction from 87.8% to 62.6% was also achieved by *Yuan and Weng (2003); Yang et al. (2005)* in municipal sludge over 41 h at a potential gradient of 5.0V/cm using a 6 cm long electrokinetic dewatering cell.

Besides the sludge dewatering area, studies have focused on electrokinetic remediation of soil, implanting two electrodes in soils vertically *(Ho et al., 1997)* or horizontally *(Ho et al., 1999)* achieving removal of more than 90% of the metals; this metal removal was also observed in an improved electrokinetic process using a cation selective membrane in front of the cathode to prevent the precipitation of metals in the vicinity of cathode *(Li and Neretnieks, 1998)*. *Cundy and Hopkinson (2005); Yang et al. (2005)* investigated a potential application of electrokinetics using a ferric iron remediation and stabilization technique to decontaminate and consolidate soil.

Studying electrokinetic applications has not been limited in soil dewatering and mineral removal areas, research has also focused on electrokinetic removal of neutral soluble or insoluble organic compounds from soil. Greater than 90% removal of hydrophobic polycyclic aromatic hydrocarbons (PAHs) by electrokinetics has been reported by *Maini et al. (2000)* in bench- and pilot-scale experiments, while in another study, *Ho et al. (1999)* reported 98% removal efficiency of *p*-nitro-phenol in one pilot unit.

However, too few of investigations were done to study the use and impact of an electrokinetic application on food materials, the only recent study was done by *Ng et al. (2011)* and on food waste to investigated the impact of electrokinetics on dewatering food waste (Brewer’s spent grain, Orange peel, Melon peel, Mango peel and Cauliflower trimmings) by applying two voltages (15V and 30V) and compared it to a control (pressure impact with no voltage). The power consumption of the application under (0V, 15V and 30V) was compared with the power consumption needed to dewater the same volume of water using conventional thermal methods.

The results indicated that a electrokinetic process combined with mechanical dewatering can reduce the percentage of moisture from 77% to 68% for orange peel, from 78% to 71% for brewer's spent grain, from 80% to 73% for mango peel, from 91% to 74% for melon peel and from 92% to 80% for cauliflower trimmings. The total moisture reduction showed a correlation with electrical conductivity ($R^2 = 0.89$). The energy consumption of every sample was evaluated and was found to be up to sixty times more economical compared to thermal processing *(Sing et al., 2011)*.

None of the previous studies discussed the principles of applying electrokinetics on different types of food materials or the impact of the factors which control this application. In addition, the impact of this application on the food quality compound is still not discussed.

2.3.3. Electrokinetics Theory

In addition to the electrokinetic impact of migrating water in the opposite direction of the ions under electrophoresis, more phenomena happen when an electrical current is applied across electrodes in an aqueous solution to maintain charge equilibrium, the main electrolysis chemical reactions at the anode are:

$M_A \rightarrow M_A{}^{n+}{}_{(aq)} + ne^-$ *(Equation 1)*

$6H_2O \rightarrow O_{2\,(g)} + 4H_3O^+{}_{(aq)} + 4e^-$ *(Equation 2)*

Where: MA is the anode metal.

These two reactions occur together during the dewatering process under an electrical current at the anode, and they have specific impacts on the electrode, suspension composition, ions, pH and both ions and water movement; in addition to these changes each reaction produces specific products. For example, the reaction described by *Equation 1*, which occurs at the anode causes the anode metal to dissolve electrochemically which reduces the electrode capacity to an extent which depends on the reaction intensity and the electrode type and material; this decrease of electrode capacity is not the only disadvantage of the dissolving effect as the dissolved metal can also contaminate the filtration cake and filters. Reaction 2 described by *Equation 2* on the other hand, generates hydronium ions, H_3O^+, which results in a reduction in the pH of the suspension in the vicinity of the anode.

Whilst reaction at the anode dissolves the anode metal and produce acids, metal ions could deposit on the cathode and alkaline liquid may be produced through the following reactions:

$2H_2O + 2e^- \rightarrow 2OH^-_{(aq)} + H_{2(g)}$ *(Equation 3)*

$2H_3O^+_{(aq)} + 2e^- \rightarrow 2H_2O + H_{2(g)}$ *(Equation 4)*

$M_C{}^{n+}_{(aq)} + ne^- \rightarrow M_C$ *(Equation 5)*

Where MC is metal that deposits on the cathode.

Equation 5 describes the deposition of the electrode metal ions on the cathode travelling from the anode; while *Equations 2 & 4* show the total gas generation (O2 at the anode and H2 at the cathode); these gases could provide an extra pressure to the system. These in-between-layer- trapped gases have been observed to create gaps between the suspension layers and reduce the contact between the electrode and the suspension surfaces which in turn reduces the efficiency of the applied field to the suspension *(Weber and Stahl, 2002)*.

Describing the electrode metal dissolving and depositing as a ‘disadvantage’ (contaminating and electrode capacity reduction factor) is not totally fair as this ion migration may be used as an efficient application to remove heavy metal contamination. This application depends on the fact that the deposited metal on the cathode shown in *Equation 5* not only comes from the dissolved anode metal *Equation 1* but also from the heavy metal contaminants which are present in the sample (soil, waste, sludge etc.). In this case applying an electric field will cause the movement of these heavy metal contaminants to the cathode creating the cathode deposit which can be collected at the end of the process for disposal *(Grundl and Michalski, 1996; Shapiro and Probstein, 1993)*.

The sum of all these electrochemical reactions in electrokinetics demonstrates to the relationship between the water and charged particles movement and electrical potential. Effects are directly related to the application of a voltage via electrodes include; heating, electrolysis of water, and other electrochemical processes.

The flow of water, under a direct current (DC) voltage, and according to the sample chemical properties from one electrode to another is shown in *Figure 2.3*. Water is removed from the electrode, where the movement direction is toward this electrode, which in turn causes some changes in the material such as a volume reduction.

The following are some of the changes during electrokinetic treatment *(Jones et al., 2008)*:

- ✓ Movement of positive and negative ions.
- ✓ Movement of particles in the water (e.g. bacteria removal).

- ✓ Oxygen production at the anode.
- ✓ Hydrogen production at the cathode (and sometimes ammonia evolution at the cathode according to the suspension composition).
- ✓ Heat production.
- ✓ Changes in pH.

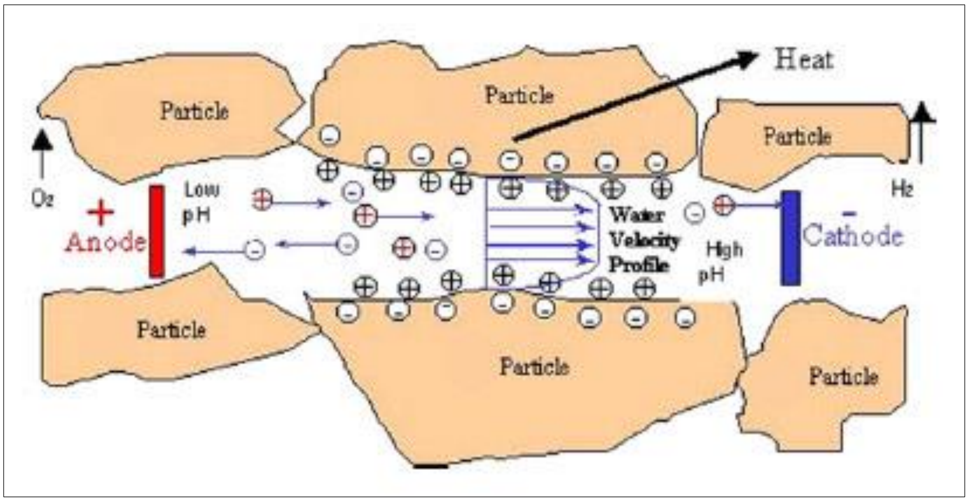


Figure 2.3. Conceptual representation of electro-osmosis[1]*-Edited from Jones et al. (2010)*

2.4. Factors Affecting Electrokinetic Dewatering

All applications of an electric field, mentioned above, can be applied either on their own or by combining them with mechanical compression.

There are different factors which influence or limit these functions. The flow rate of water through the materials is one of these factors with which electrokinetics is used to improve or treat. The difference in pressure (in case of combining with a mechanical process), identified as the hydraulic head, is also one of the most important factors affecting water flow. The rate of water flow is directly related to particles size and is determined by the permeability of the material. Practically it is very difficult to move water through and out of many materials. Many engineers from different backgrounds are concerned with controlling water content and water movement in order to influence different characteristics such as volume, strength and particulate content of the water phase such as bacteria *(Jones et al., 2010)*.

Each of these factors can affect the process negatively or positively; the combination of these factors has important effects as well. To control this application the following factors should be controlled:

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[1] Water profile direction *figure2.3* presents a specific situation, where water moves towards the cathode, which is not a general situation.

2.4.1. Residence Time

Keeping the sample in the cell for a long time gives a better result in terms of reducing the moisture content of the sample; but besides the economic loss which results from the slow production, the long duration could affect the chemical properties of the product negatively particularly the pH and the oxidation of compounds within the parent material.

2.4.2. Applied Voltage

High applied voltage leads to faster dewatering but also has many disadvantages; high voltage can cause very rapid drying out of the sample around the anode and results in a high pH rise of the effluent discharge at the cathode *(Veal et al., 2000; Lockhart, 1983)*. Electrokinetic treatment is much more energy efficient at lower voltage gradients. Starting at a low voltage gradient then gradually increasing it can make conditions further optimized as dewatering proceeds since high voltage result in more negative impact and power consumption *(Lockhart, 1983)*.

2.4.3. Applied Pressure

Higher applied pressures leads to faster drying, but the need for a higher pressure depends on different factors such as the initial moisture content of the sample, the applied voltage and the types of the filters. The lower the moisture content the greater the need for increasing the pressure. Increasing the voltage generates more gas which may form gas layers within the sample, for example, between sample and electrode. This decreases the conductivity; in this case the best solution is to increase the pressure to compress the layers and help gas to escape. Regarding filter types, the smaller the size of the filter pores the slower the process, in this case increasing pressure may help to speed up the process.

2.4.4. Filter Types

Applying EKEF to food materials needs the use of several types of filters to save the quality components (nutrients, colour, flavour and aroma). The proportion of the lost compounds depends on the filter pore size.

Filter type and material must be chosen according to the cell design, applied pressure and sample properties. The following are the filtration methods and their requirement:

- **Microfiltration:** *retains solid particles which are > 0.1 µm in size.*

This type of filtration retains solid particles which are > 0.1 µm in size. Here the particles of the liquid or the soluble fraction are affected by the filtrate flow which carries them onto the membrane by convection, and the hydrodynamic lift force which transport them away from the membrane due to the parallel shear flow.

Most of the food juice particles are smaller than 0.1 µm which makes this type of filtration inadequate to retain all needed compounds *(Ripperger et al., 2009)* in case of using one filtration step as most of these compounds and particles will exit with the discharged filtrate. However, in case of using second step as RO, microfiltration filters is enough to do the pre-treatment filtration.

- **Ultra-filtration:** *retains colloidal particles<0.1 µm. (The cut-off size= 1000- 100,000 g/mol).*

  Here the process is different, the separation is according to the size; the smaller the particles are the easier to pass through the filter to go with the filtrate while the larger particles are collected on the membrane. The cut-off size of an ultra-filtration is "*usually defined as the molar mass (in g/mol) of a test suspension.* Usually in practical application the cut-off size depends on other factors in addition to the pore size of the membrane such as the "gel" layer on the membrane consisting of retained colloids *(Ripperger et al., 2009)*.

- **Nano-filtration:** *The cut-off size= 200-1000 g/mol.*

  It is a relatively new technique; this method is a combination of features of ultra-filtration and reverse osmosis with a high selectivity. It is called Nano-filtration because of its approximate cut-off size of some nanometres or more approximate molar masses of 200 – 1000 g/mol.

  To achieved this special Nano-filtration membranes are required which still have pores of a defined size, but their retention depends on the electrostatic charge of the molecules to be separated (bivalent anions are typically retained).

- **Reverse osmosis:** *retains molecules or ions, pores size 0.0001µ. It's used for juice but pulp should be removed before (1-5 µ).*

  Reverse osmosis use selective membranes without pores to retain molecules or ions. The separation in these membranes depends on several factors such as the size of the molecules, their solubility in the membrane material and the applied pressure. Certain molecules, which are soluble in the membrane material, permeate through the membrane. Other molecules are not (or less) soluble and are retained (or concentrated) on the upstream side of the membrane. Reverse osmosis has a particular feature which is the need for high pressure to overcome the osmotic pressure of the retained molecules. This type of membrane is used in several applications such as desalination of seawater which is the most important field to use these membranes. In the food industry it is applied to concentrate juices at low temperatures *(Ripperger et al., 2009).*

2.4.5. *Sample Thickness (the Distance Between the Electrodes)*

Sample volume determines the distance between the two electrodes which called the sample thickness, different distances result in different current intensity which in turn affects the electrokinetic process.

2.4.6. *Moisture Content of the Sample*

The initial moisture content of the sample could play both a positive and negative role in the dewatering process depending on the conductivity and the composition of the dry matter present in the sample.

2.4.7. *Conductivity and Material of the Electrodes*

Different types and materials of electrodes will give different results. The materials of the electrodes differ according the use of the final product and the applied factors such as the chemical composition of the sample and the applied voltage. Usually the electrodes used are metal electrodes or/and carbon-coated metal electrodes. Awareness of the chemical interactions between the electrodes and the material under the different conditions such as the pH and the temperature is important. For example, copper is a very commonly used metal for electrodes but it dissolves very quickly under an electrical field which then contaminates the sample, this must be taken into account when dealing with food materials as this contamination with copper ions may reach high concentrations which could exceed the toxin allowance (for example: 0.1-0.4 mg/Kg for oils *(FAO/WHO, 2011)* and 2mg/l for drinking water *(EU Commission Council Directive, 1998)*; however using copper electrodes for soil application could be very efficient because of the cheap cost of copper electrodes compared with other electrodes such as titanium or platinum as well as its high conductivity.

2.4.8. Composition of the Tested Food Sample

In addition to the two types of water mentioned in *Section 2.2.1*, the composition of the food affects the water binding capacity of the sample which also determines the dewatering efficiency. For example, fibre is well known for its high water holding and binding capacity. Fibre can directly bind water and material with a high fibre content should show a higher resistance towards dewatering. Besides fibre, protein also can increase the water holding capacity of the sample. The water held within protein structure is divided into two types: a) that part which is no longer available as a solvent and bound to the molecule; and b) the remaining water, with a corresponding co-matrix (polysaccharide, fat) or trapped in the protein matrix.

2.4.9. Conductivity of the Food Sample

The electrical conductivity of foods is a relatively recent interest for researchers. This interest started to develop in the late 1980s. Starting applying electrical fields to food increased the interest in food conductivity such as using pulsed electrical field in pasteurizing foods and the use of Ohmic heating.

Samples with higher conductivity allow more ions to migrate towards the oppositely charged electrode which transfers momentum to the surrounding pore fluid molecules. This ion migration causes and electro-osmotic flow which contributes to the success of the electrokinetic dewatering process. This suggested that the conductivity measurement might be a good indicator to evaluate how well the electrokinetic method can dewater food *(Zhang, 2009)*.

2.4.10. Sample pH and Isoelectric Point

The isoelectric point (*pI, pH(I), IEP*), is the pH at which a particle is electrically neutral and carries no net electrical charge. Naturally, particle surfaces may be charged to form a double layer (H^+/OH^-); the net surface charge is affected by the pH of the liquid in which the particle is submerged.

At this pH, the overall charge on the particle is 0, but this does not mean that the particle contains no charged groups; it means that the number of the negatively charged groups is equal to the number of positively charged groups on the surface of the particle. This pH value is very important in electrical food applications especially for protein applications as proteins precipitate at the isoelectric point because of the lack of a surface charge. The pH value of the isoelectric point differs for each particle; it depends on the ratio of the negative and positive ions carried by the particle. There are many food applications that use the isoelectric point concept, for instance, in cottage cheese production; lactic acid is added to milk to bring the pH to the isoelectric point of the caseins (major milk protein). Caseins will then precipitate from the rest of the milk to give cheese *(Vaclavik and Christian, 2013)*.

2.5. The Drawbacks of the Electrokinetic Process

Just like all other processes, electrokinetics has some drawbacks which impact the process efficiency; these drawbacks result from the potential chemical reactions occurring inside the electrokinetic cell during the process or from the cell components. The negative impacts of the drawbacks could affect the product properties or the power consumption. The following are the most important drawbacks of the electrokinetic process:

2.5.1. Corrosion of the Electrodes

Different studies reported a common problem during different electrokinetic applications which results in high corrosion rates at the anode. For example, corrosion rates of 2.5g/A/day *(Bjerrum et al., 1967)*, 26g/A/day *(Sprute and Kelsh, 1982)*, and 1.1kg/dry-tonne of product *(Lockhart and Stickland, 1984)* have been reported. Significant voltage losses at the electrodes are also reported to be as much as 25%-50% by *Bjerrum et al. (1967)*. According to *Lockhart (1983)*, the energy, required to achieve a particular solids content, is affected slightly by the type of metal. Using novel metals such as Platinum and Titanium for electrodes decreases this corrosion significantly, however, the problem still exists, and the issue of whether any proportion of corrosion will be acceptable in food industry is critical for the future development of this technology.

Samaranayake et al. (2003b) carried out an investigation to compare different recommended materials to select the most suitable one for an electrode material for use in a pulsed electric field (PEF) chamber used for foodstuffs. The comparison considered electrode corrosion, electrode surface morphologies and toxicity of migrated element amounts. Several materials were tested as electrodes in this study; Titanium (Grade 2), platinised-titanium, stainless steel 316 and boron carbide.

In general titanium is known as a good corrosion-resistant material and is often used where the corrosion resistance of stainless steel is not good enough *(Peters and Leyens, 2003).* Sometimes when the corrosion resistance is the main requirement instead of strength then it is recommended to use commercially pure titanium or low-alloy titanium grades *(Samaranayake et al., 2003b)*. Platinum is well used for electrical contacts or corrosion-resistant apparatus since it is a noble material and is thus not oxidised in air at any temperature. However pure platinum is expensive which leads to the use of platinised titanium instead especially when electrochemical processes are involved *(Kamachi et al., 2000).* Grade 316 stainless steel has a better corrosion resistance than other stainless steel products *(Samaranayake et al., 2003b)*. Therefore it is widely used in food processing, transportation and architecture. Boron carbide is a refractory and non-metallic compound which makes it corrosion resistant compared with metallic alloys *(Rigaud, 2000).*

To analyse the corrosion of these electrodes the study depended on analysing the concentrations of Pt (from the platinised titanium electrodes), Ti (from the titanium electrodes), Fe (from the stainless steel electrode), and B (from the boron carbide electrodes) as migration of electrode materials into the media. Scanning electron microscopy was used to examine the effect of corrosion on the electrode surfaces.

Samaranayake et al. (2003b) concluded that the most corrosion-resistant material for use as the electrode material was titanium which was not visibly changed much on the edge while boron carbide electrodes were worn out after 12 hours of process. The amount of migrated Ti was significantly less than migrated Pt, Fe, and B in the media. Platinised titanium electrodes were not as good as titanium. However, this material showed better corrosion resistance than the stainless steel 316 and boron carbide electrodes. Boron carbide was the least corrosion-resistant material.

2.5.2. High Temperature Near to the Electrodes

During the treatment, the temperature of the material being dewatered increases especially near the electrodes because of the ion movement and the electric field, and this may affect the chemical properties of the products. When it is not possible to use mixing in the electrokinetic cell because of the design, the closest sample layers to the electrodes will be the most vulnerable to the effect of the high temperature.

2.5.3. Rise in pH

During Electrokinetic treatment pH value changes significantly due to the production of H^+ and OH^-, in the anode and cathode respectively, by the electrolysis of water *(Equations 2 & 3)*. This change affects the chemical interactions and the microbial activities especially during the storage period of non-pasteurised products, and also has a negative impact especially during juice concentration as this change of pH may reduce the product quality. Furthermore, lack of mixing in the electrokinetic cell will increase the variation of pH between the sample layers from the anode to the cathode *(Saeedi et al., 2009; Weber and Stahl, 2002)*.

2.5.4. Oxidation Due to the Released Oxygen

Electrokinetic treatment has been shown to result in oxygen gas generation at the anode, associated with the electrolysis of water *(Equation 2)*. The oxygen generated at the anode is entrapped by the water flowing into the sample which makes it difficult to escape from the sample. However, in some cases when pressure is applied, the entrapped air may escape from the sample. Oxygen production may lead to increased oxidation in some materials especially foodstuffs which contain a significant percentage of fatty acids. This production can potentially also increase the rate of aerobic microbiological activity *(Tamagnini et al., 2009)*.

2.5.5. Creation of Gas Gaps

Generated oxygen *(Equation 2)* and hydrogen *(Equations 3 & 4)* may create gas gaps within the sample layers, especially those with a dry matter of more than 30%; these gas gaps decrease the conductivity within the samples which in turn decreases the efficiency of the electrokinetic treatment and increases the power consumption. These gas gaps also increase the surface area of the sample/oxygen interface and so potentially increase the risk for oxidative damage to food components.

2.5.6. Power Consumption

The power consumption of the electrokinetic treatment depends on the comparison made. For example, electrokinetic treatment has higher power saving efficiency than thermal drying methods; it shows, on the other hand, higher power consumption compared with other normal gravitational or hydraulic filtration processes. However, electrokinetic treatment is much more energy efficient at lower voltage gradients *(Lockhart, 1983)*.

2.6. Possible Effects of Applying an Electric Field on Food

There are different uses of electric field in the food industry such as concentrating or preserving by inactivating microorganisms. One of the preserving methods that uses electric field application is the Pulsed Electric Field process (PEF) which is *"a non-thermal method of food preservation that uses short bursts of electricity for microbial inactivation and causes minimal or no detrimental effect on food quality attributes. PEF can be used for processing liquid and semi-liquid food products"* (Balasubramaniam et al., 2010).

This process is close to electrokinetics in terms of applying an electric field through foodstuff and between two electrodes. PEF uses a high voltage value for very short time (20-80 kV usually for a couple of microseconds); but this application is used to inactivate microbial growth not to dry foods. Despite the big difference between the applied voltage and the processing time between electrokinetics and PEF, the PEF impacts on food provide a good indication about the potential impact of electric field application.

Many researchers concerned with the possible impact of the PEF process on orange juice have investigated the effects on compounds, especially bioactive ones such as vitamin C, carotenoids, and flavanones, comparing PEF with some other processes especially thermal methods. *Sanchez-Moreno et al. (2005)* compared some non-thermal technologies as follows: High pressure (HP) (400MPa/40˚C/1min), pulsed electric fields (PEF) (35 kV.cm^{-1}/750s)) and some thermal ones (low pasteurisation temperature (LPT) (70˚C/30=s), high pasteurisation temperature (HPT) (90˚C/1min), HPT plus freezing (HPT+F) (-38˚C/15min), and freezing (F).

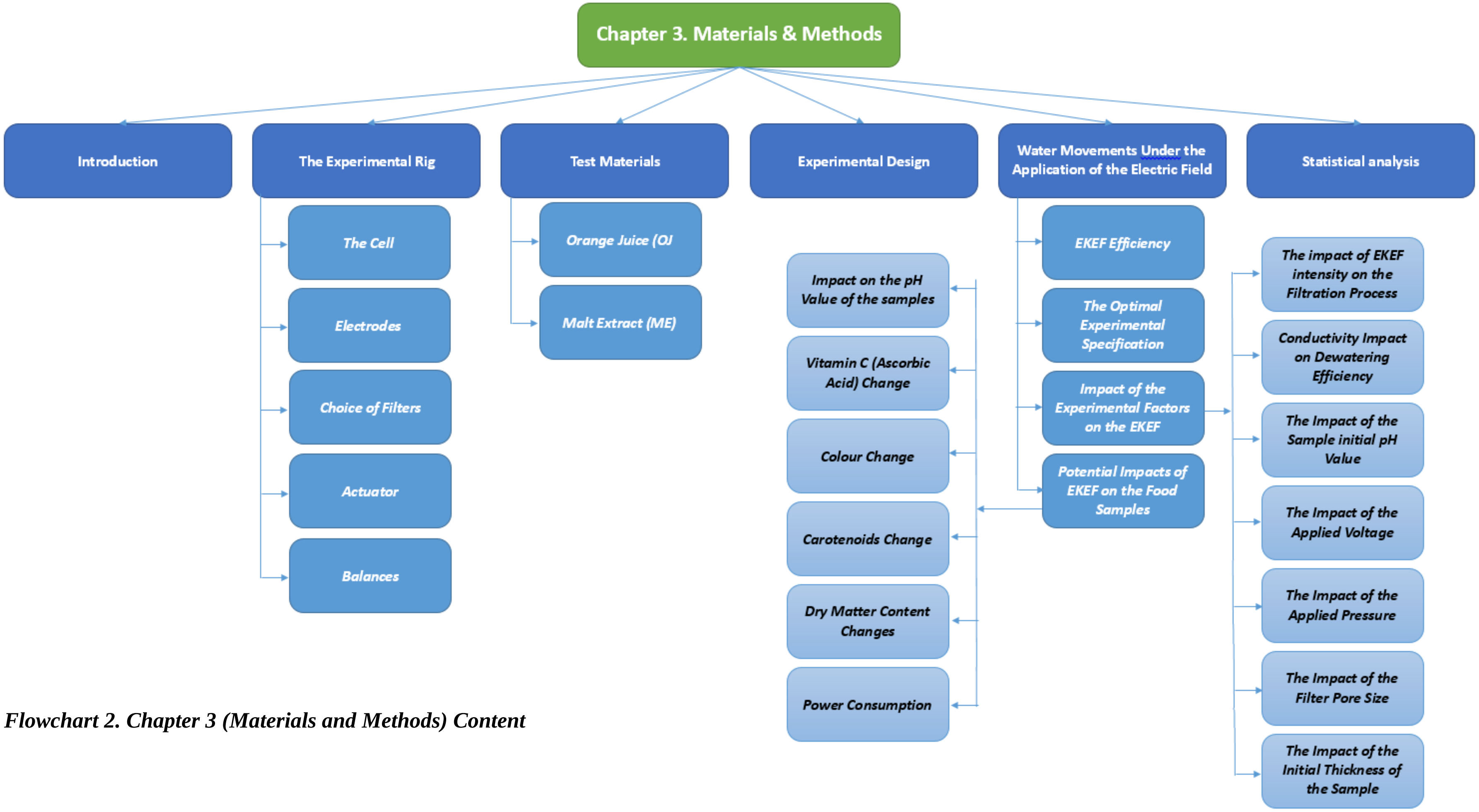
The comparison has been run between the effects of the previous methods on the properties of orange juice, considering fresh squeezed orange juice as a control, the results were as shown in *Table 2.3*.

Table 2.3. The impact of the non-thermal methods on the properties of orange juice, considering fresh squeezed orange juice as a control (Sanchez-Moreno et al., 2005)

Properties	**Changes**
Physical& Physicochemical Characterization	All of the treatments showed significantly higher Brix measurements than fresh squeezed orange juice.
Colour	Non-thermal processes showed significantly lower Chroma values, which represent colour intensity, than fresh squeezed orange juice
Vitamin C Content	A significant decrease in L-AA content (~7.79%) with all treatments including PEF (~6.98%) in comparison with fresh squeezed orange juice, except of LPT and F treatments did not exert any change. There were also decreases (~8.24%) in total vitamin C with all treatments except of HP and LPT did not exert any change. Even though the losses were <9%, treatments with the higher temperature tended to show the higher negative impact on the L-AA and L-DHAA contents. However, these losses in vitamin C are relatively low.

Carotenoids Content and Vitamin A Value	HP-Treated orange juice showed the higher vitamin A value and carotenoids content compared with other treated juices. In general, each carotenoid showed a significant increase in comparison with the control sample: R-carotene (33.76%), â-carotene (30.24%), R-cryptoxanthin (45.87%), α -cryptoxanthin (43.21%), zeaxanthin (44.52%), and lutein (75.43%). However, PEF-treated orange juice did not modify individual or total carotenoids content. Traditional thermal treatments did not show any significant impact on total carotenoids content or vitamin A value in comparison with freshly squeezed orange juice.

Chapter 3. Materials and Methods



Flowchart 2. Chapter 3 (Materials and Methods) Content

3.1. Introduction

Electrokinetics has already been used as a technique to remove moisture from slurry, slimes, sewage and soil (a process described as dewatering). Different moisture contents in these different substrates will often result in different water contents in the final products. In previous studies, different moisture contents have been tested; generally the dry matter content of samples was between 35-60 % *(Lamont-Black et al., 2007)*. Most of previous studies were done on non-food materials, the only about food materials was done by *Ng et al. (2011)* to investigate the power consumption of applying electrokinetics to dewater food waste.

This present study tested the effectiveness of the electrokinetically enhanced filtration (EKEF) process on food materials with higher water contents; the chosen materials were two types of liquid food materials with water content higher than 80%.

Furthermore, previous studies were concerned with enhancing flow rate in soil and waste materials where there was no need for consideration of the final product's sensory properties or its safety and suitability for human consumption. In this study one of the principle purposes of the experiments run was to investigate the EKEF impacts on food samples and their chemical and physical properties which affect the quality of the final products as a foodstuff; there are many parameters that change during the traditional dewatering methods (especially thermal treatment), for instance loss of vitamins (especially vitamin C and thiamine), colour, flavour, the oxidation of fatty substances, sugars and degradation in the Maillard reaction (sugars with amino acids) and the caramelisation of sugar because of the high temperature.

3.2.The Experimental Rig

Several filtration applications have been used to dry and concentrate food materials and to avoid the negative impact of thermal applications. In general these applications (including micro-filtration, ultra-filtration and reverse osmosis) showed very good results in terms of preserving food quality aspects and nutrients, but the main problem faced by manufacturers was the slowing down of filtration flow rate over time due to the caking of material on the filter and the relatively quick blockage of filters.

Traditional methods to concentrate orange juice, such as thermal processes, result in many problems including colour degradation, a loss of flavour, and a cooked taste. In addition many nutrients such as citric acid and vitamin C are damaged because of the high temperatures involved. Traditionally, these nutrients can be recovered through essence recovery, careful process control and blending to produce a good quality concentrate which is distinguishable from fresh juice, but still meets broad consumer acceptance needs.

Much research has been undertaken to overcome this problem using methods such as applying pressures or/and using pre-filtration steps in order to separate different-sized particles of material through different steps which increased the filter usage time.

For example, in 1985, FMC and DuPont began a joint research program to develop a membrane process. The aim was to develop membranes to concentrate orange juice to 58°Brix and retain the fresh juice flavour. The process, which was called *Fresh Note^{TM}*, was based on combining ultra-filtration, reverse osmosis, pasteurization and blending, and aimed to concentrate orange juice with very high quality.

Fresh Note^{TM} is used successfully to close the flavour gap between fresh and concentrated orange juice.

Pasteurisation should be applied on juice to ensure the commercial stability; but the high temperature of this process could affect other sensitive compounds negatively therefore these sensitive compound must be isolated during the pasteurisation step by separating them in advance.

This separation of key sensitive components could be done using ultra-filtration process, as shown in *Figure 3.1*.

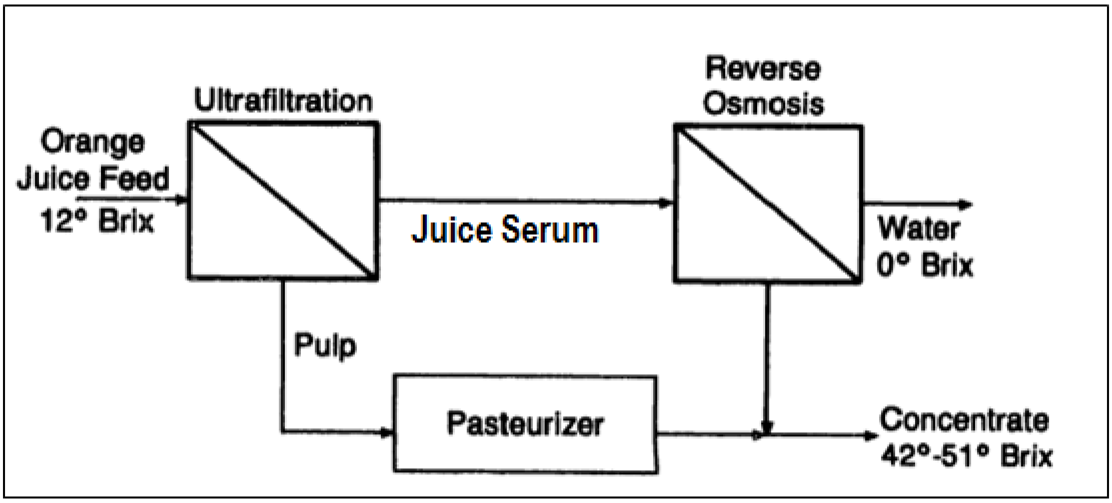


Figure 3.1. Fresh Note^{TM} System Diagram (U.S. Patent #4643902) (Cross, 1989)

Juice produced by this system scored higher in flavour and is better in quality, when the juice is reconstituted, than any thermally concentrated product, and is often indistinguishable from the fresh juice used to produce it *(Cross, 1989)*.

Despite of the good results of such systems, the filtration step still had some problems due to the juice bits and viscosity. The solid pectin content in orange juice creates a very viscous stream when concentrated. The high viscosity combined with the high osmotic pressure of concentrated sugars, results in low membrane permeate rates and low levels of concentration.

The aim of this study was to enhance the juice concentration process using filtration systems by speeding up the filtration flow rate with a combination of electric field work and pressure work.

Some test rigs and prototypes were considered for use to apply electrokinetics and evaluate its impacts on the sample; these rigs were mainly designed for use on soil in civil engineering and geology applications. However, because of the differences between food material (juice in particular) and soil in terms of physical and chemical aspects, a new test rig was designed specifically for the purposes of this research so that experimental parameters could be controlled in a better and more efficient way.

The experimental rig was developed by modifying the testing cell '*Rosli cell*' *(Figure 3.2)* mentioned in *Jones et al. (2005)*, which was used to test the effects of electrokinetics on dehydrating water from soil; these modification included many features and components, taking into account:

- Differences between the physical properties of soil and foodstuffs in terms of dry matter and viscosity. Specifically this required a better sealing system to avoid leakage and different mechanisms to hold the electrodes.

- Differences between the Chemical properties of soil and foodstuffs in terms of chemical composition, acidity and gas production. These differences led to the necessity to change the electrodes type.

- Differences between the required functions. The soil EKG cell was used to apply an electric field on soil while the foodstuffs cell required two processes, the application of an electric field on the foodstuff in addition to the filtration process. To achieve this function, two combinations of filters and meshes were added to the top and bottom outlets.

- Some other modifications to ease the rig assembly and usage.

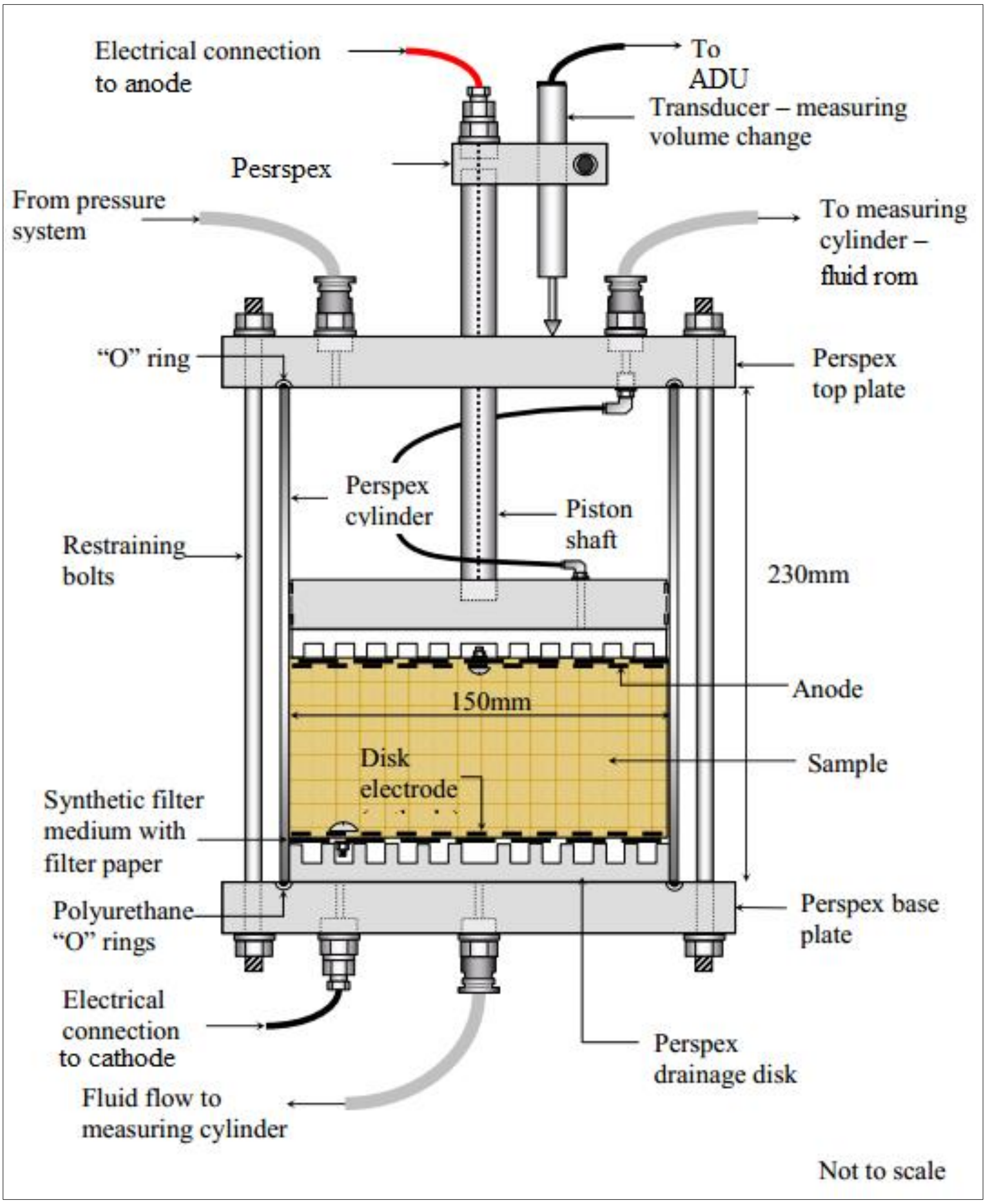
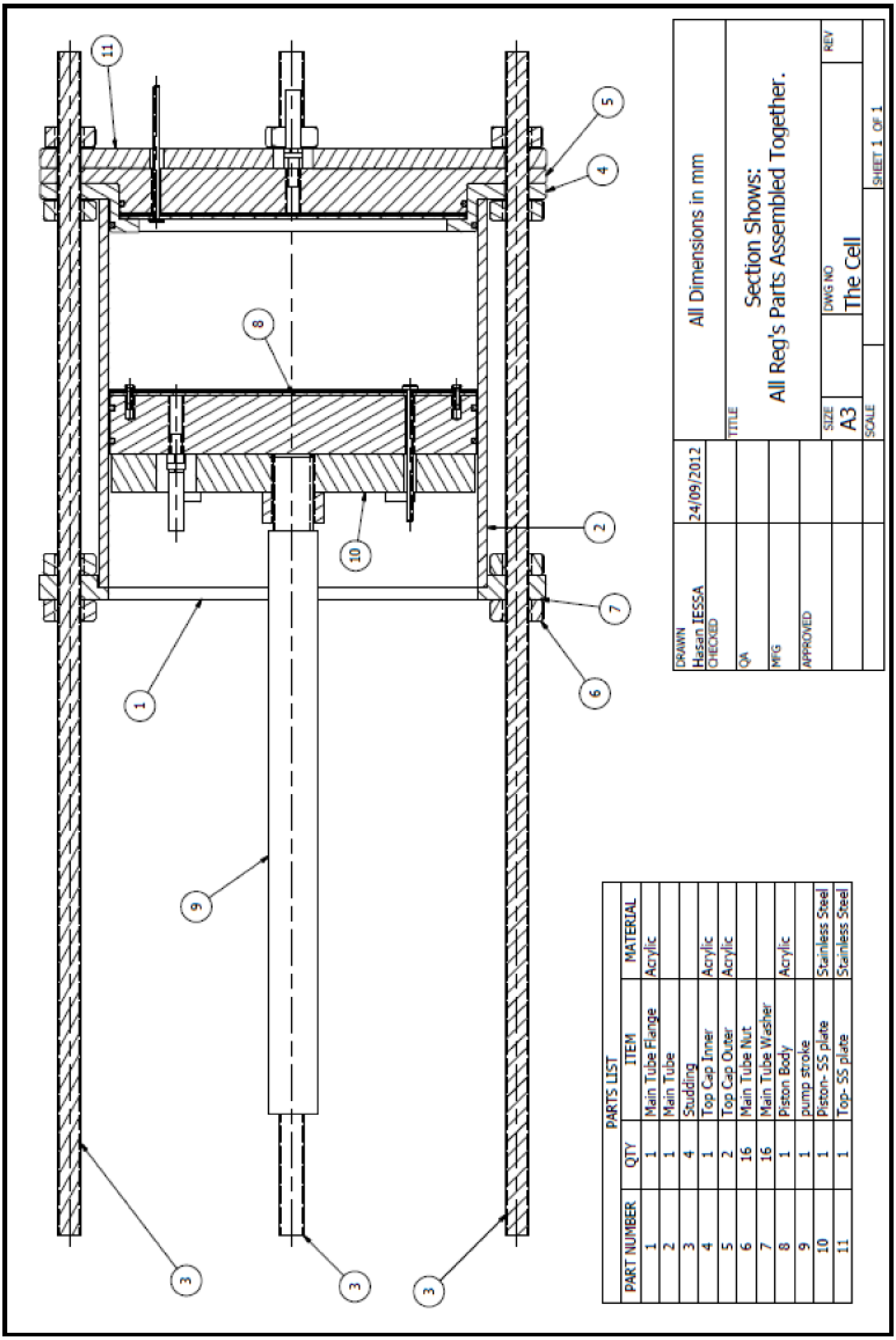


***Figure 3.2. Schematic of electro-osmotic cell** Jones et al. (2005)*

The cell shown in *Figure 3.3* was designed using Autodesk Inventor Professional 2012 (Autodesk® Inventor® 3D CAD software); and manufactured on commission by Dyer Engineering Limited (UK, County Durham, DH9 7RU). Other parts were then connected to the test cell to create the final functional rig shown in *Figure 3.4*.



PARTS LIST			
PART NUMBER	QTY	ITEM	MATERIAL
1	1	Main Tube Flange	Acrylic
2	1	Main Tube	
3	4	Studding	
4	1	Top Cap Inner	Acrylic
5	2	Top Cap Outer	Acrylic
6	16	Main Tube Nut	
7	16	Main Tube Washer	
8	1	Piston Body	Acrylic
9	1	pump stroke	
10	1	Piston- SS plate	Stainless Steel
11	1	Top- SS plate	Stainless Steel

Figure 3.3. The modified experimental Cell (the main part of the rig)

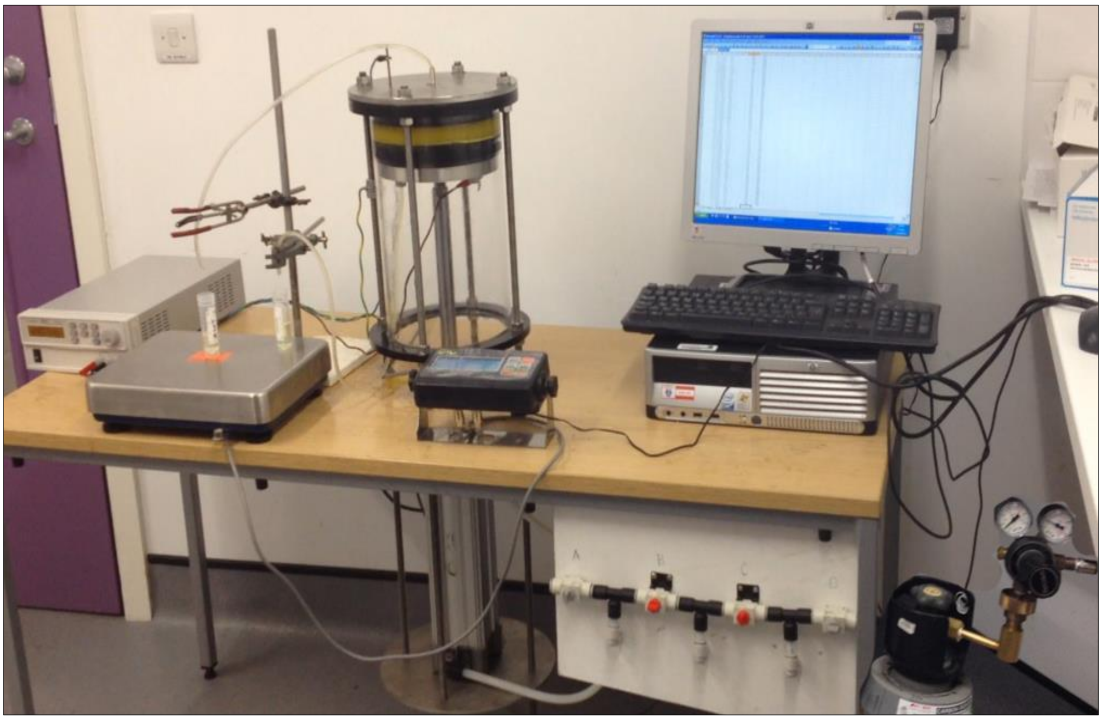


Figure 3.4. Final Rig (Total Parts)

The following are the main parts of the experimental rig (check *Appendices 7* for more details):

3.2.1. The Cell

The cell, which forms the main part of the rig, where the two processes of electrokinetics and filtration were applied on food material, is shown in *Figure 3.5*.

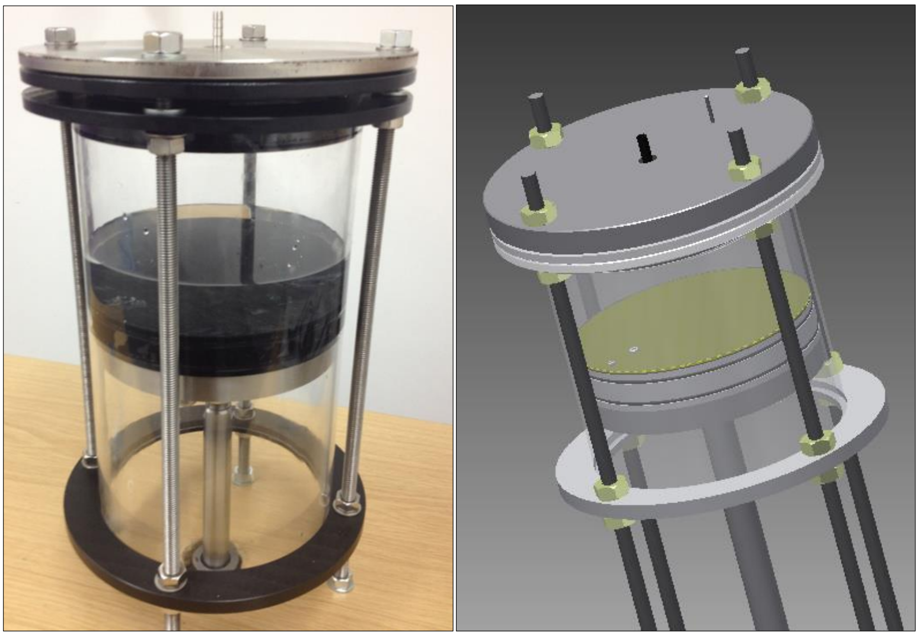


Figure 3.5. The Cell

This cell includes several parts:

- ***Main Plastic Tube:*** a transparent acrylic tube (OD=~200mm, 4mm thickness and 500mm length) *(Figure 3.6)*, this tube contained the sample with a capacity of up to 4 litres held between two horizontal electrodes forming the ends of the cell. Electrodes were fixed on the cell *top cap* at the top and on the *piston* at the bottom.

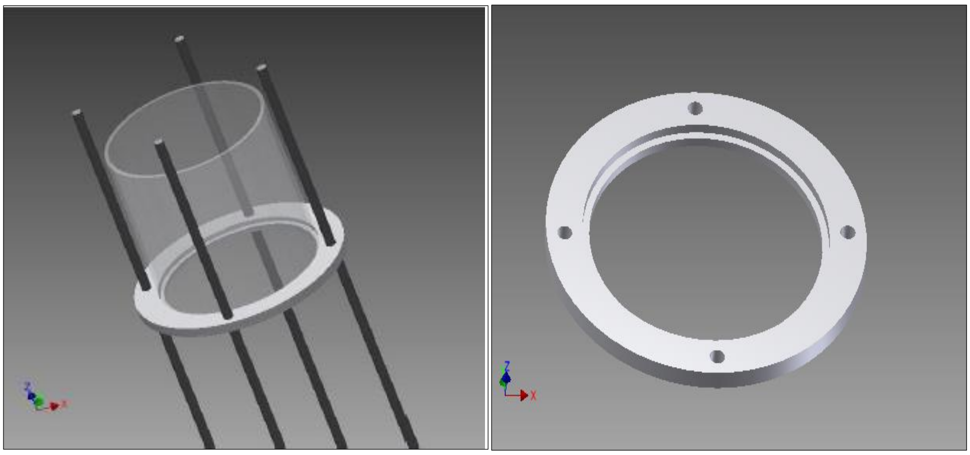


Figure 3.6. Main Plastic Tube (left) with the bottom flange (right)

- ***Top and Bottom Holding Parts:*** The acrylic tube was secured tightly between solid nylon ring mounts (*top cap* and *bottom flange as shown in Figure 3.6*. The top cap consisted of inner *(Figure 3.7)* and outer *(Figure 3.8)* parts between which the top electrode and filter combination was secured.

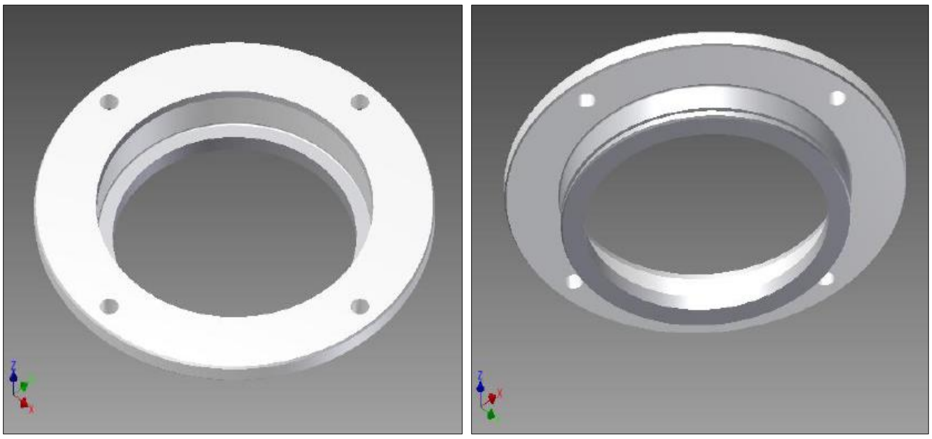


Figure 3.7. Top cap inner (Top and bottom view)

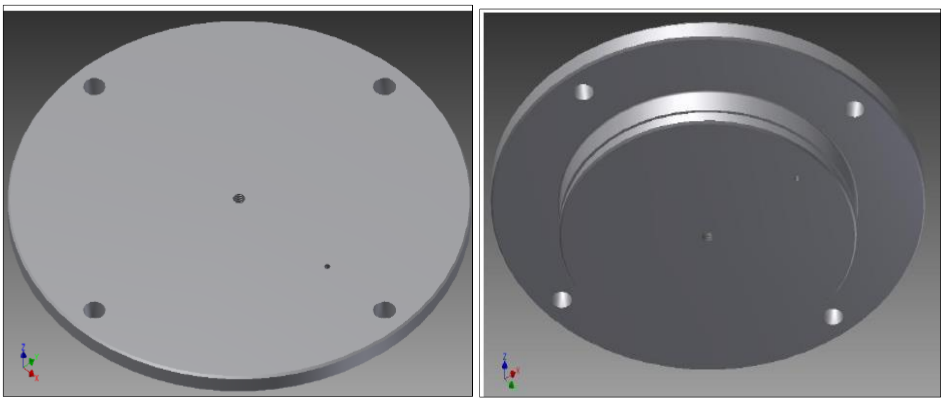


Figure 3.8. Top cap outer (Top and bottom view)

- ***Nylon Piston:*** which moves up and down vertically inside the acrylic tube. The piston held the bottom electrode and filter combination on the top while connecting to the actuator rod from the bottom as shown in *Figure 3.9*.

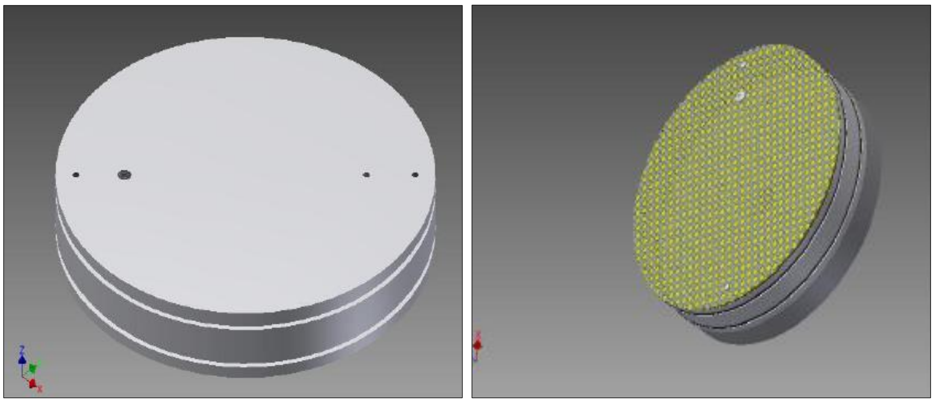


Figure 3.9. Nylon Piston; to the right the electrode on the top of the piston

- ***Stainless Steel Plates:*** Two stainless steel disc plates were added on the top and bottom of the cell to provide more support against the applied pressure *(Figure 3.10)*. Each of the top cap and the piston head had two holes, one connected to a plastic tube forming the anolyte and the catholyte outlets passing through the two steel disks and the second to allow a wire to connect the electrodes with the power supplier as shown in *Figure 3.11*.

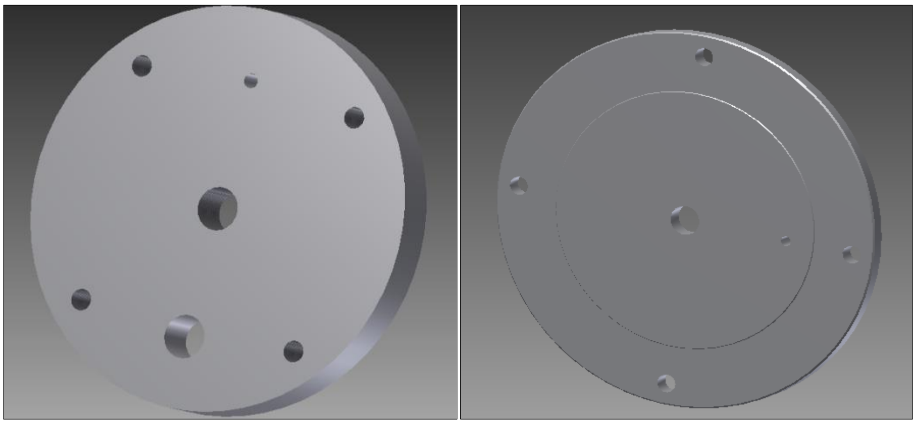


Figure 3.10. Top (right) and bottom (left) Stainless Steel disc Plates

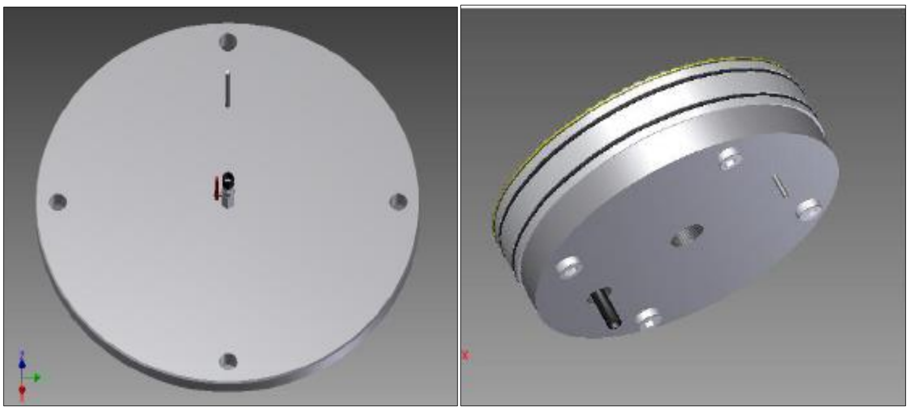


Figure 3.11. Outlet and wire through the piston (right) and through the top cap (left)

3.2.2. Electrodes

During the process of concentrating juice, the electrodes were in direct contact with the juice or other food materials. At the electrical interface, the electrode is influenced by two factors, the electric field formed between the layer of ions and the electrode, and the electrolyte charge held at the electrode.

Here we have two situations: with no applied voltage, the electronation reaction is equal to the de-electronation reaction and the net current across the interface is equal to zero *(Bockris and Reddy, 1970)*. On the other hand, when a voltage is applied to the electrode, the layer of ions increases at the interface and starts behave as a capacitor. Usually there is a certain threshold voltage; under this threshold the capacitive current does not involve any charge transfer or chemical reaction, but it results in removal or accumulation of electrical charges in the electrolyte near the interface and in the electrode. However, above this threshold voltage the electrochemical reactions could occur at the interface.

These reactions result in several chemical changes in food products and the electrodes; including the release of electrode material, electrode corrosion (at the anode) and deposit of particles on the electrode surface (at the cathode), as shown before in *Equations 1 & 5*.

These reactions lead to several problems; the released electrode material could cause contamination, colour changes or activation of some undesirable interactions in foodstuff *(Samaranayake et al., 2003b)* and they also affect the electrode by shortening its life time. There are several ways to reduce these reactions, such as selecting proper electrode materials and changing the chamber design *(Dunn and Pearlman, 1987; Jayaram and Lubicki, 1997)*.

Some researchers suggest using special materials for electrodes in food industrial applications especially the inert materials such as gold, Titanium, platinum, carbon and metal oxide *(Bushnell et al., 1996)*. Others recommend the use of electrically conductive polymers as electrode materials.

There are many factors to be taken into account before choosing the electrode material; some of these factors are related to the cell efficiency, others are related to the health terms.

The most important factors are the conductivity, the durability, the efficiency, the toxicity and the inactivity in food liquid.

In a previous study *(Iessa et al., 2011)*; copper was used as the electrode material and showed very good conductivity and efficiency. However, the copper electrodes reacted very actively with orange juice which produced different copper products with characteristic blue/green colours as shown in *Figures 3.12* and *3.13*.

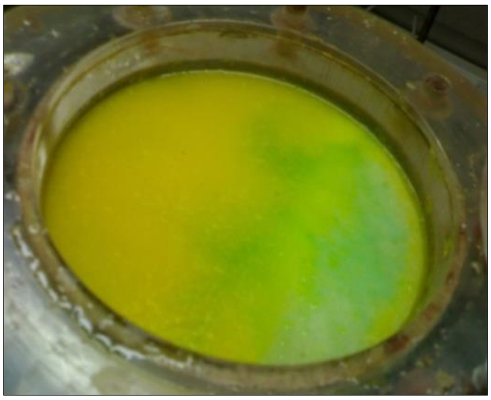


Figure 3.12. Blue-green copper salt in juice at the electrode interface

Figure 3.13. Orange juice before and after the electrokinetic treatment with copper electrodes

This contamination of the fruit juice results in inappropriate production conditions for foodstuffs, and this was due to the change in colour and contamination of the product with toxic metal salts.

Based on the literature review (*Section 2.5.1*), material cost, experimental aims and manufacturing requirements ***perforated Titanium discs*** *(Figure 3.14)* were used in the present study as anode and cathode materials, rather than using stainless steel. These discs were bought from Ti-Shop.com/ Division of William Gregor Ltd (UK, London, E14 8PX).

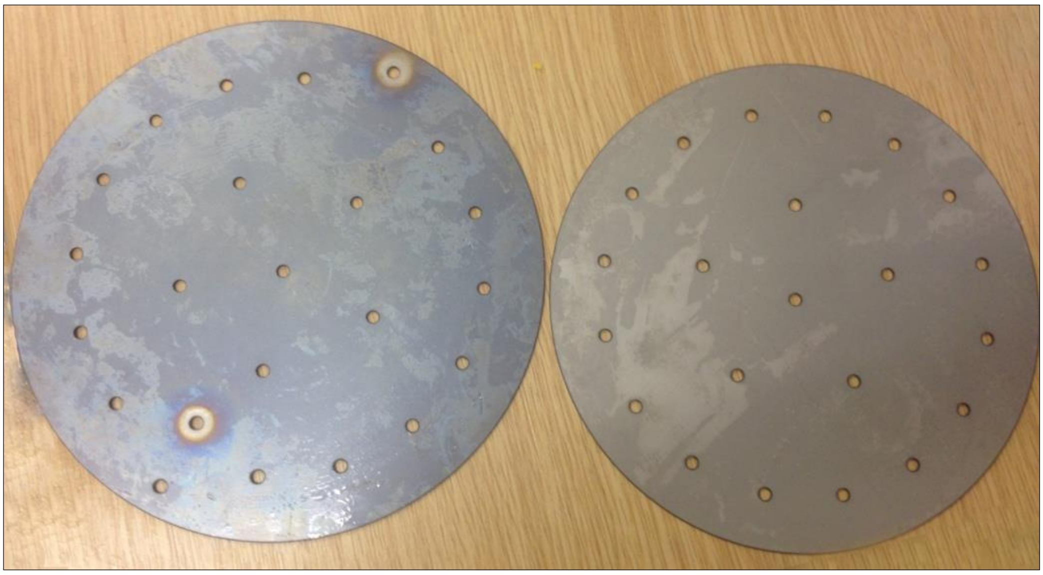


Figure 3.14. Perforated Titanium disc as electrode

3.2.3. Choice of Filters

Filter type and material must be chosen according to the cell design, applied pressure and sample properties.

In the present study the samples used as test foods were freshly squeezed orange juice and malt extract. The main aim of the first filtration step is to capture the relatively big bits of juice before going to the reverse osmosis step; the most suitable filter for this study are microfiltration and ultra-filtration filters. Cell walls can mechanically stand a maximum pressure up to 7bar; for this reason ultra-filtration processes could not be used effectively with this material. In addition, according to the cell design, filters had to be available in sheets to cut to the required size which were only available as microfiltration filters.

Taking these factors into consideration two filters were used in each experiment (either 0.2µ or 1µ). Whatman® filters were bought from Schleicher & Schuell via www.sigmaaldrich.com.

3.2.4. Actuator

The bottom piston was connected to a vertical gas actuator (ISO Cylinder 80 x 300 mm) shown in *Figure 3.15* which was connected to a CO_2 gas cylinder through a control panel as shown in *Figure 3.16*. The actuator provided the cell with the required pressure (up to 7bar); and moved the piston up and down using the designed control panel. Actuator was bought from SMC® (UK, Buckinghamshire, MK8 0AN) *via* uk.rs-online.com.

Figure 3.15. SO Cylinder 80 x 300 mm

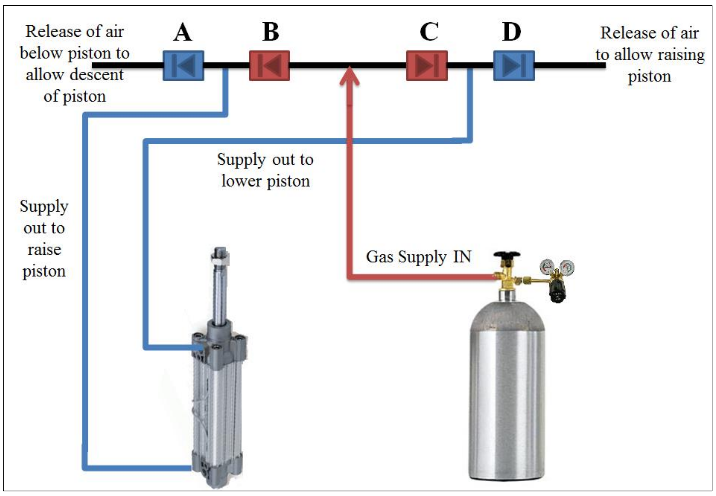


Figure 3.16. Gas circulation and control panel

3.2.5. Balances

A sensitive electronic bench scale platform (METTLER TOLEDO PBA655/PBA655X) supplied by Northern Balance LTD (The Greens Farm, Consett Rd, Gateshead, NE11 0AN) was connected to Microsoft office Excel 2003 through WinCT (RsCom / RsKey / RsWeight by A&D Company Ltd) to measure the discharged filtrate weight every 5 seconds.

3.2.6. Power Supplier

An electric field was provided by a controllable power supplier (U8002A DC Power Supply, Agilent, 30V, 5A). Power supplier was bought *from* Chauvin Arnoux Group® (UK, West Yorkshire, WF12 7TH) *via* uk.rs-online.com.

3.3. Test Materials

3.3.1. Orange Juice (OJ)

Samples were chosen from Marks and Spencer*'s* "Pure Freshly Squeezed Valencia Orange Juice/ with juicy bits, barcode number M0945257S" (UK, Newcastle upon Tyne, NE1 7AS) (16 orange fruits per 1 litre orange juice). *Table* 3.1 shows the composition of the OJ as reported on the ingredient list:

Table 3.1. The composition of OJ per 100ml (Marks and Spencer)

Water (g)	Protein (g)	Fat (g)	Carbohydrate (g)	Energy value (Kcal)	Fatty acids (g)	Total sugars (g)	Brix (°Bx)	Fibre (%)	Carotene (μg)	Vitamin C (mg)
86.1	0.8	0.2	10.7	50	0	9.3	10.5	5	28	48

OJ samples were tested directly without freezing or any other treatment. Samples were collected frequently during each experiment to be analysed later; some fresh samples were taken before treatment to be used as baseline controls (with no EKEF application), and then frequent samples were taken from every stage (at least one sample for each stage) to represent filtrate discharge of each stage. In addition at the end of each experiment further samples were taken from within the cell to represent the layers close to the cathode and the anode.

3.3.2. Malt Extract (ME)

ME samples were supplied by Muntons PLC (UK, Stowmarket, IP14 2AG) as dark dry malt then prepared in the laboratory to produce the ME with water content around 80%, using the following method suggested by Muntons PLC:

Dark malt grain was dispensed onto water at 52°C in ratio of 1 part grain: 4 parts water) and stirred at 9rpm at 52°C for 20min. Then the contents were heated to 65°C, the stirrer speed was increased to 18rpm and held at this condition for 20min; the temperature was then raised to 89°C, and the stirring speed further increased to 36 rpm for 20min. After this time the sample was allowed to cool down and was kept in the fridge at 4°C for 24 hours to analyse.

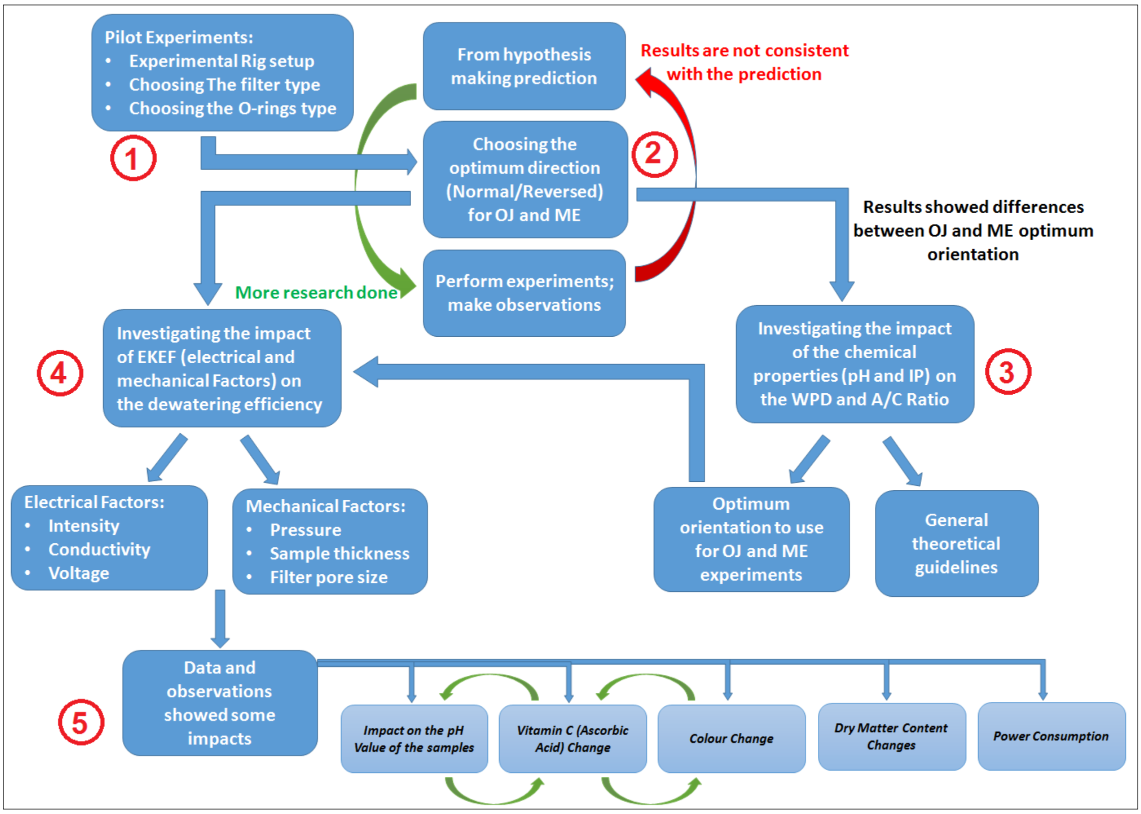
3.4. Experimental Design

Pilot experiments were done at the beginning to check the efficiency of the experimental rig in terms of holding the samples without leakage and to modify the O-rings size and the filter types for better piston movement and to slow down the filtrate to have enough time to investigate the impacts and changes *(Flowchart 3/ Step1)*.

The next step included running experiments to determine the charges of the top and bottom electrodes (anode or cathode) or what is called later the optimum orientation for each material (OJ and ME). These experiments were based on the theory of water profile direction under electrokinetics impact *(Section 2.3)* and the orientation was chosen according to the experiment and sample material (as the water profile direction WPD was different according to the food material due to the difference between each product initial chemical properties). When results were not consistent with the theory, more research were done and new experiments were run *(Flowchart 3/ Step2)*.

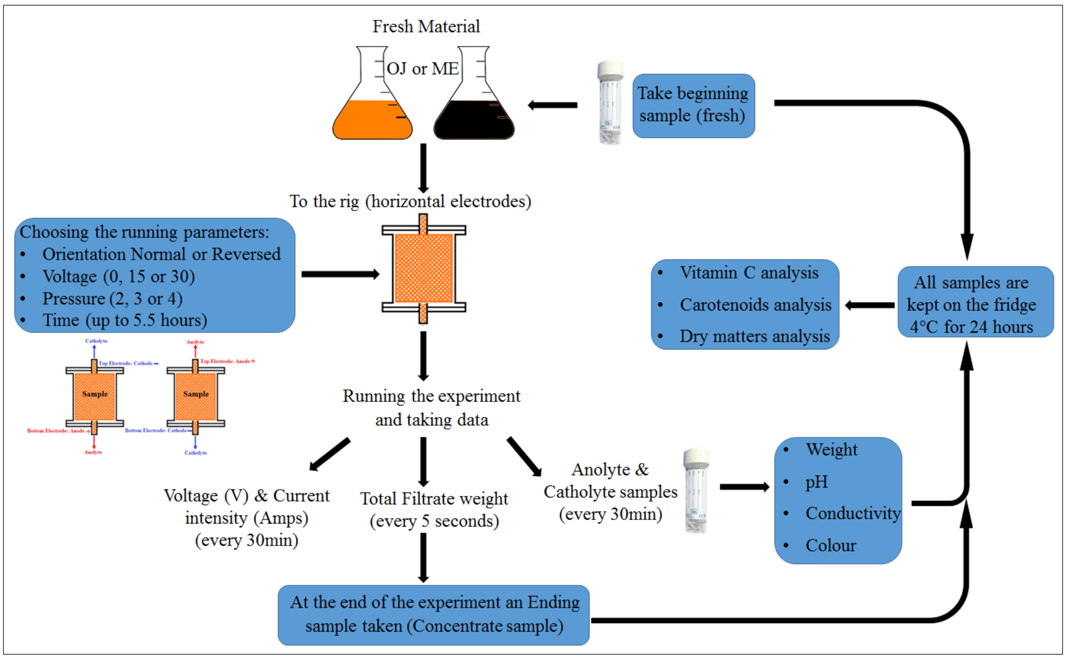
The results of the optimum orientation showed differences between OJ and ME in terms of WPD due to the difference between OJ and ME chemical properties, therefore the impact of the chemical properties (pH and IP) were investigated and some theoretical guidelines about the optimum orientation of each material according to its chemical properties were concluded *(Flowchart 3/ Step3)*.

These experiments helped to choose the optimum orientation for the next experiments *(Flowchart 3/ Step4)* which were run to investigate the impact of EKEF (electrical and mechanical factors) on the dewatering efficiency, where the electrical factors are the intensity, the voltage and the conductivity, while the mechanical ones are the pressure, sample initial thickness and filter pore size.



Flowchart 3. The methodology of running experiments and collecting samples and data

Different voltage values (0, 15 and 30V) and pressures (2, 3 and 4bar) were applied to investigate the impact of electric current, conductivity, voltage and pressure on EKEF dewatering efficiency. A sample of 20ml discharged filtrate from each of the anode and cathode outlet (Anolyte and Catholyte respectively) was taken every 30min, colour and pH analysis was done directly and samples then were kept in the fridge at 4°C for 24 hours to analyse *(Flowchart 4)*. Experiments for all tests are shown in *Appendices 6*.



Flowchart 4. The methodology of running experiments and collecting samples and data

A different control experiment was run for each test due to the difference between the tests' aims; for instance, the control condition to investigate the voltage impact was with no electrokinetics application (V=0), while the control condition to investigate the impact of the pH adjusting was the pH of the fresh sample.

The repetitions were done in different days under the same conditions. However, there was no ability to control all conditions especially the mechanical ones such as the distribution of the sample bits and particles on the filters, which resulted in a variation in the flow rate through the filters in the first 5min before the filtration cake formation. To minimise the data noise resulted from this variation the data recording started after the first 5min.

Figure shows the repetitions of one experiment before deleting the first 5min *(P-value=0.348)*, while Figure shows the repetitions of one experiment after deleting the first 5min *(P-value=0.647)*.

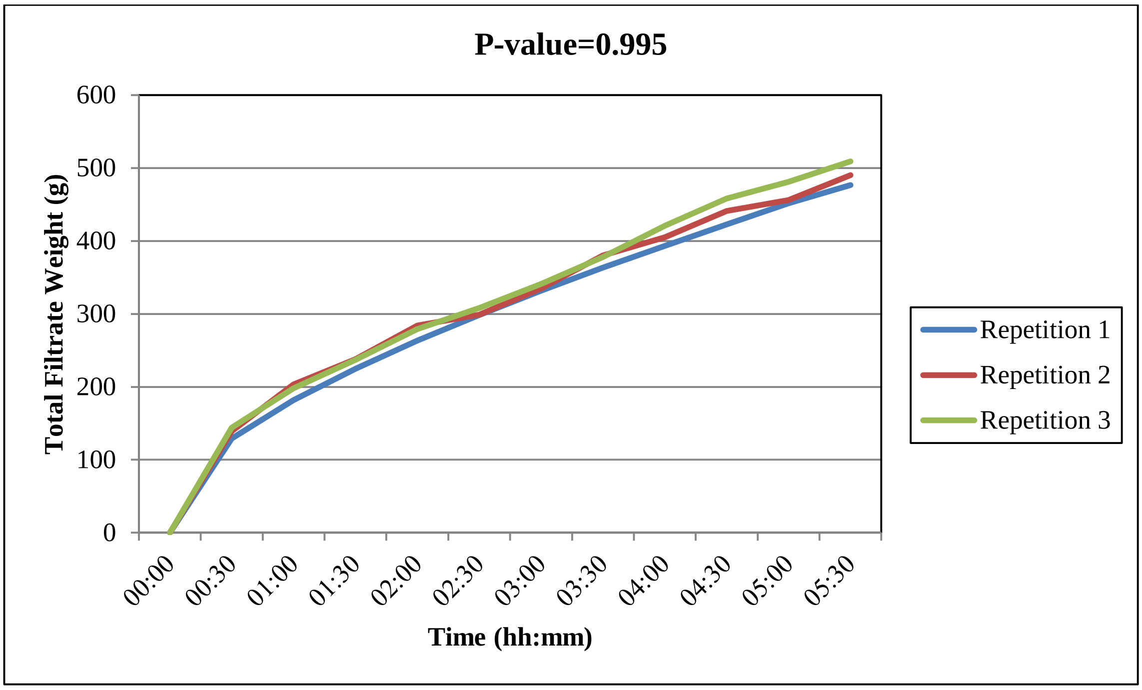


Figure 3.17. The repetitions of the OJ total filtrate weight in the control experiment (0V, 4bar, 0.2µ filter for 5.5hours) before deleting the first 5min

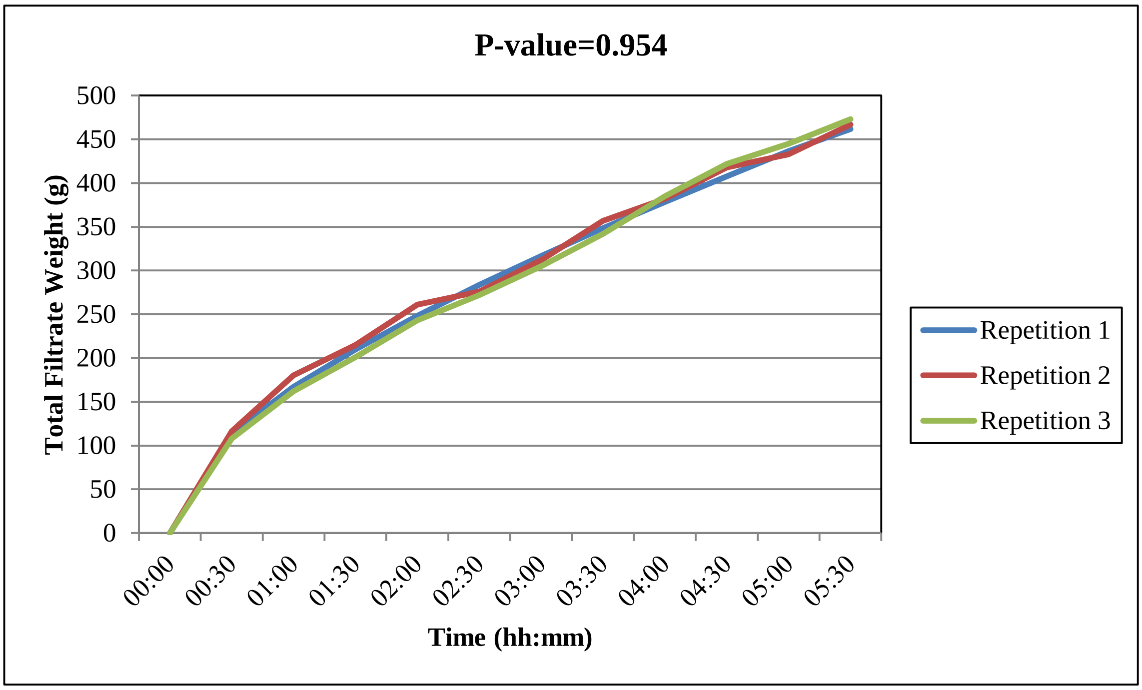


Figure 3.18. The repetitions of the OJ total filtrate weight in the control experiment (0V, 4bar, 0.2µ filter for 5.5hours) after deleting the first 5min

Collected data and observations showed that there were changes in sample pH value, vitamin C retention, colour, dry matters and power consumption over the time. These data were used to check this change and the potential reasons behind them *(Flowchart 3/ Step5)*.

The experiments were carried out to investigate the following:

3.5. Water Movements under the Application of the Electric Field

Applying an electric field between two electrodes to food samples causes two types of movement: the movement of the charged ions and particles towards the electrodes (the anode and the cathode) under the influence of electrophoresis, which in turn results in the second movement of water towards the two electrode outlets (the anolyte and the catholyte) under the effect of electrokinetics.

Negative ions and negatively charged particles, which are dissolved in water, move toward the anode (the positively charged electrode) and water then moves in the opposite direction to the cathode; in the same way, positive ions and positively charged particles move towards the cathode (the negatively charged electrode) and water moves in the opposite direction towards the anode *(Aziz et al., 2006)*.

Although electrokinetics cause movement of ions and water (discharged filtrate) towards the two outlets, the discharged filtrate volumes at each outlet are not equal.

The percentage of water volume moving towards each electrode depends on the net electrical charge; if the suspension net electrical charge is positive the direction of the major volume of discharged filtrate (water) is towards the anode as the positive ions move towards the cathode. In contrast, if the suspension net charge is negative the direction of the major volume of discharged filtrate (water) is towards the cathode.

In this study, different expressions were used to define the phenomena resulting from water movements under Electrokinetic application. The direction of the major volume of discharged filtrate (or water) towards one of the two electrodes is the *water profile direction* (WPD); and the outlet where the water profile goes to is the *main outlet*. In other words, when the sample net electrical charge is positive the WPD is towards the anode and the main outlet filtrate is the anolyte, while when the WPD is towards the cathode when the sample net electrical charge is negative, and the main outlet filtrate is the catholyte.

The *Anolyte to catholyte ratio* (A/C ratio) was used to describe the percentage (or the volume) of the anolyte relative to the percentage (or the volume) of the catholyte; where the anolyte and catholyte refer to the electro-osmotic flows corresponding to the movement under the EKEF impact and/or the applied pressure towards either the anode or the cathode respectively *(Yang et al., 2005)*.

3.5.1. EKEF Efficiency

Different data were collected to investigate the EKEF efficiency:

- *Dewatering efficiency*: is defined as the ratio of the total filtrate weight (the anolyte and the catholyte combined together) to the initial weight of the water in the sample.

  *Dewatering efficiency = (weight of total filtrate/initial weight of water in the sample)*100 Equation 6.*

  In this study, the initial moisture content was 86% for OJ and 80% for ME.

- *Net dewatering efficiency:* is defined as the total dewatering efficiency minus the efficiency of the control (without electricity).

  Net dewatering efficiency = the total dewatering efficiency (with electricity*) - the efficiency of the control (without electricity) Equation 7.*

- *Total Flow rate (TFR):* is defined as the total filtrate weight per minute; which was calculated by dividing the total discharged filtrate coming out from anode and cathode (anolyte and catholyte) by the total time (per min).
- *Anolyte and catholyte weight:* each of anolyte and catholyte filtrates was collected separately in different bottles to identify the total amount eluted from each electrode outlet; these two values were summed later to give the total flow rate.
- *The change of total filtrate weight over time*: total filtrate weight value was collected each 5 sec over the experiment duration by the electronic scale, see *Section 3.2.5*.

3.5.2. The Optimal Experimental Specification (Optimal Orientation)

The movement of discharged filtrate (water) under the application of EKEF is toward the opposite direction of the charged ions and particles dissolved in the water. The movement of these charged ions and particles depends on the net electrical charge of each particle, when the net electrical charge is positive the particle moves toward the cathode (-), while it moves toward the anode (+) when the net electrical charge is negative. The net electrical charge of any particle depends on the isoelectric point (IEP) of the particle and the pH of the medium.

The isoelectric point is the pH value at which a particle is electrically neutral and carries no net electrical charge. Naturally, the particle surfaces charge to form a double layer (H^+/OH^-), the net surface charge is affected by the pH of the liquid in which the particle is submerged.

At the pH of IEP, the overall charge (net electrical charge) on the particle is 0, but this does not mean that the particle contains no charged group; it means that the number of the negative charges is equal to the positive ones. This pH value is very important in electrical food applications especially as the conductivity decreases when the total ion content decreases by becoming neutral.

Foodstuffs are a complex mixture of different compounds and particles which have different IEPs. Most of the devices that are usually used to measure the IEP are calibrated for specific (or single) compounds such as a protein, lipid, or polymer, etc. However, using the definition of IEP, another point was measured in samples to show the potential pH where the net charge of the complex material (the whole foodstuff) becomes almost neutral before turning to the opposite charge. Analysis showed that at this specific pH value, the measured conductivity was at its lowest value; this pH value is called in this study the lowest conductivity point (LCP). According to the LCP and the pH values of foodstuff, the majority of the discharged filtrate volume moves toward one of the two electrodes and eluted through its outlet, this outlet was called the main outlet.

A *JENWAY 4510 Conductivity meter* was used to measure the lowest conductivity point (LCP) of the initial samples of OJ and ME; the titration solutions were made up manually. pH titration included ten 200ml adjusted pH samples of OJ and ME covering the pH range between 1.5 and 6, using 10% *NaOH* (5g in 50 ml) and 10% *HCl*, as shown in *Table 3.2*, and then the Conductivity value was measured using the conductivity meter for each solution.

Table 3.2. Titration solutions used to measure the LCP of OJ and ME

Solution	OJ			ME		
	NaOH 10% (ml)	HCl 37% (ml)	pH	NaOH 10% (ml)	HCl 37% (ml)	pH
Fresh	-	-	3.6	-	-	4.55
Solution 1	-	7.1	1.5	-	6.3	1.5
Solution 2	-	5.5	2.09	-	5.5	2.1
Solution 3	-	4	2.5	-	3.5	2.35
Solution 4	-	2.5	3	-	2.5	3.1
Solution 5	-	1.5	3.45	-	1.5	3.66
Solution 6	1	-	4	-	1	4.23
Solution 7	3.5	-	4.46	1.7	-	5.17
Solution 8	5.3	-	5	2.5	-	5.7
Solution 9	6.8	-	5.5	3.3	-	6.1
Solution 10	8.2	-	6			

The position of the main outlet (top or bottom) had an important role in this process in the vertical set up as it determined whether the water profile movement was with or against the gravity. In this study and for ease of orientation, the orientation when the anode (+) was the bottom electrode and the cathode (-) was the top electrode will be called Normal, while the opposite orientation when the anode was the top and the cathode was the bottom electrode will be called reversed; while the control orientation was when the EKEF effect was switched off (0V) *(Table 3.3* and *Figure 3.19)*.

Table 3.3. Electrodes in the three different positioning orientations (control, normal and reversed)

Orientation	Top electrode	Top Outlet	Bottom electrode	Bottom Outlet
Normal	Cathode	Catholyte	Anode	Anolyte
Reversed	Anode	Anolyte	Cathode	Catholyte
Control	No Charge	Top	No Charge	Bottom

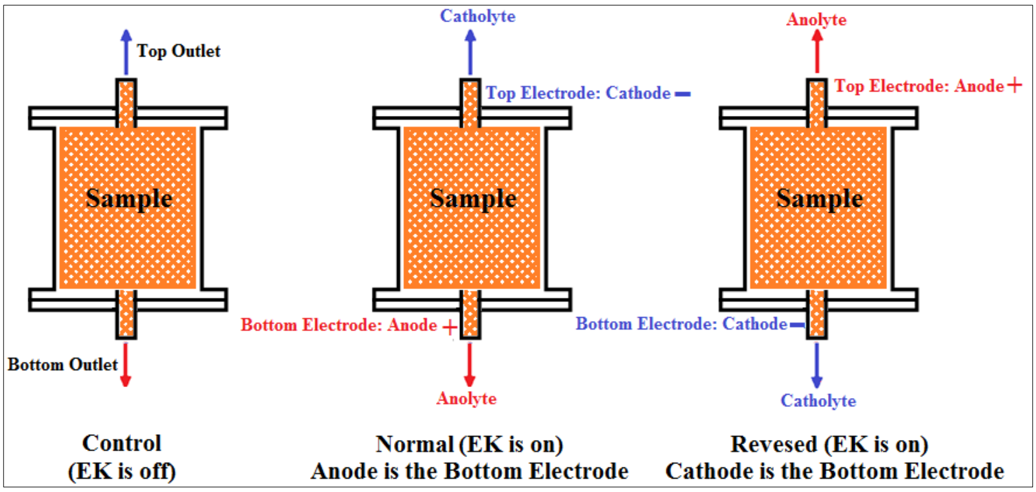


Figure 3.19. Electrodes in the three different positioning orientations (control, normal and reversed)

To determine the optimum orientation of each food material (OJ and ME), experiments were divided into three sets (one with OJ and two with ME) and each set included three experiments one for each orientation (normal, reversed and control); experiments for each set were run under the same voltage (30V in all experiments), the same pressure (4bar in all experiments), and one of two pores sizes of filters (0.2µ or 1µ). *Table 3.4* shows the experimental running conditions.

Table 3.4. Running conditions of the three experiments sets

Exp. Set	Sample	Position	Sample volume (L)	Time (hour)	Voltage (V)	Pressure (bar)	Filter Size (µ)	Top Electrode	Bottom Electrode
1	OJ	Control	1	5.5	0	4	0.2	X	X
		Normal	1	5.5	30	4	0.2	Cathode	Anode
		Reversed	1	5.5	30	4	0.2	Anode	Cathode
2	ME	Control	1	3.5	0	4	1	X	X
		Normal	1	3.5	30	4	1	Cathode	Anode
		Reversed	1	3.5	30	4	1	Anode	Cathode
3	ME	Control	1	2	0	4	0.2	X	X
		Normal	1	2	30	4	0.2	Cathode	Anode
		Reversed	1	2	30	4	0.2	Anode	Cathode

The values of the total filtrate weight and dewatering efficiency (total and net) were compared. In addition, A/C ratio was used to refer to the WPD and the main outlet, as well as to estimate the intensity of the EKEF effect as the following:

- A/C ratio ≈1 means that the anolyte volume ≈ the catholyte volume, and the same volume (or weight) of discharged filtrate is exiting from both outlets.
- A/C ratio >1 means that the anolyte volume > the catholyte volume, WPD towards the anode and more volume (or weight) of discharged filtrate is exiting from the outlet connected to the anode.
- A/C ratio <1 means that the anolyte volume < the catholyte volume, WPD towards the cathode and more volume (or weight) of discharged filtrate is exiting from the outlet connected to the cathode.

In addition to the chemical properties, other factors impacts were investigated such as voltage and pressure by running the experiments shown in *Table 3.5*:

Table 3.5. Experiments of the voltage and pressure impact on WPD and A/C ratio

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
OJ	Control	1	0	2	5.5	0.2
OJ	Normal	1	15	2	5.5	0.2
OJ	Normal	1	30	2	5.5	0.2
OJ	Control	1	0	3	5.5	0.2
OJ	Normal	1	15	3	5.5	0.2
OJ	Normal	1	30	3	5.5	0.2
OJ	Control	1	0	4	5.5	0.2
OJ	Normal	1	15	4	5.5	0.2
OJ	Normal	1	30	4	5.5	0.2

3.5.3. Impact of the Experimental Factors on the EKEF Efficiency

Several experiments were run to investigate the impacts of different factors:

3.5.3.1. The impact of EKEF intensity on the Filtration Process

All experiments were undertaken to investigate the impact of EKEF in enhancing the filtration efficiency. The EKEF process happens by applying an electric field between two electrodes through the sample layers, therefore the change in the electric current intensity under a constant voltage can be used as a representative parameter for the intensity of the EKEF intensity change. Thus, the impact of EKEF on the filtration flow rate can be shown through the relationship between the electric current intensity and the flow rate value, where the flow rate is the filtrate weight per minute. *Table 3.6* shows the experimental running conditions.

Table 3.6. Experiments of EKEF intensity impact on the filtration process

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
OJ	Normal	1	15	4	5.5	0.2
OJ	Normal	1	30	4	5.5	0.2
ME	Reversed	1	15	4	3.5	1
ME	Reversed	1	30	4	3.5	0.2

3.5.3.2. Conductivity Impact on Dewatering Efficiency

The process conductivity was calculated using the change in the electric current intensity under a stable voltage using the *Equations 8 & 9*:

$\sigma = L/(A.R) = (L/A)(I/V)$ *(Equation 8)*

Where:

σ: is the specific electrical conductance (Siemens/m, S/m)

L: is the electrode gap or length of sample (m)

A: is the area of cross section of the sample (m^2)

R: is the resistance of the sample (Ω)

V = electrical potential (V)

I = current flow (ampere) *(Zhang, 2009)*

In the present study, A=0.0289 m^2 and L=0.0346m, L decreases over the experiment which make L/A≈1 and in this case:

(Siemens, S) $\approx 1/R = I/V$ *(Equation 9)*

Under a stable voltage value, the change in the electric current intensity over time represents the conductivity change, and both could be used to represent the change in the EKEF intensity.

To investigate the impact of the initial conductivity, three experiments were run on OJ samples under three different initial conductivity values (0.22, 0.33 and 0.55S), pressure 4bar, and filter 0.2µ for duration of 5.5hours. Initial conductivity values were set using the power supplier. Total filtrate weight of these three experiments was compared over time to show the impact of the three initial conductivities. *Table 3.7* shows the experimental running conditions:

Table 3.7. Experiments of the conductivity impact on dewatering efficiency

Material	Orientation	Volume (L)	Conductivity (S)	Pressure (Bar)	T (Hour)	Filter (µ)
OJ	Normal	1	0.22	4	5.5	0.2
OJ	Normal	1	0.33	4	5.5	0.2
OJ	Normal	1	0.50	4	5.5	0.2

3.5.3.3. The Impact of the Sample initial pH Value

To investigate the impact of different pH value on the WPD and the dewatering efficiency, the pH of OJ samples (1L) were adjusted using NaOH 20% and HCl 37%, as shown in *Table 3.8:*

Table 3.8. Titration solutions

Solution	NaOH 20% (ml)	HCl 37% (ml)	pH
Solution 1	-	10.5ml	1.5
Fresh	-	-	3.6
Solution 2	15ml	-	5
Solution 3	35ml	-	6.5

Then the experiments were run under 30V and 4bar pressure using 1µ filter and compared to the results of fresh juice (pH=3.6, 30V and 4bar pressure) and the control (pH=3.6, 0V and 4bar pressure) under the same conditions, as shown in *Table 3.9*:

Table 3.9. Experiments of the sample initial pH Value impact on the dewatering efficiency

Material	Orientation	pH	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
OJ	Control	3.6	1	0	4	3	1
OJ	Normal	1.5	1	30	4	3	1
OJ	Normal	3.6	1	30	4	3	1
OJ	Normal	5	1	30	4	3	1
OJ	Normal	6.5	1	30	4	3	1

Total filtrate weight and WPD of these experiments was compared over time to show the impact of the different initial pH of samples.

3.5.3.4. The Impact of the Applied Voltage

To investigate the impact of the applied voltage, three different voltages (0, 15 and 30V) were applied on OJ samples under the same conditions (normal orientation anode is the bottom electrode, 0.2µ filter, and 5.5 hours), these experiments were repeated three times under different pressures (2, 3 and 4bar) (*Table 3.5*). These three voltages were also applied on ME samples under the same conditions (reversed orientation cathode is the bottom electrode, 4bar pressure, 1µ filter, 3.5 hours) *(Table 3.10)*:

Table 3.10. Experiments of the applied voltage on the dewatering efficiency

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
ME	Reversed	1	0	4	3.5	1
ME	Reversed	1	15	4	3.5	1
ME	Reversed	1	30	4	3.5	1

The impact of the voltage values (15 and 30V) on the values of the total filtrate weight, A/C ratio, and dewatering efficiency (total and net) was compared with the control (same material and condition but under 0V, no EKEF).

3.5.3.5. The Impact of the Applied Pressure

Three different pressures (2, 3 and 4bar) were applied to OJ samples under the same condition (normal orientation anode is the bottom electrode, 0.2µ filter, 5.5 hours), OJ experiments were repeated three times under three different voltage values (0, 15 and 30V) (*Table 3.5*).

The impact of pressure on the dewatering process was presented by the change in the values of the total filtrate weight, A/C ratio, and dewatering efficiency (total and net) under the three different pressure values.

3.5.3.6. The Impact of the Filter Pore Size

In order to investigate how filter pores size affected the EKEF process, two types of filters were used (0.2µ and 1µ) on the two material (OJ, where three voltage values were applied 0, 15 and 30V) and (ME, where two voltage values were applied 0, and 30V) under the same pressure of 4bar, as shown in

Table 3.11:

Table 3.11. Experiments of the filter pore size on the dewatering efficiency

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
OJ	Control	1	0	4	5.5	0.2
OJ	Normal	1	15	4	5.5	0.2
OJ	Normal	1	30	4	5.5	0.2
OJ	Control	1	0	4	5.5	1
OJ	Normal	1	15	4	5.5	1
OJ	Normal	1	30	4	5.5	1
ME	Control	1	0	4	2.5	0.2
ME	Reversed	1	30	4	2.5	0.2
ME	Control	1	0	4	2.5	1
ME	Reversed	1	30	4	2.5	1

The impact of the filter pore size on the values of the total filtrate weight, A/C ratio, and dewatering efficiency (total and net) was compared.

3.5.3.7.The Impact of the Initial Thickness of the Sample (the initial distance between the electrodes)

All experiments were run in the same cell with the same inner and outer diameters; therefore the only variable dimension is the distance between the two electrodes where the sample is captured. The bottom electrode was connected to the piston and moves up and down freely. The initial distance between the two electrodes depends on the sample volume; this distance is considered as the sample initial thickness. To investigate the impact of the initial thickness, three volumes of the ME sample were used (0.5, 1 & 1.5L) which resulted in thicknesses of 17.3, 34.6 & 51.9mm, respectively for inner diameter of 0.192m, as shown in *Table 3.12*:

Table 3.12. Experiments of the sample initial thickness on the dewatering efficiency

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
ME	Control	0.5	0	4	2	0.2
ME	Reversed	0.5	30	4	2	0.2
ME	Control	1	0	4	2	0.2
ME	Reversed	1	30	4	2	0.2
ME	Control	1.5	0	4	2	0.2
ME	Reversed	1.5	30	4	2	0.2

All these samples have same dry matter content equal to 20% and each of them was used in a set of two experiments, under two voltages 0 and 30V, one pressure of 4bar, and for two hours to compare the net dewatering efficiencies of these sets.

3.5.4. Potential Impacts of EKEF on the Food Samples

In addition to the EKEF efficiency parameters described above, some other parameters were tested to investigate the potential impact of the EKEF on food quality. pH value, Vitamin C, colour and dry matter content were measured for previous purpose. Several devices were used to analyse these compounds in the collected samples:

3.5.4.1. Impact on the pH Value of the samples

The pH values of the collected sample (every half hour over time) were measured and compared with the pH of the fresh sample to investigate the change in the pH of (Anolyte, Catholyte and the total filtrate) of OJ experiments over time under two voltage values (15V and 30V) and the control (0V), (normal orientation, 4bar pressure, 0.2µ and 5.5hr duration), and ME sample (Anolyte, Catholyte and the total filtrate) under (0V, 15V and 30V) (reversed orientation, 4bar pressure, 1µ and 3.5hr duration), as shown in *Table 3.13*:

Table 3.13. Experiments of the pH change

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
OJ	Control	1	0	4	5.5	0.2
OJ	Normal	1	15	4	5.5	0.2
OJ	Normal	1	30	4	5.5	0.2
ME	Control	1	0	4	3.5	1
ME	Reversed	1	15	4	3.5	1
ME	Reversed	1	30	4	3.5	1

A standard pH meter (Martini Instruments Mi 150) was used to measure these values; the meter was calibrated daily using calibration standards (pH4, pH7 & pH10) before use.

3.5.4.2. Vitamin C (Ascorbic Acid) Change

Vitamin C is highly sensitive to degradation and has very low stability in solution and it is oxidized readily in light, air and when heated. Because it is also water soluble, heating in water (like thermal treatment) causes the vitamin to be oxidized and to leach out of the food into the water *(Tannenbaum and Walstra, 1985; Lee et al., 2004)*.

To compare between the EKEF process and the conventional methods for their impact on vitamin C content, several experiments were run on fresh OJ samples with vitamin C content of 46.79 ±6.83mg/100g *(Marks and Spencer)*. Two different voltages were applied (15 and 30V) under 4bar pressure and a duration of 5.5 hours and then compared with the control (0V, 4bar pressure and 5.5hr duration), as shown in *Table 3.14*:

Table 3.14. Experiments of the vitamin C retention change

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (μ)
OJ	Control	1	0	4	5.5	0.2
OJ	Normal	1	15	4	5.5	0.2
OJ	Normal	1	30	4	5.5	0.2

Samples were collected and vitamin C retention was calculated according to *Equation 10*:

% Retention= [Concentration of vitamin C in processed juice / Concentration of vitamin in un-processed juice (fresh)] x 100 (Equation 10) (Vikram et al., 2005)

At the end of each experiment, fresh OJ was divided into two parts: the concentrated juice (juice left inside the EK cell at the end of the experiment), and the total filtrate (all filtrate exiting from the two outlets through the filters), total filtrate also consists of two streams (the anolyte and the catholyte).The retention of vitamin C of the two juice components under (0, 15 and 30V) was compared with the initial vitamin C content.

Vitamin C content was measures in all samples using an ESA CoulArray HPLC system as described in the next section.

Vitamin C Protocol

Vitamin C analysis was carried out on the ESA CoulArray HPLC. The column was a Hypersil GOLD AX with properties shown in *Table 3.15*, and column oven set at 37℃. The flow rate was 0.75mL/min, injection volume 20μl. The mobile phase was 100mM Ammonium Acetate pH 6.8/ Acetonitrile [MeCN] (30:70), vitamin C contents of all samples were measured as mg vitamin C/100g juice.

Table 3.15. Hypersil GOLD AX column properties (Thermo Fisher Scientific, 2012)

Manufacturer	Thermo Fisher Scientific
Model	Hypersil GOLD AX
Diameter	4.6mm
Length	250mm
Particle Size	5µm
Packing Material	AX

Sample Preparation:

OJ samples were deproteinized by mixing 500µL of each sample with 500µL 1% meta-phosphoric acid (MPA) in a micro-centrifuge tube on ice. The samples were left on ice for 5min after thorough mixing. Then the tubes were centrifuged for 5min at 16,000 rpm and the supernatant diluted and used for HPLC analysis.

The samples needed dilution in order to reduce the MPA concentration in samples to maintain HPLC column life and to bring the vitamin C concentration into the required range for analysis. Assuming that the vitamin C concentration in fresh OJ is around 0.50 mg/ml, this 1ml extracted solution contained 0.25mg/ml vitamin C in 0.5% MPA. A 400µL aliquot from this solution was taken and diluted to 5ml with 0.043% MPA to give a final MPA concentration of 0.08% and a vitamin C concentration of around 0.02mg/ml.

Preparation of Standards:

Vitamin C concentration in the OJ sample was expected to be around 0.02mg/ml in the extracted sample. Therefore, the standards range must cover potential vitamin C concentration values; standards of vitamin C were prepared using 0.05 mg/ml ascorbic acid (AA) in 0.08% MPA according to *Table 3.16*:

Table 3.16. Vitamin C analysis standards

Concentration (mg AA: ml MPa(0.08%))	**Dilution**	**0.05mg/ml AA:MPa (0.08%)**	**MPa (0.01%)**
0.005 mg/ml	0.0005%	1ml	9 ml
0.01 mg/ml	0.001%	2 ml	8 ml
0.015 mg/ml	0.0015%	3 ml	7 ml
0.02 mg/ml	0.002%	4 ml	6 ml
0.025 mg/ml	0.0025%	5 ml	5 ml
0.03 mg/ml	0.003%	6 ml	4 ml

3.5.4.3. Colour Change

The change in the colour was monitored only in OJ samples as the colour of ME samples was black and did not visibly change over the experiment; the main discolouration was in the OJ samples.

At the end of the process, fresh OJ was divided into two streams (concentrate and filtrate). Due to the design of the device it was not possible to monitor the colour change in the concentrated juice over time; therefore, only the colour of the final concentrate at the end of the experiment was measured. On the other hand, the change in the filtrate colour was measured every half hour during the experiment.

Measuring colour change was based on the Hunter colour parameters (a, b and L). Samples were put into Petri dishes (60 x 15mm); the colour of samples was measured before and after concentrating using *Minolta Chroma Meter CR-200*. The instrument was standardized each time using a calibration plate (No.20533076 C: Y=92.7, x=.3132 and y=.3192). colour of each sample was measured and L, a, and b values of the measurements were recorded, where L represents the light–dark spectrum with a range from 0 (black) to 100 (white); a represents the green–red spectrum with a range from−60 (green) to +60 (red); and b represents the blue–yellow spectrum with a range from−60 (blue) to +60 (yellow) (*Fazaeli et al., 2013*).

The three measured colour parameters were reported as the browning index (BI). "*BI represents the purity of the brown colour and is considered an important parameter associated with browning*". Browning index was calculated according to *Equations 11 & 12 (Diamante et al., 2010; Fazaeli et al., 2013)*:

$BI = [100 (x - 0.31)] / 0.17$ *(Equation 11)*

Where: $x = (a + 1.75L) / (5.645L + a - 3.012b)$ *(Equation 12)*

The measurements of the concentrated juice and the filtrate samples under three voltages (0, 15 and 30V) and three pressures (2, 3 and 4bar) shown in *Table 3.5*, were compared with measurements of fresh OJ.

3.5.4.4. Carotenoids Change

Fresh, filtrate and concentrate OJ samples were dried using vacuum freeze dryer to use the dried juice powder in the carotenoids analysis method.

Samples preparation:

70 mg of freeze dried sample was homogenized with 3.5 ml of ethyl acetate in 10 mL centrifuge tubes, samples were then vortexed for a few minutes, covered to keep them dark and placed in the refrigerator at 4°C overnight. The next day the samples were centrifuged for 10 minutes at a speed of 4000 g and the supernatant put into a new screw-top test tube. 1 mL of ethyl acetate was added to the residue tube and mixed. Again, the samples were covered and put in the refrigerator for 1 hour. After that the samples were centrifuged and the supernatant transferred to screw-top test tube for extraction, and 0.5 mL of ethyl acetate was added to the residue tube and kept for 1hour again. Samples were then centrifuged and the supernatant removed. In total, the extraction procedure was carried out three times with the supernatant being placed into the same screw-top test tubes. They were then filtered using 1mL syringe and filtered (0.2µm). The extracts were combined and directly analysed by high performance liquid chromatography (HPLC).

OJ samples experienced a problem during freeze drying due to a fault in the equipment, which resulted in a random loss in the samples weight; this loss made analysing carotenoids concentration in these samples meaningless. However a visual detection test was run on some samples to detect whether there is carotenoids in the filtrate samples or not; as carotenoids dissolves in ethyl acetate turning its colour from blank to yellow. 100 g of dried juice sample were dissolved and homogenized with 1.5 ml of water and then mixed with 3.5 ml of ethyl acetate in 10 mL centrifuge tubes, samples were then vortexed for a few minutes, covered to keep them dark and placed in the refrigerator at 4 °C overnight. Samples then will be divided into two phases, acetate at the bottom (0.897 g/cm^3) and water at the bottom.

3.5.4.5. Dry Matter Content Changes

Due to the design of the cell, it was not possible to collect concentrated juice samples over time; therefore, the change in the dry matter content of the concentrated juice was calculated from the change in the total filtrate weight according to *Equation 13*:

$$W_{FJ} \times DM_{FJ} = W_{CJ} \times DM_{CJ} + W_{TF} \times DM_{TF} \quad \text{(Equation 13)}$$

Where: W: is the weight (g)
DM: is the dry matter content (%)
FJ: Fresh Juice
CJ: Concentrated Juice
TF: Total Filtrate

The dry matter content of the samples was determined after drying for 24 hours at 75°C to measure the change of dry matter content over time under the application of three different voltages (0, 15 and 30V) in OJ (normal orientation, 4bar pressure and 0.2µ filter for a duration of 5.5 hours) and ME (reversed orientation, 4bar pressure and 0.2µ filter for a duration of 3 hours), as shown in *Table 3.17*:

Table 3.17. Experiments of the dry matter content change

Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	T (Hour)	Filter (µ)
OJ	Control	1	0	4	5.5	0.2
OJ	Normal	1	15	4	5.5	0.2
OJ	Normal	1	30	4	5.5	0.2
ME	Control	1	0	4	3	0.2
ME	Reversed	1	15	4	3	0.2
ME	Reversed	1	30	4	3	0.2

To determine whether dry matter contents were influenced by the electrode charge (anode or cathode) or the electrode position (top or bottom), the two orientations (normal and reversed) were used in ME experiments under 15V, 4bar and through 1µ filter for 2.5 hours.

3.5.4.6. Power Consumption

The power consumption during the application of EKEF was measured to compare with estimated power consumption during thermal processing.

The total power consumption of dewatering using EKEF can be calculated as the sum of electrical power consumed by the applied voltage across the sample and the mechanical power applied by the pneumatic pressure ram.

Total theoretical energies required for the dewatering process under EKEF application are calculated according to *Equations 14* to *16 (Ng et al., 2011)*.

The energy used to apply pressure to the sample during the process was estimated based upon the distance travelled by the pneumatic ram and the volume displacement from the cell. Mechanical power consumption was calculated for the maximum pressure value (4bar), using *Equation 14*:

$$E_1 = P \times \frac{V}{A} \text{ (Equation 14)}$$

Where E = Energy (J), P = Pressure (KPa) (here P=400Kpas), V= Volume displacement of the cell (m^3) (here V= 0.001m^3, A= Cross sectional area of the cell (m^2) (here A=0.0289 m^2 for inner diameter= 0.192m) :

$$E_1 = 400 \times \frac{0.001}{0.0289} = 13.84\, j = 0.384 \times 10^{-5} KWh$$

The electrical power consumed by applying voltage across the sample was calculated for two voltage values (the minimum 15V and the maximum 30V) using *Equation 15*:

$$E_2 = \int \frac{(V \times I)}{1000} dt \text{ (Equation 15)}$$

Where E = Energy (KW h), V= voltage (V) (V=15 and 30V), I= current (A) and dt= Processing time period (h) (every half hour).

Total energy consumed over the process is the sum of the electrical and mechanical Energy:

$$E = E_1 + E_2 \text{ (Equation 16)}$$

After calculating energies consumed over the experiments, a comparison between the energy consumption of EKEF application (electrokinetics 0&30V and pressure) and the normal microfiltration (with pressure only) was carried out.

In addition, a comparison between the energy consumed during the EKEF application under 4bar pressure and three voltage values (0, 15 and 30V) and the estimated thermal energy required to remove same amount of water by evaporation for OJ and ME was made. The theoretical calculated energy required for thermal removal of water was determined using a value of 43kJ.mol^{-1}, as quoted by *Al-Asheh et al. (2004)*. This theoretical value assumes 100% energy transfer efficiency by the heating mechanism (oven, hot plate etc.) although the efficiencies are in reality much less than this. Some studies claimed even higher values for the required energy to evaporate water using thermal methods such as *Murraya and Lagrange (2011)* who claimed that the required energy was 3873.8 to 8234 kJ/kg product.

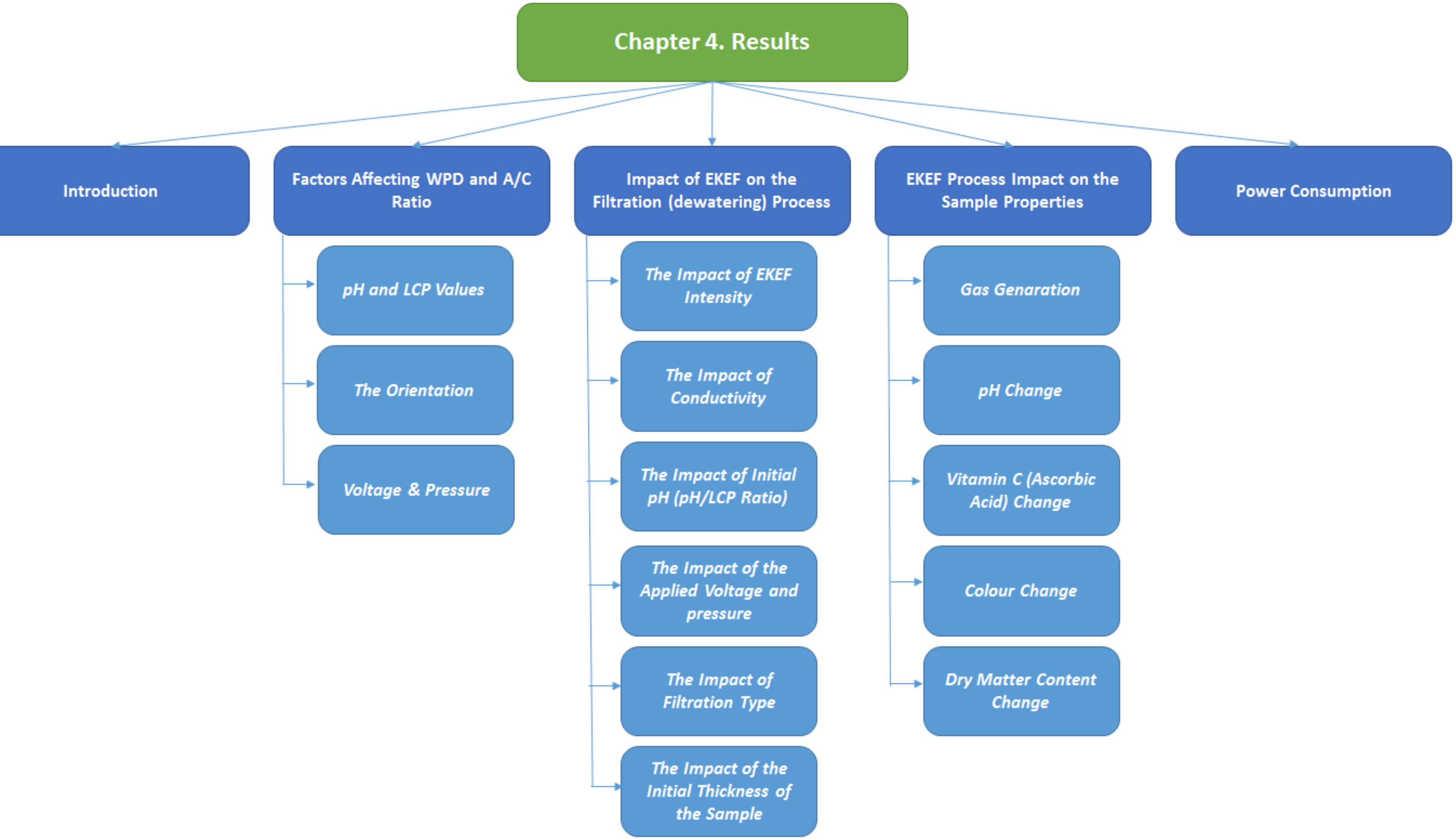
3.6. Statistical analysis

All main results were presented as means ± standard deviation (SD). Analysis of variance was performed on the data taken from triplicates. Differences were detected by one-way analysis for variance (ANOVA) and Time Series analysis. Correlations were obtained by using the Pearson correlation coefficient and R-squared values. Significance of differences between means was determined at the level of 0.05 ($p<0.05$). Overall P-values are presented in tables for all experiments and for comparisons within an experiment.

Due to the limitations on time some of the complementary experiments were done only once, therefore results of these experiments were presented as values not as means ± standard deviation.

All the statistical tests were completed using Minitab 16 Statistical Software and Microsoft Office Excel 2007.

Chapter 4. Results



Flowchart 5. Chapter 4 (Results) Content

4.1. Introduction

The EKEF process is based on the movement of ions and charged particles under an applied voltage gradient between two electrodes (electric force) in addition to the pressure applied by the actuator (mechanical force). These ions and particles arise mainly from the sample ions and particles, and partly from the dissolved electrode.

From the literature review, this movement under a certain voltage gradient creates a constant electric current. The value of this current depends upon several conditions such as the voltage value, conductivity and material of electrodes, conductivity of the samples, amount and types of charged ions and particles (the type of the major ions has the main impact on the water profile movement direction).When these conditions change during the process, they have an impact on both the electric current value and the filtrate flow rate value. In addition to these conditions, there are some other conditions that have a significant impact on the filtrate flow rate and in turn on the dewatering process outcome, such as the applied pressure, the sample volume, the filter pore size and the generated gases within the sample inside the cell.

In this chapter, results are divided into three parts as shown in *Flowchart 5*: the first part presents data to show the impact of the sample chemical properties on the water movement direction and to justify the reasons for choosing different orientation (normal or reversed) for OJ and ME, respectively. The second part presents data about the impacts of the EKEF conditions (conductivity, voltage, pressure, filter pore size and the sample volume) on the filtrate flow rate and the dewatering efficiency. The third part is about the impact of EKEF on the food sample properties (pH, vitamin C, colour and dry matter content). Finally, the power consumption of EKEF process was calculated to estimate the potential of power saving of this process.

4.2. Factors Affecting WPD and A/C Ratio

As mentioned in *Section 3.5* different expressions were used to define the phenomena resulting from water movements under the EKEF application; such as *water profile direction* (WPD), *the main outlet* and the *Anolyte to catholyte ratio (A/C ratio).*

There are several factors affecting the direction and the volume of discharged filtrate (water) moving towards each outlet. These factors are related to the sample chemical properties (pH and isoelectric point IEP/ the lowest conductivity point LCP values), experimental cell design (the position of the outlet, top or bottom), or experiment conditions (applied voltage and pressure). The following shows the impact of these factors on WPD and A/C ratio:

4.2.1. pH and LCP Values Impact on WPD and A/C Ratio:

LCP values of OJ and ME were measured using the conductivity meter *(Section 3.5.2)* for each solution of the titration *(Table 3.2)*; results are shown in *Figure 4.1*.

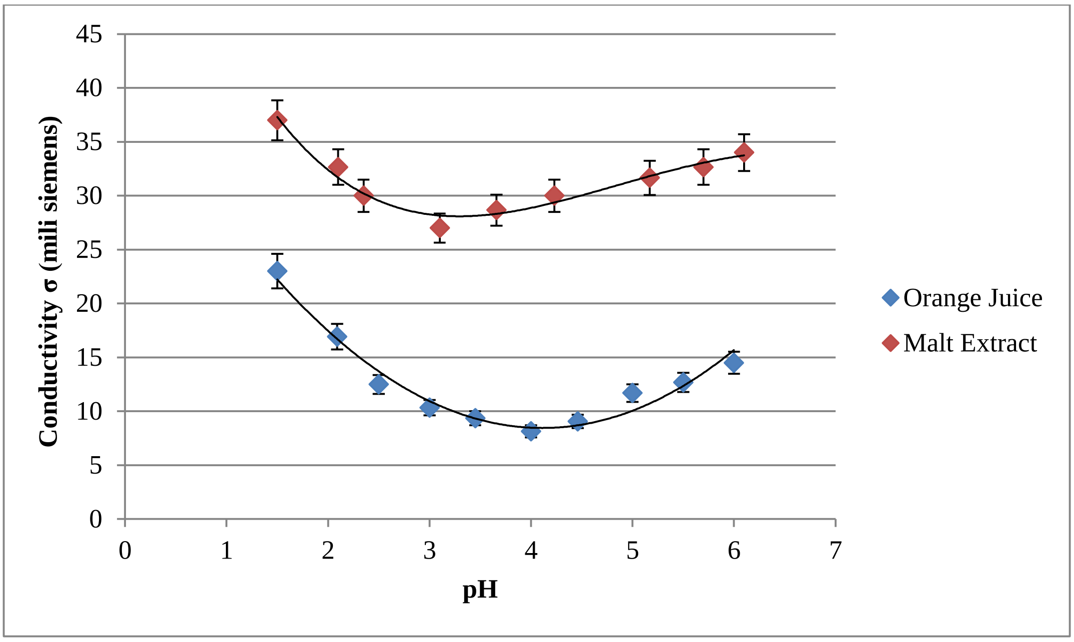


Figure 4.1. Change in conductivity value of OJ and ME under different pH values, where LCP is the pH of the lowest conductivity. Each value is expressed as mean ± standard deviation (n=3)

LCP values of fresh OJ and fresh ME were determined as the pH values where the material has the lowest conductivity; *Table 4.1* shows pH value and LCP value of fresh OJ and fresh ME:

Table 4.1. The pH and LCP values of fresh OJ and fresh ME samples

sample	**pH**	**LCP**
Fresh OJ	3.6	~ 4.0
Fresh ME	4.55	~ 3.2

Several experiments were run on the two contrasting samples (OJ and ME) to investigate the impact of pH and LCP values on WPD and A/C ratio, which later enabled choice of the optimal experimental specification (normal or reversed orientation) for each sample.

Experiments were done according to the method mentioned in *Section 3.5.2*. *Table 4.2* shows the impact of the sample chemical properties (pH and LCP) and the orientation (normal/anode is the bottom electrode or reversed/cathode is the bottom electrode) on WPD and A/C ratio; the discharged filtrate moved almost equally toward the anode and the cathode when the EKEF was not applied (voltage=0V). However, the bottom outlet showed a small increase. When the electric field was switched on, the difference between two outlets filtrates started to increase; examining the A/C ratio data, *Table 4.2* shows that the A/C ratio was > 1 for OJ in all experiments with EKEF and for ME was < 1 in all experiments with EKEF.

Table 4.2. Impact of the sample's chemical properties (pH and LCP) and the orientation (normal or reversed) on WPD and A/C ratio[2]

Experimental conditions	**Anolyte (g)**	**Catholyte (g)**	**Weight of total filtrate (g)**	**Anolyte (%)**	**Catholyte (%)**	**A/C Ratio**
Experiment Set (1): 1 litre, OJ, 0.2µ filter, 30V, 4bar, 5.5 hours						
Control	239.3(B)	222(T)	461.3	51.88	48.12	1.08
Normal / Cathode Top	329.9	192	521.9	63.21	36.79	1.72
Reversed / Cathode Bottom	192.9	115.6	308.5	62.53	37.47	1.67
Experiment Set (2): 1 litre, ME, 1µ filter, 30V, 4bar, 3.5 hours						
Control	202.40(B)	170.80(T)	373.20	54.23	45.77	1.19
Normal / Cathode Top	139.30	187.70	327.00	42.60	57.40	0.74
Reversed / Cathode Bottom	220.50	246.70	467.20	47.20	52.80	0.89
Experiment Set (3): 1 litre, ME, 0.2µ filter, 30V, 4bar, 2 hours						
Control	80.70(B)	77.50(T)	158.20	51.01	48.98	1.04
Normal / Cathode Top	90.40	110.10	200.50	45.09	54.91	0.82
Reversed / Cathode Bottom	98.60	121.60	220.20	44.78	55.22	0.81

4.2.2. The Impact of the Orientation on WPD and EKEF Process Efficiency

In addition to the impact of the sample chemical properties on the WPD and A/C ratio, the orientation could have a potential impact on the volume of the total discharged and in turn on the total process outcome.

Table 4.3 shows the impact of choosing the correct orientation (normal or Reverse) for each material (OJ and ME) by comparing the dewatering efficiency of previous experiments mentioned in *Table 4.2*.

Looking at data in *Table 4.3*, it can be seen that in experiments using OJ the weight of total filtrate after 5.5hours was 461.3g (average flow rate = 1.40g/min, dewatering efficiency = 59.6%) under the control orientation; but when an electric field was applied in the normal orientation, the weight of total filtrate increased to 521.9g (average flow rate = 1.59g/min, dewatering efficiency = 67.43%), achieving a net dewatering efficiency (NDE) = 7.83%.

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[2] In control situation there is no electric field which means the outlets' filtrates cannot be called anolyte and catholyte; but in the table, the bottom filtrate for the bottom is put under the anolyte and mentioned by letter (B) next to the value, while the top filtrate for the bottom is put under the catholyte and mentioned by letter (T) next to the value.

On the other hand, the weight of total filtrate was 308.5g (average flow rate = 0.93/min, dewatering efficiency = 39.86%) under the reversed orientation which decreased the dewatering process efficiency, achieving NDE = -19.74%, and much lower compared with the normal orientation.

In the ME experiments, applying an electric field in the reversed orientation improved the NDE by 11.75%, while the normal orientation decreased the dewatering efficiency by -5.78% comparing with the control orientation using filter 1µ. Using 0.2µ in both reversed and normal orientations improved the efficiency comparing with the control values, however, the reversed orientation showed an improvement of 7.75% compared with the 5.29% improvement achieved by the normal orientation.

Table 4.3. Impact of the orientation on the EKEF dewatering efficiency[3]

Experimental conditions	**Weight of total filtrate (g)**	**Flow Rate (g/min)**	**Dewatering efficiency (%)**	
			Total	**Net**
Experiment Set (1): 1 litre, OJ, 0.2µ filter, 30V, 4bar, 5.5 hours				
Control	461.3	1.40	59.60	
Normal / Cathode Top	521.9	1.58	67.43	7.83
Reversed / Cathode Bottom	308.5	0.93	39.86	-19.74
Experiment Set (2): 1 litre, ME, 1µ filter, 30V, 4bar, 3.5 hours				
Control	373.20	1.78	46.65	
Normal / Cathode Top	327.00	1.56	40.88	-5.78
Reversed / Cathode Bottom	467.20	2.22	58.40	11.75
Experiment Set (3): 1 litre, ME, 0.2µ filter, 30V, 4bar, 2 hours				
Control	158.20	1.32	19.78	
Normal / Cathode Top	200.50	1.67	25.06	5.29
Reversed / Cathode Bottom	220.20	1.84	27.53	7.75

The impact of the orientation (normal or reversed) on the total filtrate weight is shown in *Figures 4.2* to *4.4*, which also show a significant difference between the normal and the reversed orientation ($P<0.001$) for OJ, ME (1µ) and ME (0.2µ). These figures show that for orange juice applying EKEF in the normal orientation played a positive role in increasing total filtrate weight over time in comparison with the control while the total filtrate weight was less using the reversed orientation. In contrast the reversed orientation was the optimal one to increase the

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[3] Dewatering efficiency = (weight of total filtrate/initial weight of water in the sample) *100 (take the initial moisture content as 86% for orange juice and 80% for malt extract). Net dewatering efficiency is the total dewatering efficiency minus the efficiency of the control (without electricity). Weight of the total filtrate is the total weight of the discharged filtrate going out through the two outlets, and the flow rate is the total filtrate weight per minute.

total filtrate weight over time comparing with normal and control orientation in ME experiments (1 and 0.2 µ filters).

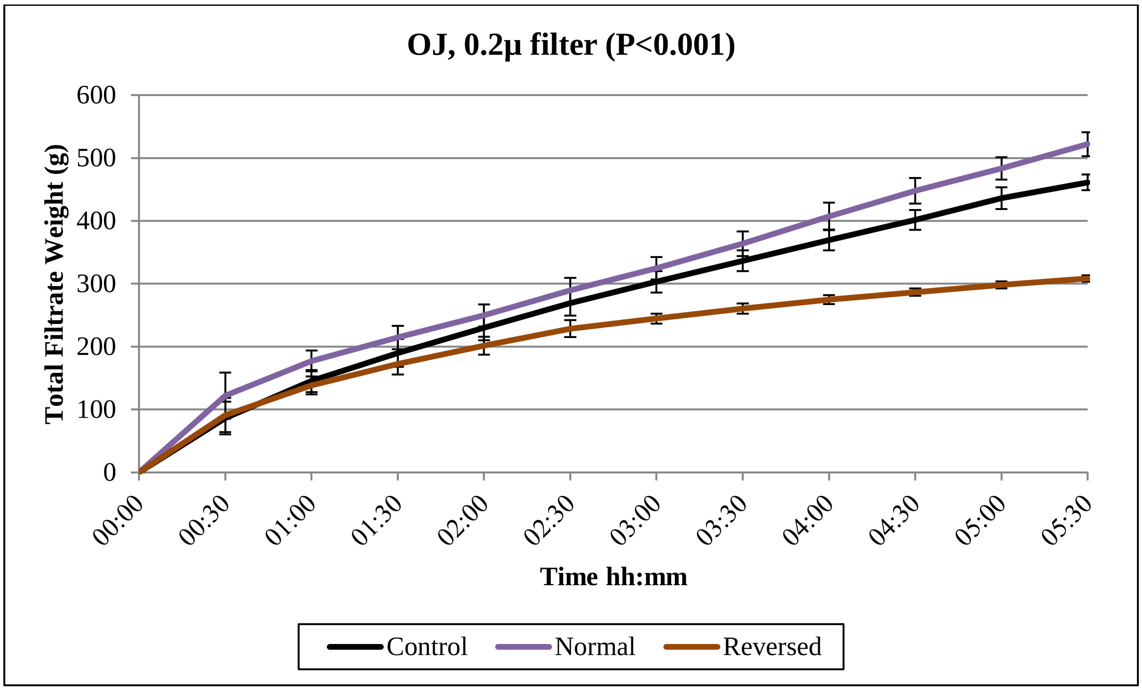


Figure 4.2. Impact of the orientation (control, normal and reversed) on the EKEF dewatering efficiency (Experiment Set (1): 1 litre, OJ, 0.2µ filter, 30V, 4bar, 5.5 hours). Each value is expressed as mean ± standard deviation (n=3)

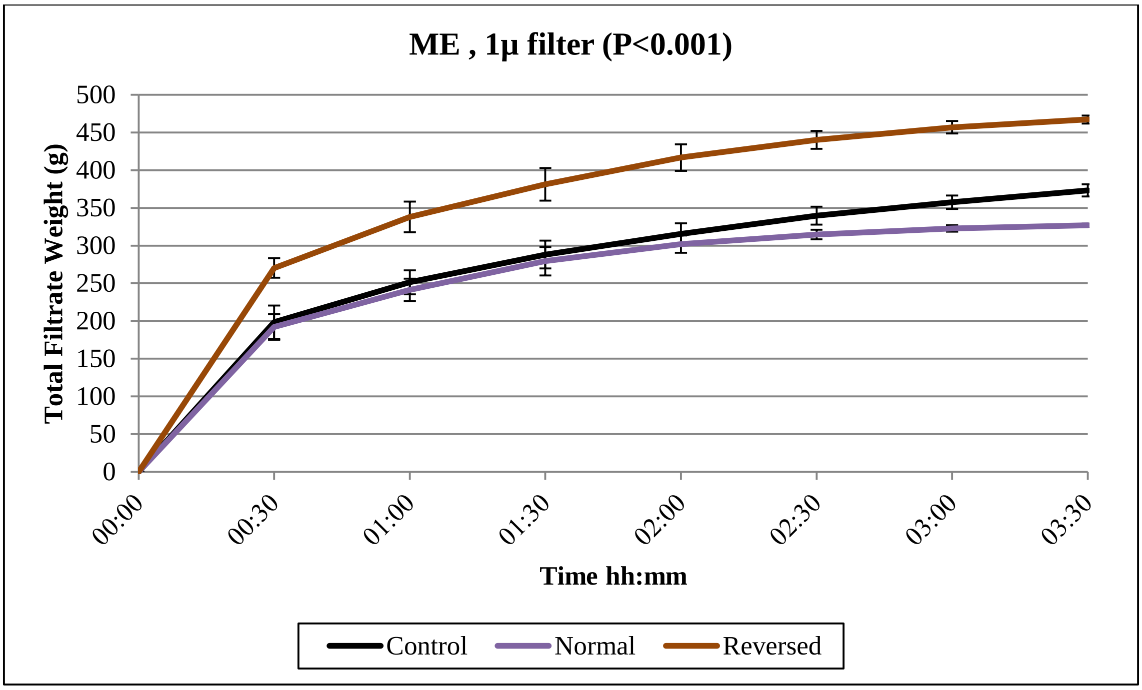


Figure 4.3. Impact of the orientation (control, normal and reversed) on the EKEF dewatering efficiency (Experiment Set (2): 1 litre, ME, 1µ filter, 30V, 4bar, 3.5 hours). Each value is expressed as mean ± standard deviation (n=3)

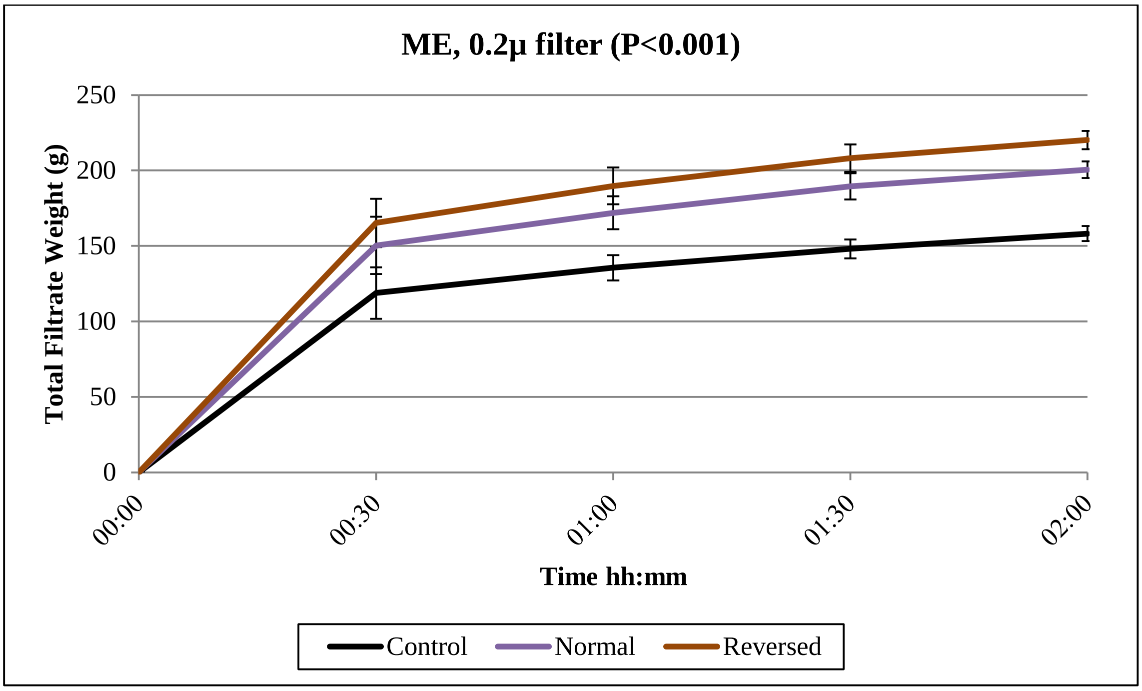


Figure 4.4. Impact of the orientation (control, normal and reversed) on the EKEF dewatering efficiency (Experiment Set (3): 1 litre, ME, 0.2μ filter, 30V, 4bar, 2 hours). Each value is expressed as mean ± standard deviation (n=3)

For more explanation about the impact of EKEF on water movement and why it is important to choose the correct orientation, the weight of anolyte and catholyte was measured each half an hour to see the change in weight over time compared with the control (0V, 4bar).

The EKEF process was applied to OJ under 30V, 4bar and 0.2M filter for 5.5h in two orientations (normal and reversed) *(Figures 4.5* to *4.7)*.

When the electric field was off the two filtrates were almost equal (anolyte=239.3g and catholyte=222g), with a small increase seen at the bottom *(Figure 4.5)*, and the difference between them was not significant (P=0.679, One Way ANOVA). When the electric field was switched on, (voltage=30V) in the normal orientation, the anolyte showed a higher flow rate and the final anolyte weight after 5.5hours was significantly higher than the final catholyte weight (anolyte/bottom filtrate=329.9g, catholyte/top filtrate=192g and P=0.013) *(Figure 4.6)*.

Under the reversed orientation, the two outlets have also performed differently compared to the control orientation with higher anolyte weight compared with the catholyte. However, this difference between the two outlets was not significant after 5.5 hours (anolyte/top filtrate=192.9g, catholyte/bottom filtrate=115.6g and P=0.260) *(Figure 4.7)*.

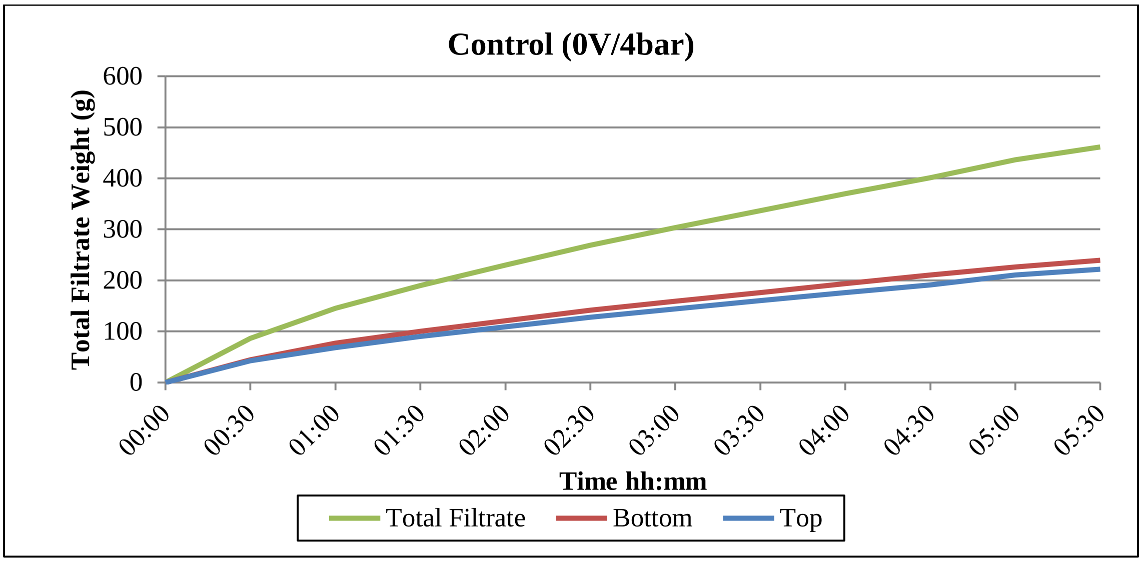


Figure 4.5. Change in the weight of OJ bottom, top and total filtrates over time under the control orientation (0.2µ filter, 0V, 4bar, 5.5 hours)

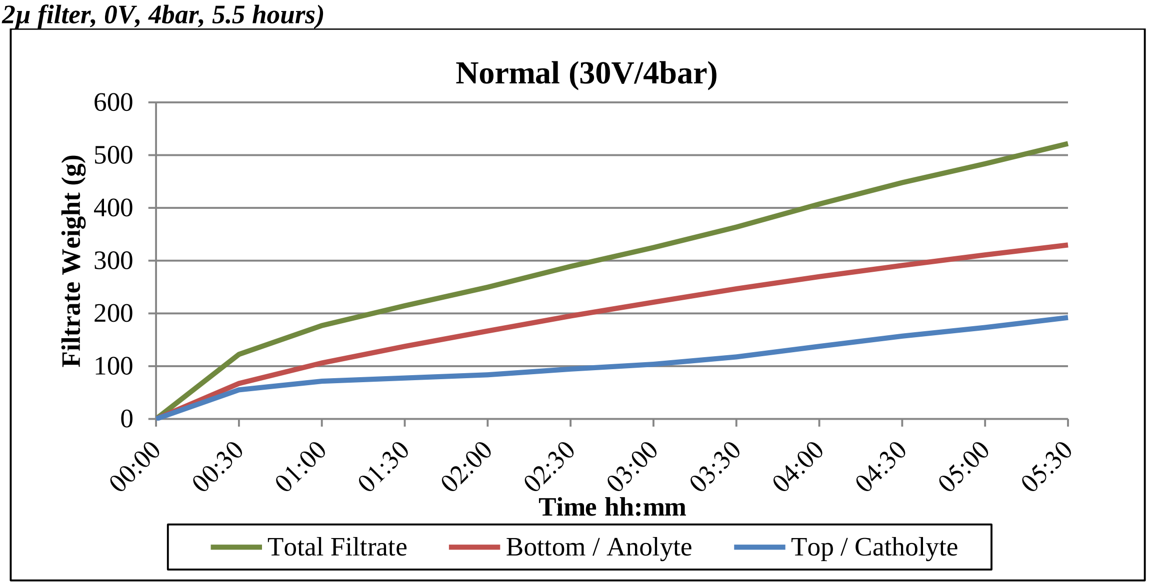


Figure 4.6. Change in the weight of OJ anolyte, catholyte and total filtrate over time under the normal orientation (0.2µ filter, 30V, 4bar, 5.5 hours)

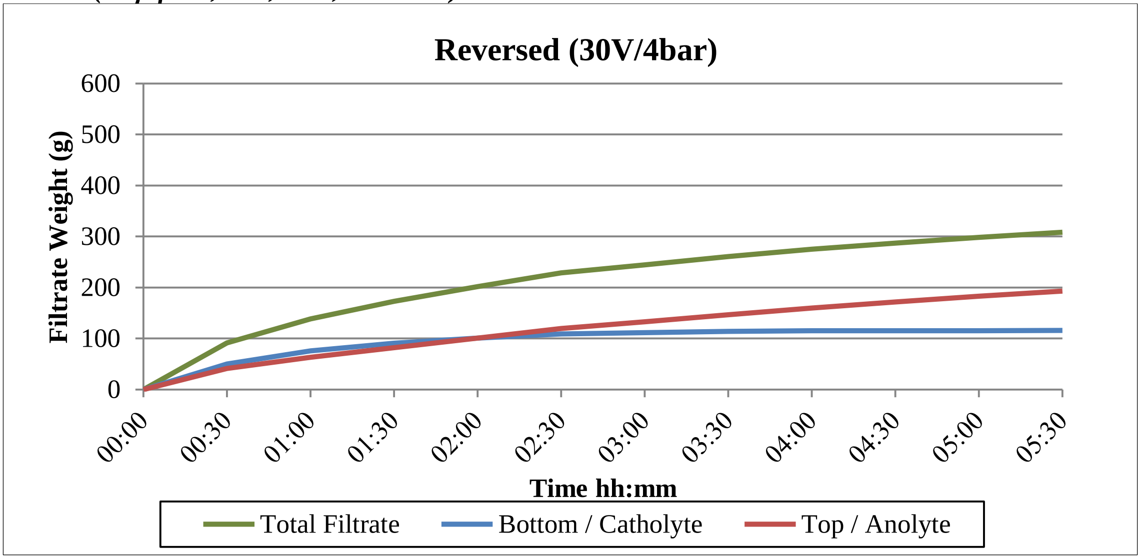


Figure 4.7. Change in the weight of OJ anolyte, catholyte and total filtrate over time under the reversed orientation (0.2µ filter, 30V, 4bar, 5.5 hours)

4.2.3. *Other Factors Affecting WPD: (Voltage and Pressure)*

Furthermore, there are other factors affecting WPD and the discharged filtrate volume such as the applied voltage and pressure.

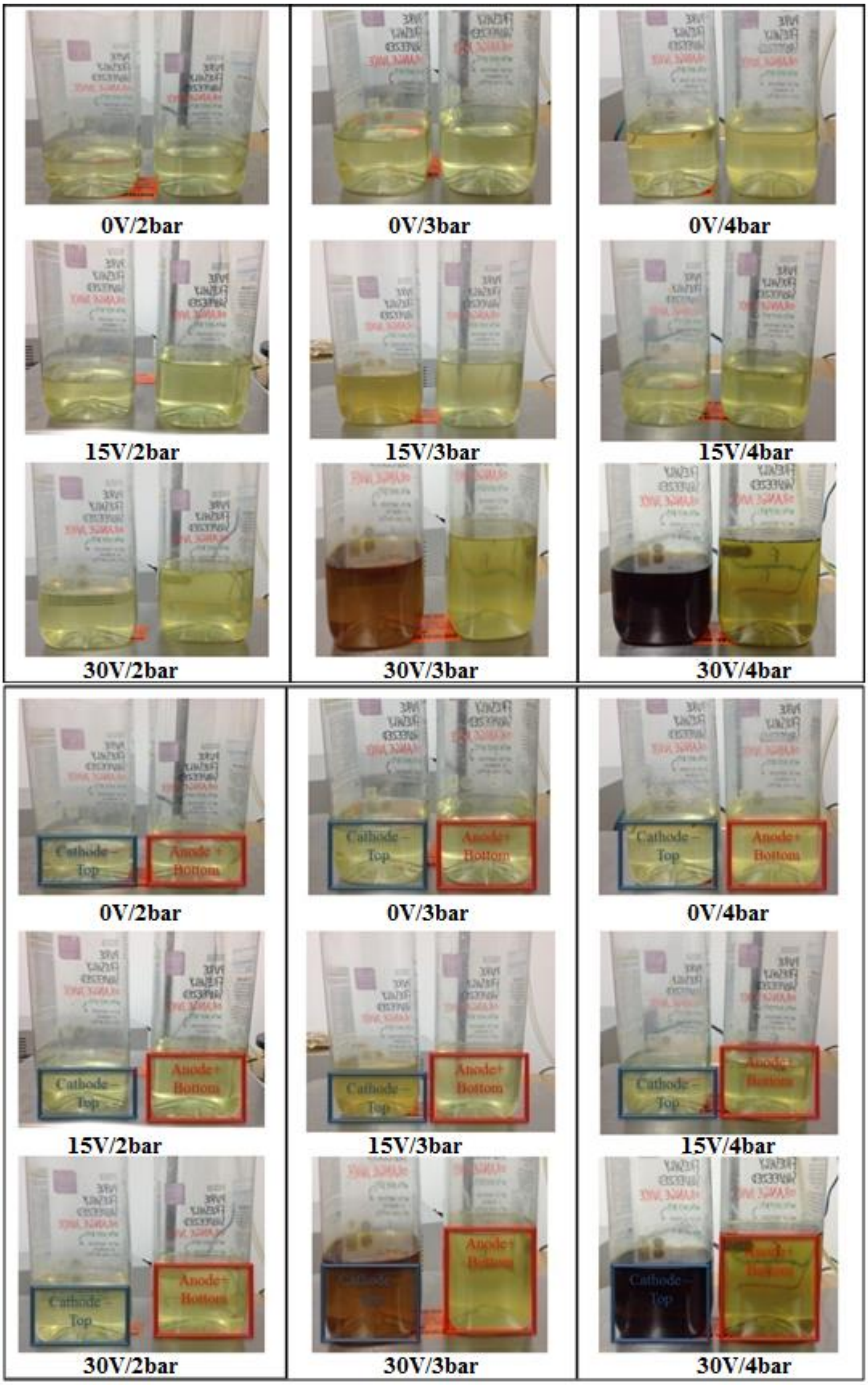
The impact of applying three values of voltage (0, 15 and 30V) and pressure (2, 3 and 4bars) on WPD and A/C ratio of OJ samples is shown in *Figure 4.8* and *Table 4.4*.

Anolyte and catholyte have almost equal volume (A/c ratio ~1) at the end of the control experiments (when voltage= 0V) under the three values of pressure (2, 3 and 4bar). However, the higher the pressure, the bigger the anolyte, the catholyte and total filtrate weight.

When an electric field was applied (15V and 30V), water movement toward the bottom anolyte became greater (A/C ratio > 1); and this ratio increased as the voltage increased with greater movement towards the anode under 30V compared with 15V.

Table 4.4. Impact of voltages (0, 15 and 30V) and pressures (2, 3 and 4bars) on WPD and A/C ratio (normal orientation, 0.2μ filter, 5.5 hours)

Experimental conditions	**Anolyte (g)**	**Catholyte (g)**	**Total Weight of Filtrate (g)**	**Anolyte (%)**	**Catholyte (%)**	**A/C Ratio**
At 2 bar						
Control (0V)	193.8(B)	173.1(T)	366.9	52.8	47.2	1.12
15V	248.4	156.1	404.5	61.4	38.6	1.59
30V	244.1	169.1	413.2	59.1	40.9	1.44
At 3 bar						
Control (0V)	194.1(B)	192.7(T)	386.8	50.2	49.8	1.01
15V	243.8	162.2	406.0	60.0	40.0	1.50
30V	259.4	167.6	427.0	60.7	39.3	1.55
At 4 bar						
Control (0V)	232.5(B)	229.2(T)	461.7	50.4	49.6	1.01
15V	288.0	185.1	473.1	60.9	39.1	1.56
30V	327.2	192.0	519.2	63.0	37.0	1.70



0V/2bar 0V/3bar 0V/4bar

15V/2bar 15V/3bar 15V/4bar

30V/2bar 30V/3bar 30V/4bar

0V/2bar 0V/3bar 0V/4bar

15V/2bar 15V/3bar 15V/4bar

30V/2bar 30V/3bar 30V/4bar

Figure 4.8. Impact of voltages (0, 15 and 30V) and pressures (2, 3 and 4bars) on WPD and A/C ratio (0.2µ filter, 5.5 hours)

For more details, the weight and the flow rate of anolyte, catholyte and total filtrate were observed over time under the application of three voltages and represented in *Appendices 1 & 2*.

Under 0V (control), both anolyte and catholyte extraction changed according to the normal filtration curve and ended with almost the same volume ($P=0.679$) *(Appendices 1.1* and *2.1)*.

After applying an electric field, water moved towards the anode resulting in bigger-anolyte volume comparing with the catholyte. The difference between anolyte and catholyte volume was not significant under 15V ($P= 0.123$) while it was significant under 30V ($P=0.013$) *(Appendices 1.2* and *1.3)*.

Elution of the anolyte under an electric field of 15V and 30Vchanged over time according to the normal filtration curve while the catholyte fluctuated over time; this fluctuation was higher with the higher voltage (30V) due to higher gas generation *(Appendices 2.2* and *2.3)*.

4.3. Impact of EKEF on the Filtration (dewatering) Process

Impact of EKEF on the dewatering process (dewatering efficiency and filtrate flow rate) depends on the experimental conditions of applying EKEF. These conditions are: EKEF intensity (represented by the electric current intensity), process conductivity, applied voltage, applied pressure, filter pore size and the sample volume:

4.3.1. The Impact of EKEF Intensity on the Filtration Process

According to *Section 3.5.3.1*, the impact of EKEF on the filtration flow rate can be shown through the relationship between the electric current intensity, which represents the EKEF intensity, and the flow rate value, where the flow rate is the filtrate weight per minute.

Table 4.5 shows the correlation between filtration efficiency (represented by the flow rate value) and EKEF intensity (represented by in the electric current intensity under a stable voltage gradient), from different experiments. This correlation is presented by R^2 values and Pearson r correlation. In addition this correlation was shown in *Appendices 3*.

The EKEF intensity (represented by the electric current intensity under a stable voltage gradient) and the filtration efficiency change over time (represented by the changes flow rate value) has a positively correlated relationship (r = 0.911 and 0.874 for OJ experiments under 15V/4bar, 30V/4bar respectively, and 0.813, 0.986 for ME experiments under 15V/4bar, 30V/4bar respectively); this demonstrated that filtration efficiency increased as the EKEF value increased.

Table 4.5. The correlation between electric field intensity and filtrate flow rate under different condition. Each value is expressed as mean ± standard deviation (n=3)

Experiment	**Pearson r correlation**
OJ (15V/4bar)	0.911
OJ (30V/4bar)	0.813
ME (15V/4bar)	0.986
ME (30V/4bar)	0.978

4.3.2. Conductivity Impact Upon Dewatering Efficiency

The process conductivity was calculated using the change in the electric current intensity under a stable voltage using *Equation 9, Section 3.5.3.2*; under a stable voltage value, the change in the electric current intensity over time represents the conductivity change, and both can be used to represent the change in the EKEF intensity of the EKEF applied. *Figure 4.9* shows the change of the process conductivity over time under 15V and 30V. Process conductivity fluctuated and dropped over time under both voltages. This fluctuation was larger under 30V while the conductivity dropping was steadier under 15V.

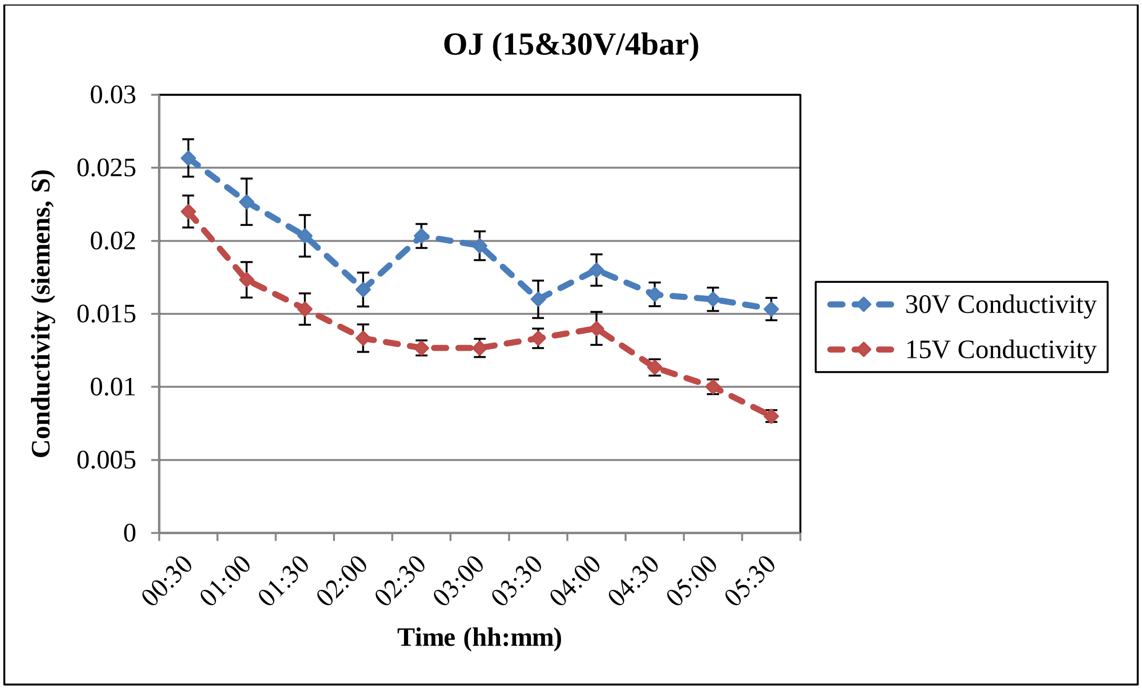


Figure 4.9. Change in the conductivity in OJ samples over the experiment time under two stable voltages (15V&30V), a pressure of 4bar, 0.2µ filter and for 3.5 hours. Each value is expressed as mean ± standard deviation (n=3)

Figure 4.10 shows the impact of different initial conductivity values (0.22, 0.33 and 0.5S) on the filtration efficiency (represented by the weight of the total filtrate) experiments were done as written in *Section 3.5.3.2*. The initial conductivity value played a significantly positive impact ($P<0.001$) on increasing the filtration efficiency.

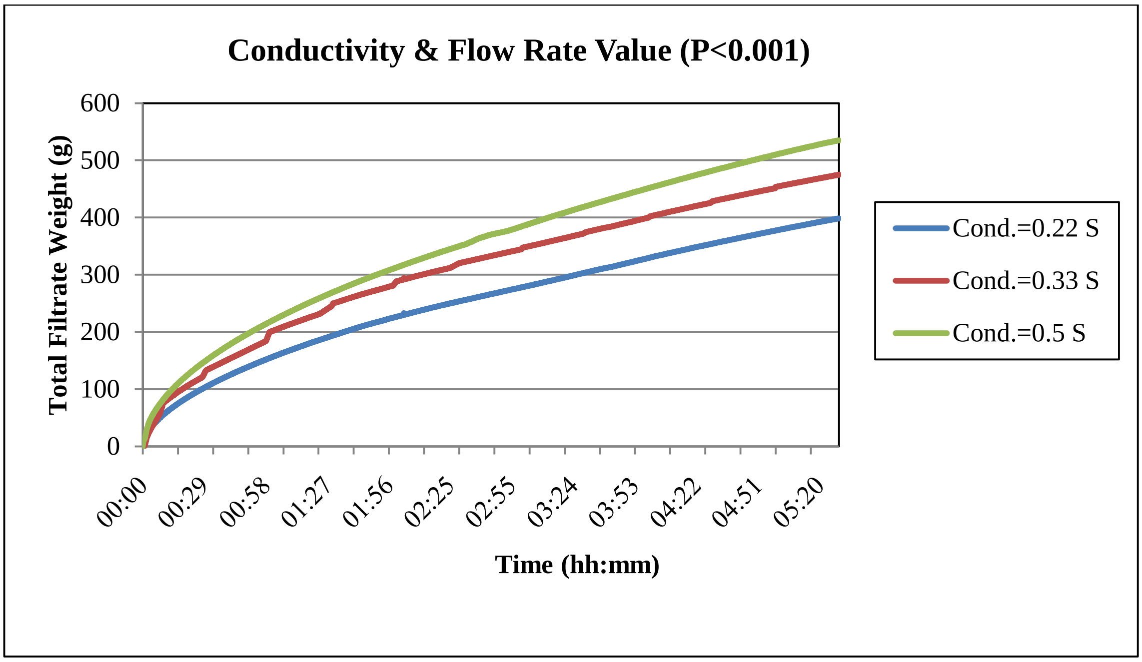


Figure 4.10. Impact of different initial conductivity values (0.22, 0.33 and 0.5S) on the weight of the total filtrate in OJ using 0.2µ filter, a pressure of 4bar and for 5.5 hours

4.3.3. Initial pH Impact (pH/LCP Ratio)[4] upon WPD and Dewatering Efficiency

As shown in *Section 3.5.3.3*, the initial pH of OJ samples were adjusted to (1.5, 3.6, 5 and 6.5) and then the experiments were run under 30V and 4bar pressure using 1µ filter and compared to the results of fresh juice (pH=3.6, 30V and 4bar pressure) and the control (pH=3.6, 0V and 4bar pressure) under the same conditions.

The results in *Figure 4.11* and *Table 4.6* show that the bigger the difference between the pH value and LCP value (LCP=~4), the higher the final outcome. Increasing the difference between pH an LCP is effective in both sides (either increasing the acidity (pH/LCP ratio<<1) or the alkalinity (pH/LCP ratio>>1)), the highest total filtrate weight was in OJ sample with pH=1.5 and then with pH=6.5.

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[4] In this study, LCP is used instead of IEP due to the difficulty of measuring the IEP of the experimental materials; however, findings are correct for IEP if it is measurable in the sample.

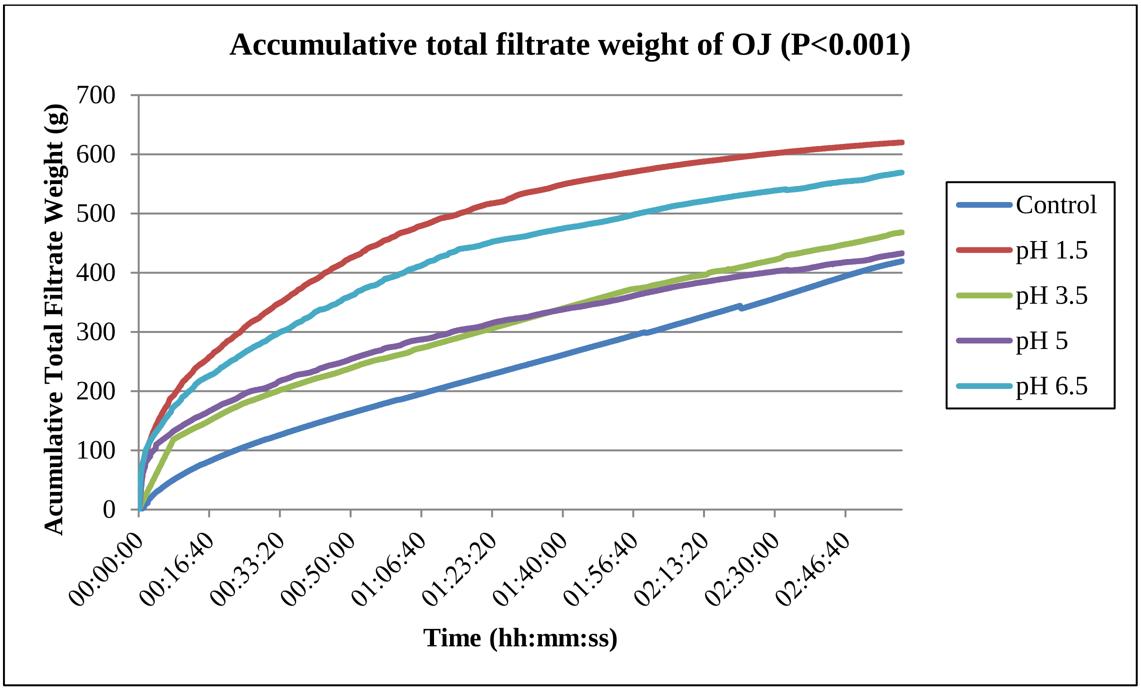

Figure 4.11. Accumulative total filtrate weight of OJ samples with different initial pH values (1.5, 3.6, 5.0 &6.5)under the application of electrokinetic dewatering (normal orientation, 30V,4bar,1µ filter and 3hours) in comparison with the control orientation (pH=3.6, 0V, 4bar, 1µ filter and 3hours)

Table 4.6. Impact of adjusting OJ initial pH value (1.5, 3.6, 5.0 &6.5) on the electrokinetic dewatering efficiency (normal orientation, 30V, 4bar, 1µ filter and 3hours) in comparison with the control orientation (pH=3.6, 0V, 4bar, 1µ filter and 3hours)

Experimental conditions	Anolyte (g)	Catholyte (g)	Total Filtrate Weight (g)	Anolyte (%)	Catholyte (%)	Flow Rate (g/min)	Dewatering efficiency (%) Total	Net
Control (0V) (pH=3.6)	218	203	421.0	51.78	48.22	2.34	54.39	
pH=1.5	342.6	277.4	620.0	55.3	44.7	3.44	80.10	25.71
pH=3.6	262.5	202	464.5	56.5	43.5	2.58	60.01	5.62
pH=5.0	210.5	222.2	432.7	48.6	51.4	2.40	55.90	1.51
pH=6.5	242.6	326.5	569.1	42.6	57.4	3.16	73.53	19.14

Figure 4.12 illustrates the process final total filtrate weight of the previous samples with different pH values; in addition the outcome of the control experiment (fresh juice, pH=3.6, 0V & 4bar) is shown in this figure.

This figure shows that the total filtrate weight increased as the difference between the pH and the LCP values increased; according to the curve, the lowest outcome was around the LCP in the range of pH between 4 and 5.

The total outcome of the control experiment was close to the outcome of the LCP, which demonstrates that when pH is close to the LCP (pH/LCP ratio≈1) the suspension is mostly neutral which reduces or cancels the impact of EKEF.

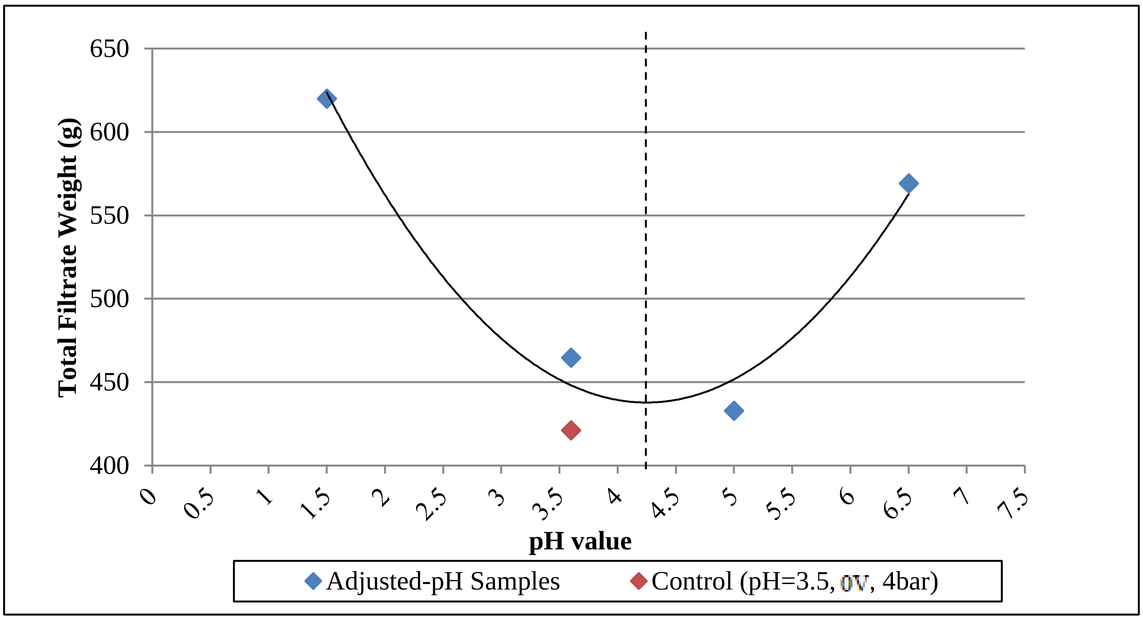

Figure 4.12. Total filtrate weight of OJ samples with different initial pH values (1.5, 3.6, 5.0 &6.5)

To explain the impact of increasing the difference between the pH and the LCP, the change of the current intensity of each pH experiment was measured over time and is shown in *Figure 4.13*.

The larger the difference between the sample pH and the LCP the higher the current intensity.

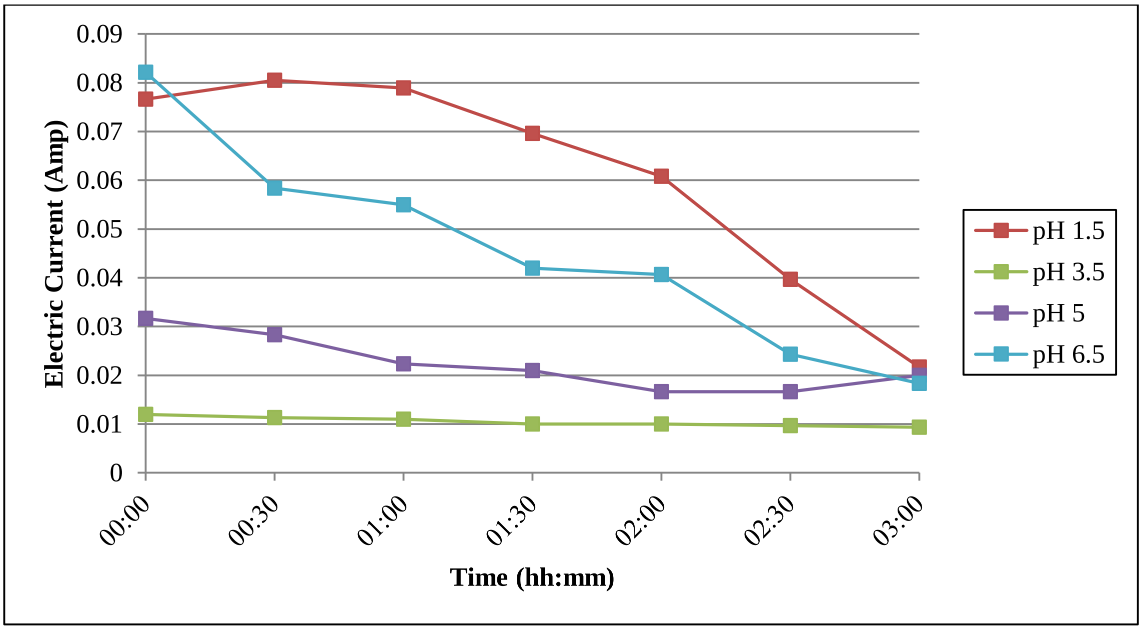

Figure 4.13. Change in electric current intensity through OJ samples with different initial pH values(1.5, 3.6, 5.0 &6.5)under the application of electrokinetic dewatering (normal orientation, 30V, 4bar,1µ filter and 3hours) in comparison with the control orientation (pH=3.6, 0V, 4bar, 1µ filter and 3hours)

In addition, changing the pH/LCP ratio changed the flow rate which was higher with larger pH/LCP difference, and also affected water profile direction which was toward the anode with pH/LCP ratio < 1 (resulting in higher anolyte A/C ratio > 1) *(Figure 4.15)*, and toward the cathode with pH/LCP ratio > 1 (resulting in higher catholyte A/C ratio < 1), as shown in *Figure 4.16*. The change in the flow rate of the OJ samples with pH values (3.5 and 5) is shown in *Appendices 4*.

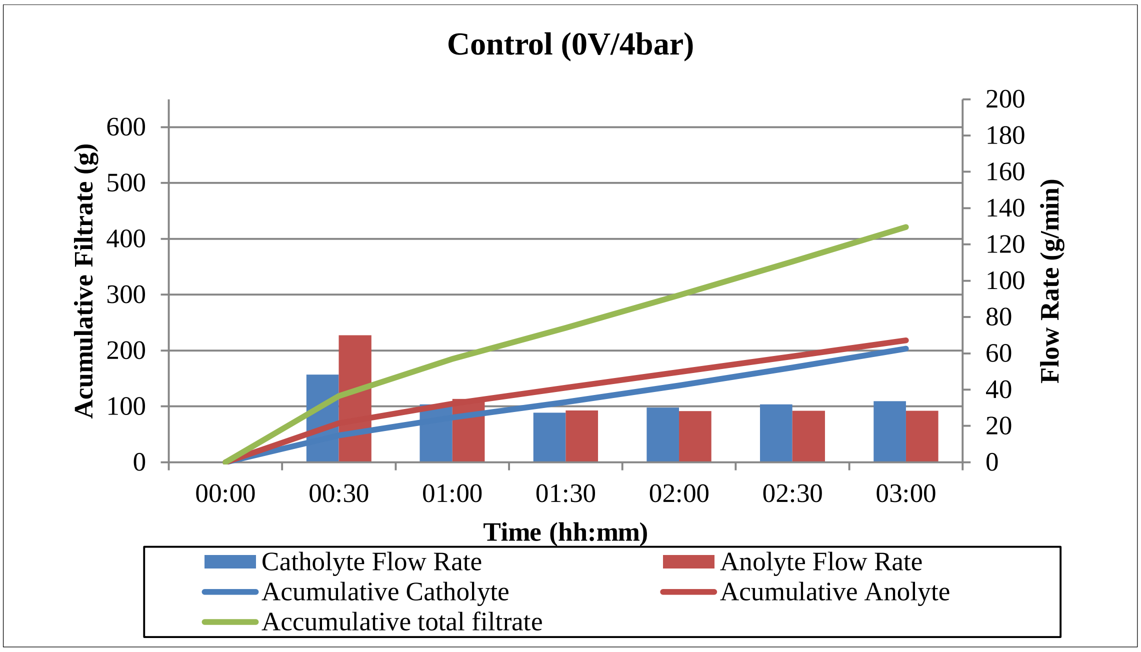

Figure 4.14. Change in OJ accumulative total and filtrate flow rate over time under the control orientation (pH=3.6, 0V, 4bar, 1μ filter and 3hours duration)

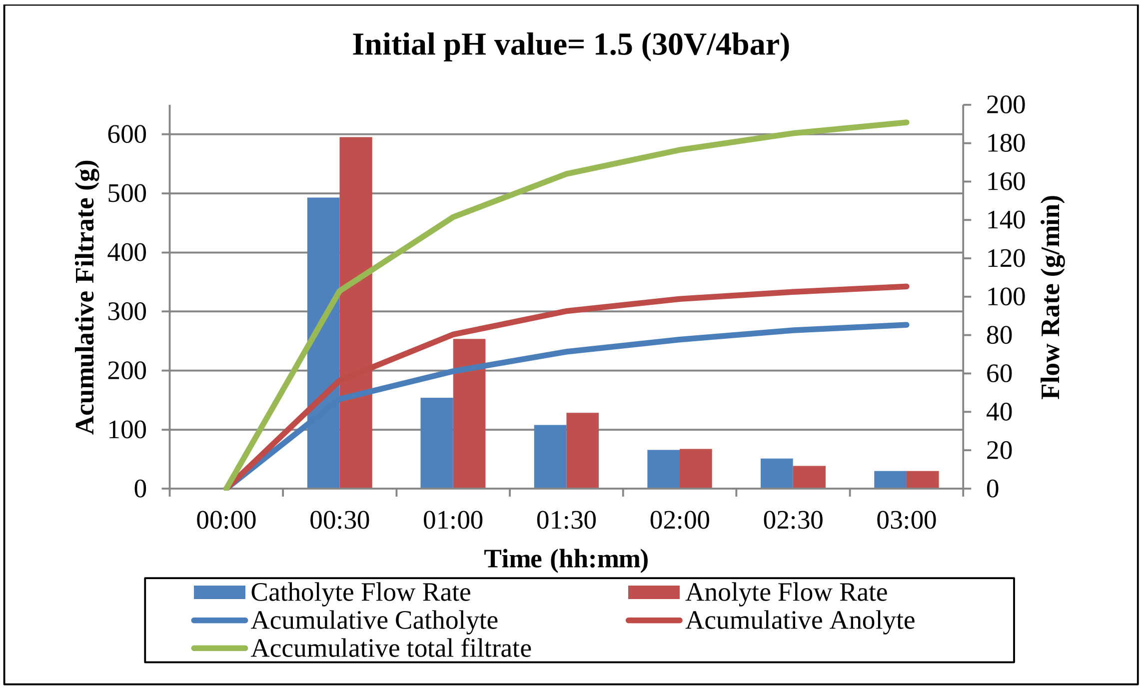

Figure 4.15. Change in OJ accumulative filtrates and filtrate flow rate over time under the application of EKEF with initial pH value=1.5 (normal orientation, 30V, 4bar,1μ filter and 3hoursduration)

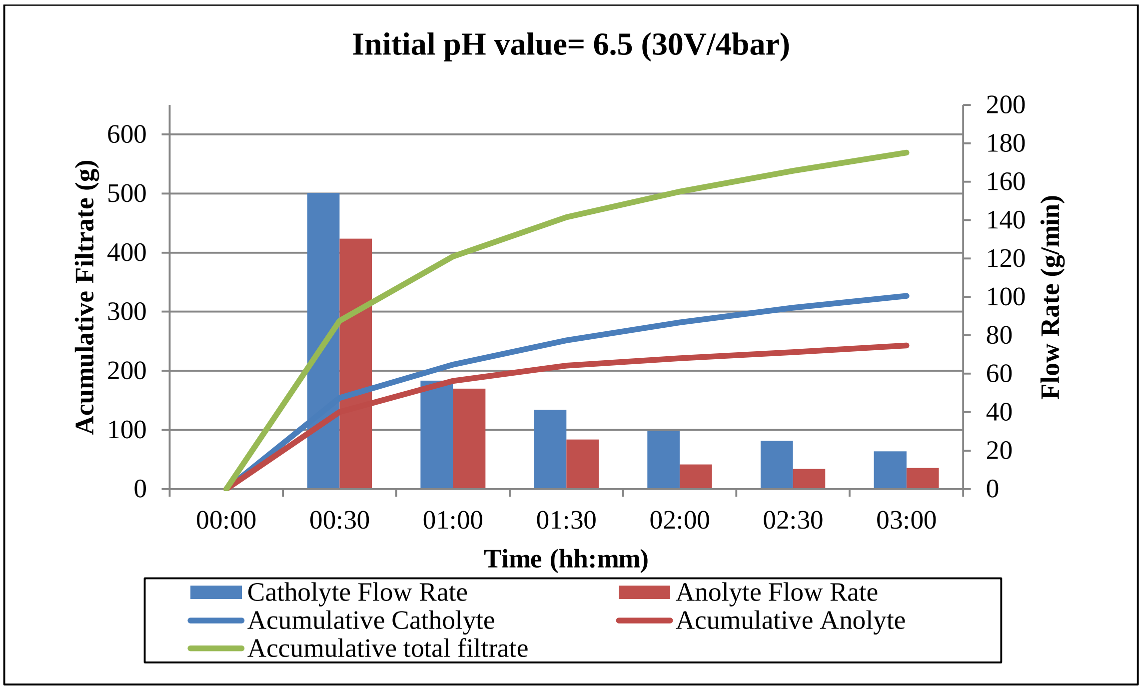

Figure 4.16. Change in OJ accumulative filtrates and filtrate flow rate over time under the application of EKEF with initial pH value=6.5 (normal orientation, 30V, 4bar,1μ filter and 3hours duration)

4.3.4. The Impact of the Applied Voltage and pressure

Applying voltage plays a role in WPD as water starts to move towards one of the electrodes after energising the electric field. In addition, voltage has an impact on the total filtrate weight and the EKEF dewatering efficiency. To investigate this impact, experiments on OJ and ME were run as described in *Section 3.5.3.4*; the results are shown in *Table 4.7* and *4.8* for OJ and ME respectively.

The data shows that there was a positive net dewatering efficiency (NED) after applying EKEF of up to 7.43% in OJ experiments and to 10.68% in ME experiments, this NED was significant higher with 30V compared with 15V under a pressure of 2 and 3bar as the total filtrate weight increased when the voltage was increased, however the difference between 15V and 30V was not significant under 4bar pressure (P-value=0.093). In addition, when voltage was applied the filtrate started moving towards the anode with (A/C ratio >1) in OJ samples, and towards the cathode with (A/C ratio <1) in the ME samples and this movement was larger with 30V applied compared with 15V, while the difference between the anolyte and catholyte was smaller under 0V (control) with a slight increase at the bottom outlet.

Table 4.7. Impact of voltage (0, 15 and 30V) on the EKEF dewatering efficiency (OJ, in the normal orientation, 5.5hours, 0.2μ filter, under the application of three pressure values (2, 3& 4bar))[5]

Experimental conditions	**Anolyte (g)**	**Catholyte (g)**	**Total Filtrate Weight (g)**	**Anolyte (%)**	**Catholyte (%)**	**A/C Ratio**	**Flow Rate (g/min)**	**Dewatering efficiency (%)**	
								Total	**Net**
At 2 bar									
Control (0V)	193.8(B)	173.1(T)	366.9	52.8	47.2	1.12	1.11	47.4	
15V	248.4	156.1	404.5	61.4	38.6	1.59	1.23	52.26	4.86
30V	244.1	169.1	413.2	59.1	40.9	1.44	1.25	53.39	5.99
At 3 bar									
Control (0V)	194.1(B)	192.7(T)	386.8	50.2	49.8	1.01	1.17	49.97	
15V	243.8	162.2	406	60	40	1.5	1.23	52.45	2.48
30V	259.4	167.6	427	60.7	39.3	1.55	1.29	55.17	5.2
At 4 bar									
Control (0V)	232.5(B)	229.2(T)	461.7	50.4	49.6	1.01	1.4	59.65	
15V	288	185.1	473.1	60.9	39.1	1.56	1.43	61.12	1.47
30V	327.2	192	519.2	63	37	1.7	1.57	67.08	7.43

Table 4.8. Impact of voltage (0, 15 and 30V) on the EKEF dewatering efficiency (ME, reversed orientation, 1μ filter, 3.5 hours, and a pressure of 4bar)

Experimental conditions	Anolyte (g)	Catholyte (g)	Total Filtrate Weight (g)	Anolyte (%)	Catholyte (%)	A/C Ratio	Flow Rate (g/min)	Dewatering efficiency (%)	
								Total	Net
Control (0V)	202.4(B)	170.80(T)	373.2	45.8	54.2	1.19	1.78	42.41	
15V	206.4	221.9	428.3	48.2	51.8	0.93	2.04	48.67	6.26
30V	220.50	246.70	467.2	47.2	52.8	0.89	2.22	53.09	10.68

The positive impact of voltage on the dewatering process is also represented by *Figures 4.17* to *4.19* which present the change in the total filtrate weight of OJ over time under the three different voltage conditions; each figure presents the results obtained under a different pressure. Change in the total filtrate weight of ME experiment over time under the three different voltage values and 4bar pressure is also presented in *Figure 4.20*.

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[5] Dewatering efficiency = weight of total filtrate/initial weight of water in the sample (take the initial moisture content as 86% for orange juice). Net dewatering efficiency is the total dewatering efficiency minus the efficiency of the control (without electricity). Weight of the total filtrate is the total weight of the discharged filtrate going out through the two outlets, and the flow rate is the total filtrate weight per minute (experiments were run for 5.5h).

In addition, means and P-values were calculated using One Way ANOVA, and shown in *Tables 4.9* and *4.10*, which show that the impact of the applied voltage on the total filtrate weight was significantly positive ($P<0.001$) in comparison with the control experiment. The final total filtrate weight was higher under 30V compared with 15V in both OJ and ME experiments. However, the mean value of OJ total filtrate over time was higher under 15V than its value under 30V in the OJ experiments, while it was higher under 30V in the ME experiments *(Figures 4.21* and *4.22)*.

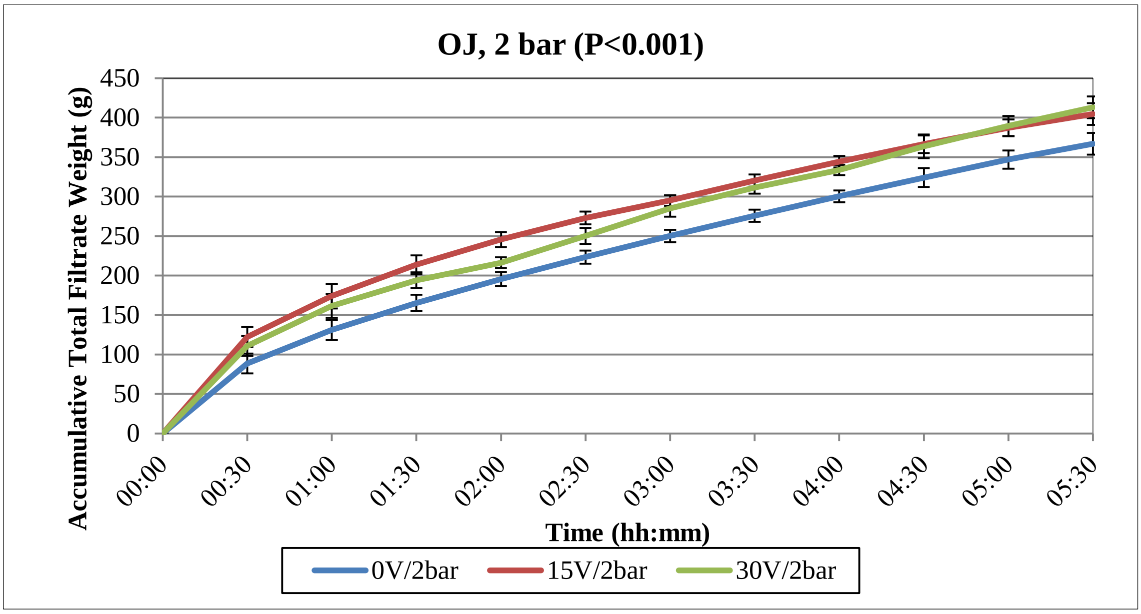

Figure 4.17. Change in OJ total filtrate over time under three voltage values (0, 15 &30V), (normal orientation, 5.5 hours, 0.2µ filter, and 2bar). Each value is expressed as mean ± standard deviation (n=3)

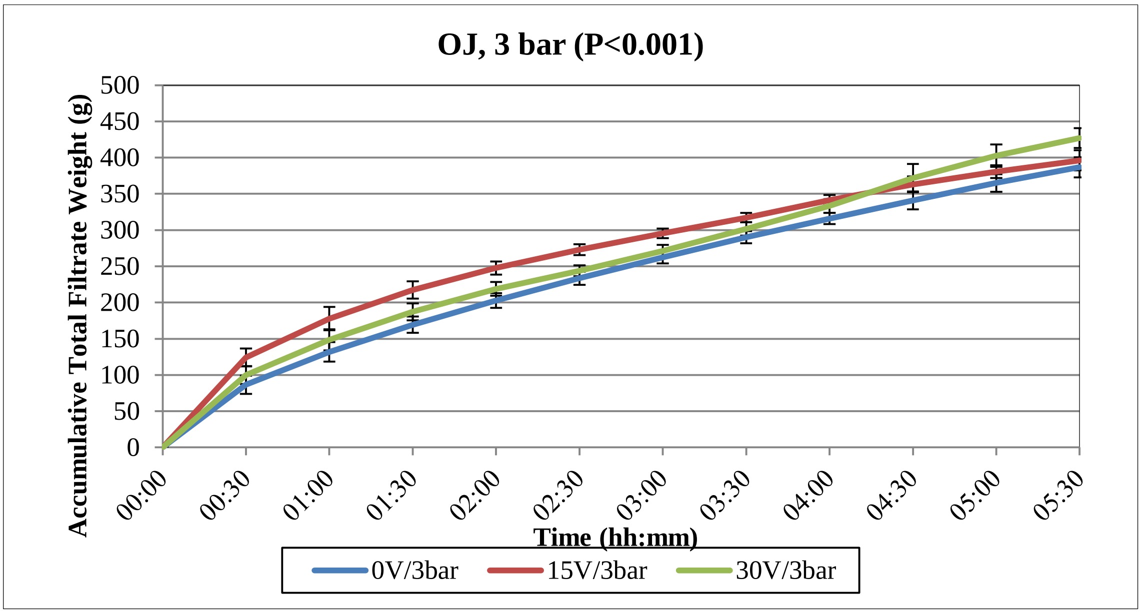

Figure 4.18. Change in OJ total filtrate over time under three voltage values (0, 15 &30V), (normal orientation, 5.5 hours, 0.2µ filter, and 3bar).Each value is expressed as mean ± standard deviation (n=3)

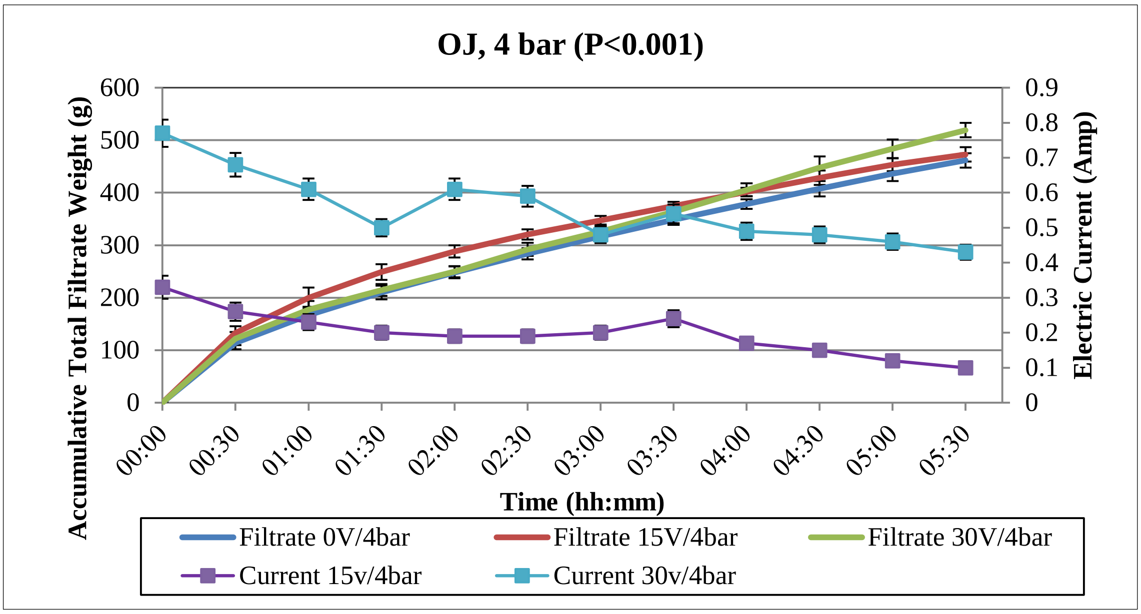

Figure 4.19. Change in OJ total filtrate over time under three voltage values (0, 15 &30V), (normal orientation, 5.5 hours, 0.2μ filter, and 4bar).Each value is expressed as mean ± standard deviation (n=3)

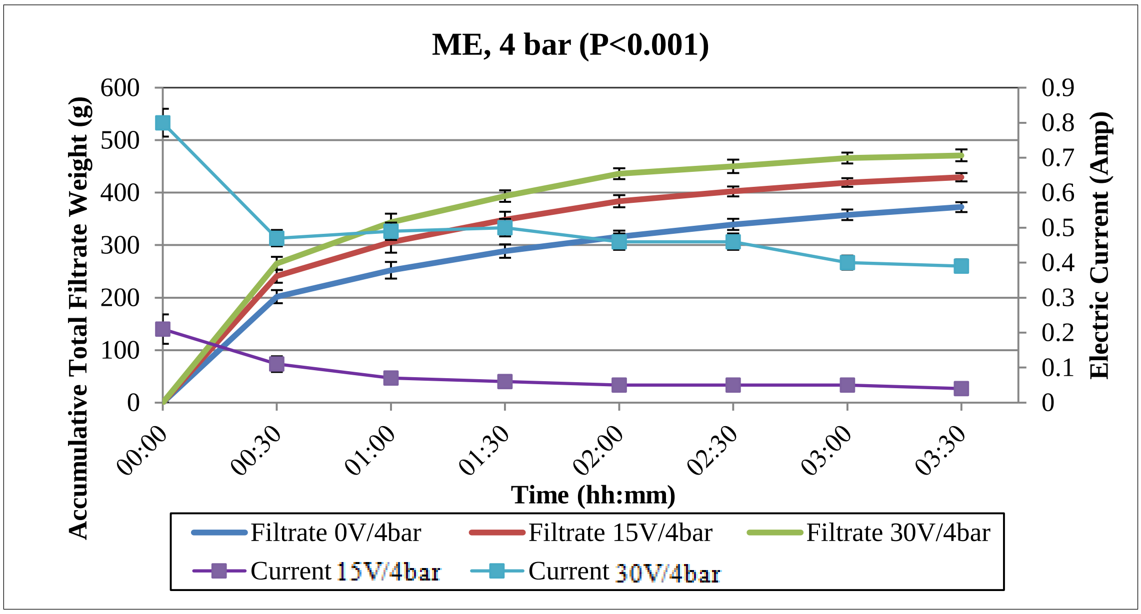

Figure 4.20. Change in ME total filtrate over time under three voltage values (0, 15 &30V), (reversed orientation, 3.5 hours, 1μ filter, and 4bar). Each value is expressed as mean ± standard deviation (n=3)

Table 4.9. P-values of the change of OJ total filtrate over time under three voltage values (0, 15 &30V), (normal orientation, 5.5hours, 0.2μ filter, and a pressure of 4bar)

	P-value of the accumulative total filtrate			
	0V-15V-30V	**0V-15V**	**15V-30V**	**0V-30V**
2bar	<0.001	<0.001	<0.001	<0.001
3bar	<0.001	<0.001	<0.001	<0.001
4bar	<0.001	<0.001	0.093	<0.001

Table 4.10. P-values of the change in ME total filtrate over time under three voltage values (0, 15 &30V), (reversed orientation, 3.5hours, 1μ filter, and a pressure of 4bar)

	P-value of the accumulative total filtrate			
	0V-15V-30V	**0V-15V**	**15V-30V**	**0V-30V**
4bar	<0.001	<0.001	<0.001	<0.001

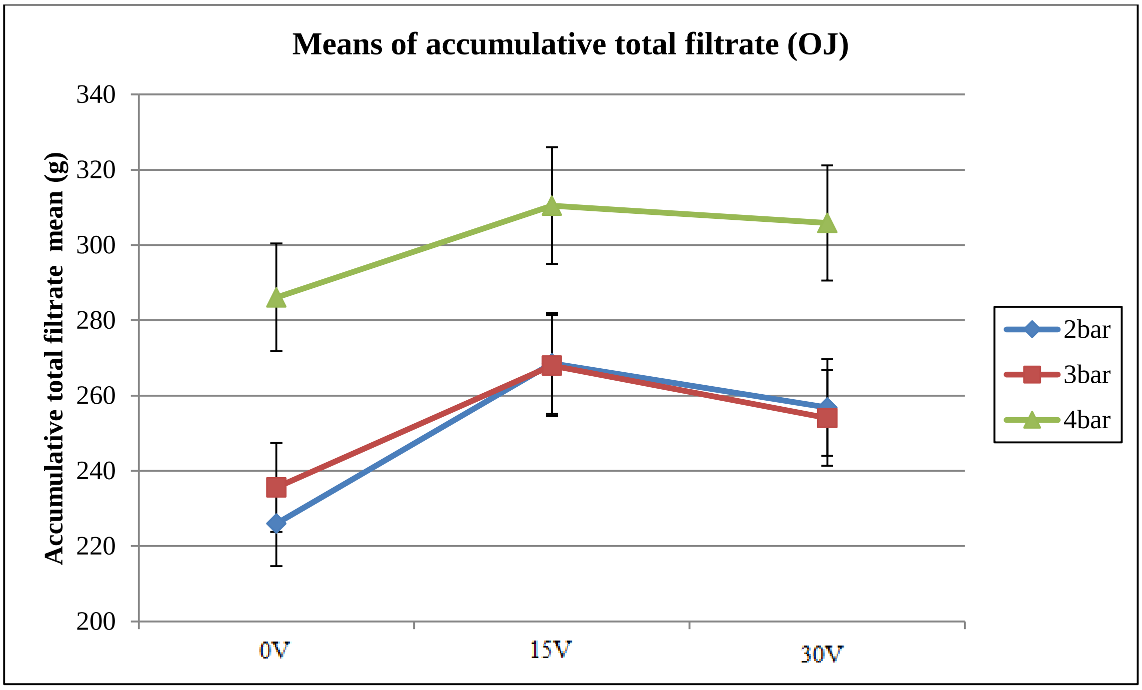

Figure 4.21. Means of the accumulative OJ total filtrate over time under three voltage values (0, 15 &30V), normal orientation, 5.5hours, 0.2μ filter, and (2,3&4bar) pressure

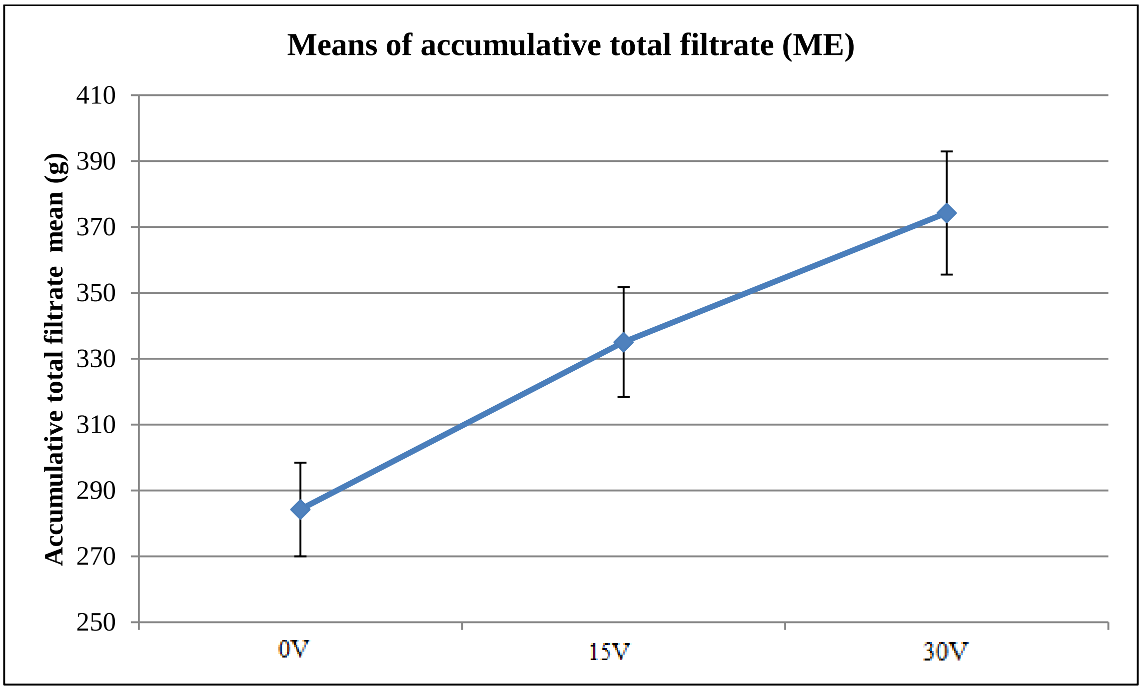

Figure 4.22. Means of the accumulative ME total filtrate over time under three voltage values (0, 15 &30V), (reversed orientation, 3.5hours, 1μ filter, and a pressure of 4bar)

Figures 4.17 to *4.19* show that during the processing of OJ, 30V achieved a higher outcome after 5.5hours running, but up to a specific time the outcome of applying 15V was greater before the 30V impact accelerated and crossed the 15V line in what is called the voltage crossing point (VCP). This phenomenon is also shown in *Figures 4.23* to *4.25* which show the time series plots of the flow rates under the three voltages and pressures.

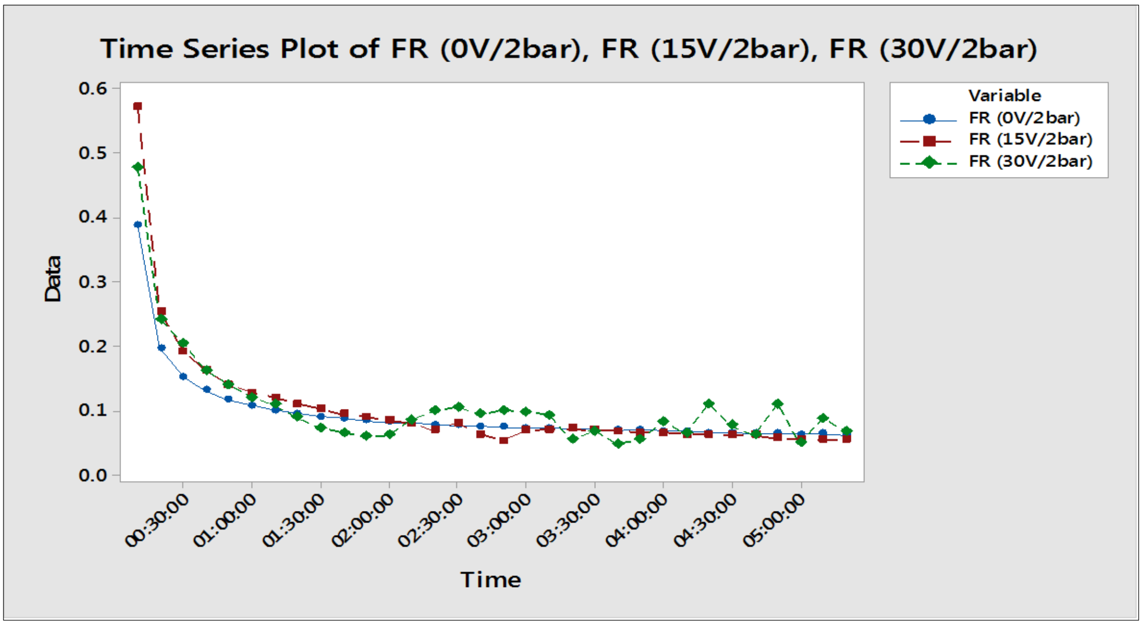

Figure 4.23. The Time series plot of the OJ filtrate flow rates under three voltage values (0, 15 &30V), (normal orientation, 5.5 hours, 0.2µ filter, and 2bar).Each value is expressed as mean ± standard deviation (n=3)

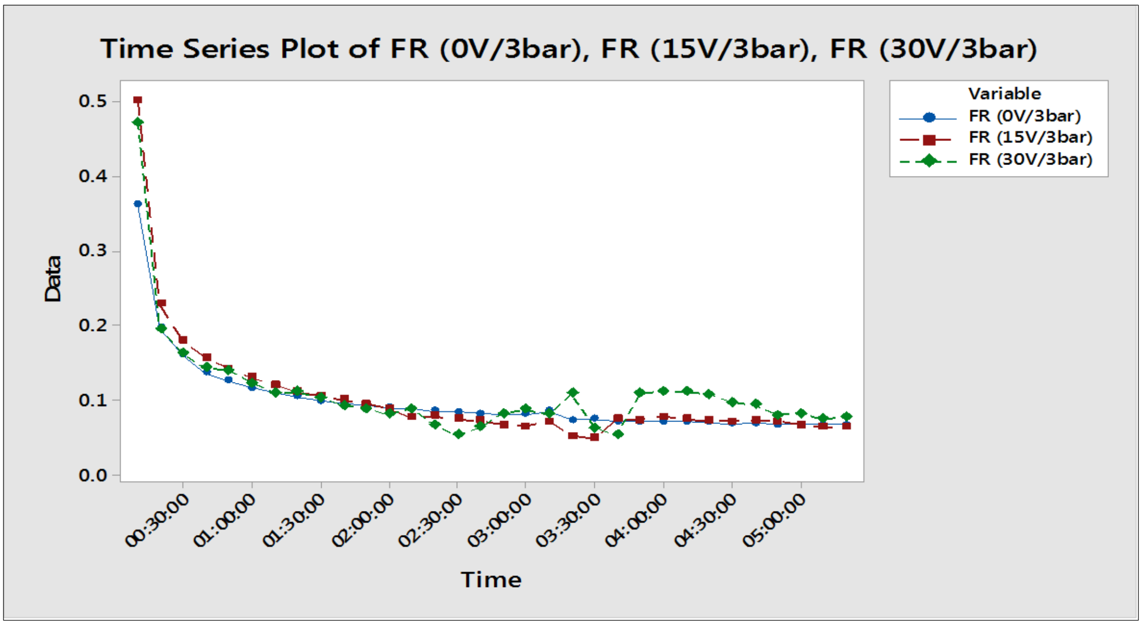

Figure 4.24. The Time series plot of the OJ filtrate flow rates under three voltage values (0, 15 &30V), (normal orientation, 5.5 hours, 0.2µ filter, and 3bar).Each value is expressed as mean ± standard deviation (n=3)

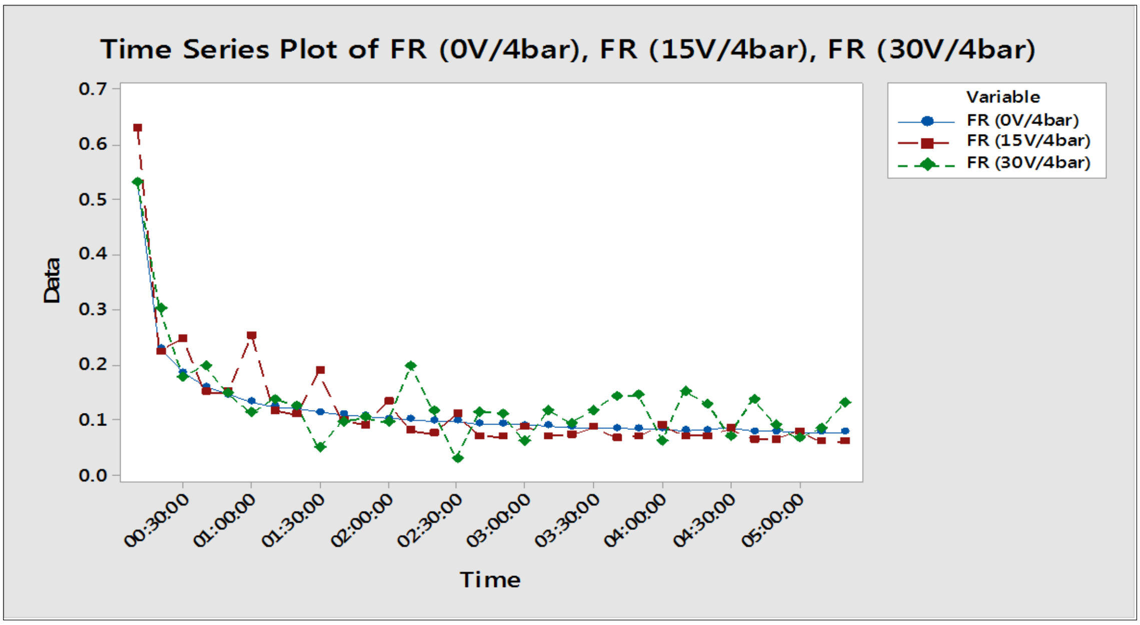

Figure 4.25. The Time series plot of the OJ filtrate flow rates under three voltage values (0, 15 &30V), (normal orientation, 5.5 hours, 0.2µ filter, and 4bar).Each value is expressed as mean ± standard deviation (n=3)

Using data in *Figures 4.17* to *4.19* the details of the VCP under the three pressures were obtained and shown in *Table 4.11,* these details are: time of VCP, the weight and the percentage of the total filtrate and the concentrate at this point as well as the moisture content of the concentrate and the distance between the electrodes.

Table 4.11. Voltage crossing points (VCP) properties under different pressures, (2, 3 and 4bar) normal orientation (0, 15 &30V, 4bar, 0.2µ and 5.5h duration)

Pressure (bar)	Time (hh:mm)	Total Filtrate (g)	Total Filtrate (%)	Concentrate (%)	Concentrate Dry matter (%)	Distance between Electrodes (mm)
2	~04:45	377	41.9	58.1	24.09	18.097
3	~04:15	351	39.0	61.0	22.95	18.997
4	~03:40	384	42.7	57.3	24.42	17.855

To investigate the improvement of using low voltage at the beginning then increasing it later, 15V was applied to an OJ sample for 3hours under 3bar pressure then increased later to 30V and then let to run for about 2.5 hours. Results were compared with the previous results of applying 0V, 15V and 30V with 3bar and are shown in *Figure 4.26*.

In the first three hours total filtrate weight was very close to the 15V total filtrate weight then later it accelerated after applying the 30V, acting better than 30V in the first 3hours and better than 15V in the last 2.5hours.

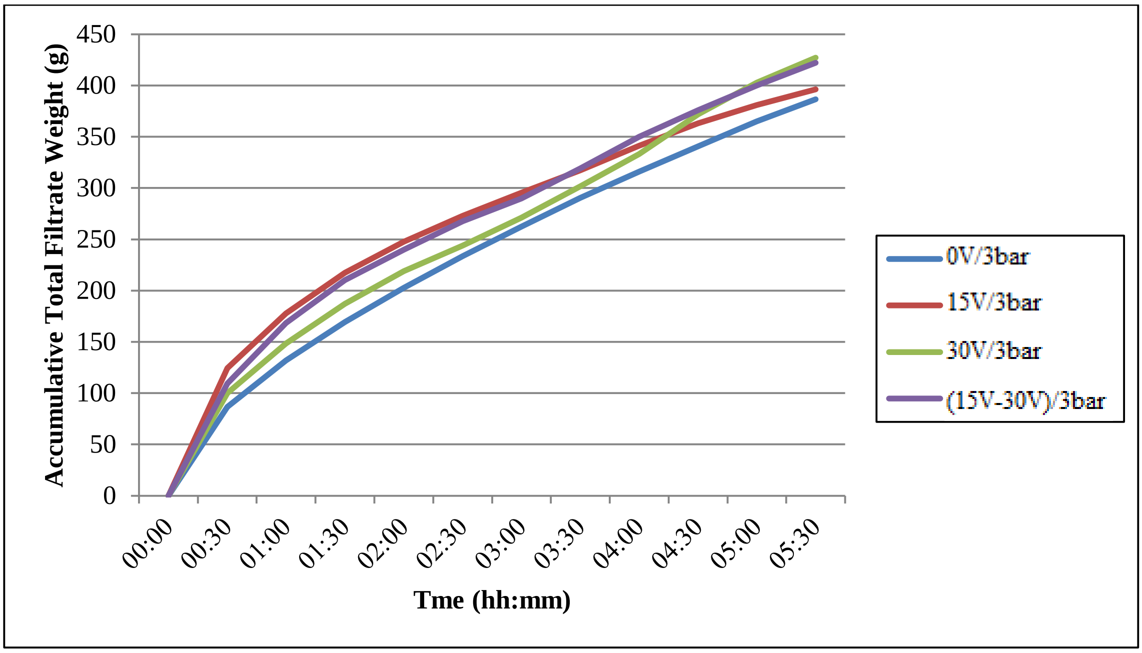

Figure 4.26. Change in OJ total filtrate over time under gradual voltage increase in comparison with constant 0V, 15V and 30V normal orientation (3bar, 0.2µ and 5.5h duration)

The impact of pressure on the dewatering process was also investigated according to *Section 3.5.3.5*. *Table 4.7* shows also the change in the discharged filtrate weight over time under the three different pressure values 2, 3 and 4bar. Looking at this table, increasing applied pressure increased the flow rate over time and improved the net dewatering efficiency by up to 13.69%.

Means and P-values were and shown in *Table 4.12* and *Figure 4.27*.

The impact of increasing pressure was significantly higher with 4bar compared with 3 and 2bar in all experiments ($P<0.000$). However, the pressure impact was significant only between 2 and 3bar in the control experiment while it was not significant when the voltage was applied.

Table 4.12. P-values of the change in OJ total filtrate over time under three pressure values (2, 3 &4bar), normal orientation, (0, 15&30V), duration of 5.5hours and 0.2µ filter)

	P-value			
	2-3-4bar	**2-3bar**	**3-4bar**	**2-4bar**
0V	<0.001	<0.001	<0.001	<0.001
15V	<0.001	0.777	<0.001	<0.001
30V	<0.001	0.232	<0.001	<0.001

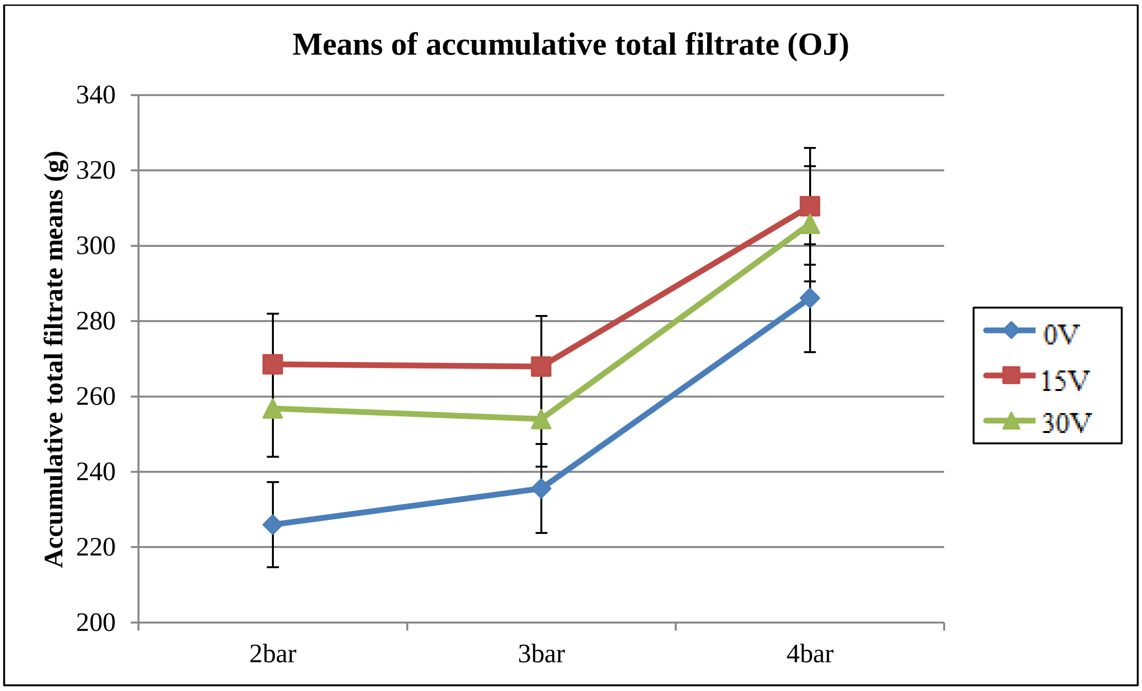

Figure 4.27. Means of the accumulative OJ total filtrate over time under three pressure values (2, 3 &4bar), normal orientation, (0, 15&30V), duration of 5.5hours and 0.2µ filter)

4.3.5. The Impact of Filtration Type (Filter Pores Size)

In addition to previous conditions, filter pore size can also affect the filtration process outcome and efficiency. In order to investigate how filter pores size affected the EKEF process, the method described in *Section 3.5.3.6* was used and results are shown in *Tables 4.13* and *4.14*.

The change of the accumulative total filtrate under previous conditions is also shown in *Figures 4.28* and *4.30*, P-values and total filtrate means were calculated by – to show the significance of the effects, these values are shown in *Figures 4.29, 4.31* and *Tables 4.15* to *4.18*.

Comparing the net dewatering efficiency (NDE) between the two sizes of the filter in *Tables 4.13* and *4.14, Figures 4.28* and *4.30*, it can be seen that the larger the filters pore size the higher the NDE. NDE was 1.47% under 15V and 7.43% under 30Vin the OJ experiments and 7.81% under 30V in the ME experiments with 0.2µ filters, while it increased to 11.07% under 15V and 19.37% under 30V in OJ experiments and 10.68% under 30V in ME experiments when 0.2µ filters were replaced with 1µ filters. The impact of the filter pore size was significant (P<0.005) as shown in *Figures 4.29, 4.31* and *Tables 4.15* to *4.18*.

Table 4.13. Impact of filters pore size (0.2µ and 1µ) on the EKEF dewatering efficiency (OJ, normal orientation, 0, 15 & 30V, a pressure of 4bar, and a duration of 5.5h)

Experimental conditions	Anolyte (g)	Catholyte (g)	Filtrate Weight (g)	Anolyte (%)	Catholyte (%)	A/C Ratio	Flow Rate (g/min)	Dewatering efficiency (%)	
								Total%	Net%
0.2 μ Filter									
Control (0V)	232.5(B)	229.2(T)	461.7	50.36	49.64	1.01	1.4	59.65	
15V	288	185.1	473.1	60.88	39.12	1.56	1.43	61.12	1.47
30V	327.2	192	519.2	63.21	36.79	1.71	1.57	67.08	7.43
1 μ Filter									
Control (0V)	267(B)	254.3(T)	521.3	51.22	48.78	1.05	1.58	67.35	
15V	373.49	233.51	607	61.53	38.47	1.6	1.84	78.42	11.07
30V	376.8	294.4	671.2	56.14	43.86	1.28	2.03	86.72	19.37

Table 4.14. Impact of filters pore size (0.2μ and 1μ) on the EKEF dewatering efficiency (ME, reversed orientation, 0 & 30V, a pressure of 4bar, and a duration of 2.5h)

Experimental conditions	Anolyte (g)	Catholyte (g)	Filtrate Weight (g)	Anolyte (%)	Catholyte (%)	A/C Ratio	Flow Rate (g/min)	Dewatering efficiency (%)	
								Total	Net
0.2 μ Filter									
Control (0V)	95.50(B)	70.20(T)	165.70	57.63	42.37	1.36	1.10	20.71	
30V	98.90	129.30	228.20	43.34	56.66	0.76	1.52	28.53	7.81
1 μ Filter									
Control (0V)	185.4(B)	154.30(T)	339.70	45.42	54.58	1.20	2.26	42.46	
30V	220.5	246.7	467.2	47.2	52.8	0.89	2.22	53.09	10.68

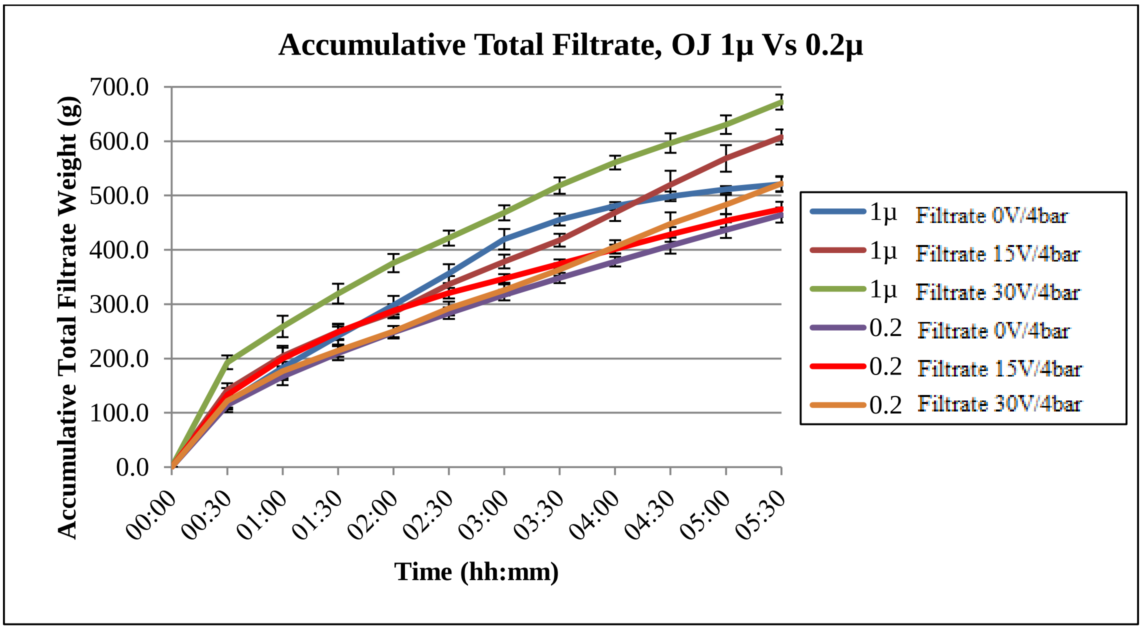

Figure 4.28. Impact of filters pore size (0.2μ and 1μ) on OJ accumulative total filtrate (normal orientation, 0, 15 & 30V, 4bar pressure, and a duration of 5.5h). Each value is expressed as mean ± standard deviation (n=3)

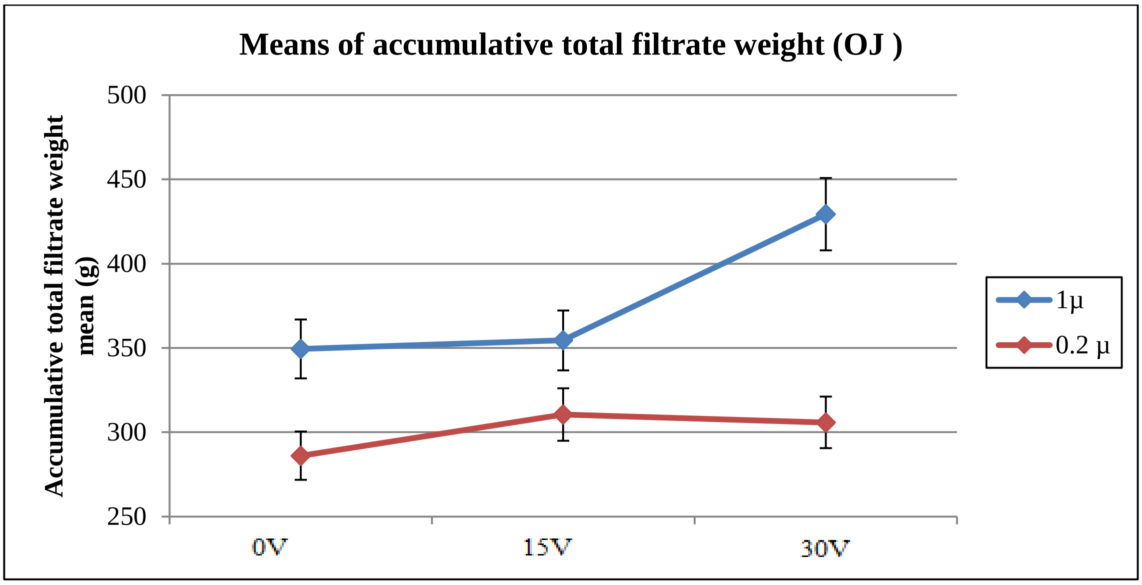

Figure 4.29. Means of the accumulative OJ total filtrate over time using two filter sizes (0.2μand 1μ) (normal orientation, 0, 15 & 30V, 4bar pressure and a duration of 5.5h)

Table 4.15. P-values of the change in OJ total filtrate over time under three voltages values (0, 15& 30V) under the same filter sizes (0.2μor 1μ), (normal orientation, 4bar pressure and duration of 5.5hours)

		P-value of the accumulative total filtrate			
		0V-15V-30V	**0V-15V**	**15V-30V**	**0V-30V**
0.2μ	**4bar**	<0.001	<0.001	0.093	<0.001
1μ	**4bar**	<0.001	0.191	<0.001	<0.001

Table 4.16. P-values of the change in OJ total filtrate over time using two filter sizes (0.2μand 1μ) under the same voltage value (0, 15 or 30V), (normal orientation, 4bar pressure and duration of 5.5hours)

P-value of the accumulative total filtrate		
0V(0.2μ)-0V(1μ)	**15V(0.2μ) -15V(1μ)**	**30V(0.2μ) -30V(1μ)**
<0.001	<0.001	<0.001

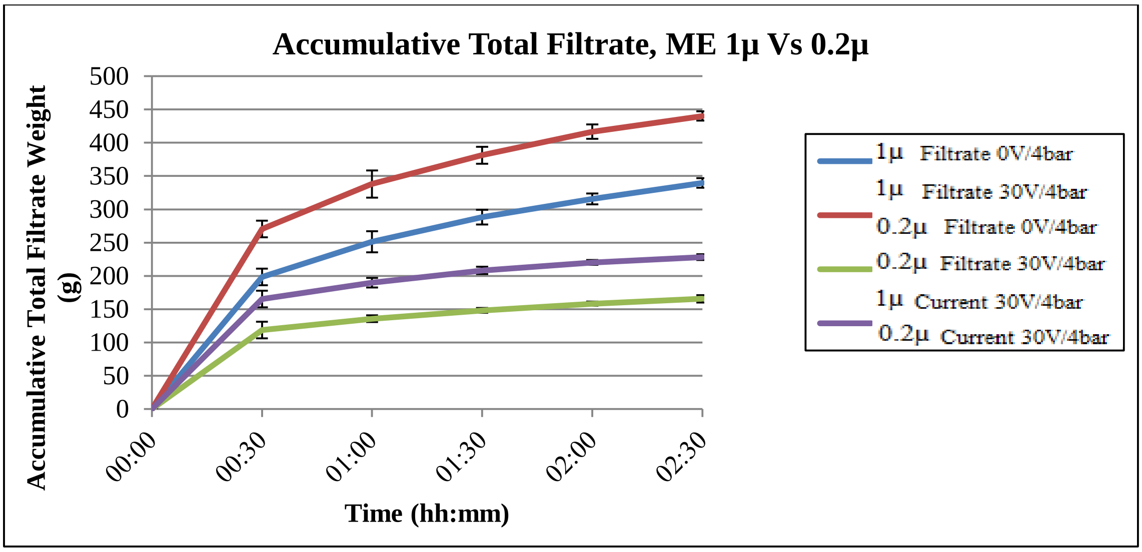

Figure 4.30. Impact of filters pore size (0.2μ and 1μ) on ME accumulative total filtrate and electric current intensity (reversed orientation, 0 & 30V, 4bar and 2.5h). Each value is expressed as mean ± standard deviation (n=3)

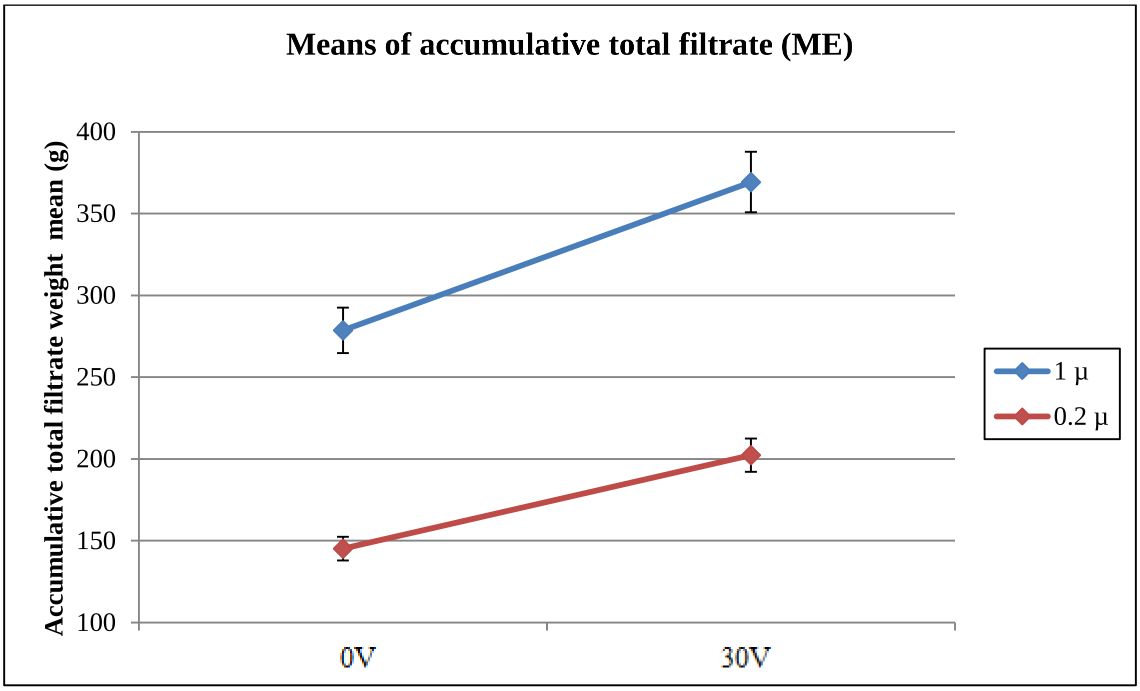

Figure 4.31. Means of the accumulative ME total filtrate over the time using two filter sizes (0.2µ and 1µ) (reversed orientation, 0 & 30V, 4bar pressure and 2.5h duration)

Table 4.17. P-values of the change in ME total filtrate over time under two voltages values (0& 30V) under the same filter sizes (0.2µor 1µ), (reversed orientation, 4bar pressure and 2.5hr duration)

		P-value of the accumulative total filtrate			
		0V-15V-30V	**0V-15V**	**15V-30V**	**0V-30V**
0.2µ	**4bar**	<0.001	<0.001	<0.001	<0.001
1µ	**4bar**	<0.001	<0.001	<0.001	<0.001

Table 4.18. P-values of the change in ME total filtrate over time using two filter sizes (0.2µ and 1µ) under the same voltage value (0 or 30V), (reversed orientation, 4bar pressure and 2.5hr duration)

P-value of the accumulative total filtrate		
0V(0.2µ)-0V(1µ)	**15V(0.2µ) -15V(1µ)**	**30V(0.2µ) -30V(1µ)**
<0.001	<0.001	<0.001

4.3.6. Impact of the Initial Thickness of the Sample (the Distance Between the Electrodes)

The impact of three different sample volumes (0.5, 1 & 1.5L), producing sample thickness of 17.3, 34.6 & 51.9mm, was measured according to method in *Section 3.5.3.7*, results are shown in *Table 4.19*; The change of the accumulative total filtrate and electric current under previous conditions is also shown in *Figure 4.32*.

Table 4.19 and *Figure 4.32* show that the larger the initial sample thickness the smaller the EKEF dewatering efficiency as the NED dropped from 21.34% in the 0.5L volume sample to 7.75% and 2.03% in 1L and 1.5L volumes respectively. In addition, *Figure 4.32* shows that the electric current intensity was higher with smaller thickness.

Table 4.19. Impact of sample's initial thickness (0.5, 1 & 1.5L) on the EKEF dewatering efficiency of ME (reversed orientation, 0 & 30V, 4bar pressure, 0.2 µ, 2hr duration)

Experimental conditions	Anolyte (g)	Catholyte (g)	Total Weight of Filtrate (g)	Anolyte (%)	Catholyte (%)	A/C ratio	Flow Rate (g/min)	Dewatering efficiency (%) Total	Dewatering efficiency (%) Net
0.5L: 17mm									
Control (0V)	68.1	72.8	140.9	48.3	51.7	0.9	1.17	32.62	
30V	103.7	129.4	233.1	44.5	55.5	0.8	1.94	53.96	21.34
1L: 34.5mm									
Control (0V)	90.7(T)	67.5(B)	158.2	57.3	42.7	1.3	1.32	19.78	
30V	98.6	121.6	220.2	44.8	55.2	0.8	1.84	27.53	7.75
1.5L: 52mm									
Control (0V)	98.6(T)	61.7(B)	160.3	61.5	38.5	1.6	1.34	13.36	
30V	88.8	95.9	184.7	48.1	51.9	0.9	1.54	15.39	2.03

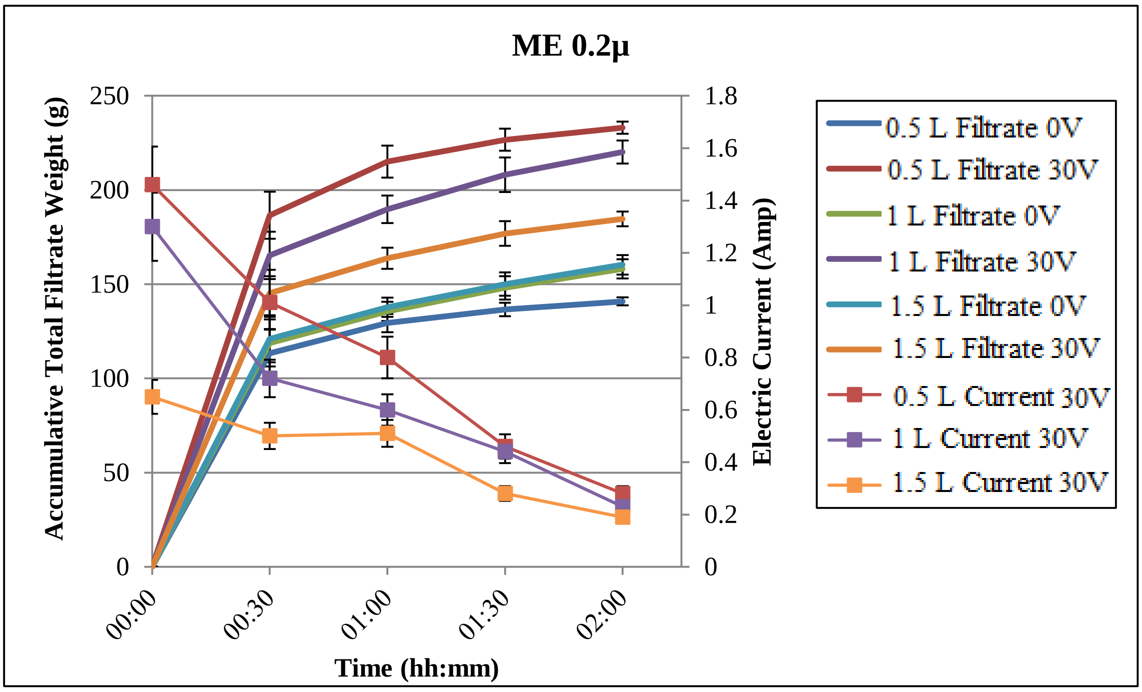

Figure 4.32. Impacts of the sample's initial thickness (0.5, 1 & 1.5L) on ME accumulative total filtrate and electric current intensity (reversed orientation, 0 &30V, 4bar pressure, 2hr duration). Each value is expressed as mean ± standard deviation (n=3)

4.4. EKEF Process Impact on the Sample Properties

The following experiments were run to investigate the potential impact of EKEF on the chemical properties of the samples including pH value, vitamin C content, colour, and dry matter content.

4.4.1. Gas Generation

Gas generation occurs next to the electrodes as shown in *Equations 2 & 4* where two types of gases are generated: oxygen at the anode and hydrogen at the cathode, and then these gas bubbles gathered to create bubble clusters which flowed out from the top outlet (*Figure 4.33*).

Figure 4.33. The bubbles clusters flow out from the top outlet

Table 4.20 presents gas generation time (from experiment start to the first appearance of the gas bubbles) in OJ experiments under different voltages and pressure values. Data in this table show that gas generation time and amount depends mainly on the applied voltage value; the higher the voltage the sooner and the larger the gas generation which explained the earlier time and the bigger fluctuation in the conductivity value under 30V compared with 15V.

Table 4.20. Gas generation time (from experiment start to the first appearance of the gas bubbles) in OJ experiments under different voltages and pressure values (to the result)

Experiment	Voltage (V)	Pressure (bar)	Time of Gas first appearance (min)
Exp1	30	2	27
Exp2	15	2	145
Exp3	0	2	-
Exp4	30	3	30
Exp5	15	3	190
Exp6	0	3	-

4.4.2. pH Change

Figure 4.34 presents the change in pH value of Anolyte, Catholyte and the total filtrate of OJ experiments over time under two voltage values (15V and 30V) and the control (0V), normal orientation was used (anode is the bottom electrode).

The initial pH value of OJ samples was around 3.65; this value was stable over the whole process in the case of not applying any EKEF (the control orientation); but it started to change during the experiment when a voltage was applied; Increasing at the cathode (the top electrode) with a maximum value of 10.87 for the catholyte under 15V and 11.77 under 30V. On the other hand, the pH value under EKEF dropped at the anode (the bottom electrode) with a minimum value of 2.58 for the anolyte under 15V and 2.21 under 30V. However, the pH value of the total filtrate (the anolyte and the catholyte combined together) showed a maximum value of 5.21under 15V and 6.74 under 30V.

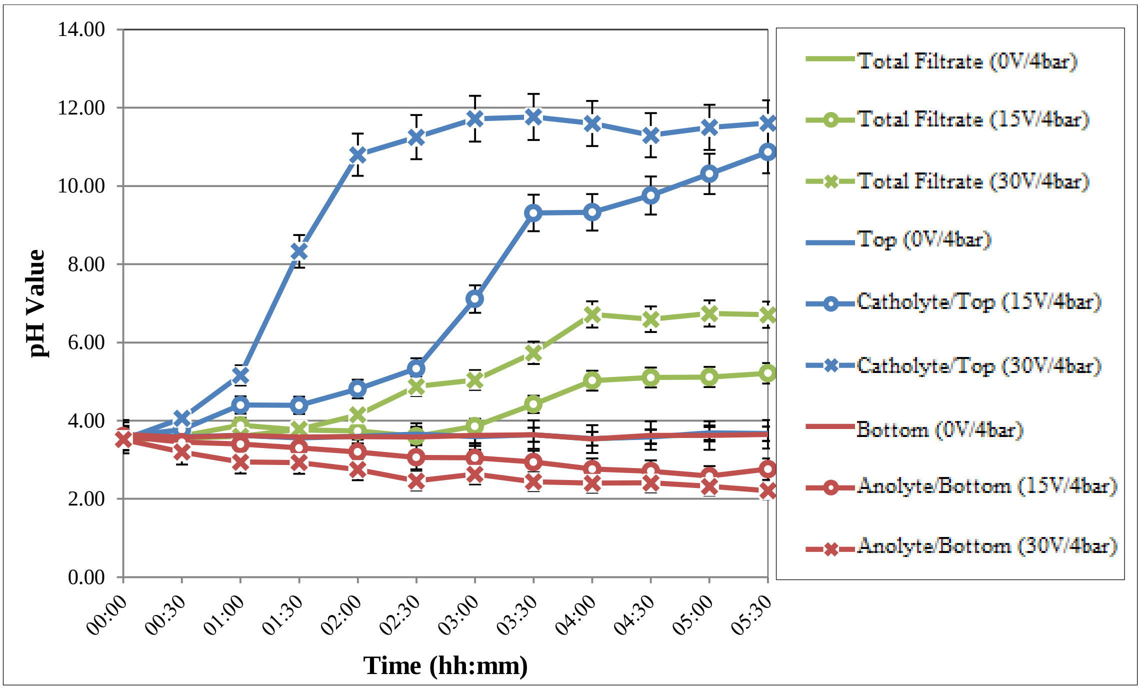

Figure 4.34. Change in pH value of (Anolyte, Catholyte and the total filtrate) of OJ experiments over time under three voltage values (0, 15 & 30V) (normal orientation, 4bar pressure, 0.2μ and 5.5hr duration). Each value is expressed as mean ± standard deviation (n=3)

The same experiment was repeated on ME sample, and monitoring the change in the Anolyte, Catholyte and the total filtrate pH value over time under the reversed orientation (cathode is the bottom electrode) as shown in *Figure 4.35*.

In the ME experiments, the pH value of the anolyte, catholyte and total filtrate changed over time very similarly to OJ pH. Under control experiments (voltage =0V), the pH value stayed stable around the fresh sample pH value of 4.55, but after applying EKEF, the pH value started to change during the experiment; Increasing in the catholyte (the bottom filtrate) with a maximum value of 10.94 under 15V and 11.3 under 30V. On the other hand, the anolyte (the top filtrate) pH value under EKEF dropped with a minimum value of 2.56 under 15V and 1.87

under 30V. However, the pH value of the total filtrate (the anolyte and the catholyte combined together) showed a maximum value of 8.80 under 15V and 9.31 under 30V.

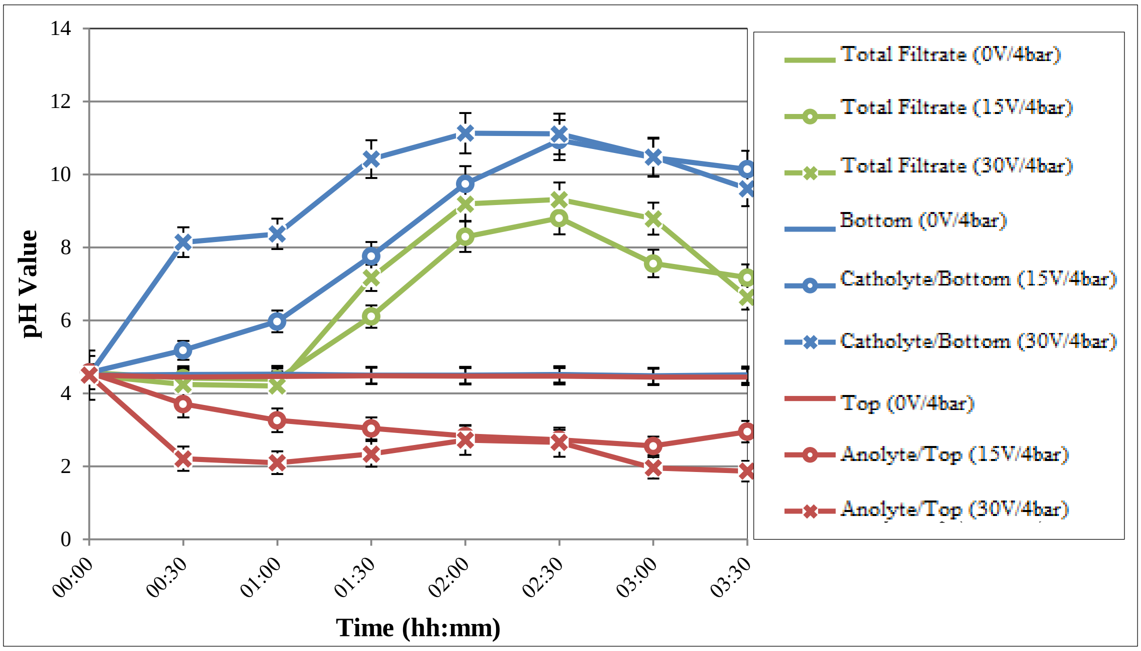

Figure 4.35. Change in pH value of (Anolyte, Catholyte and the total filtrate) of ME experiments over time under three voltage values (0, 15 & 30V) (reversed orientation, 4bar pressure, 1µ and 3.5hr duration)

Figures 4.36 and 4.37 present the final pH value of the total filtrate and final concentrate of OJ with ME respectively, comparing with fresh samples under (0V, 15V and 30V); change in the pH value of the concentrate under different voltages was not significantly different compared with the fresh sample pH. However the total filtrate pH value was clearly higher than the fresh one.

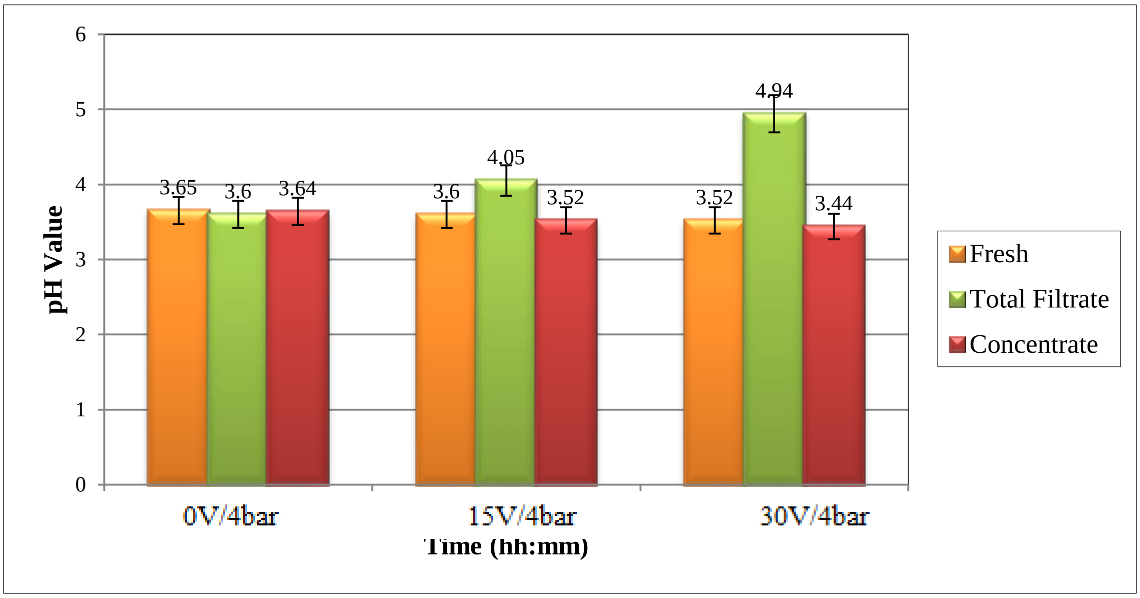

Figure 4.36. Final pH value of the OJ total filtrate and final concentrate comparing with fresh sample under three voltage values (0, 15 & 30V) normal orientation (4bar pressure, 0.2µfilter, 5.5hr duration). Each value is expressed as mean ± standard deviation (n=3)

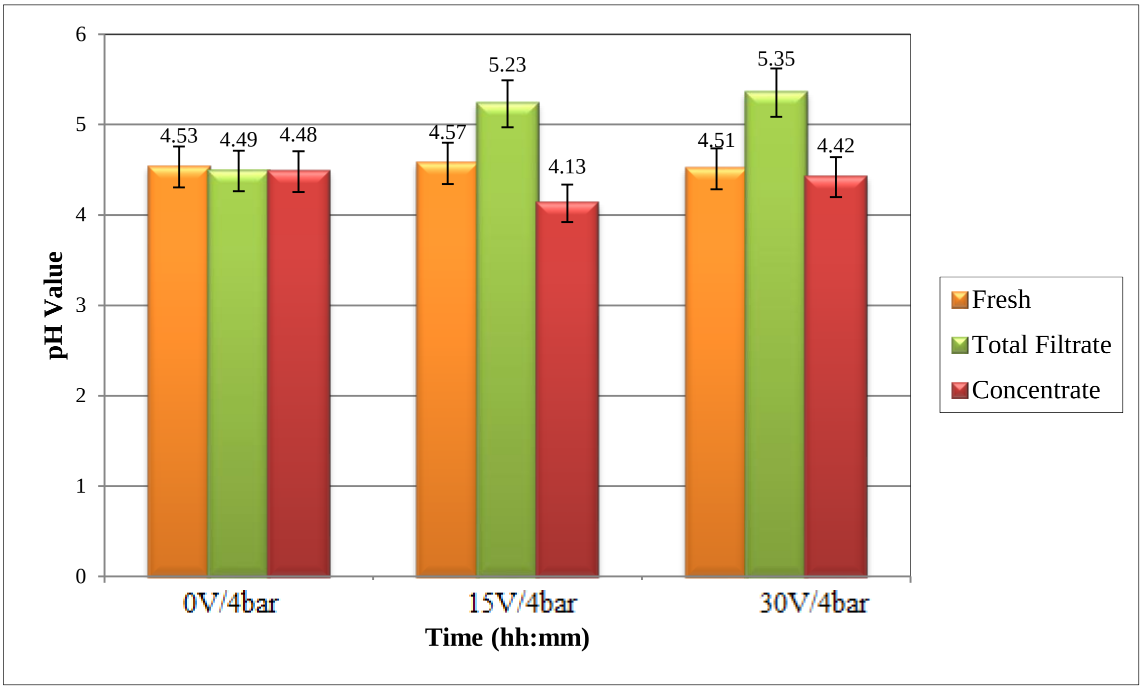

Figure 4.37. Final pH value of the ME total filtrate and final concentrate comparing with fresh sample under three voltage values (0, 15 & 30V) reversed orientation (4bar pressure, 1µ filter, 3.5hr duration). Each value is expressed as mean ± standard deviation (n=3)

In addition to the previous data, the correlation between pH change and the flow rate change over the time was calculated using *Pearson correlation,* and *Table 4.21*shows this significant relationship (see *Appendices 5* for more details).

Table 4.21. Person correlation between pH change and the flow rate change over the time

Sample	pH & FR
Catholyte (15v/4bar)	-0.634066293
Anolyte (15v/4bar)	0.77132684
Total Filtrate (15v/4bar)	-0.535820252
Catholyte (30v/4bar)	-0.603912256
Anolyte (30v/4bar)	0.865773256
Total Filtrate (30v/4bar)	-0.460697015

4.4.3. Vitamin C (Ascorbic Acid) Change

To compare between the EKEF process and the conventional methods for their impact on vitamin C content, several experiments were carried out on OJ samples according to the methods in *Section 3.5.5.2*. Samples were collected and vitamin C retentions were then calculated according to *Equation 10*.

The retention of vitamin C of the two juice parts (the final concentrated juice and the total filtrate) under (0, 15 and 30V) was compared with the initial vitamin C content. *Figure 4.38* shows that there was no degradation in vitamin C content of the control concentrated juice while the control total filtrate lost about 11% of its vitamin C content.

However, after applying EKEF for 5.5hours, vitamin C retention of the concentrated juice dropped to 41% under 15V and 7.14% under 30V. Also the retention of the total filtrate was influenced by the impact of the EKEF, where it dropped to 47.8% under 15V and 35.45% under 30V.

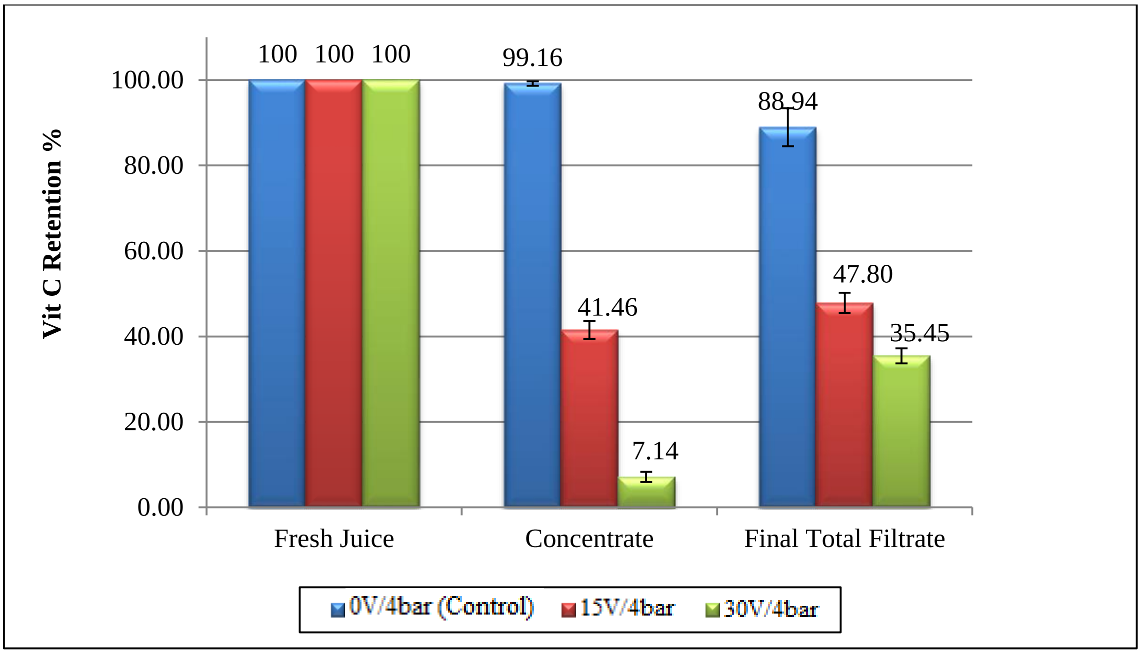

Figure 4.38. Vitamin C retention of OJ total filtrate and final concentrate compared to vitamin C content of fresh sample under three voltage values (0, 15 & 30V)(normal orientation, 4bar pressure, 0.2µ filter and 5.5hr duration). Each value is expressed as mean ± standard deviation (n=3)

Due to the design of the rig it was difficult to monitor vitamin C content of the concentrated juice over the experiment period as concentrated juice was retained inside the cell during the entire experiment. However, the vitamin C content of the total filtrate and its two components was monitored and analysed every half an hour.

Figure 4.39 shows that the vitamin C content of the total filtrate decreased to half (50%) after (2:50h) under 15V and (1:15h) under 30V compared with the initial values.

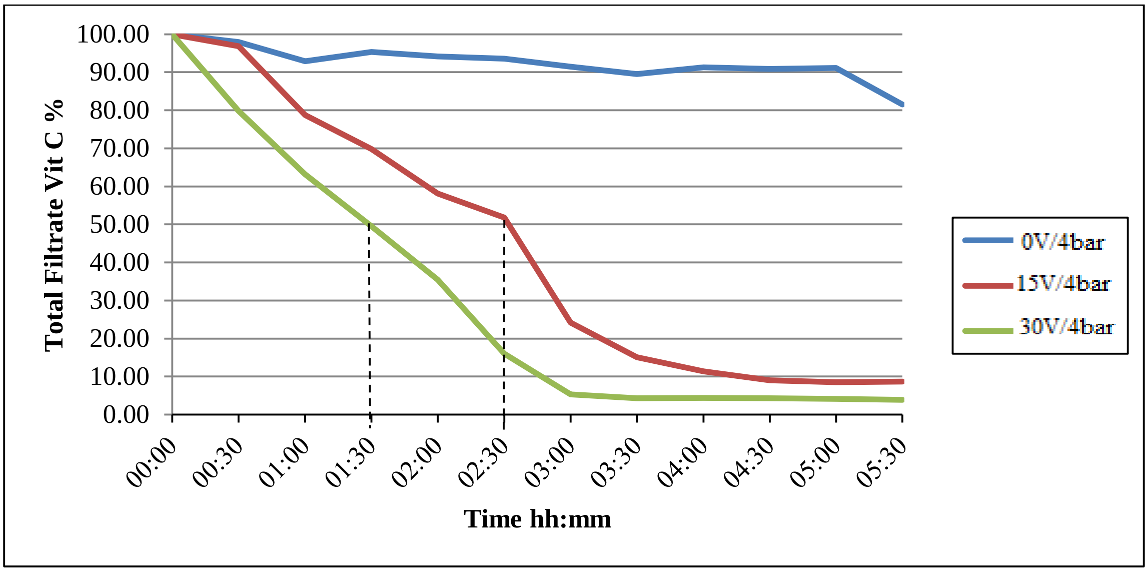

Figure 4.39. Change in vitamin C retention of OJ total filtrate over time under three voltage values (0, 15 & 30V) (normal orientation, 4bar pressure, 0.2µ filter and 5.5hr duration)[6]

To investigate whether the two electrodes were equal in terms of their impact on vitamin C, the change in the anolyte, catholyte and total filtrate vitamin C retention were measured over time under the following voltages, 0, 15 and 30V.

Figures 4.40 to *4.42* show that when EKEF was applied the main degradation of vitamin C content occurred in the catholyte which lost its content very rapidly in comparison with the anolyte. Since vitamin C retentions of the anolyte and catholyte are different, the volume percentage of each of them in the total filtrate affects the final vitamin C content in the total filtrate, as shown in *Table 4.22*.

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[6] This figure (*figure 4.39*) shows the change in the total filtrate vitamin C retention over the time while in the previous figure (*figure 4.48*) the value of the final total filtrate retention presents vitamin C retention of the whole amount of the total filtrate eluted over the experiment.

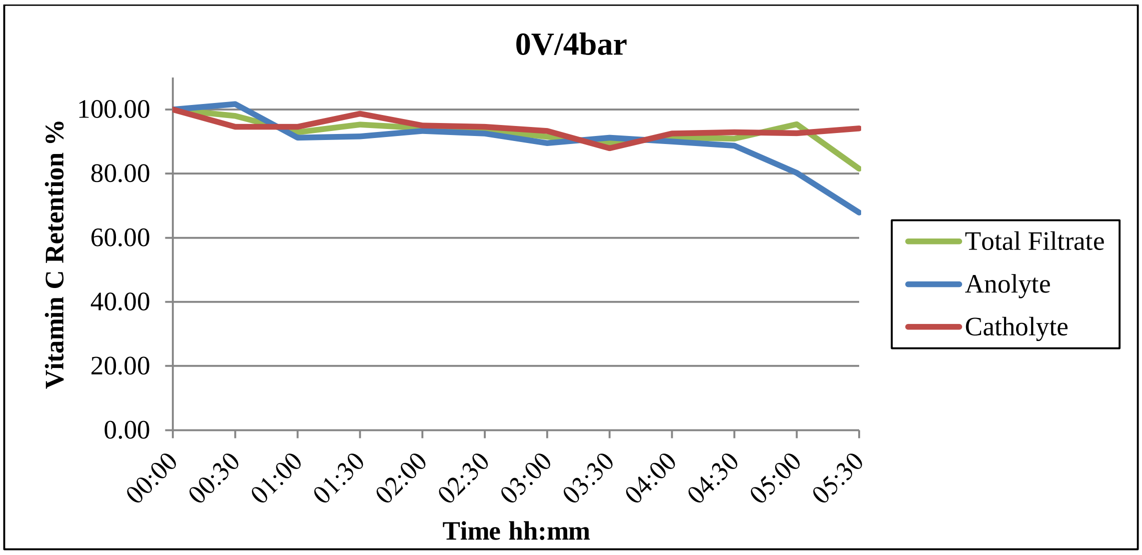


Figure 4.40. Change in anolyte, catholyte and total filtrate vitamin C retention over time under (control orientation, 0V, 4bar pressure, 0.2μ filter and 5.5hr duration)

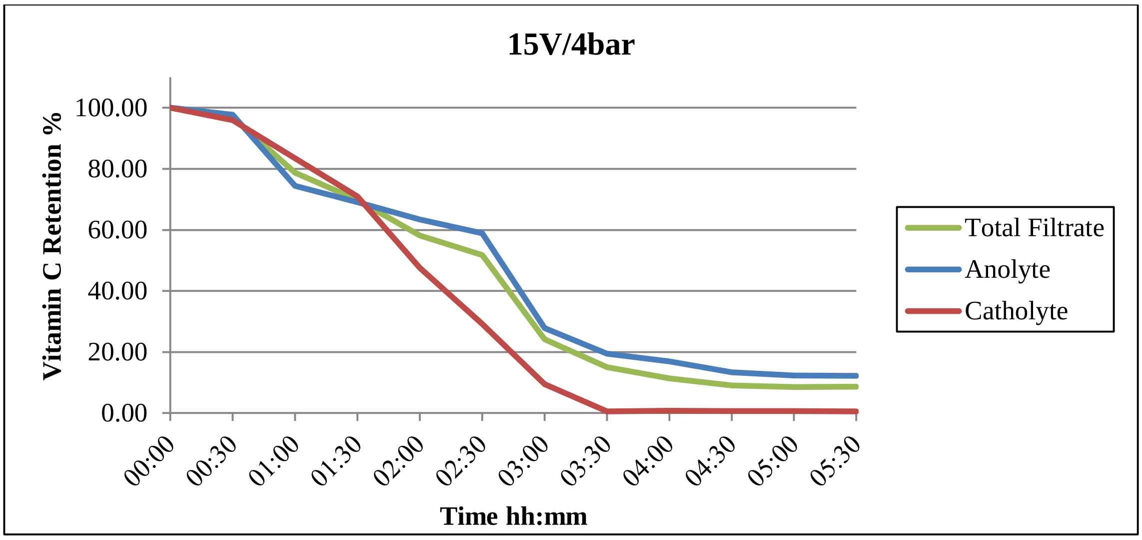


Figure 4.41. Change in anolyte, catholyte and total filtrate vitamin C retention over time under (normal orientation, 15V, 4bar pressure, 0.2μ filter and 5.5hr duration)

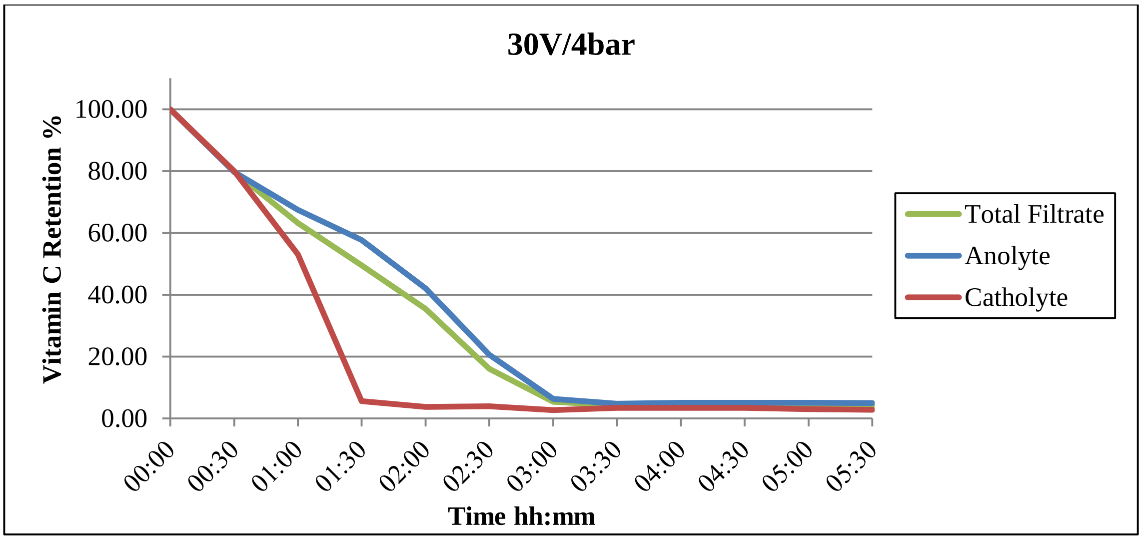


Figure 4.42. Change in anolyte, catholyte and total filtrate vitamin C retention over time under (normal orientation, 30V, 4bar pressure, 0.2μ filter and 5.5hr duration)

Table 4.22. Change in vitamin C retention (Vit. CR%) in OJ anolyte, catholyte and total filtrate (TF) in the first 3hours with respect for the percentage of anolyte and catholyte in the total filtrate (normal orientation, 0, 15 &30V, 4bar pressure, 0.2µ filter and 3hr duration)

	Time (hh:mm)								
	01:00			**02:00**			**03:00**		
	Control (0V)	**15V**	**30V**	**Control (0V)**	**15V**	**30V**	**Control (0V)**	**15V**	**30V**
TF Weight (g)	64	66.5	55.1	50	39	35.1	44.1	27.1	35.5
Anolyte %	51.41	51.88	70.42	51.40	66.67	82.62	51.70	80.07	73.52
Catholyte %	48.59	48.12	29.58	48.60	33.33	17.38	48.30	19.93	26.48
Anolyte Vit. C R%	91.22	74.45	67.45	93.31	63.44	42.11	89.55	27.86	6.30
Catholyte Vit. C R%	94.57	83.46	53.08	94.99	47.52	3.73	93.31	9.49	2.71
TF Vit. C R%	92.94	78.79	63.20	94.17	58.13	35.44	91.50	24.20	5.35

To investigate the impact of the filter pore sizes on the vitamin C content, experiments on OJ samples were run using 0.2µ and 1µ filters (normal orientation, 30V, 4bar and 5.5h duration) and compared with the contents under the control experiment (0.2µ, 0V, 4bar and 5.5h duration); *Figure 4.43* shows vitamin C contents of fresh juice, final concentrate and total filtrate under these experiments.

Using filters with a bigger pore size had a positive impact on the vitamin C retention of OJ total filtrate, as the vitamin C retention of the total filtrate going through the 1µ filters was 57.4% in comparison with 35.45% using 0.2µ filters after 5.5h of process.

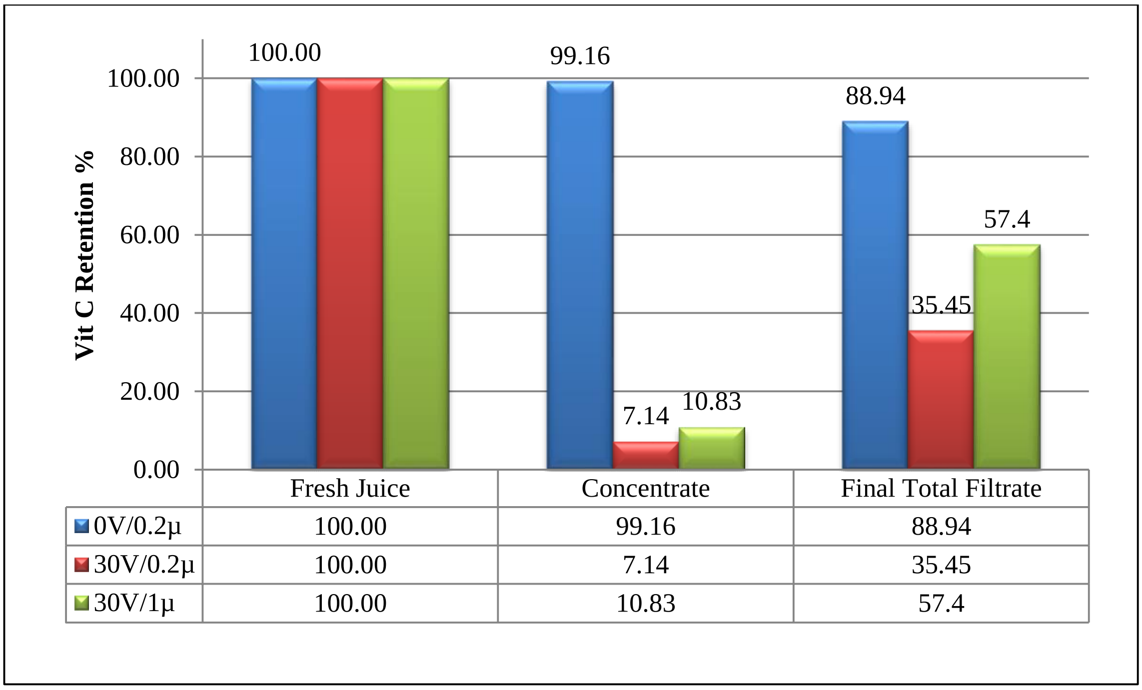


Figure 4.43. Vitamin C contents of fresh juice, final concentrate and total filtrate using two filters 0.2µ and 1µ (normal orientation, 30V, 4bar and 5.5hduration) in comparison with them under the control experiment (0.2µ, 0V, 4bar and 5.5hduration).

The change of total filtrate vitamin C retention over time was shown in *Figure 4.44*; the degradation of the 50% of vitamin C retention was after 1.30 hours using 0.2µ filters, while it was extended to 2 hours using a 1µ filters.

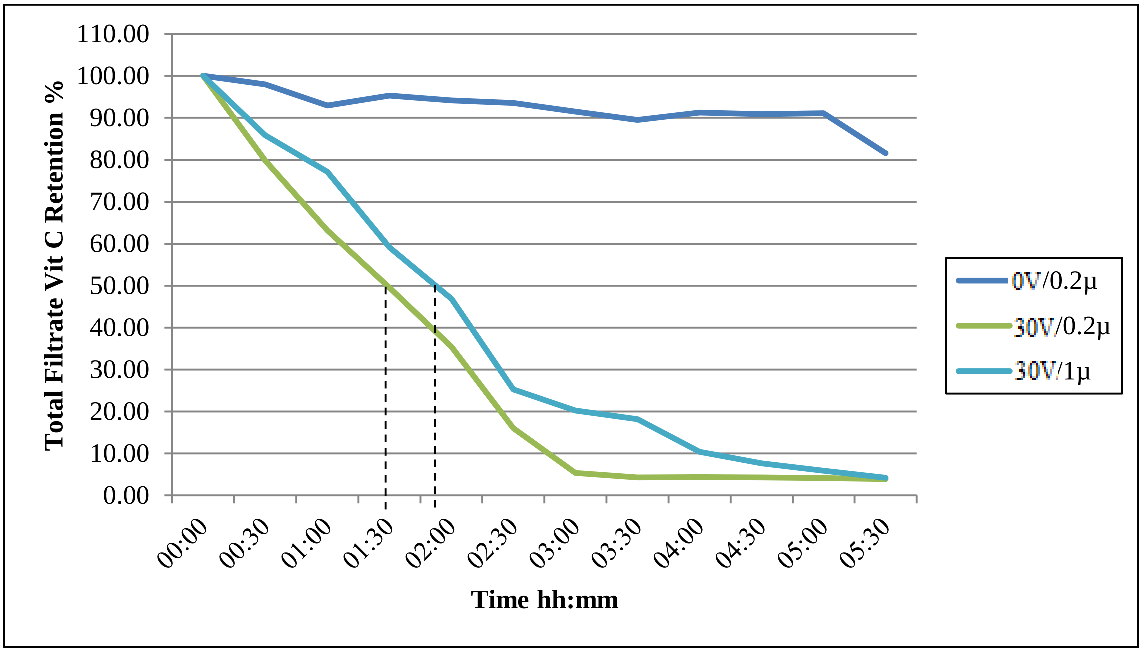


Figure 4.44. Change in vitamin C retention of OJ total filtrate using two filters 0.2µ and 1µ (normal orientation, 30V, 4bar and 5.5h duration) in comparison with the control experiment (0.2µ, 0V, 4bar and 5.5hduration).

To investigate the potential reasons behind the vitamin C loss, the correlation between the change of the vitamin C retention and the pH change was calculated using *Pearson correlation*, in addition the correlation between the change of vitamin C retention and flow rate change was checked in the anolyte, catholyte and total filtrate.

Table 4.23 shows that there is a significant relationship between pH change and the change of vitamin C retention as well as between vitamin C retention and the flow rate, although that the relationship with the flow rate change is less significant.

Table 4.23. The correlation of the vitamin C retention with pH change and the flow rate change

	pH & Vit C	Vit C & FR
Catholyte (15v/4bar)	-0.915561448	0.840017889
Anolyte (15v/4bar)	0.955441309	0.844178159
Total Filtrate (15v/4bar)	-0.841744547	0.829244011
Catholyte (30v/4bar)	-0.94929524	0.791428654
Anolyte (30v/4bar)	0.961743796	0.845705373
Total Filtrate (30v/4bar)	-0.86274467	0.726273699

However, *Figures 4.45* to *4.50* show that the maximum loss of vitamin C does not happen at the same time of the maximum change of pH, while *Figures 4.51* to *4.56* show that the maximum loss of vitamin C occurs when the flow rate is at its lowest value.

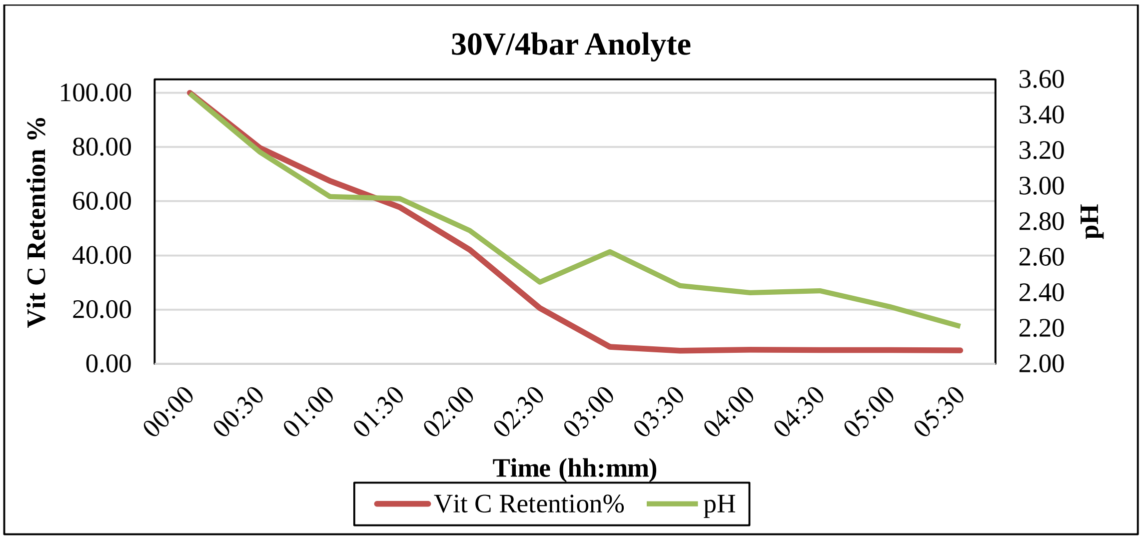


Figure 4.45. The change of vitamin C retention and pH of OJ anolyte over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

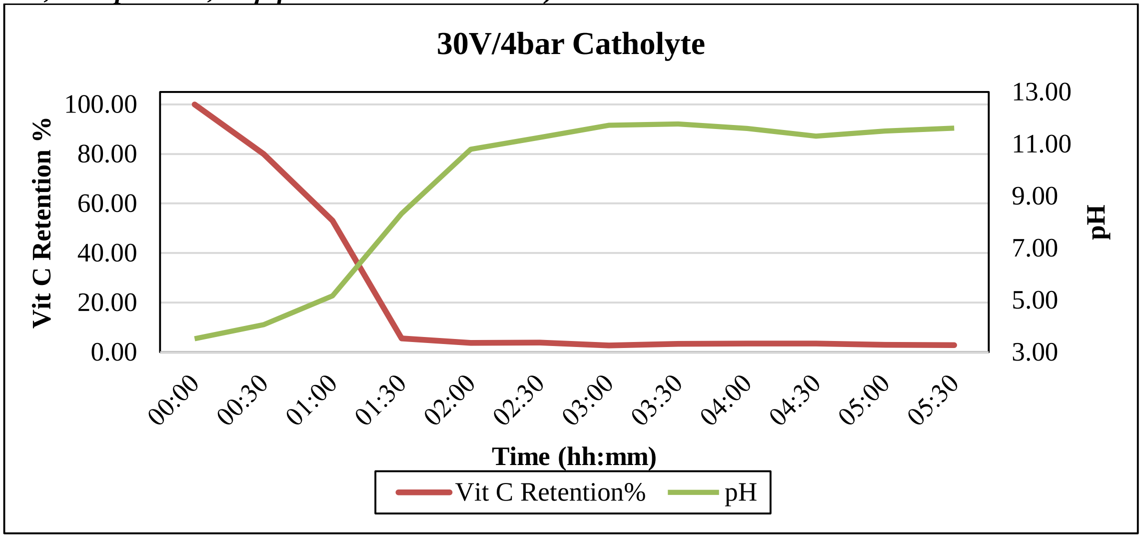


Figure 4.46. The change of vitamin C retention and pH of OJ catholyte over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

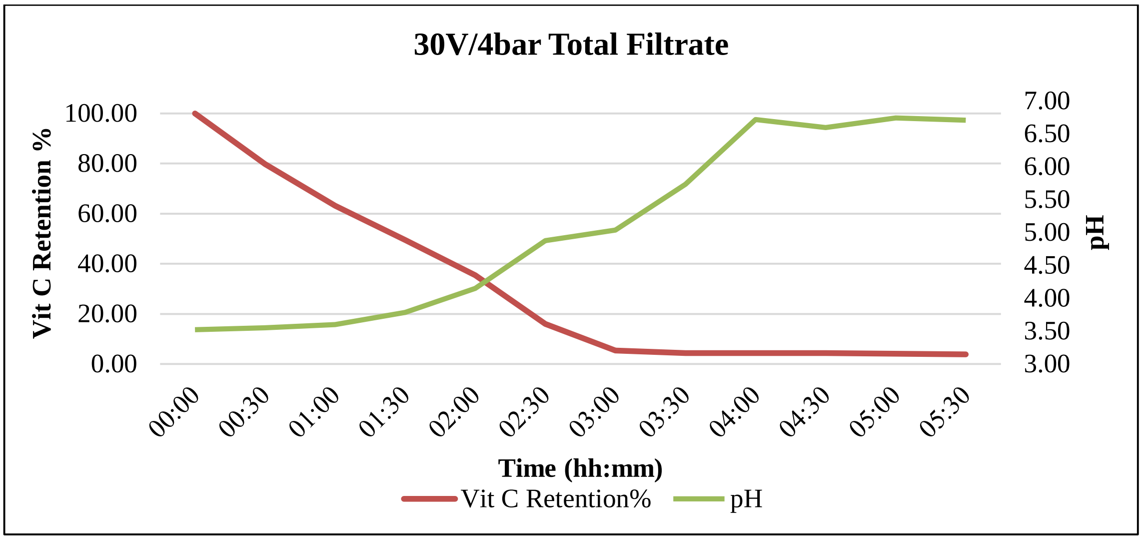


Figure 4.47. The change of vitamin C retention and pH of OJ total filtrate over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

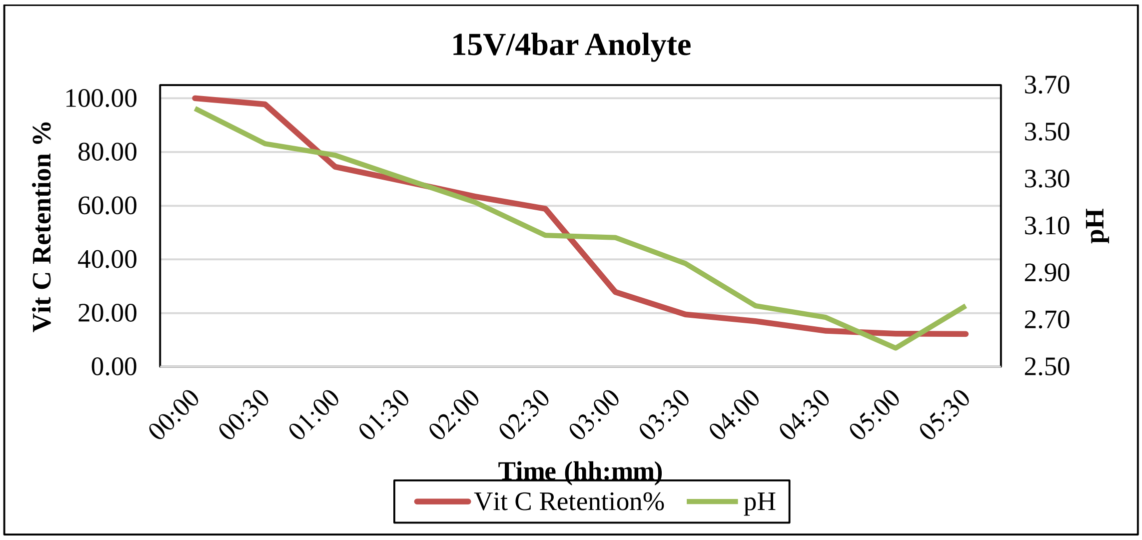


Figure 4.48. The change of vitamin C retention and pH of OJ anolyte over time under (normal orientation, 15V, 4bar pressure, 0.2µ filter and 5.5hr duration)

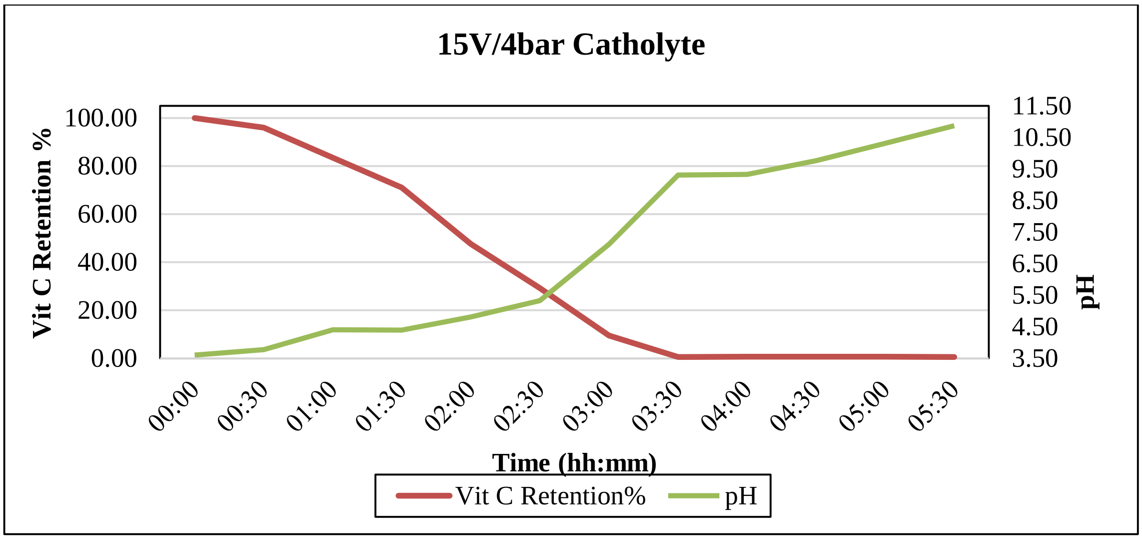


Figure 4.49. The change of vitamin C retention and pH of OJ catholyte over time under (normal orientation, 15V, 4bar pressure, 0.2µ filter and 5.5hr duration)

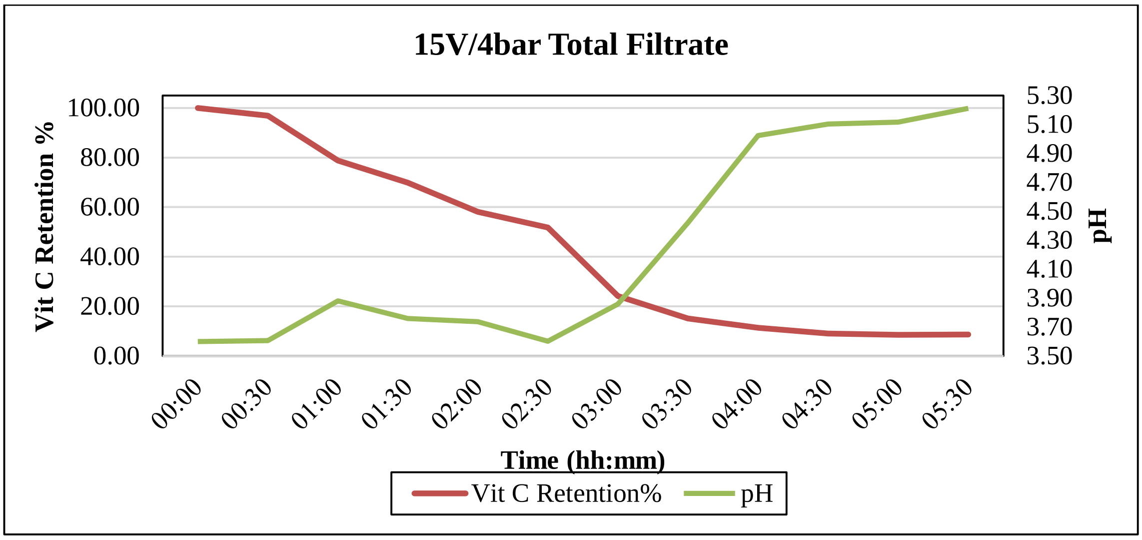


Figure 4.50. The change of vitamin C retention and pH of OJ total filtrate over time under (normal orientation, 15V, 4bar pressure, 0.2µ filter and 5.5hr duration)

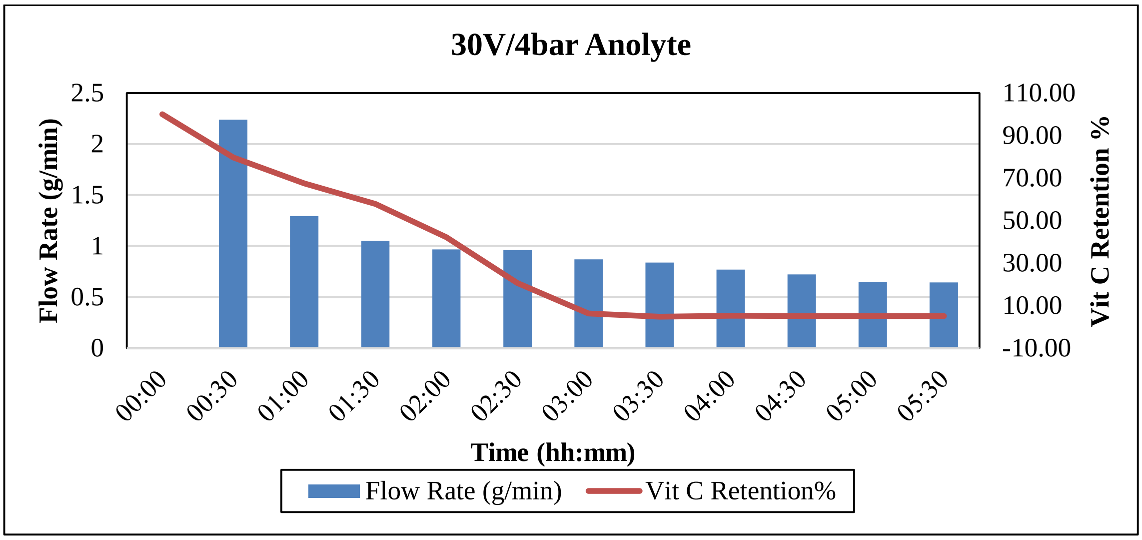


Figure 4.51. The change of vitamin C retention and the flow rate change of OJ anolyte over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

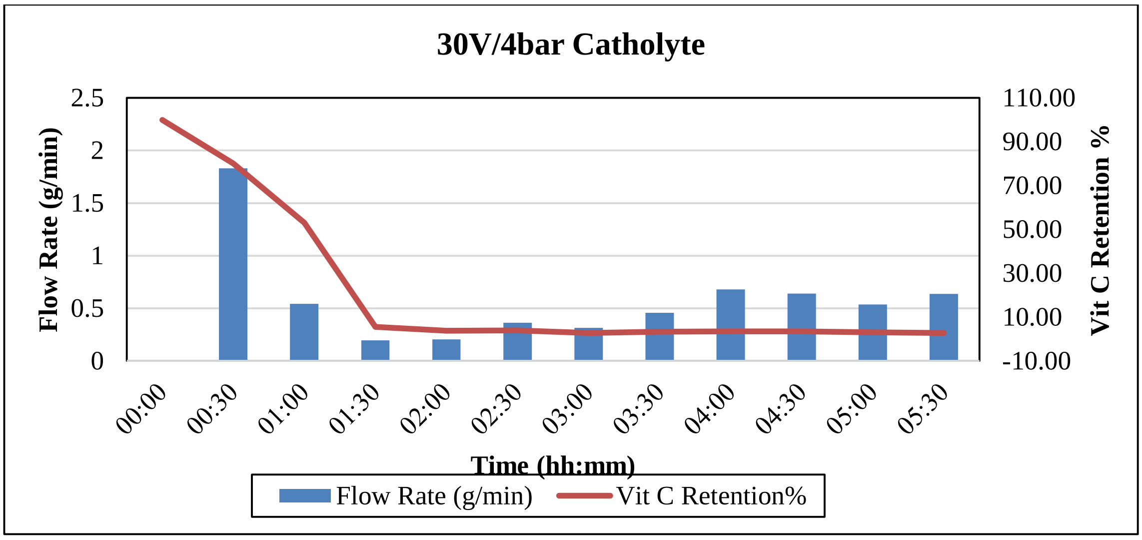


Figure 4.52. The change of vitamin C retention and the flow rate change of OJ catholyte over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

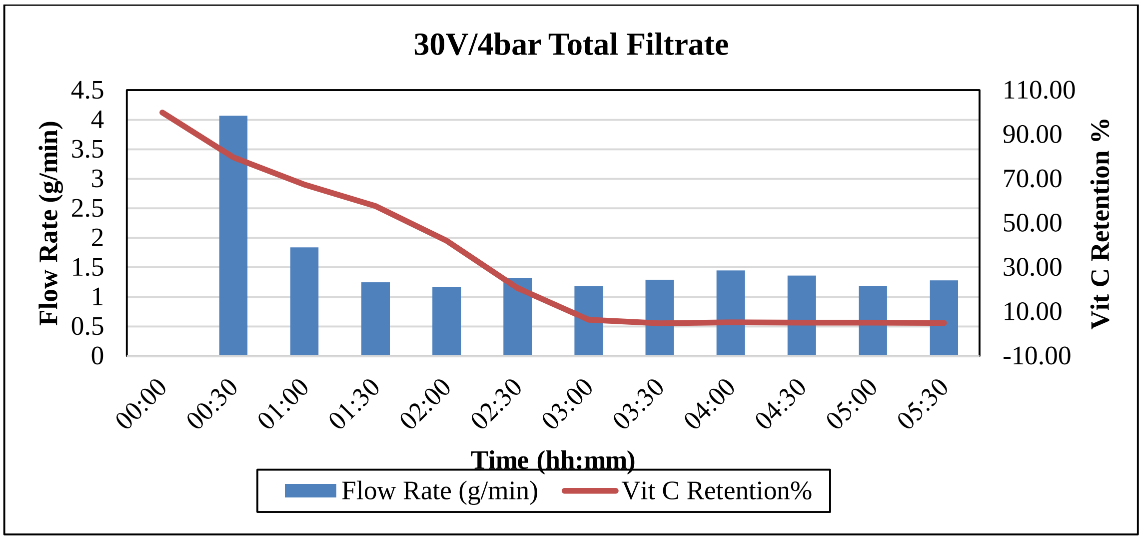


Figure 4.53. The change of vitamin C retention and the flow rate change of OJ total filtrate over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

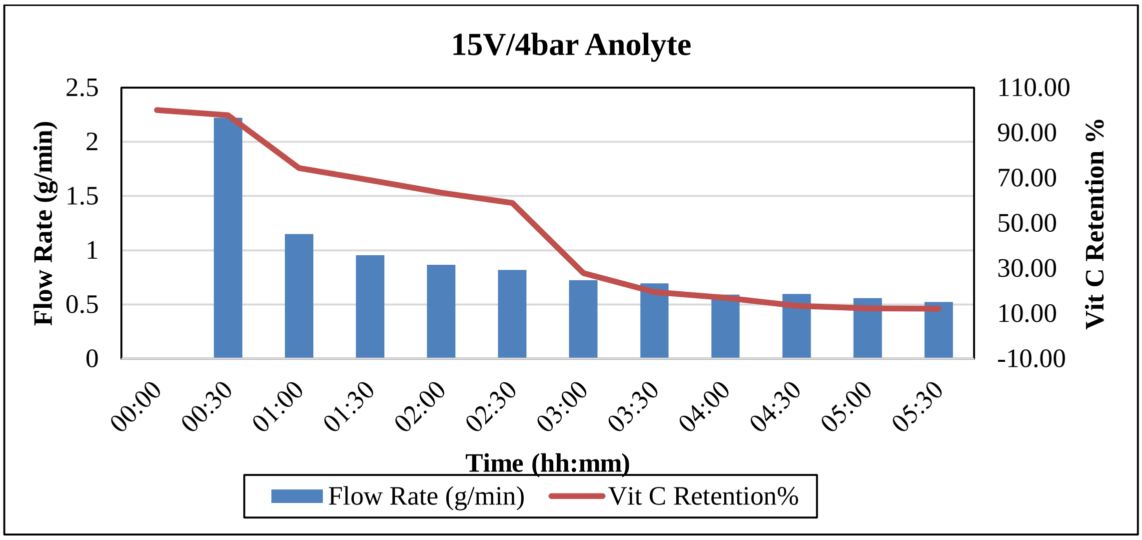


Figure 4.54. The change of vitamin C retention and the flow rate change of OJ anolyte over time under (normal orientation, 15V, 4bar pressure, 0.2µ filter and 5.5hr duration)

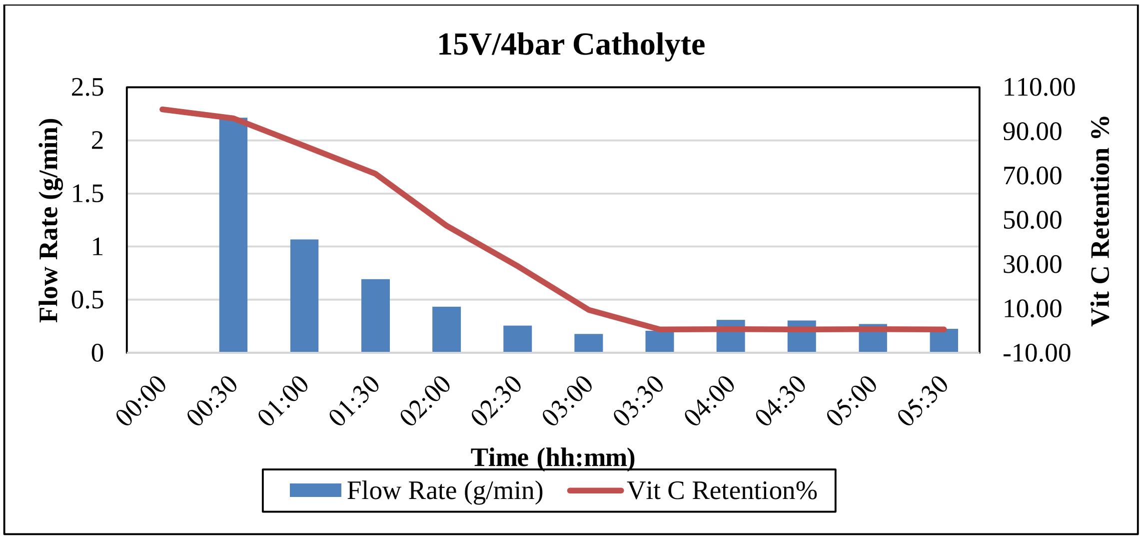


Figure 4.55. The change of vitamin C retention and the flow rate change of OJ catholyte over time under (normal orientation, 15V, 4bar pressure, 0.2µ filter and 5.5hr duration)

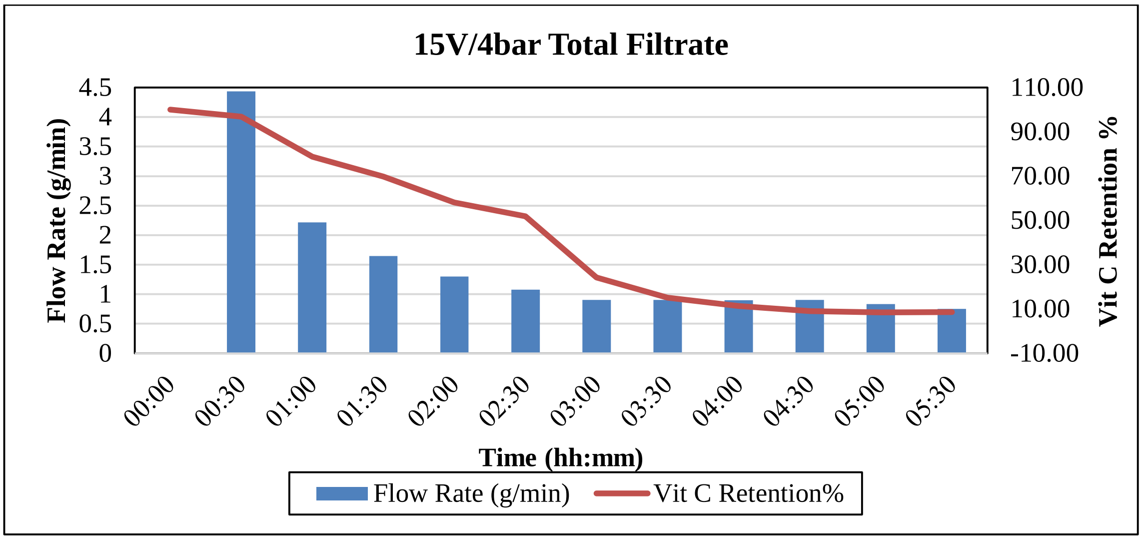
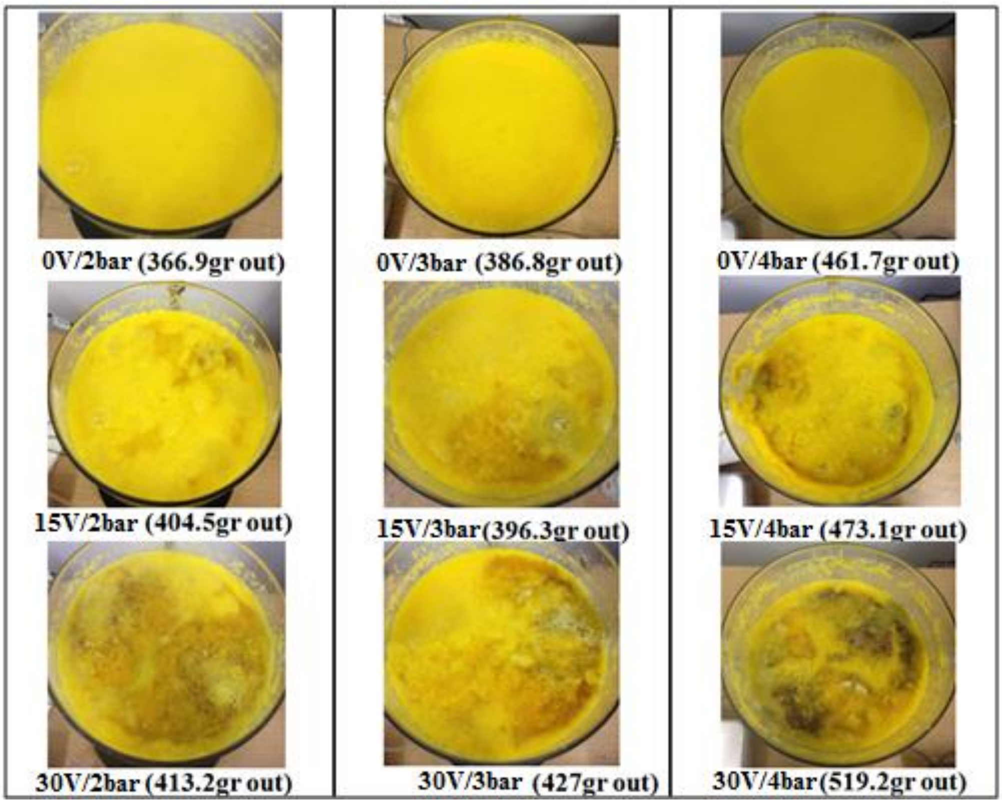


Figure 4.56. The change of vitamin C retention and the flow rate change of OJ total filtrate over time under (normal orientation, 15V, 4bar pressure, 0.2µ filter and 5.5hr duration)

4.4.4. Colour Change

Figure 4.57 is a view of the top surface of the concentrate, which was in contact with the cathode, at the end of the experiment under three different voltages (0, 15 and 30V), three pressures (2, 3 and 4bar) and normal orientation.

No change in the concentrated juice colour was observed under 0V (the control). However, discoloration started to appear when voltage was applied and increased when voltage and pressure values increased, this discolouration was more under 30V comparing with 15V.



0V/2bar (366.9gr out)
0V/3bar (386.8gr out)
0V/4bar (461.7gr out)
15V/2bar (404.5gr out)
15V/3bar(396.3gr out)
15V/4bar (473.1gr out)
30V/2bar (413.2gr out)
30V/3bar (427gr out)
30V/4bar(519.2gr out)

Figure 4.57. Top surface of concentrated OJ, under three voltages (0, 15 & 30V) and three pressures (2, 3 & 4bar) (normal orientation, 0.2μ filter, 5.5hr duration)

The discoloration was predominantly at the top layer next to the top electrode (the cathode) only. Blending the concentrated juice reduced this discoloration as the rest of concentrated juice had no change in the colour under the maximum condition of 30V and a pressure of 4bar, as shown in *Figure 4.58* which shows fresh juice at the left and the filtrate at the right and the blended concentrate in the middle at the end of the experiment.

Figure 4.58. Fresh juice (at the left) the filtrate (at the right), and the blended concentrate (in the middle) at the end of the experiment (normal orientation, 30V, 4bar, 0.2μ filtrate and 5.5hr duration)

The colour of the blended concentrated juice under the application of three different voltages (0, 15 and 30V) was compared with the fresh juice colour using Petri dishes (60x15mm) as shown in *Section 3.5.5.3*. The colour of the concentrate got darker as the voltage was increased *(Figure 4.59)*.

Fresh Concentrate (0V) Concentrate (15V) Concentrate (30V)

Figure 4.59. Colour of the blended concentrated juices under three different voltages (0, 15 and 30V) comparing with the fresh sample colour (normal orientation, 30V, 4bar, 0.2µ, 5.5h)

In addition to the visual comparison, change in the concentrate colour values were also compared under the three different voltages, where L/L0 presents the change in the sample's light–dark spectrum of the sample (when L/L0<1 that means the concentrate is darker than the control which is the fresh sample) *(Figure 4.60)*. The browning index presents the purity of the brown colour or the browning resulting from the process; results were shown as BI/BI0 *(Figure 4.61)*, where BI0 is the browning index of fresh OJ.

These figures show that the colour of samples decreased by around 5%, 10% and 16% under 0V, 15V and 30V respectively on the light–dark spectrum which means that the concentrate got darker under the influence of EKEF and this discoloration increased under higher voltages. This result is shown again when the BI/BI0 was compared as the browning index of the concentrate increased by 9%, 10% and 13% under 0V, 15V and 30V respectively compared with the fresh OJ browning index.

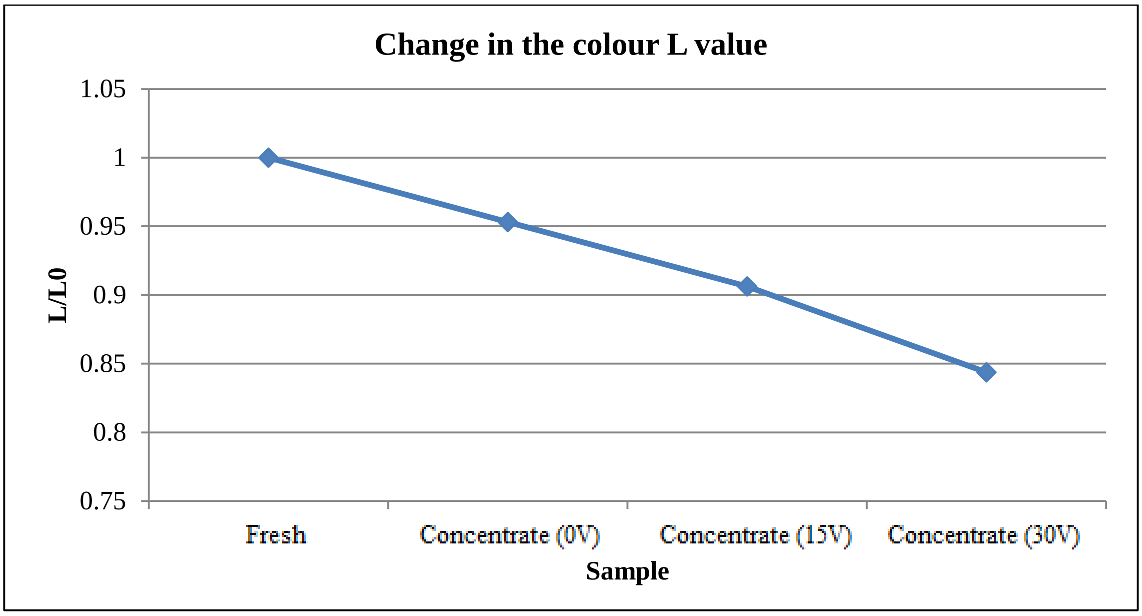


Figure 4.60. Change in colour L value of the blended concentrated juices under three different voltages (0, 15 and 30V) comparing with the fresh sample colour (normal orientation, 30V, 4bar pressure, 0.2µ filter, 5.5hr duration)

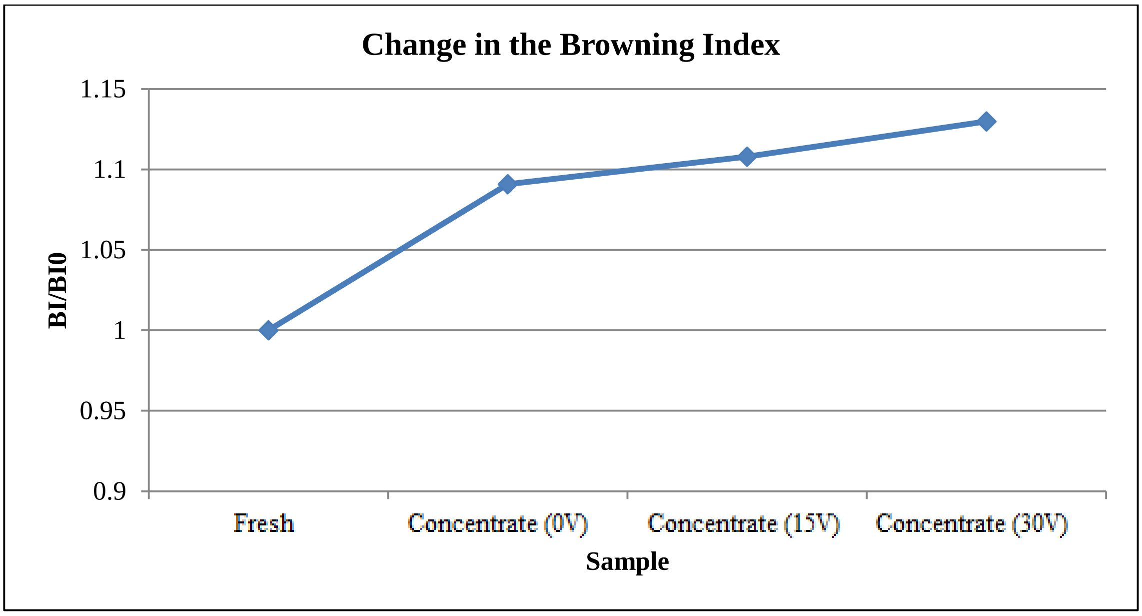


Figure 4.61. Change in Browning Index of the blended concentrated juices under three different voltages (0, 15 and 30V) comparing with the fresh sample colour (normal orientation, 30V, 4bar pressure, 0.2µ filter, 5.5hr duration)

In contrast, monitoring the change in the colour over time was easier in the filtrate due to the ability to collect sample every half hour and the transparent nature of the colour of the initial filtrate.

Figure 4.62 shows the change in the anolyte and catholyte under three different voltages (0, 15 and 30V); this change is compared to first collected filtrate sample.

Filtrates showed a minimal colour for the control samples and early stages of the 15V and 30V samples. Colour started to appear in the catholyte after 3hours and 1.5 hours of the experiment under 15V and 30V respectively. While in the anolyte, colour appeared clearly only in the 30V catholyte after 4hours.

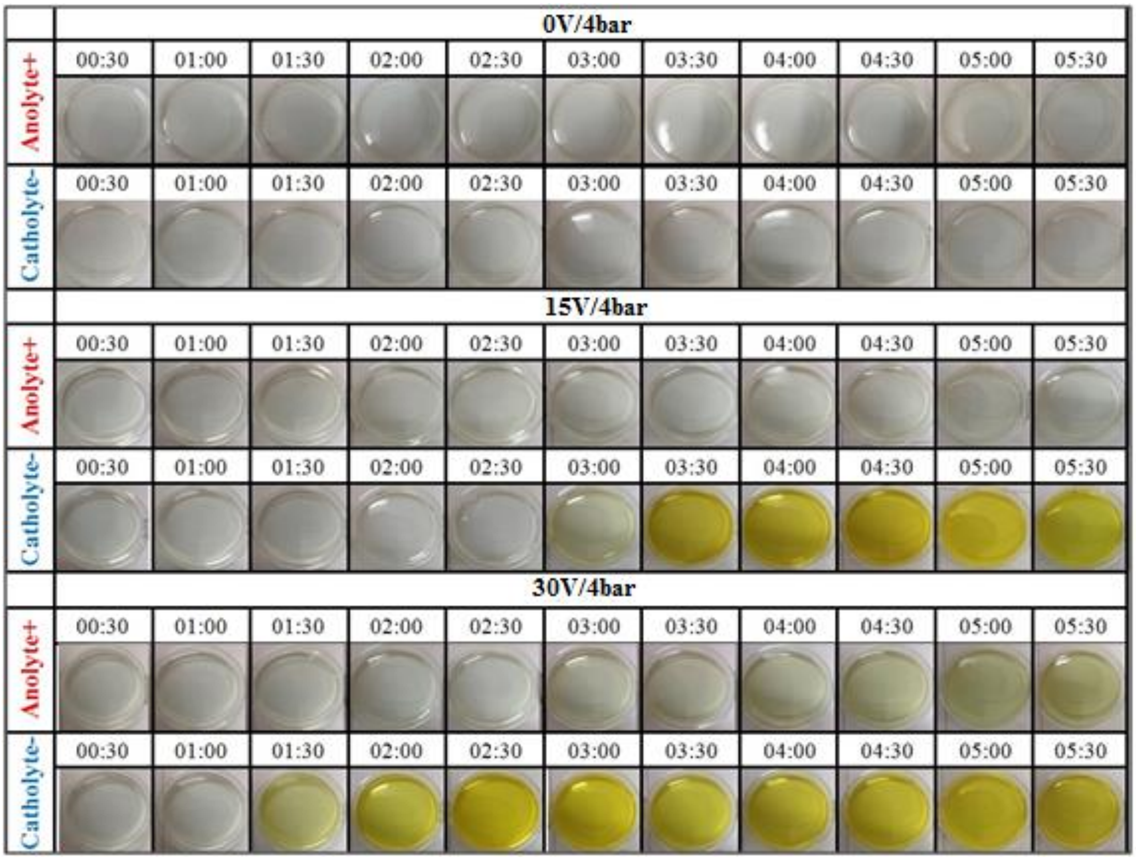
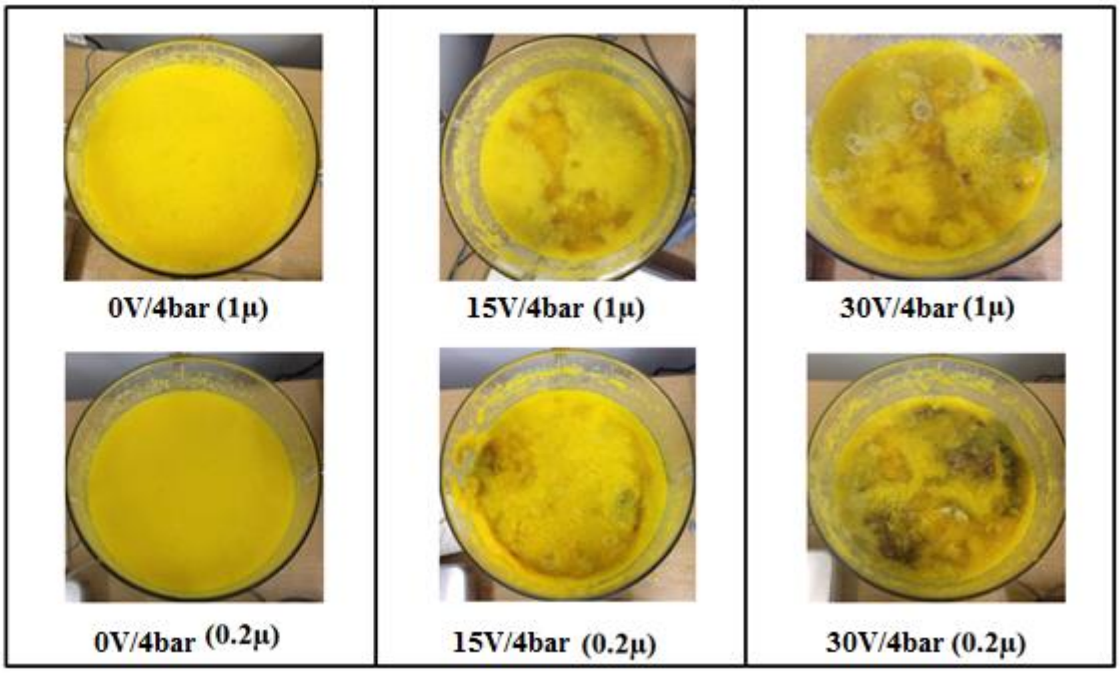


Figure 4.62. Change in the filtrate colour over the experiment time under three different voltages (0, 15 and 30V) (normal orientation, 30V, 4bar pressure, 0.2μfilter, 5.5hr duration)

Filter pores size not only played a positive role for the outcome and vitamin retention only, but also had a positive impact on the juice discoloration. *Figure 4.63* shows that the discolouration of the concentrate top surface was lower under the three different voltages 0V, 15V and 30V using the 1μ filters in comparison with the discolouration using the 0.2μ filters.

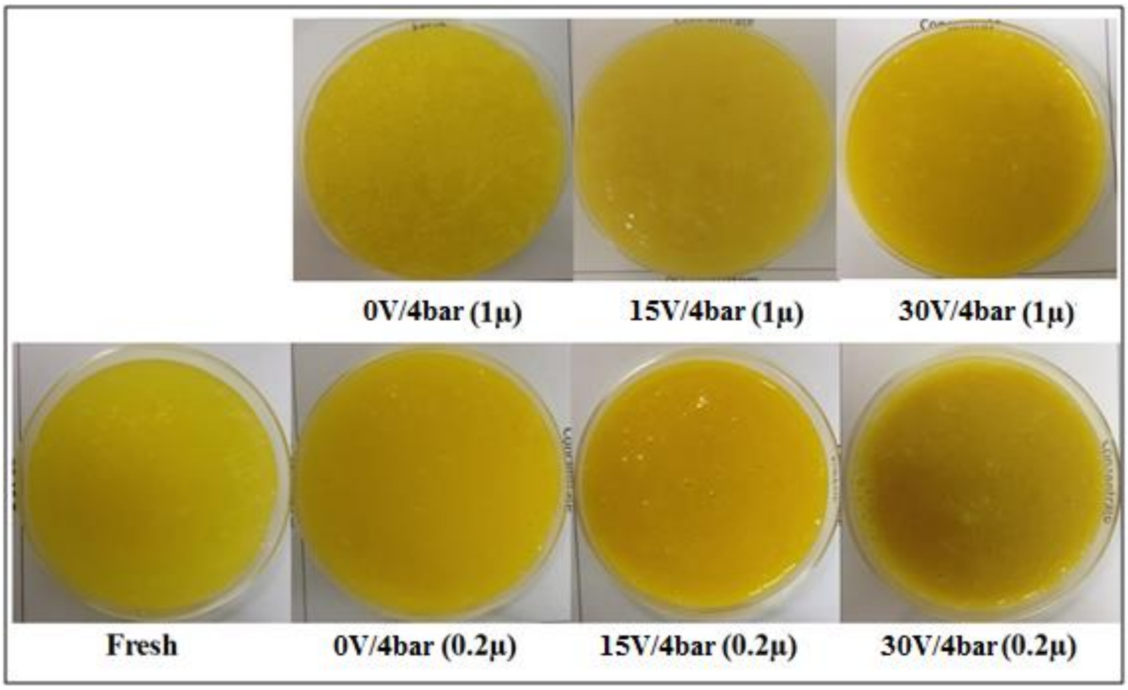


0V/4bar (1µ) 15V/4bar (1µ) 30V/4bar (1µ)

0V/4bar (0.2µ) 15V/4bar (0.2µ) 30V/4bar (0.2µ)

Figure 4.63. Top surface of concentrated OJ, under three voltages (0, 15 & 30V) using two filters 0.2µ and 1µ (normal orientation, 4bar, 5.5h duration)

The discolouration of the total blended concentrate was also lower using the 1µ filters as shown in *Figure 4.64*, with lower browning index *(Figure 4.65)*.



0V/4bar (1µ) 15V/4bar (1µ) 30V/4bar (1µ)

Fresh 0V/4bar (0.2µ) 15V/4bar (0.2µ) 30V/4bar (0.2µ)

Figure 4.64. Colour of the blended concentrated juices; under three voltages (0, 15 & 30V) using two filters 0.2µ and 1µ (normal orientation, 4bar, 5.5h duration)

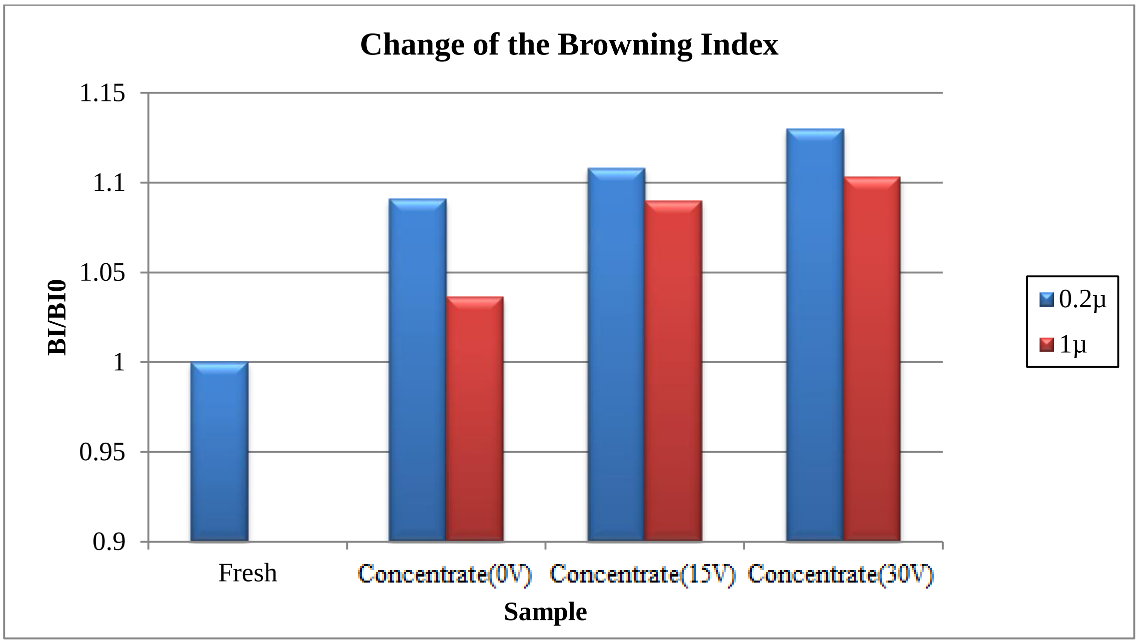


Figure 4.65. Change in Browning Index of the blended concentrated juices under three voltages (0, 15 & 30V) using two filters 0.2µ and 1µ (normal orientation, 4bar, 5.5h duration)

In terms of the starting time of discolouration, the catholyte samples showed that the discolouration started to occur after 4.5hours under 15V and after 1.5hours under 30V through 1µ filters, while it started after 3hours under 15V and after 1.5hours under 30V through 0.2µ filters *(Figure 4.66)*. Even though the discolouration started at the same time through both filters under 30V it was light in the catholyte that exited through 1µ filters until 2.5hours of the experiment.

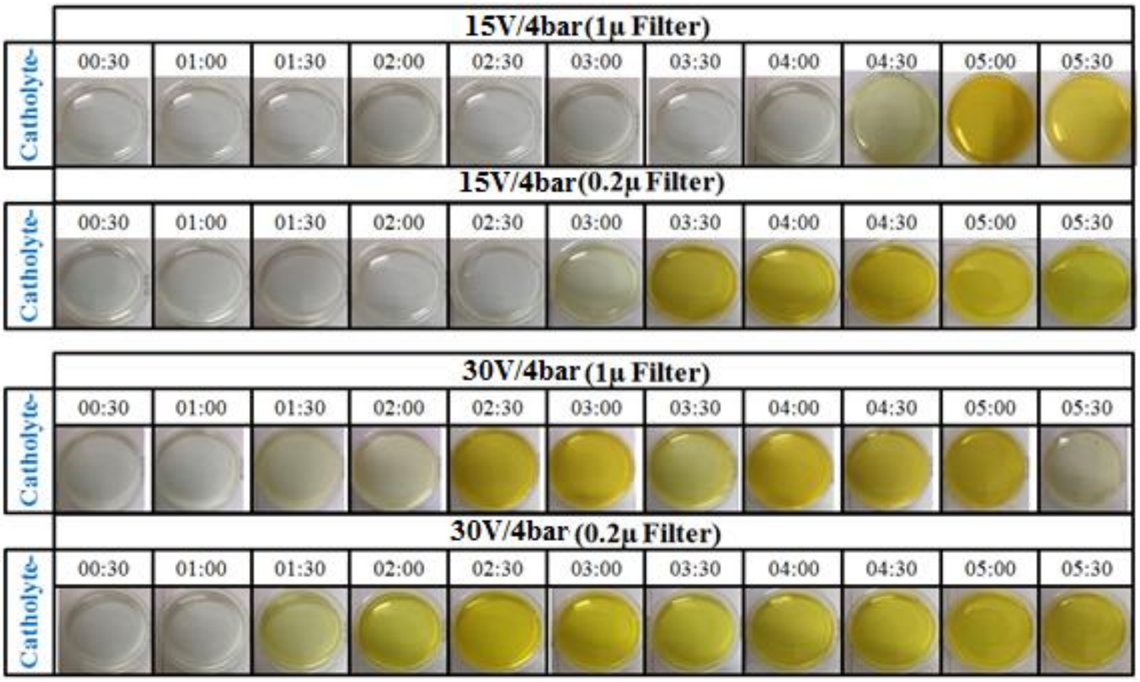


Figure 4.66. Change in the catholyte colour over the experiment time under two voltages (15 & 30V) using two filters 0.2µ and 1µ (normal orientation, 4bar, 5.5hduration)

As mentioned in *Section 3.5.5.4* a visual detection test was run on some discoloured samples to detect whether there is carotenoids in the filtrate samples or not, as carotenoids dissolves in ethyl acetate turning its colour from blank to yellow.

Figure 4.67 shows that the ethyl acetate phase turned into yellow in both fresh and concentrated samples while it remained blank in the anolyte and catholyte samples which means that there is no carotenoids in the anolyte and catholyte samples.

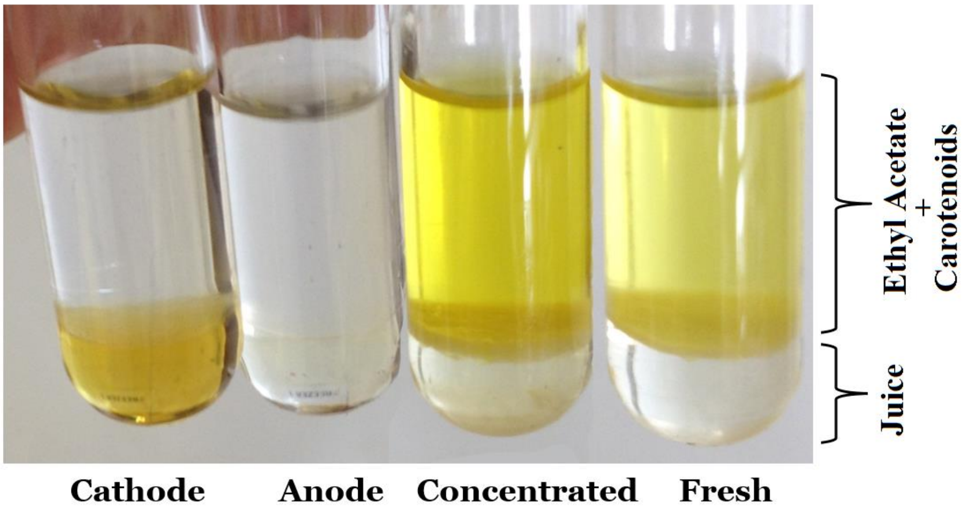


Figure 4.67. Carotenoids visual detection on fresh, concentrated, anolyte and catholyte OJ sample under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

4.4.5. Dry Matter Content Change

Figures 4.68 and *4.69* show the change in the concentrate dry matter content over time under the application of three different voltages (0, 15 and 30V) in OJ and ME respectively. Applying EKEF helped to increase dry matter content from 14% to 29.52% and 33.09% for OJ concentrate and from 20% to 32.14% and 34.2% for ME concentrate after 5.5hrs under 15V and 30V respectively while the maximum dry matter content was 28.75% and 29.63% under the control orientation after the same time for OJ concentrate and ME concentrate respectively.

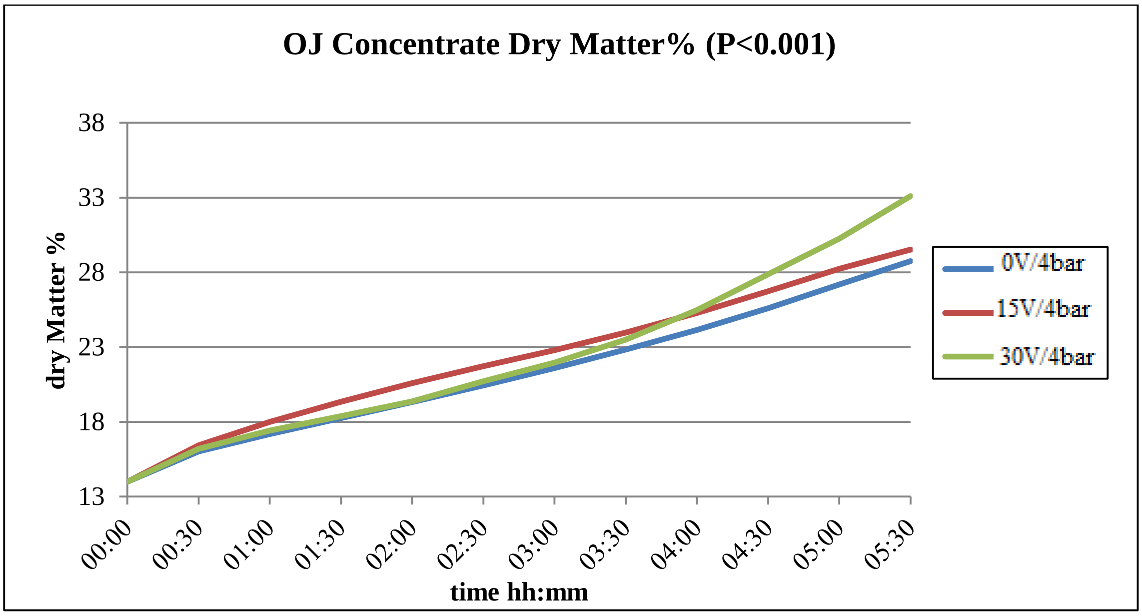


Figure 4.68. Change in OJ concentrate dry matter content over time, under three different voltages (0, 15 and 30V) (normal orientation, 4bar pressure, 0.2μ filter and 5.5hr duration)

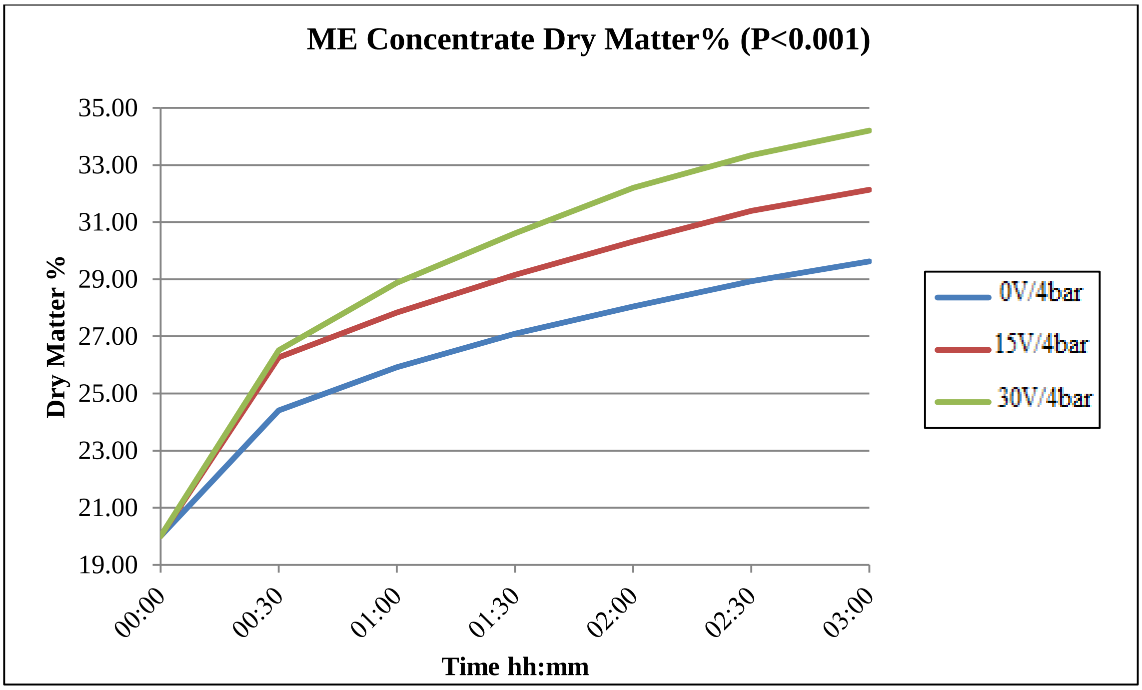


Figure 4.69. Change in ME concentrate dry matter content over time, under three different voltages (0, 15 and 30V) (reversed orientation, 4bar pressure, 0.2μ filter and 3hr duration)

Water exits through the outlets creating the filtrate stream which has its own dry matter content that depends on the fresh juice dry matter content and the filter pore size.

Figures 4.70 and *4.72* show the change in total filtrate dry matter content over time under three different voltages (0, 15 and 30V) in OJ and ME respectively, while *Figures 4.71, 4.73, Tables 4.24* and *4.25* show the means and P-values of this change:

The voltage values did not have a significant impact on dry matter content of the total filtrate unlike on the concentrate dry matter content where it was significant with $P<0.001$. However OJ filtrate dry matter content was significantly lower under 30V ($P<0.001$) in comparison with 0V and 15V.

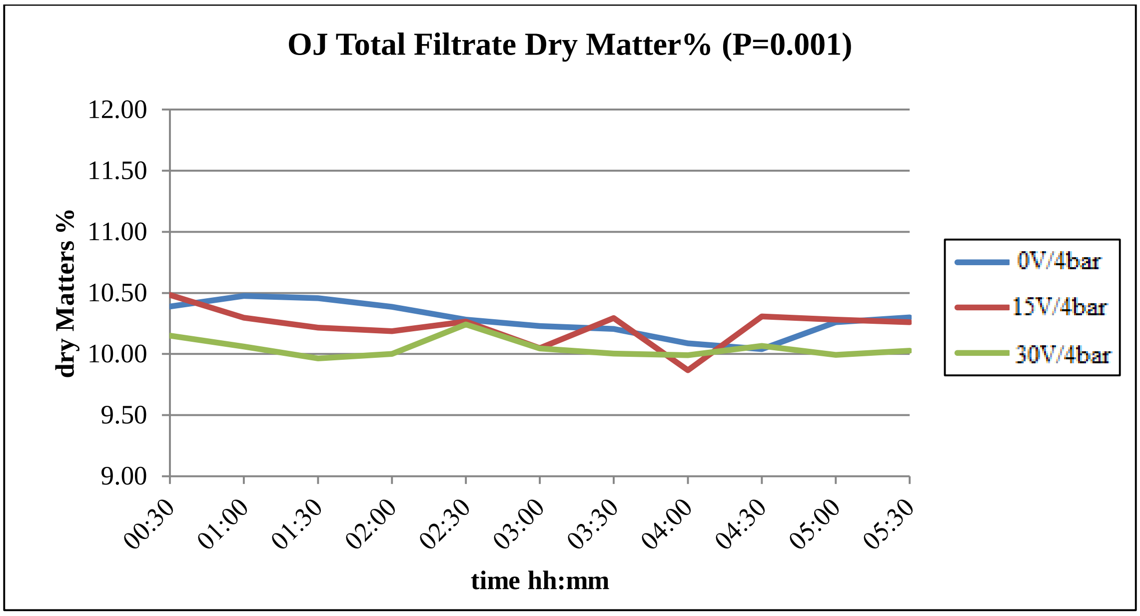


Figure 4.70. Change in OJ total filtrate dry matter content over time, under three different voltages (0, 15 and 30V) (normal orientation, 4bar pressure, 0.2µ filter, 5.5hr duration)

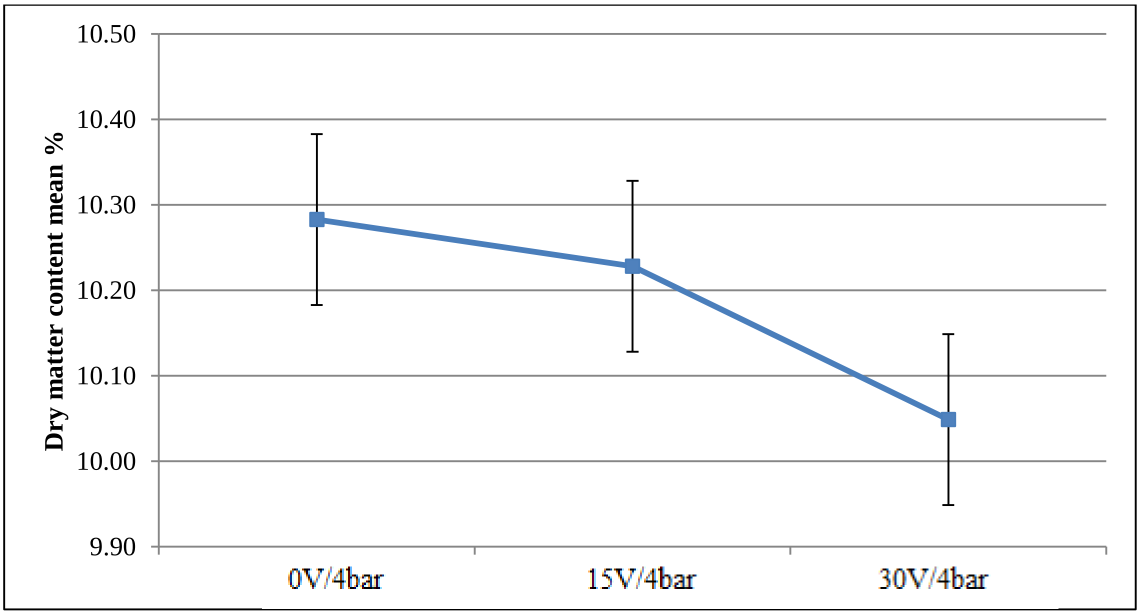


Figure 4.71. Means of the dry matter content of OJ total filtrate over time under three different voltages (0, 15 and 30V) (normal orientation, 4bar pressure, 0.2µ filter, 5.5hr duration)

Table 4.24. P-values of the change in the dry matter content of OJ total filtrate over time under three different voltages (0, 15 and 30V) (normal orientation, 4bar pressure, 0.2µ filter, 5.5hr duration)

		P-value of the accumulative total filtrate			
		0V-15V-30V	**0V-15V**	**15V-30V**	**0V-30V**
0.2µ	**4bar**	0.001	0.401	0.003	<0.001

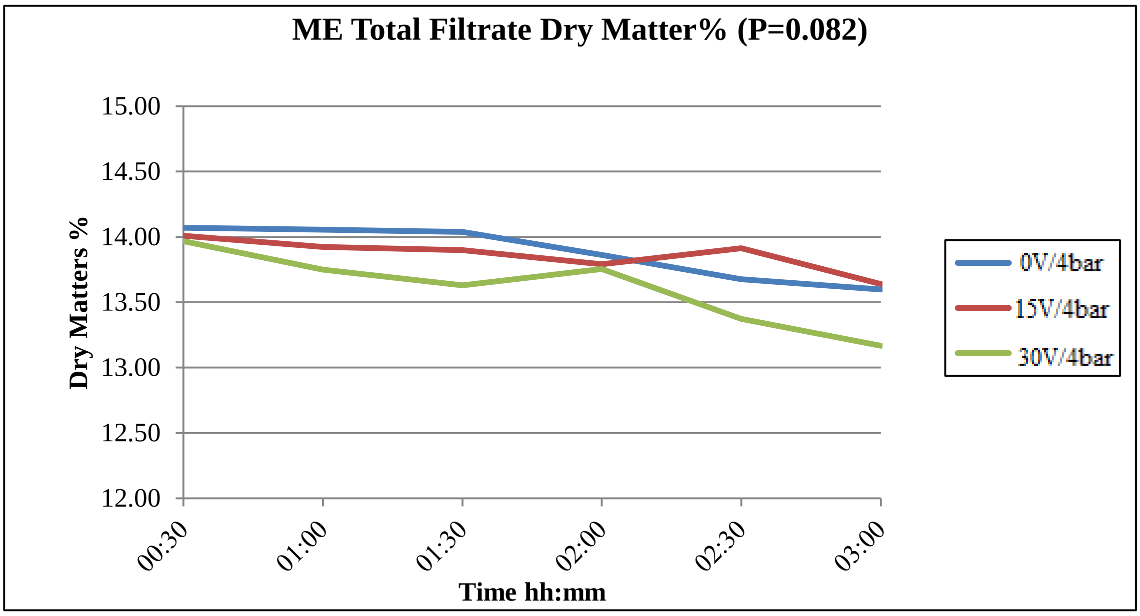


Figure 4.72. Change in ME total filtrate dry matter content over time, under three different voltages (0, 15 and 30V) in (reversed orientation, 4bar pressure, 0.2µ filter, 3hr duration)

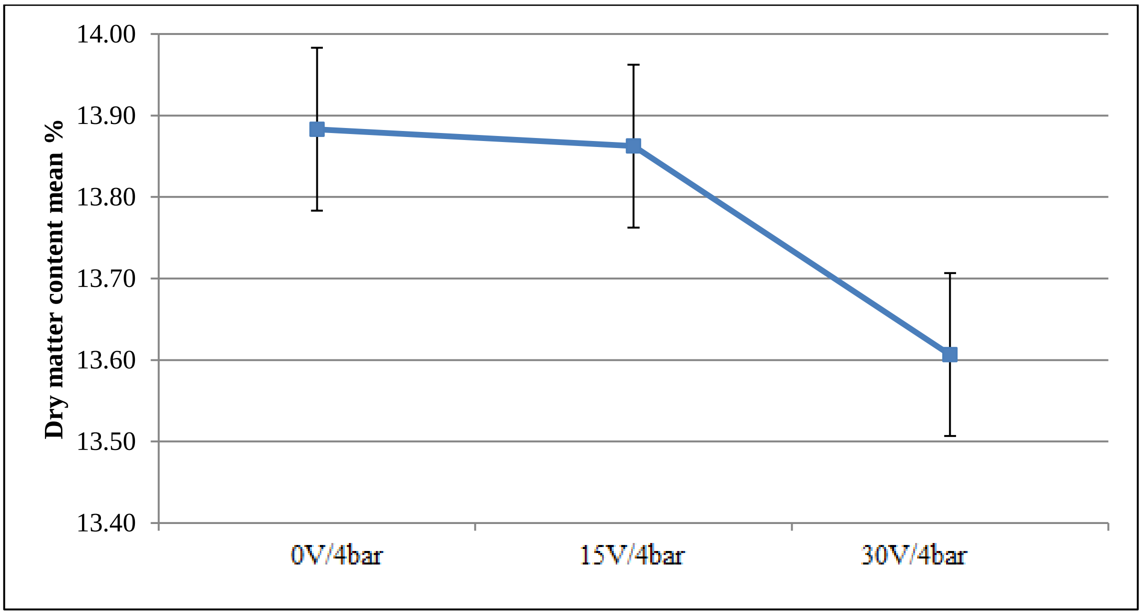


Figure 4.73. Means of the dry matter content of ME total filtrate over time, under three different voltages (0, 15 and 30V) in (reversed orientation, 4bar pressure, 0.2µ filter, 3h r duration)

Table 4.25. P-values of the change in dry matter content of ME total filtrate over time, under three different voltages (0, 15 and 30V) in (reversed orientation, 4bar pressure, 0.2µ filter, 3hr duration)

		P-value of the accumulative total filtrate			
		0V-15V-30V	**0V-15V**	**15V-30V**	**0V-30V**
0.2µ	**4bar**	0.082	0.840	0.077	0.087

The main factor that impacted on filtrate dry matter content was the filter pore size, the larger the pore size the more of the dry matter passed through them. *Figure 4.74* and *Table 4.266* shows the impact of filter pore size on the total filtrate dry matter content over time under the application of three different voltages (0, 15 and 30V) in ME. Filter with the larger pore size (1µ) allowed more particles to pass through which in turn resulted in significantly higher dry matter content compared with the small pore size filter (0.2µ) (P<0.001).

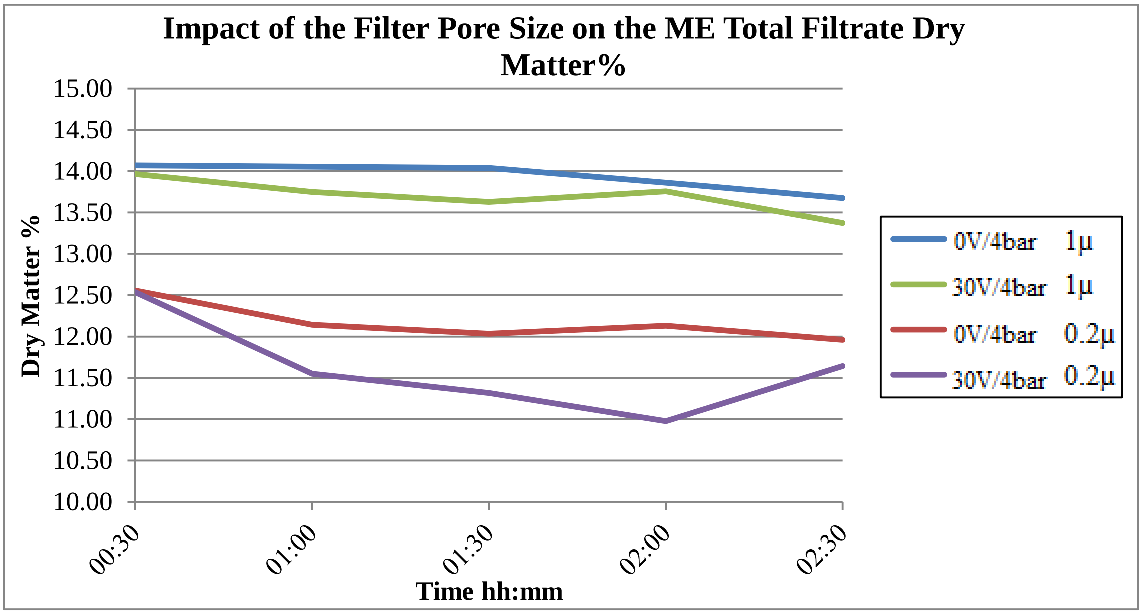


Figure 4.74. Impact of the filter pore's size (0.2&1µ) on ME total filtrate dry matter content over time, under (0 &30V, reversed orientation, 4bar pressure, and 2.5hr duration)

Table 4.26. P-values of the filter pore's size (0.2& 1µ) on ME total filtrate dry matter content over time, under (0 &30V, reversed orientation, 4bar pressure, and 2.5hr duration)

P-value of the accumulative total filtrate	
0V(0.2µ)-0V(0.2µ)	**30V(0.2µ) -30V(1µ)**
<0.001	<0.001

To investigate the potential impact of the electrode type and position on the dry matter content, the anolyte and catholyte were collected every half hour then the dry matter content was measured for each sample. *Figure 4.75* shows the difference between the anolyte and catholyte dry matter content over time, under three voltages (0, 15 and 30V); these experiments were run under the reversed orientation (cathode is the bottom). In addition *Figure 4.76* and *Table 4.27* show the means and P-values of this change

The data show that there was a significant difference between the anolyte and the catholyte dry matter contents under 0V and 15V (P<0.001 and 0.002 respectively), where it was more stable over the experiment time in the catholyte while it dropped over time in the anolyte. However, the difference between the anolyte and the catholyte dry matter contents under 30V was not significant, (P=0.087).

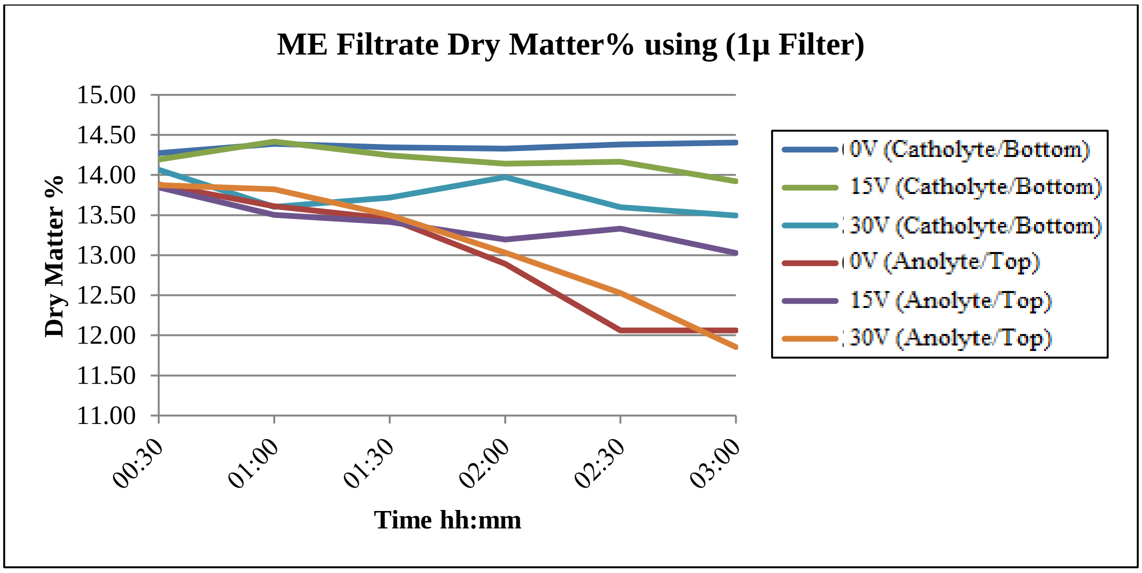


Figure 4.75. Change in the ME anolyte and catholyte dry matter content over time, under three different voltages (0, 15 and 30V) (reversed orientation, 4bar pressure, 1µ filter, 3hr duration)

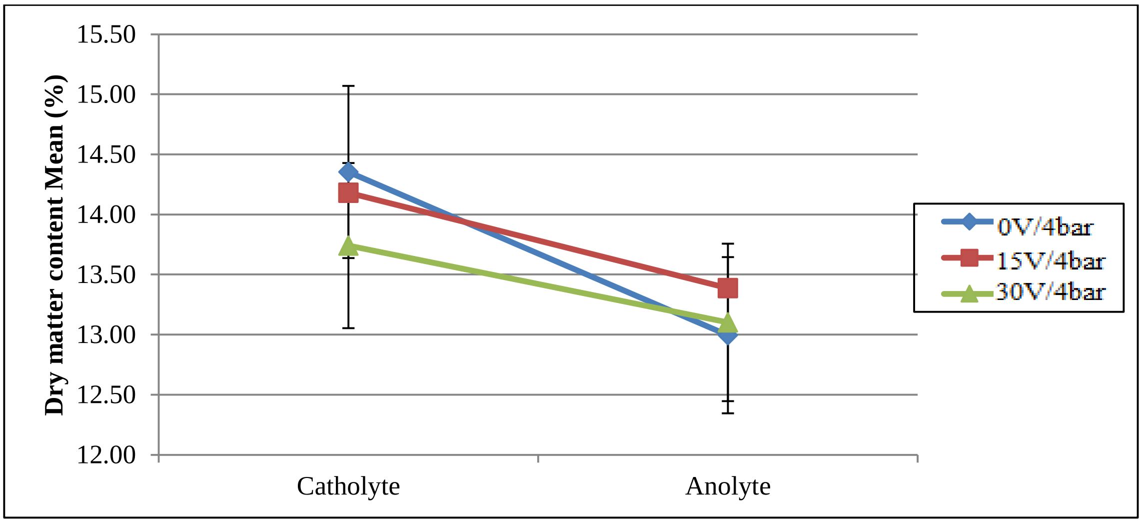


Figure 4.76. Means of the dry matter content of ME anolyte and catholyte dry matter content over time, under three different voltages (0, 15 and 30V, reversed orientation, 4bar pressure, 1µ filter, 3hr duration)

Table 4.27. P-values of the electrode type impact on ME total filtrate dry matter content over time, under (0 &30V, reversed orientation, 4bar pressure, and 2.5hr duration)

P-value of the accumulative filtrates		
0V(Anolyte)-0V(Catholyte)	**15V(Anolyte)-15V(Catholyte)**	**30V(Anolyte)-30V(Catholyte)**
0.002	<0.001	0.087

All previous experiments were set as reversed orientation (cathode at the bottom); to check whether dry matter contents were influenced by the electrode charge (anode or cathode) or the electrode position (top or bottom), results of the two orientations (normal and reversed) under 15V, 4bar and through 1µ filter were compared as shown in *Figure 4.77*. This figure shows that the lowering in dry matter content was in the normal catholyte (the top outlet stream) and the reversed Anolyte (the top outlet stream).

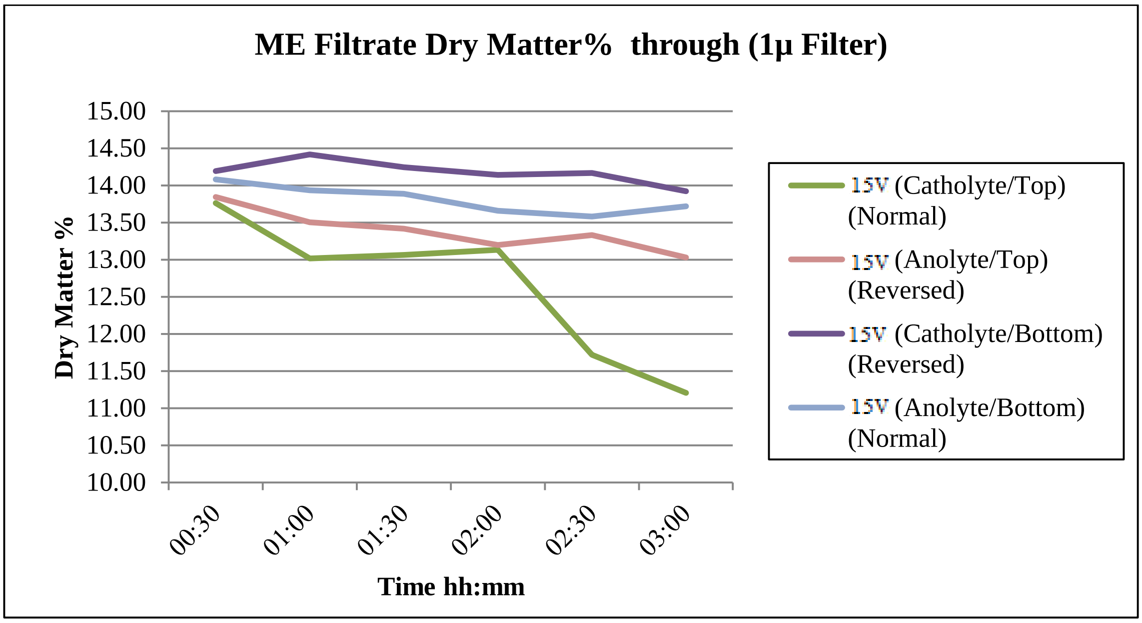


Figure 4.77. Change in the ME anolyte and catholyte dry matter content over time, under two orientations (reversed orientation/ Cathode is the bottom and normal orientation/ Anode is the bottom) (15V, 4bar pressure, 1µ filter and 3hr duration)

4.5. Power Consumption

As mentioned in *Section 3.5.5.6*, the total power consumption (*E*) during the application of EKEF can be calculated as the sum of the mechanical power applied by the pneumatic pressure ram (*E1*) and the electrical power consumed by the applied voltage across the sample (*E2*).

E1 was calculated using *Equation 14* as the following:

$$E_1 = 13.84\,J = 0.384 \times 10^{-5} KWh$$

For pressure = 400Kpas, volume displacement of the cell = 0.001m³, and cross sectional area of the cell = 0.0289 m² for inner diameter = 0.192m.

Tables 4.28 and *4.29* show the values of the electrical current and the electrical energy consumption (*E1*) every half hour during the EKEF application under 15V (the minimum) and 30V (the maximum) in OJ and ME respectively:

Table 4.28. Values of the electrical current and electrical energy consumption (E) every half hour during the EKEF application under (15 and 30V), in OJ samples (normal orientation, 0.2µ filter, 4bar pressure & 5.5hr duration)

T (min)	Electric Current (Amps)		Voltage (V)		dE2(KW h)	
	15V	30V	15V	30V	15V	30V
00:00	0	0	0	0	0	0
00:30	0.33	0.77	15	30	0.00248	0.01155
01:00	0.26	0.68	15	30	0.00195	0.0102
01:30	0.23	0.61	15	30	0.00173	0.00915
02:00	0.2	0.5	15	30	0.0015	0.0075
02:30	0.19	0.61	15	30	0.00143	0.00915
03:00	0.19	0.59	15	30	0.00143	0.00885
03:30	0.2	0.48	15	30	0.0015	0.0072
04:00	0.24	0.54	15	30	0.0018	0.0081
04:30	0.17	0.49	15	30	0.00128	0.00735
05:00	0.15	0.48	15	30	0.00113	0.0072
05:30	0.12	0.46	15	30	0.0009	0.0069
		Total E (EKEF)			0.0171	0.09315

Table 4.29. Values of the electrical current and electrical energy consumption (E) every half hour during the EKEF application under (15 and 30v), in ME samples (reversed orientation, 1µ filter, 4bar pressure &3.5hr duration)

T (min)	Electric Current (Amps)		Voltage (V)		dE2(KW h)	
	15V	30V	15V	30V	15V	30V
00:00	0	0	0	0	0	0
00:30	0.22	0.47	15	30	0.00165	0.00705
01:00	0.13	0.49	15	30	0.00098	0.00735
01:30	0.11	0.5	15	30	0.00083	0.0075
02:00	0.1	0.46	15	30	0.00075	0.0069
02:30	0.09	0.46	15	30	0.00068	0.0069
03:00	0.08	0.4	15	30	0.0006	0.006
03:30	0.07	0.39	15	30	0.00053	0.00585
		Total E (EKEF)			0.006	0.04755

After calculating energies consumed over the experiments, a comparison between the energy consumption of EKEF application (electrokinetics and pressure) and the normal microfiltration (with pressure only) was carried out.

Tables 4.30 and *4.31* show that EKEF application under 4bar pressure and two voltage values (15 and 30V) results in a large additional power consumption compared with applying pressure only with a relatively small increase in dewatering efficiency.

The required additional energy needed to achieve the same increase in dewatering efficiency by increasing the pressure only without EK can be calculated as follows:

$$P_{\text{Additional}} = \frac{W_{EKEF} - W_P}{W_P} \text{x} P \quad \textit{(eq. 15)}$$

$$E_{\text{Additional}} = \frac{P_{\text{Additional}} \text{ x } E}{P} \quad \textit{(eq. 16)}$$

Where:

$P_{\text{Additional}}$: is the required additional pressure needed to achieve the same increase in dewatering efficiency.

$E_{\text{Additional}}$: is the energy consumption resulted from applying the $P_{\text{Additional}}$.

P: is the original pressure applied; here P=4 bar.

E: is the energy consumption resulted from applying the original pressure (P); here E= $3.8378 \text{x} 10^{-6}$ KWh.

W_P: the amount of water removed by applying the original pressure (P) (*Table 4.32*).

W_{EKEF}: the amount of water removed by applying EKEF application (*Table 4.32*).

For OJ this equates to an additional $9.47 \text{x} 10^{-8}$ and $4.78 \text{x} 10^{-7}$ KWh of energy expenditure for the equivalent of applying EKEF at 15V and 30V, respectively. For ME this equates to an additional $5.66 \text{x} 10^{-7}$ and $9.66 \text{x} 10^{-7}$ KWh for the equivalent of applying EKEF at 15V and 30V, respectively.

Table 4.30. Comparison between the energy consumed during EKEF application on OJ under 4bar pressure and three voltage values (0, 15 and 30V) and the thermal energy required to remove same amount of water by evaporation

Energy (Type)	Orange Juice Normal (0.2µ, 4bar)			
	Dewatering Efficiency (%)	Energy (KWh) in 5.5h of process	Energy (KJ/mol water)	$E_{\text{Additional}}$ (KWh)
Pressure +0V	59.65	$3.8378 \text{x} 10^{-6}$	0.001	-
Pressure +15V	61.12	0.017	2.343	$9.47 \text{x} 10^{-8}$
Pressure +30V	67.08	0.093	11.626	$4.78 \text{x} 10^{-7}$

Table 4.31. Comparison between the energy consumed during EKEF application on ME under 4bar pressure and three voltage values (0, 15 and 30V) and the thermal energy required to remove same amount of water by evaporation

Energy (Type)	Malt Extract Reversed (1µ, 4bar)			
	Dewatering Efficiency (%)	Energy (KWh) in 3.5h of process	Energy (KJ/mol water)	$E_{Additional}$ (KWh)
Pressure +0V	42.41	0.0000038378	0.001	-
Pressure +15V	48.67	0.006	0.908	$5.66x10^{-7}$
Pressure +30V	53.09	0.048	6.596	$9.66x10^{-7}$

Table 4.32. Moisture content of concentrated OJ and ME under 4bar pressure and different voltages (0, 15 and 30V)

Sample	Time (h)	Moister Content (%)				Removed Water (g)		
		Initial	Pressure +0V	Pressure +15V	Pressure +30V	Pressure +0V	Pressure +15V	Pressure +30V
OJ Normal (0.2µ, 4bar)	5.50	86.10	71.25	70.48	66.91	461.7	473.1	519.2
ME Reversed (1µ, 4bar)	3.50	80.00	69.73	67.25	65.23	373.2	428.3	467.2

In addition a comparison between the energy consumption of EKEF and different thermal methods was carried out. *Tables 4.33, 4.34* and *Figures 4.78* to *4.80* show the comparison between the energy consumed during EKEF application under 4bar pressure and three voltage values (0, 15 and 30V) and the thermal energy required to remove the same amount of water by evaporation for OJ and ME. Amounts of removed water are shown in *Table 4.32* for experiments concerning the application of EKEF. The theoretical calculated energy required for thermal removal of water (shown in *Tables 4.33* and *4.34*) was determined using a value of 43kJ.mol^{-1}, as quoted by *Al-Asheh et al. (2004)*. This theoretical value assumes 100% energy transfer efficiency by the heating mechanism (oven, hot plate etc.) although the efficiencies are in reality much less than this.

Tables 4.33 and *4.34* show that the electrical power consumed in applying pressure is very small and almost negligible compared with the electrical power consumed by the applied voltage, and the difference between the highest voltage and lowest voltage in terms of power consumption is very significant *(Figures 4.78* and *4.79)*. The application of EKEF saves energy compared with the power consumption of the thermal method required to evaporate an equal amount of water. This saving was up to 18.35 times and 47.34 times in OJ and ME dewatering respectively *(Figure 4.80)*.

Table 4.33. Comparison between the energy consumed during EKEF application on OJ under 4bar pressure and three voltage values (0, 15 and 30V) and the thermal energy required to remove same amount of water by evaporation

		OJ Normal (0.2µ, 4bar)		
Energy (Type)	Dewatering Efficiency (%)	Energy (KWh)	Energy Saving %	$E_{Thermal}/E_{EEF}$
Thermal	61.12	0.31	94.55	18.35
Pressure +15V		0.02		
Thermal	67.08	0.34	72.96	3.70
Pressure +30V		0.09		

Table 4.34. Comparison between the energy consumed during EKEF application on ME under 4bar pressure and three voltage values (0, 15 and 30V) and the thermal energy required to remove same amount of water by evaporation

		ME Reversed (1µ, 4bar)		
Energy (Type)	Dewatering Efficiency (%)	Energy (KWh)	Energy Saving %	$E_{Thermal}/E_{EEF}$
Thermal	48.67	0.28	98.86	47.34
Pressure +15V		0.006		
Thermal	53.09	0.31	84.66	6.52
Pressure +30V		0.048		

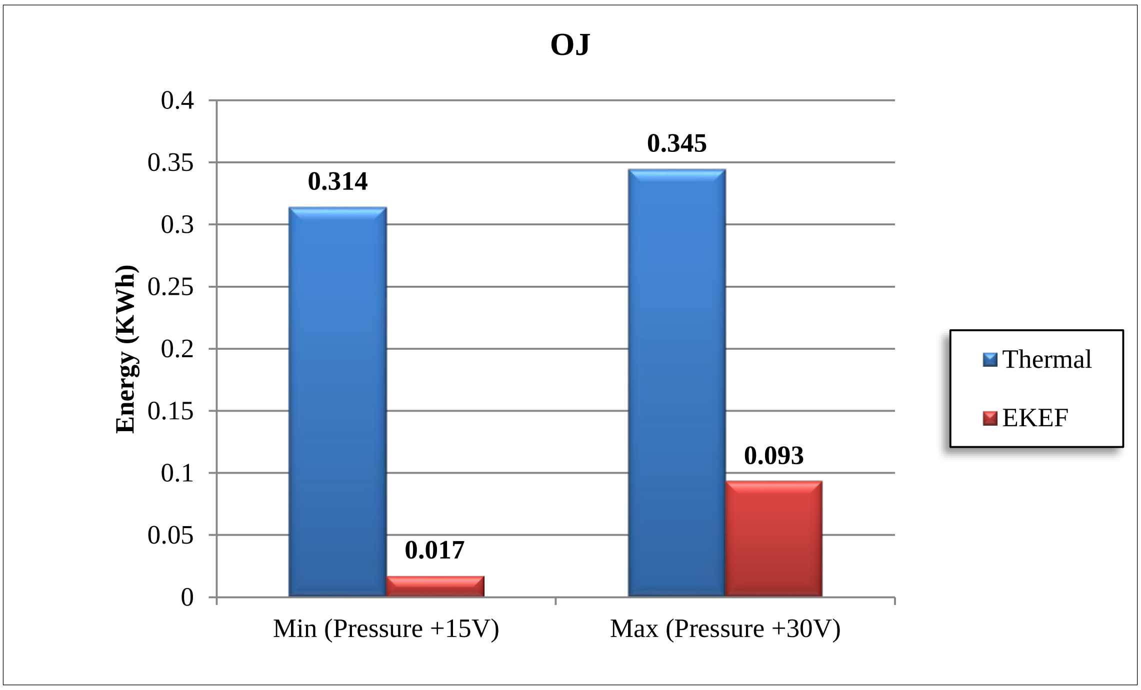


Figure 4.78. Comparison between the energy consumed during the application of EKEF to OJ under 4bar pressure and three voltage values (15 and 30V) and the thermal energy required to remove same amount of water by evaporation

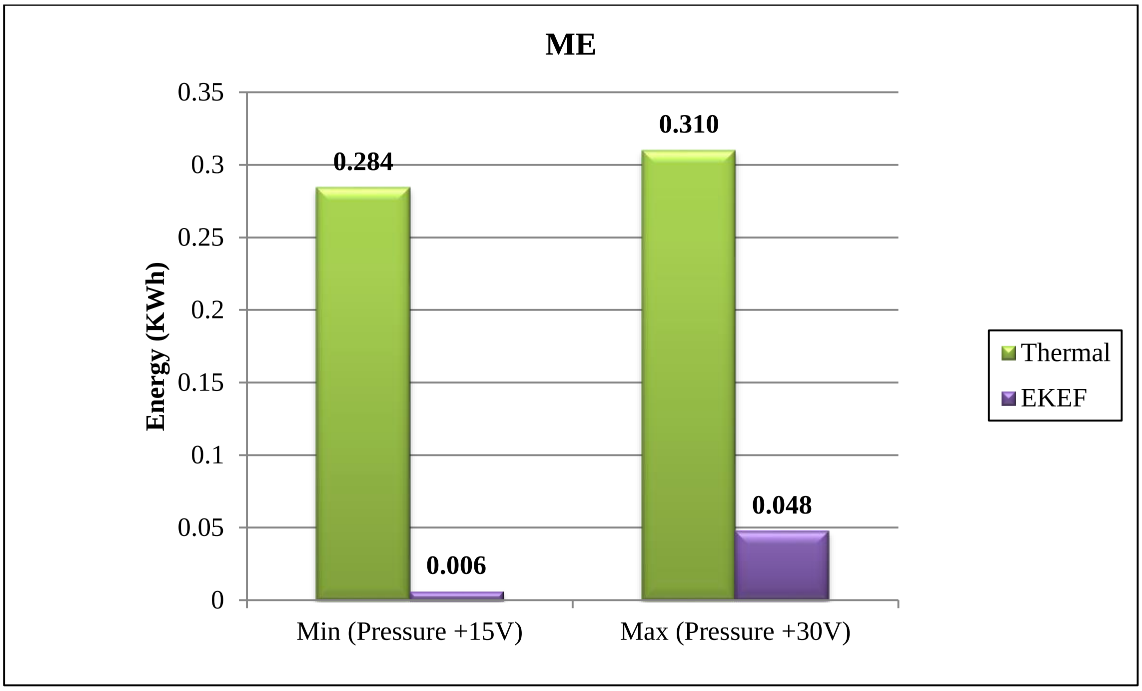


Figure 4.79. Comparison between the energy consumed during the application of EKEF to ME under 4bar pressure and three voltage values (15 and 30V) and the thermal energy required to remove same amount of water by evaporation

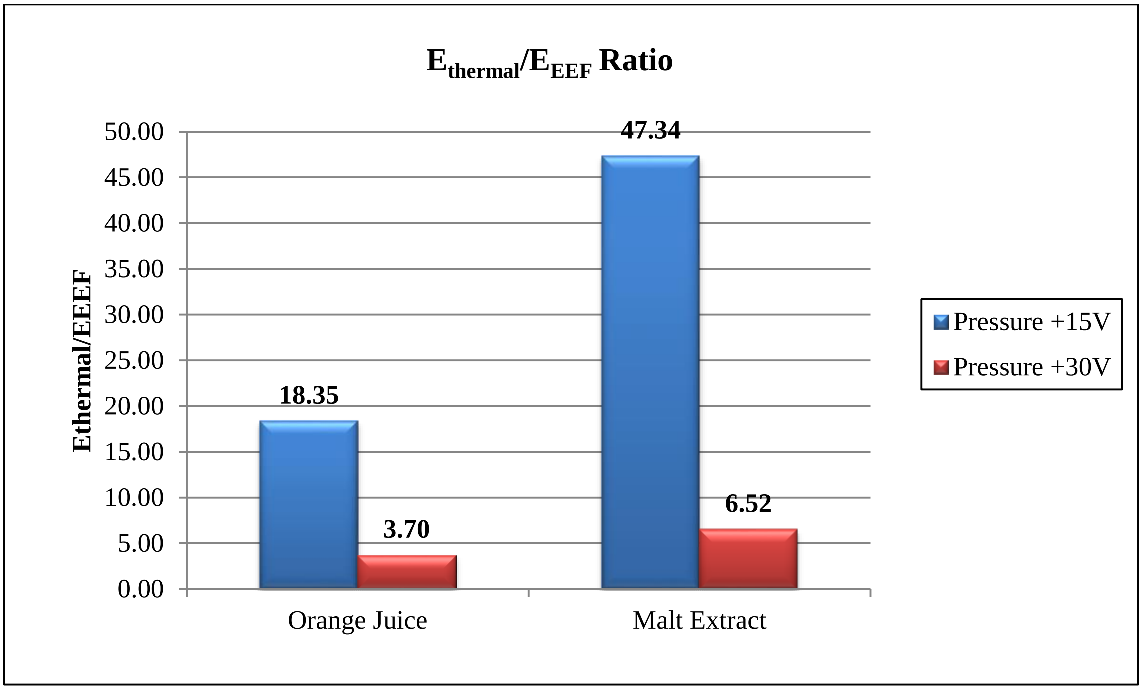


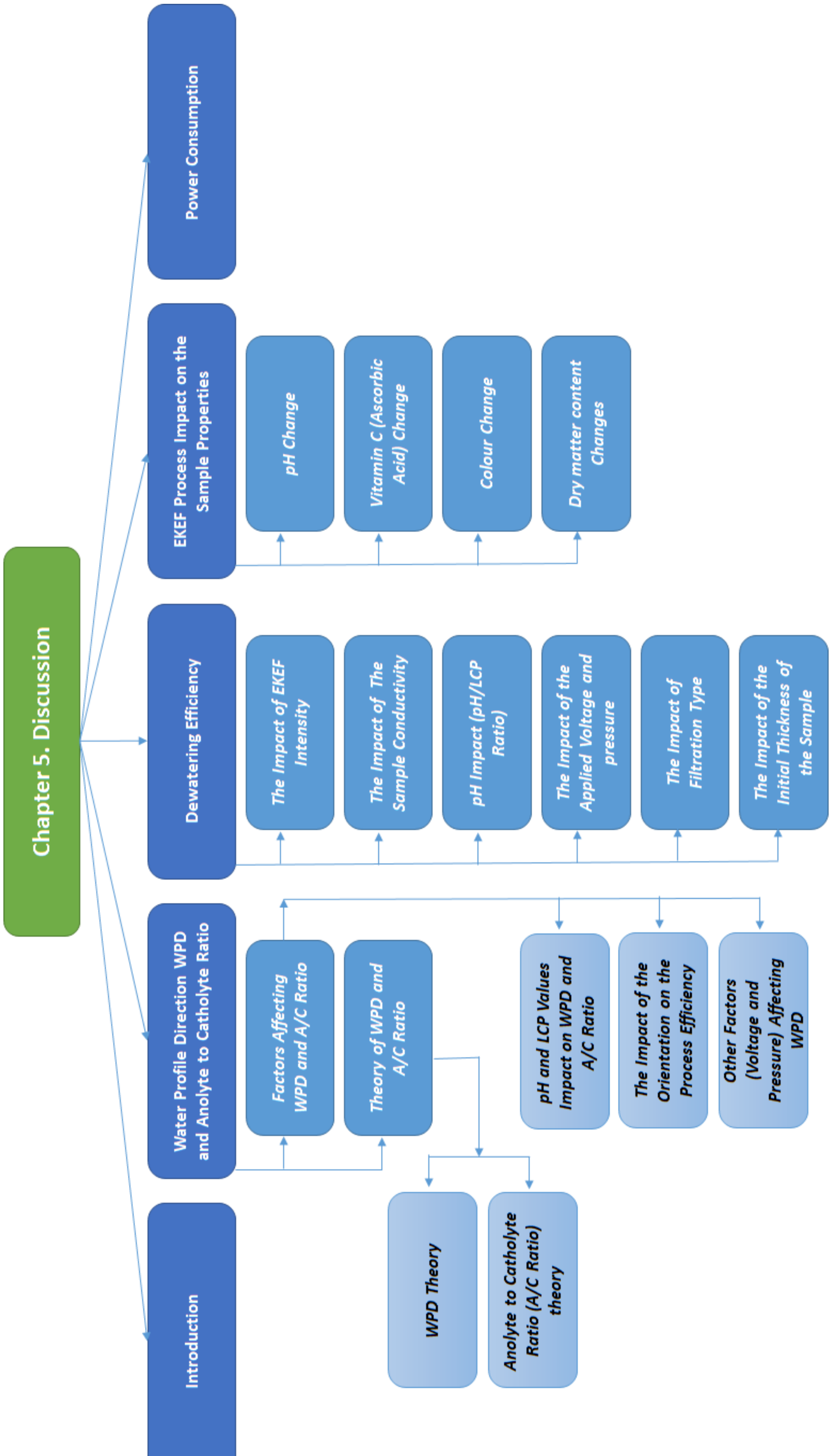
Figure 4.80. Energy saving ratio ($E_{Thermal}/E_{EEF}$) gained by concentrating OJ and ME using the application of EKEF under 4bar pressure and three voltage values (15 and 30V) in comparison with the thermal energy required to remove same amount of water by evaporation

For more details, energy consumption under previous methods was calculated in 4 different units: (KWh, KJ, KJ/mol water and KJ/Kg water), *Table 4.35*:

Table 4.35. Energy consumed during EKEF application on OJ and ME under 4bar pressure and three voltage values (15 and 30V) and the thermal energy required to remove same amount of water by evaporation (in 4 units)

	Energy (KWh)		Energy (KJ)		Energy (KJ/mol water)		Energy (KJ/Kg water)	
Method	OJ	ME	OJ	ME	OJ	ME	OJ	ME
Thermal	0.306	0.248	1102.95	891.53	43.000	43.000	2388.88	2388.88
Pressure +0V	$3.83\text{x}10^{-6}$	$3.83\text{x}10^{-6}$	0.01	0.01	0.001	0.001	0.030	0.037
Thermal	0.314	0.284	1130.18	1023.16	43.000	43.000	2388.88	2388.88
Pressure +15V	0.017	0.006	61.57	21.61	2.343	0.908	130.150	50.464
Thermal	0.345	0.310	1240.31	1116.09	43.000	43.000	2388.88	2388.88
Pressure +30V	0.093	0.048	335.35	171.19	11.626	6.596	645.905	366.425

Chapter 5. Discussion



Flowchart 6. Chapter 5 (Discussion) Content

5.1. Introduction

All experiments were centred on two main investigations:

(A) The major factors that influence the efficacy of EKEF.

(B) The changes and impacts that the application of EKEF has on the quality and integrity of the samples.

Results showed that there are several factors and conditions that control the EKEF process which change positively or negatively as they are modified; where positive changes mean a higher flow rate, less negative impact on the sample quality, and less power consumption.

The major factors investigated in these experiments were: EKEF intensity (represented by the electric current intensity), sample conductivity, applied voltage, applied pressure, filters pore size, initial sample thickness, as well as the sample chemical properties of the pH and the LCP values.

There were some impacts of EKEF on the process and the sample properties, most of these changes were investigated. The investigation centred on the impact of EKEF on the filtration flow rate (total, anolyte and catholyte), sample pH, sample colour, sample dry matter content and moisture content.

In addition, WPD between the two electrodes, the main filtration outlet and the optimal orientation for each material were thoroughly investigated; and the power consumption of this application was calculated and compared with alternative conventional thermal methods and their power consumption.

5.2. Water Profile Direction WPD and Anolyte to Catholyte Ratio (A/C Ratio)

In this present study, the occurrence of the two streams (anolyte and catholyte), corresponding to the respective movements toward either the anode or cathode, was observed and measured in all experiments. It is interesting to observe that many of the previous studies which investigated these movements observed only one stream (mostly the catholyte); for instance *Yuan and Weng (2003)* observed only catholyte during dewatering of municipal sludge cake and similarly *Habibi (2004)* also observed the movement of liquid toward the cathode during EKEF dewatering of oily sludge from a crude oil storage tank.

However, some other studies have investigated two streams such as *Yang et al. (2005)* who measured the two filtrates and found that applying electrokinetics to oily sludge increased both streams and resulted with unequal-weighted streams where the balance was in favour of the anolyte.

5.2.1. Factors Affecting WPD and A/C Ratio

The results of this present study showed that there were different factors affecting the direction and the volume of discharged filtrate (water) moving towards each outlet.

These factors were related to the sample chemical properties (pH and LCP values), the orientation of the main outlet and electrodes (normal or reversed orientation), or experimental conditions (applied voltage and pressure).

5.2.1.1. pH and LCP Values Impact on WPD and A/C Ratio

WPD depends on the net electrical charge of the suspension, when the net electrical charge is positive, the percentage of the positive ion content is higher than the negative ion percentage, thereby the WPD is toward the anode and A/C ratio is >1, while it is toward the cathode when the net charge is negative and A/C ratio is <1.

The net electrical charge of any particle depends on its pH and IEP values, where it is positive when pH value <IEP value and negative when pH value >IEP value.

A foodstuff is a combination of many compounds and particles which have different IEPs; therefore there is no specific pH value where the net charge of the whole material is 0 because of the interaction of the charges associated with the various components of the foodstuff. However, there is a pH value where most of the compounds are neutral and at that pH the conductivity of the material has the lowest value as most of the ions and particles net electrical charge is neutral. This pH value is referred to as the lowest conductivity point (LCP). When the pH value is < LCP value the net electrical charge of the material is positive, as the majority of its particles have a positively charged surface (pH < IEP), while it is negative when the pH values > LCP value as the majority of its particles have a negatively charged surface (pH > IEP).

Conductivity values for the titration solutions were shown in *Figure 4.1* for OJ and ME samples, and the LCP of OJ was 4 while it was 3.2 for ME samples *(Table 4.1)*; which means that the net charge of fresh OJ is positive as the OJ pH value of 3.5 is less than the OJ LCP value, while it is negative in ME as ME pH value= 4.55> ME LCP value.

Theoretically, the WPD would be expected to be towards the anode with an A/C ratio more than 1 in OJ samples and towards the cathode with an A/C ratio less than1 in ME samples when the EKEF is on according to the pH and LCP values of these two experimental materials as described by *Aziz et al. (2006)*.

This theory was demonstrated by *Table 4.2* which shows the data of several experiments on the two foodstuff materials; under the normal orientation (anode is the bottom electrode) and in the reversed orientation (Cathode is the bottom electrode). Data in this table show that the discharged filtrate moves almost equally toward the anode and the cathode when the EKEF is not applied (voltage=0V) due to the equal pressure on both outlets. However, the bottom outlet showed a small increase in discharge due to the gravity impact that pushes water down to the bottom anode.

Later when the electric field was switched on, the difference between the two outlets filtrates started to increase; the A/C ratio was > 1 for OJ in all experiments and for ME was < 1 in all experiments.

The fact that the WPD was toward the same electrode in both orientations (normal and reversed) regardless the position of the electrode whether it was at the top or bottom, confirms that when the electric field is on, WPD is related to the electrode charge (anode or cathode) not to the electrode position (either top or bottom).

5.2.1.2. The Impact of the Orientation on the Process Efficiency

WPD depends on the suspension net electrical charge and the electrode charge, but this fact does not mean that the main outlet position (top or bottom according to the orientation being normal or reversed *(Figure 3.19* and *Table* 3.3*)* has no impact on the total process outcome.

Determining the main outlet is important especially when the equipment is designed with one open electrode; in this case it is important to have the main water movement towards the open one. When the water movement is vertical (like in the rig used for this study), in this case it is important to choose the main outlet in a way that makes all other factors, which affects water movement and pushes water toward the main outlet, such as having the main outlet at the bottom to have the water movement supported by the impact of gravity.

The importance of choosing the correct main outlet position (top or bottom) by using the correct orientation was shown in *Table 4.3* and *Figures 4.2* to *4.4* where experiments were run under the same conditions but in two orientations (normal and reversed).

This filtrate discharge, moving to the anode in the experiments using OJ is because its pH value is less than the LCP; while in experiments using ME, it moves to the cathode as the pH value is higher than the LCP. Thus the best orientation to apply an electric field is the normal orientation for OJ when the anode (the main outlet) is the bottom outlet, and the reversed orientation is required for ME when the cathode (the main outlet) is the bottom outlet. In other words, WPD depends on the food material type (mainly its chemical properties), and the main outlet is best located at the bottom to benefit from the gravity impact in the same direction as the main WPD.

To conclude, in both orientations normal and reversed, water moved towards the anode (which is the bottom electrode with normal and the top with reversed orientation) in the OJ sample as pH value was < LCP value and towards the cathode in the ME samples as pH value was > LCP value. The normal orientation increased the dewatering efficiency in OJ experiments as the WPD was towards the bottom anode supported by the gravity, whereas the reversed orientation decreased it as it pushed water to the top anode working against gravity. In contrast reversed orientation increased the dewatering efficiency in ME experiments as the WPD was towards the bottom cathodes, whereas the normal orientation decreased it as it pushed water to the top cathode working against gravity.

5.2.1.3. Other Factors (Voltage and Pressure) Affecting WPD

In addition to the pH and LCP of food samples and the orientation, there are other factors affecting the WPD and the discharged filtrate volume such as the applied voltage and pressure.

The data shows that pressure had a positive impact on the dewatering process as rising pressure increases the mechanical force to push water through the filters; this increase was shown in both the anolyte and the catholyte which in turn increased the total filtrate. Increasing applied voltage also had a positive impact on the dewatering process but not equally on anolyte and catholyte.

5.2.2. Theory of WPD and A/C Ratio

Some previous studies have investigated water movements in non-food materials under the application of electrokinetics and observed only one stream (mostly the catholyte); looking at their experimental sample properties could give an insight about the reason for focusing on the catholyte. *Fourie et al. (2007)*, for instance, used an applied electric field to dewater mineral sands tailings (kaolinite and quartz) with a pH value of 6.4. The IEP of kaolinite varies between 3.3 to 5 *(Brian and Garrison, 1997)* This basic medium (pH>IEP) causes water to move toward the cathode, which was the basis of the rationale behind using six anodes in the outdoor test tank to push a higher amount of water toward a single cathode which was used as the extracting outlet *(Figure 5.1).*

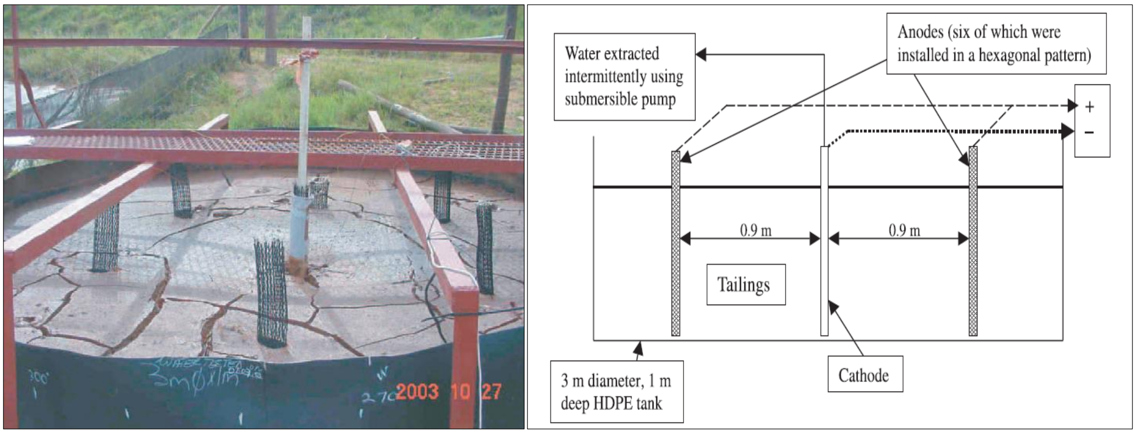


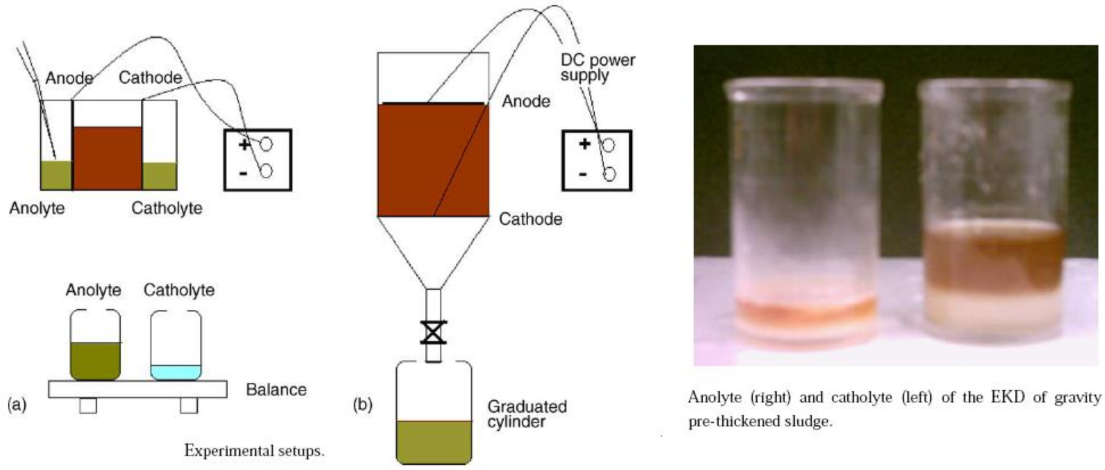
Figure 5.1. Cross section of (Fourie et al., 2007) outdoor experimental layout

Lamont-Black et al. (2007) also applied EKEF on three types of thickened kimberlite slimes from diamond mines in southern Africa. The pH value of these three types were 9.53, 7.88 and 8.63 (high pH values/ basic medium); and again the test equipment used the cathode as an outlet where the equipment provided an irrigated anode (bottom electrode) and open draining cathode (top electrode).

Some previous studies adjusted the sample pH to increase the alkalinity of the medium, for example the study done by *Aziz et al. (2006)* who adjusted the suspension pH to 10, using concentrated sodium hydroxide (NaOH). In this experiment, the effect of making the suspension alkaline was that the liquid then migrated toward the cathode, which also acted as a permeable medium.

However, some other studies experienced two streams (anolyte and catholyte) such as *Yang et al. (2005)* who measured the two filtrates and found that applying an electric field to an oily sludge increased both streams and resulted in unequal-weighted streams where the larger proportion was the anolyte.

Yang et al. (2005) worked on oily sludge with a pH value of 5.3-5.6 and IEP value of 5.9; most of the electro-osmotic flow was towards the anode as the pH of the sludge was lower than its isoelectric point which means that the positive charges on sludge particles predominated. *Yang et al. (2005)* focused on the two flows (the anolyte and the catholyte) *(Figure 5.2)*, and reported their volumes under several voltage values.



Experimental setups.

Anolyte (right) and catholyte (left) of the EKD of gravity pre-thickened sludge.

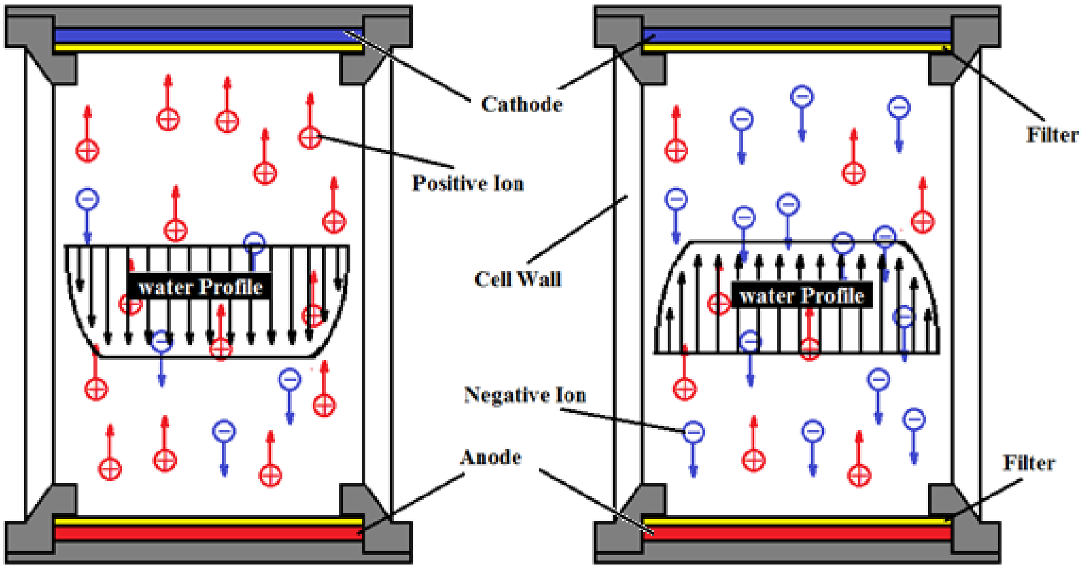
***Figure 5.2. Anolyte and catholyte of the electrokinetic dewatering of gravity pre-thickened sludge** (Yang et al., 2005)*

A significant part of the present study focused on the WPD and A/C ratio with a view to identifying guidelines on how to set up the electrodes and the outlets in cells designed to utilise EKEF.

Previously in *Section 5.2*, it was shown that the WPD depends on the net electrical charge of the sample, which depends on the sample pH and LCP value (or IEP value if it is measurable):

- When pH value<LCP (IEP) value: the net charge of sample is positive (and the surfaces of particles are positively charged): WPD is towards the anode and the A/C ratio is > 1.
- When pH value>LCP (IEP) value: the net charge of sample is negative (and the surfaces of particles are negatively charged): the WPD is towards the cathode and the A/C ratio is < 1.
- When pH value ≈ LCP (IEP) value: the net charge of sample is almost zero (and the surfaces of particles are neutral): there is no impact of electric field, water moves under pressure and the impact of gravity only. WPD is almost equal towards both electrodes and the A/C ratio is ≈ 1

Figure 5.3 shows the WPD under positive and negative net charge.



pH < IEP
A/C ratio > 1
Positive Net Electrical Charge

pH > IEP
A/C ratio < 1
Negative Net Electrical Charge

Figure 5.3. The impact of pH and LCP (IEP) values on WPD and A/C Ratio

Before deciding on the orientation of any equipment designed to use EKEF the chemical properties as well as the impact of all chemical and electrical forces that affect particle movement in such foodstuffs must be taken into account.

The following example will help to explain how to study all these factors:

5.2.2.1.WPD Theory

To concentrate foodstuffs in a vertical cell, two types of forces are used: (A) an electrical force by applying a voltage between two horizontal electrodes across the sample, and (B) a mechanical force applied by a pneumatic pressure ram. In addition to these two forces gravity also has an impact on juice particles and bits.

Figure 5.4 shows the setting up of the electrodes and the forces that affect water movement under three orientations (Control, normal and reversed) and in two mediums (acidic pH<IEP (or LCP) and basic pH>IEP (or LCP)).

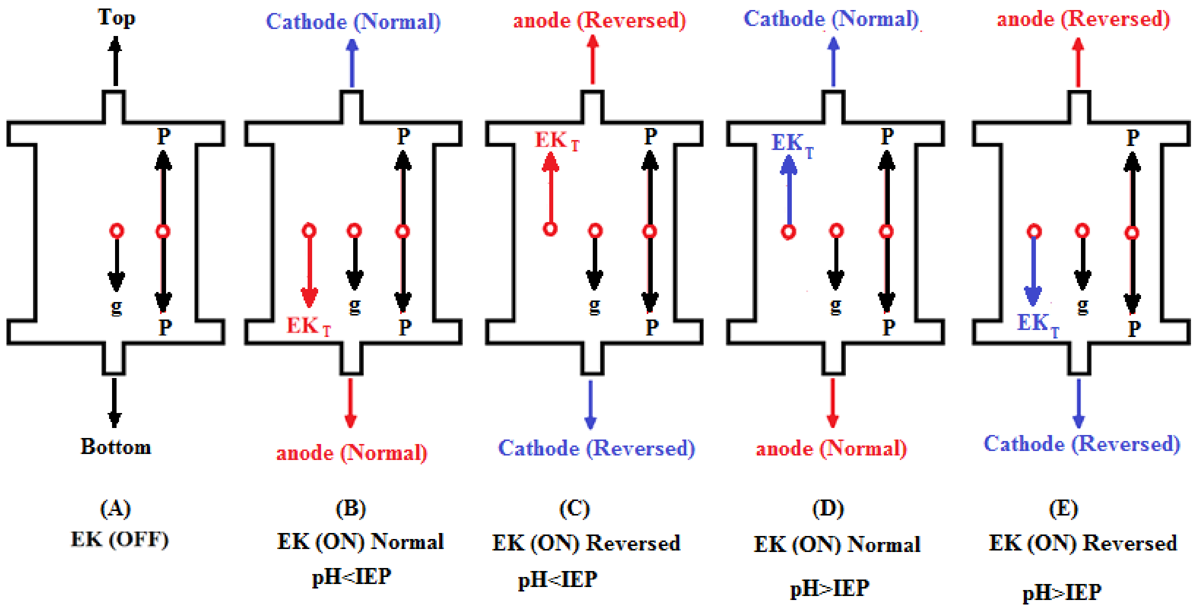


Figure 5.4. Forces affecting water movement inside an experimental cell under different orientations[7]

F_C: sum of the forces pushing particles to the cathode.

F_A: sum of the forces pushing particles to the Anode.

P: Pressure.

g: Gravity.

EK_C: EKEF toward the cathode.

EK_A: EKEF toward the anode.

EK_T (Total EKEF) = $|EK_C - EK_A|$

IEP: Iso-electric Point.

A: Anolyte.

C: Catholyte.

Three differing scenarios are considered to explain the significance of these factors on WPD:

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[7] A. Control situation, B. Normal situation and acidic medium when pH<IEP, C. Reversed situation and acidic medium when pH<IEP, D. Normal situation and basic medium when pH>IEP, and E. Normal situation and basic medium when pH>IEP.

Case (1): Electric Field is off (Control)

Water is influenced by the impact of pressure which pushes water equally toward both outlets and gravity additionally impacts by forcing water towards the bottom *Figure 5.4(A)*

$F_{Top} = P - g$

$F_{Bottom} = P + g$

WPD is almost equal toward both outlets with a small increase for the bottom outlet.

Anolyte ≈ Catholyte ⇒ A/C ratio≈ 1

Case (2): Electric Field is on and pH < IEP (LCP)

This scenario is equivalent to using OJ. In addition to previous forces (pressure and gravity), water is influenced by the electric field which pushes (via EKEF) water towards either the anode or the cathode according to the ratio of the positive and negative ion content. Since the net electrical charge is positive (pH<IEP (LCP)), $EK_A > EK_C$ and the EK_T is towards the anode.

- WPD is toward the anode (the bottom in the normal orientation and the top in the reversed orientation) as the net electrical charge is positive and the ions movement is toward the cathode.
- Anolyte > Catholyte ⇒ A/C ratio > 1
- Normal orientation (anode at the bottom) *Figure 5.4(B)*

  $F_C = P - EK_T - g$

  $F_A = P + g + EK_T$
- Reversed orientation (cathode at the bottom) *Figure 5.4(C)*

  $F_C = P + g - EK_T$

  $F_A = P - g + EK_T$

In this case choosing the normal orientation where the anolyte (the main outlet) in the bottom is the optimal choice as it is focusing the three forces (EKEF, gravity and pressure) to push water through it.

Case (3): The Electric Field is on and pH > IEP (LCP)

This scenario is equivalent to using ME. Since the net electrical charge is negative (pH>IEP (LCP)), $EK_A < EK_C$ and the EK_T is towards the cathode.

- WPD is toward the cathode (the Top in the normal orientation and the bottom in the reversed) as the net electrical charge is negative and the ions movement is toward the anode.
- Anolyte < Catholyte ⇒ A/C ratio< 1
- Normal orientation (anode at the bottom) *Figure 5.4(D)*

  $F_C = P - g + EK_T$

  $F_A = P + g - EK_T$
- Reversed orientation (cathode at the bottom) *Figure 5.4(E)*

  $F_C = P + g + EK_T$

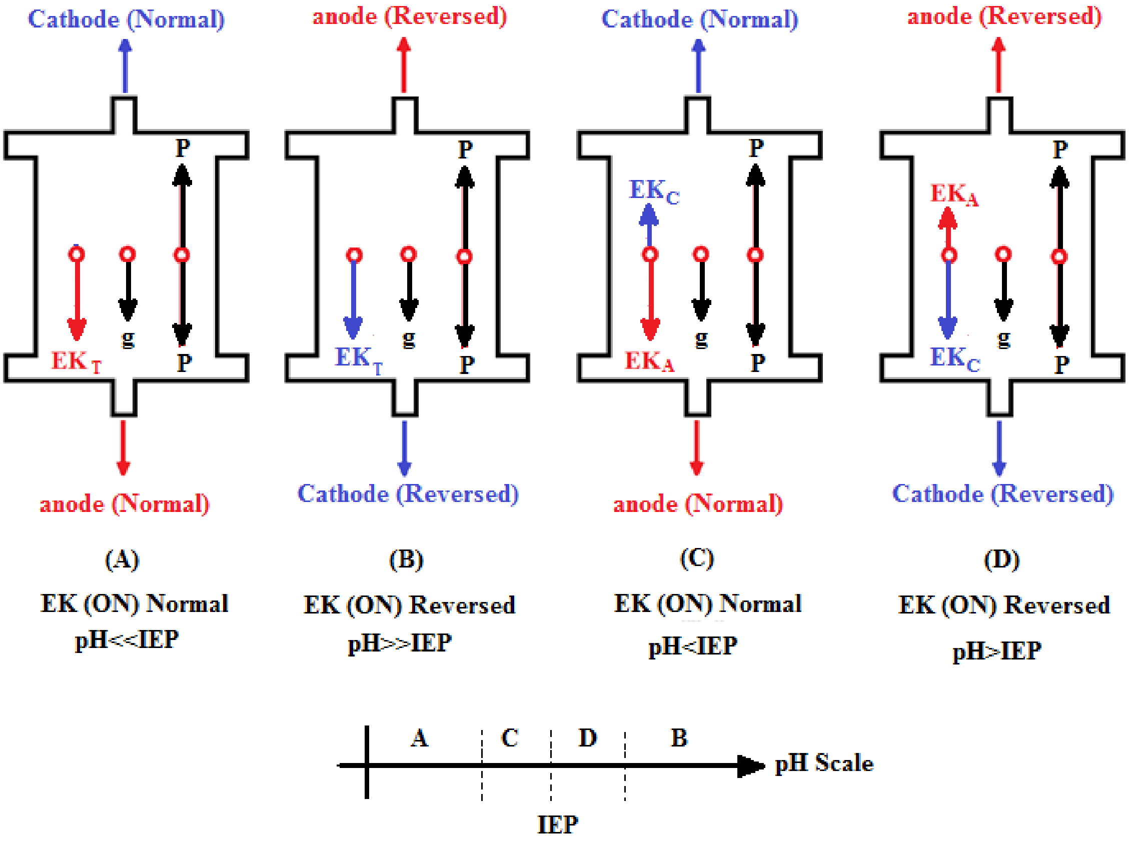
  $F_A = P - g - EK_T$

In this case choosing the reversed orientation where the catholyte (the main outlet) in the bottom is the optimal choice as it focuses the three forces (EKEF, gravity and pressure) to push water through it.

5.2.2.2. Anolyte to Catholyte Ratio (A/C Ratio) theory

Measuring the pH value of a suspension is required not only to compare it with the IEP (or LCP) to determine the WPD but also to have insight about the change induced in filtrate volumes.

Choosing the optimal orientation (the normal orientation when pH<IEP (or LCP) and the reversed when pH>IEP (or LCP)), does not mean that the whole filtrate will flow through the main outlet (the anolyte when pH<IEP (or LCP) and the catholyte when pH>IEP (or LCP)), as a part of the total filtrate will exit through the secondary outlet according to the A/C ratio. The difference between pH value and IEP (or LCP) controls the A/C ratio according to the orientations. If the optimal orientation is chosen (normal orientation in the acidic medium and reversed in the basic medium), four differing scenarios are considered to explain the impact of the difference between pH and IEP (or LCP) on the A/C ratio:



(A) EK (ON) Normal pH<<IEP

(B) EK (ON) Reversed pH>>IEP

(C) EK (ON) Normal pH<IEP

(D) EK (ON) Reversed pH>IEP

Figure 5.5. Impact of the A/C ratio on WPD in the optimal two orientations[8]

1) If the difference between pH value and LCP is relatively large, the experimental material contains significant amounts of either negative or positive ions and negligible amount of the opposite charged ions. The ions with the highest amount move to the electrode with the opposite polarity resulting in an EK_T impact in the opposite direction with an A/C ratio significantly higher or lower than 1, and here we have two cases:

   a) If the pH value is much less than LCP, the EK_C is almost negligible as the anions (negative ions) content is too low, thereby the EK_T will be mostly towards the anode which results in the anolyte volume being larger than the control outlet flow, and the catholyte volume is less than the control outlet flow depending upon the EK_T intensity. *Figure 5.5(A)* shows the normal orientation when the anode is at the bottom and the anolyte is supported by gravity in addition to the EK_T when the A/C ratio is significantly higher than 1.

b) If pH value is much more than the LCP, EK_A is almost negligible as the cation (positive ions) content is too low, thereby the EK_T will be mostly towards the cathode which

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[8] A. acidic medium, Normal orientation and the difference between pH and IEP is relatively large, B. basic medium, Reversed orientation and the difference between pH and IEP is relatively large, C. acidic medium, Normal orientation and the difference between pH and IEP is relatively small, D. acidic medium, Normal orientation and the difference between pH and IEP is relatively small,

results in the anolyte volume being less than the control outlet flow, and the catholyte volume is larger than the control outlet flow depending on the EK_T intensity. *Figure 5.5(B)* shows the reversed orientation when the cathode is at the bottom and the catholyte is supported by the gravity in addition to the EK_T with the A/C ratio being significantly lower than 1.

2) The difference between pH value and LCP is relatively small, the experimental material contains significant amounts of both the negative and positive ions, which move to the electrode with the opposite polarity after applying an electric field resulting in an EK_T impact in both directions depending on the positive to negative charge ratio and resulting in the A/C ratio being relatively close to 1, and here we also have two cases:

  a) If the pH value is a relatively lower than the LCP, the EK_A is relatively larger than EK_C, thereby the EK_T will be towards the anode and cathode in a percentage depending on the ratio of the positive and negative ions contained in solution which is relatively larger than 1, resulting in anolyte volume being significantly higher than the control outlet flow, and the catholyte volume flow a little higher than the control outlet flow depending on the EK_T intensity. *Figure 5.5(C)* shows the normal orientation when anode is at the bottom and the anolyte is supported by the gravity in addition to the EK_T with A/C ratio a little larger than 1.

b) If pH value is a relatively higher than LCP, the EK_A is relatively smaller than EK_C, thereby the EK_T will be towards the anode and cathode in a percentage that depends on the ratio of the positive and negative ions contained in solution which is less than 1, resulting in the anolyte volume being a little higher than the control outlet flow, and catholyte volume significantly higher than the control outlet flow depending on the EK_T intensity. *Figure 5.5 (D)* shows the reversed orientation when cathode is at the bottom and the catholyte is supported by the gravity in addition to the EK_T.

5.3. Dewatering Efficiency

Several data were collected to measure the EKEF process efficiency, one of the most important data was the dewatering efficiency, see *Section 3.5.1*.

Dewatering efficiency of any application is measured as the ratio of the total filtrate weight to the initial weight of water; where the total filtrate weight is the total weight exiting the outlets (the anolyte and the catholyte combined together).

Evaluating the impact of any condition on the dewatering efficiency gives a good idea about its impact on the EKEF process.

The impact of several conditions on the dewatering and EKEF efficiencies were investigated, these conditions, are related, either to the sample properties, or the parameters of the experiments, and are described in the following sections:

5.3.1. The Impact of EKEF Intensity on the Dewatering Process

Regardless of the variable conditions that affect the EKEF process, this process in general has a positive impact on dewatering efficiency. This impact has been investigated earlier by researchers on some of the soil dewatering applications.

Raats et al. (2002) reported that the solids contents in drinking water sludge increased from 17% to 24% by combining electrokinetic dewatering with a belt press in a gravity-driven thickening belt combined with an additional energy consumption of 60 kWh/t of sludge. A high water content reduction from 87.8% to 62.6% was also achieved by *Yuan and Weng (2003)* with municipal sludge in 41h at a potential gradient 5.0 V/cm using a 6 cm long electrokinetic dewatering cell.

Besides the sludge dewatering, studies have focused on electrokinetic remediation of soil, implanting two electrodes in the soil vertically *(Ho et al., 1997)* or with horizontal-electrodes *(Ho et al., 1999)* which achieved removal of more than 90% of metal contamination.

Studying the application of EKEF has not been limited to soil dewatering and mineral removal areas; research has also focused on electrokinetic removal of neutral soluble or insoluble organic compounds from soils. Greater than 90% removal of hydrophobic polycyclic aromatic hydrocarbons (PAHs) by EKEF has been reported by *Maini et al. (2000)* in bench- and pilot-scale experiments, while in another study, *Ho et al. (1999)* reported 98% removal efficiency of *p*-nitro-phenol in one pilot unit *(Yang et al., 2005)*.

In this study the impact of EKEF was divided into two types, firstly: the relationship between filtration efficiency change over time (represented by the change of the flow rate value) and the change of the EKEF intensity (represented by the electric current intensity), and secondly: the impact on the total dewatering efficiency.

The impact on the total dewatering efficiency was investigated under several conditions due to the variation of this impact under different orientation and factors. The relationship between the flow rate and the EKEF intensity was positive *(Table 4.5)*. This positive relationship means that a higher EKEF intensity will result in higher flow rates and in turn a higher dewatering efficiency; and all factors that increase the EKEF intensity play a positive role in improving the process.

5.3.2. Sample Conductivity: Change and Impacts on Dewatering Efficiency

The total conductivity of the process depends on both the electrode conductivity and the food sample conductivity, and it changes as they change. The electrode material was chosen to be Titanium to minimise the electrode corrosion and the degradation of the electrodes which helps to avoid any contamination, and produce relatively stable electrode conductivity. Therefore, the main change in the conductivity process comes from the changes that occur in the food sample conductivity during the process.

Figure 4.9 shows that conductivity fluctuated over time due to the fluctuation of the electric current intensity; in addition it was noted during the experiments that this fluctuation always started when the gas bubble clusters started to flow out through the top outlet. This fluctuation is due to the gas generation, which was also higher under 30V than 15V as shown in *Figure 4.33* and *Table 4.20*, creating gas layers between the sample layers. These gas layers separate sample layers and decrease the connection between them which in turn decreases the sample conductivity and the electrical current flowing.

Different materials under the same voltage values show different initial conductivity, and since the electrode conductivity is stable, the variation between materials depends mainly on the sample conductivity which depends on the content of charged ions and particles.

In addition, higher voltage values result in higher electric current intensity in the same material at the same conductivity.

Change in the initial conductivity not only affect the shape of the flow rate change, but also affect the total amount of the final filtrate which represents the filtration outcome, as shown in *Figure 4.10*, which represents the impact of different initial conductivity values (0.22, 0.33 and 0.5S) on the dewatering efficiency (represented by the weight of the total filtrate). This figure shows that the initial conductivity value plays a significantly positive impact ($P < 0.001$) on increasing the dewatering efficiency, which means that the higher the initial conductivity value is the higher the dewatering efficiency.

5.3.3. *pH Impact (pH/LCP Ratio)[9] Upon WPD and Dewatering Efficiency*

Each food material has its own natural chemical properties, but adjusting the initial pH could play a positive role in improving the process. Some other studies adjusted the sample pH to increase the alkalinity of the medium; one of these studies was done by *Aziz et al. (2006)* who adjusted the suspension pH to 10 using concentrated sodium hydroxide (NaOH), the liquid then migrates toward the cathode, which also acts as a permeable medium.

The results of adjusting the initial pH of OJ samples show that the bigger the difference between the pH value and LCP value (LCP=~4), the higher the final outcome *(Table 4.6* and *Figure 4.11)*. Increasing the difference between pH an LCP is effective in both sides (either increasing the acidity (pH/LCP ratio<<1) or the alkalinity (pH/LCP ratio>>1)), the highest total filtrate weight was in OJ sample with pH=1.5 and then with pH=6.5.

This figure shows that the total filtrate weight increased as the difference between the pH and the LCP values increased; according to the curve, the lowest outcome was around the LCP in the range of pH between 4 and 5.

The total outcome of the control experiment was close to the outcome of the LCP, which demonstrates that when pH is close to the LCP (pH/LCP ratio≈1) the suspension is mostly neutral which reduces or cancels the impact of EKEF.

The larger the difference between the sample pH and the LCP the higher the current intensity, due to the increase of the amount of the charged particles and ions, which increases the net electrical charge of the suspension; this high current resulted in a greater impact of EKEF which in turn increased the total filtrate weight.

In addition, changing the pH/LCP ratio changed affected water profile direction which was toward the anode with pH/LCP ratio < 1 (resulting in higher anolyte A/C ratio > 1) *(Figure 4.15)*, and toward the cathode with pH/LCP ratio > 1 (resulting in higher catholyte A/C ratio < 1), as shown in *Figure 4.16*.

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[9] In this study, LCP is used instead of IEP due to the difficulty of measuring the IEP of the experimental materials; however, findings are correct for IEP if it is measurable in the sample.

5.3.4. The Impact of Applied Voltage and Pressure

Voltage application plays a role in WPD as water starts to move towards one of the electrodes after applying the voltage. In addition, the voltage has an impact on the total filtrate weight and the EKEF dewatering efficiency.

The impact of the applied voltage on the dewatering efficiency for non-food materials has been previously discussed in several studies. For example, the impact of the electro-osmotic dewatering was investigated on a commercial scale with sludge containing a high content of aluminium ions, sludge which originated from the production of drinking water *(Buijs et al., 1994)*. In that study, applying a voltage improved the dewatering process and led to an increase in the solids content from 4% to 6.8% m/m (at 10 V DC across the slurry on top of the belt) and to 8.8% m/m at 20V.

Raats et al. (2002) also investigated the effect of EKEF on the dewatering of waste slurry. Their results showed that without the application of an electric DC-field, the final dry solids contents was less than 17% m/m, while it increased to 19.5% and 20.5% m/m after applying a DC voltage of 15 or 30 V respectively.

This impact was also investigated in reducing the weight of a protein-rich sludge; the dewatering efficiency increased from 51.9% to 56.3% when the applied voltage increased from 20V to 30V while it was 27.2% without applying any voltage *(Yang et al., 2005)*.

In this present study, the voltage also played a positive role on increasing the process efficiency. There was a positive net dewatering efficiency (NED) after applying EKEF of up to 7.43% in OJ experiments (5.5 hours) and to 10.68% in ME experiments (3.5 hours), this NED was higher with 30V compared with 15V in all the experiments as the total filtrate weight increased when the voltage was increased.

This positive impact of increasing voltage can be related to its impact on the ions movement. Since water movement depends on the ions movement; increasing voltage applied on the same material (same conductivity) increases the movement of the charged particles and ions to the opposite electrode which in turn increases the electric current intensity and speeds up the water movement, as well as to the other electrode. The effect of voltage is also influenced by other conditions in the experimental material, pressure filter pore size, sample's initial thickness.

This impact on the dewatering efficiency happened because the final total filtrate weight was higher under 30V compared with 15V in both OJ and ME experiments. However, the mean value of OJ total filtrate over time was higher under 15V than its value under 30V in the OJ experiments, while it was higher under 30V in the ME experiments *(Figures 4.21* and *4.22)*. Analysing data using Time Series analysis showed that up to a specific time the outcome of applying 15V was greater before the 30V impact accelerated and crossed the 15V line in what is called the voltage crossing point (VCP). This phenomenon is also shown in *Figure 4.23* to *4.25*. Data in *Table 4.11* shows that VCPs happened after around 40% of juice had exited as filtrate. The distance between the electrodes was 34.5mm with 1L volume at the beginning of the experiments and decreased over time; and it was interesting that the VCPs occurred when the distance between electrodes became around 18mm in the three experiments which could be the reason behind this phenomenon as the impact of voltage is influenced by the distance between the electrodes as shown in *Section 5.3.6*. The other reason could be the concentrate dry matter content which was around 24% when the VCPs occurred.

One of the ways to improve the process and decrease the negative impact on food quality compounds, as well as decreasing the power consumption, is using lower voltage values and then increase the voltage gradually if/when needed.

In addition to the voltage positive impact *Table 4.7* shows that increasing applied pressure value improved the net dewatering efficiency by up to 13.69% and was higher with 4bar compared with 3 and 2bar in all experiments.

5.3.5. Impact of the Filter Pore Size

Filter pore size plays an important role in the filtration process, as it controls the particle size of the filtrate that can go through it. The smaller the pores the smaller the particles passing into the filtrate stream. When the process goal is to concentrate foodstuff (juices), the concentrate then is the important part and any compounds that pass through with the filtrate are considered as a loss unless they are captured later in other steps. On the other hand, if the filter pores are too small then the required pressure to push filtrate through will need to be increased.

In addition to the pressure, filter pore size can affect the filtration process outcome and efficiency as well. Comparing the net dewatering efficiency (NDE) between the two sizes of the filter pore used in this study (0.2µ and 1µ), it is clear that the larger the filters pore size the higher the NDE, and that this is also affected by the applied voltage. Bigger pores allow more particles to go through the filters which in turn slow down the cake formation and reduce the negative impact of cake formation on the flow rate. Under the same conditions, the pressure values used to push water through the larger pores carrying all particles with sizes smaller than the cut off was faster than the smaller pores which capture more particles and form a filtration cake faster, and this slows down the filtration process sooner. However, the bigger the pores are the more the loss of particles which exit with the filtrate, and this could be a disadvantage to the overall concentrate product quality.

5.3.6. The Impact of the Sample's Initial Thickness (the Distance Between the Electrodes)

The impact of the sample thickness (the distance between the electrodes) on the performance of EKEF was investigated in earlier studies on non-food materials; these studies reported the negative impact of sample thickness in the filtration enhancement under the application of electrokinetics.

Lamont-Black et al. (2007) investigated the impact of EKEF on soil dewatering of two sample thicknesses of 35mm and 50mm under three voltage values 0V, 15V and 30V, where the percentage of the solid content was measured after 35min. The results showed that the solid content increased from ~59% to ~73% in the 50mm thickness under the higher voltage 30V compared with the control, while it increased from ~62% to ~83% in the 35mm thickness under the higher voltage 30V compared with the control, suggesting that the lower thickness was better for dewatering.

Yang et al. (2005) also investigated this impact by comparing the net dewatering efficiency under 0V, 10V, 20V and 30V in three thicknesses 4, 6 and 8cm. the NED was only 13% at 8cm while it increased to 16.1% and 29.1% in 6cm and 4 cm respectively, confirming the benefit of reducing distance between electrodes.

Since all experiments in this present study were run in the same cell with the same inner and outer diameters, then the only variable dimension is the distance between the two electrodes where the sample is captured; one of these electrodes (the bottom one) is connected to the piston and moves up and down freely. The initial distance between the two electrodes depends on the sample volume; this distance is considered as the sample's initial thickness.

Three volumes of the ME sample were used (0.5, 1 & 1.5L) which resulted in thicknesses (17.3, 34.6 & 51.9mm) respectively for inner diameter of 192mm. The results clearly demonstrated similar effects to those described above, with a decreasing NED with increased distance between the electrodes. This was most likely due to the higher electric current intensity with smaller thickness as the electrodes are closer, which in turn increases the impact of EKEF.

This impact on electric current is because increasing the distance between the two electrodes reduces the capacitance according to the *Equation 17*:

$$C = \varepsilon_r \frac{\varepsilon_0 A}{d}$$ *(Equation 17)(Terzic et al., 2012)*

Where:

C: is the capacitance in farads (F),

ε_r: is the relative static permittivity (dielectric constant) of the material between the electrodes,

ε_0: is the permittivity of free space,

A: is the area of each electrode, in square meters and

d: is the separation distance (in meters) of the two electrodes.

This capacitance reduction results in dropping in the electric current intensity, according to *Equation 18*:

$$I = C \frac{dV}{dt}$$ *(Equation 18)(Chaniotakis and Cory, 2006)*

Where:

C: is the capacitance in farads (F),

I: is the electric current intensity (Amp),

V: is the voltage, and

t: is the time; for DC (constant in time) signals (dV/dt =0)

5.4. EKEF Process Impact on the Sample Properties

Just like any other method, EKEF has an impact on the chemical properties and the quality compounds of the foodstuffs; these impacts are because the EKEF phenomenon happens due to the ions movements which create an electric current, some heat and gas.

In this part of the study the focus was on the potential impact of the application of EKEF and comparison of it with the impact of the more commonly used thermal dewatering methods.

5.4.1. pH Change

The pH values of the samples are very interesting when considering the EKEF process, it is not only that the initial sample pH affects the EKEF process by affecting the main direction of the water or WPD and determining the main outlet (anode or cathode); but also that the EKEF process has an impact on the sample pH which changes over time under the electric current application.

This change was reported before by *Yang et al. (2005)*, who stated that the distribution of pH along the length of the sludge was increased as soon as the cell was connected to the DC power supply, this distribution increased from around 5.4 to above 6.0 except for the interface of sludge and anode; and pH value changed from ~7 next to the cathode to ~2 next to the anode, while the initial pH was 5.3-5.6.

In the present study, pH values were measured for the anolyte and the catholyte every half hour which represents the pH value next to the anode and cathode (the maximum and minimum values). Since there is no way to measure the distribution of pH along the sample due to the design of the cell, the final concentrate pH was measured after blending.

Figures 4.34 and *4.35* show that the anode had the lowest pH value while the cathode had the highest in the two orientations (normal and reversed) regardless of the electrode position (top or bottom), which means that the pH value is related to the electrode charges.

Going back to *Equations 2 & 3*, when the electric field was applied, it can be surmised that water electrolysis started at the anode and the cathode producing cations (H_3O^+) at the anode *(Equation 2)*, and anions (OH^-) at the cathode *(Equation 3)*; this ion production results in an acidic anolyte (with low pH value) and basic catholyte (with high pH value) exiting through the anode and the cathode respectively.

The pH value of the final total filtrate (all the filtrate combined at the end of the process) depends on the pH of both the anolyte and the catholyte and their percentages in the total filtrate.

Figures 4.36 and *4.37* shows that the change in the pH value of the concentrate under different voltages was insignificant compared with the fresh sample pH, as the ions produced, which cause the pH change, move to the opposite electrode to exit the cell with one of the flows (either the anolyte or the catholyte depending on the type of the ions), which also resulted in a significant change in the total filtrate pH value especially when applying 30V.

In addition to the ions movement impact, *Table 4.21* and *Appendices 5* show that there is a significant relationship between the change of the pH and the flow rate over the time as the amount of the immigrant ions increased with the high flow rate which speeds up the pH change to more acidity in the anolyte and more alkalinity in the catholyte.

5.4.2. Vitamin C (Ascorbic Acid) Change

Thermal methods have a negative impact on the heat sensitive compounds of fruit juice; degradation of these compounds depends on the type, temperature and the other conditions of pressure and oxygen presence. One of the most important heat sensitive compounds in OJ is vitamin C.

Vitamin C is highly sensitive to degradation and has very low stability in solution. In addition, vitamin C is oxidized readily in light, air and when heated. Because it is also water soluble, heating in water (like thermal treatment) causes the vitamin to be oxidized and also to leach out of the food into the water *(Tannenbaum and Walstra, 1985; Lee et al., 2004)*.

Vitamin C degradation in OJ at various temperatures heated by different methods was investigated previously by *Vikram et al. (2005)*; manually squeezed orange samples were processed by different methods (conventional, ohmic, infrared and microwave) under different time and temperature combinations as shown in *Section 2.2.2*, *Figure 2.1*.

Lower temperatures have also had a negative impact on vitamin C content, the thermal degradation of vitamin C in OJ was analysed over in a 20-45°C temperature range by *Manso et al. (2001) Section 2.2.2*, *Figure 2.2)*.

In the present study there was no degradation in vitamin C content of the Control concentrated juice while the control total filtrate lost about 11% of its vitamin C content *(Figure 4.38)* due to the contact with air unlike the concentrated juice which was inside the cell and was not exposed to the air. However, after applying EKEF for 5.5hours, vitamin C retention of the concentrated juice dropped to 41% under 15V and 7.14% under 30V. Also the retention of the total filtrate was influenced by the impact of the EKEF, where it dropped to 47.8% under 15V and 35.45% under 30V.

The maximum temperature of the electrodes due to the application of EKEF was around 25-35ºC, and according to *Figure 2.2* after 5.5hours the vitamin C content drops to 95% at 20 ºC, 85% at 30 ºC, and 55% at 45 ºC *(Manso et al., 2001)*, which means that the vitamin C content drops more rapidly at a specific range of temperatures under the influence of EKEF compared with the same temperature impacts without the use of EKEF. This negative impact could be due to the combination of heat and oxidization resulting from the gas generation by EKEF, this gas generation contributes to vitamin C degradation due to the high sensitivity of vitamin C to the oxygen which oxidises ascorbic acid to dehydroascorbic acid as shown in *Figure 5.6*.

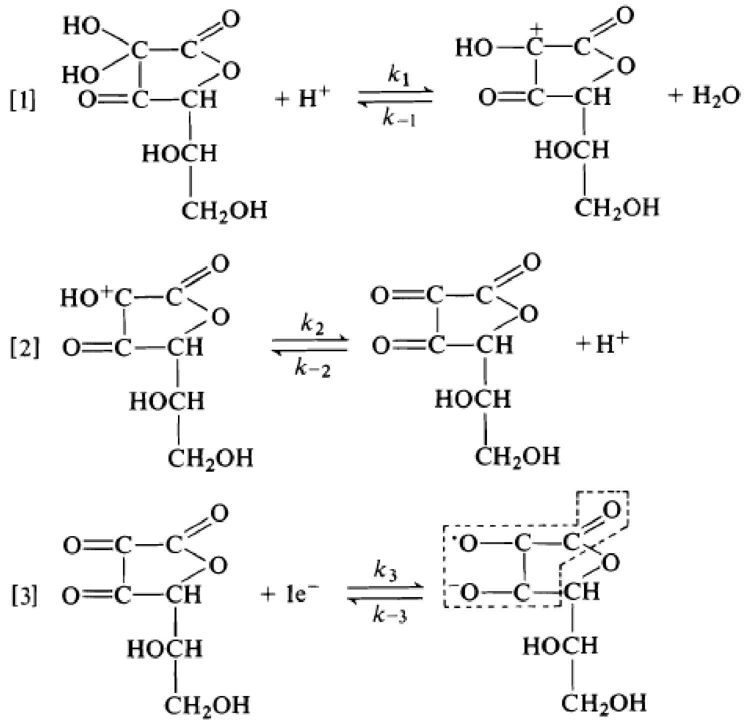

Figure 5.6. Ascorbic acid oxidisation mechanism (Ruiz et al., 1977)

However vitamin C retention was higher under the EKEF process compared with its retention value under conventional thermal methods, as it needed about 10-15min at 50-75ºC and 3min at 90ºC to drop to 50% or less as shown in *Table 2.2*, while it took 2:50h under EKEF 15V and 1:15h under 30V for OJ to drop to just 50% of vitamin C retention *Figure 4.39*.

The main degradation of vitamin C content occurred in the catholyte which lost its content very rapidly in comparison with the anolyte. This difference between the anolyte and the catholyte could have resulted from the slow flow rate of the catholyte which results in a thinner juice layer and longer contact time between juice and the electrode which in turn increase the negative impact of the heat effects on the Vitamin C in the juice as well as the direct ionisation of vitamin C at the electrode. However, the main outlet from the application of EKEF to OJ in OJ experiments is the anolyte which means that the main volume of the total filtrate exited through the anolyte which in turn decreased the total loss of the vitamin C content in the final total filtrate, as shown in *Table 4.22*.

Using filters with bigger pore size had a positive impact on the vitamin C retention of OJ total filtrate, as the vitamin C retention of the total filtrate going through the 1µ filters was 57.4% in comparison with 35.45% using 0.2µ filters after 5.5h of process. However, there was no significant difference in the vitamin C retention of the concentrates captured by both filters; this insignificance in the vitamin C retention of the concentrate was because these filters were in a direct contact with the electrodes in both experiments, and reached the same temperature when the electric field was switched on, and therefore, the impact of heat on the vitamin C was the same for both experiments with 1µ and 0.2µ filters.

To investigate the potential reasons behind the vitamin C loss, the correlation between the change of the vitamin C retention and the pH change was calculated using *Pearson correlation*, in addition the correlation between the change of vitamin C retention and flow rate change was checked in the anolyte, catholyte and total filtrate.

Table 4.23 shows that there is a significant relationship between pH change and the change of vitamin C retention as well as between vitamin C retention and the flow rate, although that the relationship with the flow rate change is less significant. However, *Figures 4.45* to *4.50* show that the maximum loss of vitamin C does not happen at the same time of the maximum change of pH, while *Figures 4.51* to *4.56* show that the maximum loss of vitamin C occurs when the flow rate is at its lowest value. In addition, vitamin C retention dropped in both the anolyte and catholyte where pH decreased and increased respectively, which means that the relationship between the pH change and the vitamin C change is because both of them are related to the flow rate change as shown in *Table 4.21*, *Appendices 5* and *Figures 4.51* to *4.56*.

To conclude, EKEF application has a negative impact on the vitamin C content but this impact is significantly smaller in comparison to the impact of conventional thermal methods. The impact of EKEF is influenced by the processing time and the temperature of the electrodes, that increase with higher voltage values, as this negative impact increases with a longer processing time and higher temperature (higher voltage values).

5.4.3. Colour Change

Colour is one of the most important visual sensorial quality parameters, and one critic component of first impression formation. Thermal methods have a negative impact on the heat-sensitive colour compounds of OJ; the degradation of these compounds depends on the type of juice and the temperatures used in processing.

There appeared to be no change in the concentrated juice colour under 0V. However, discoloration started to appear when voltage was applied and increased when voltage and pressure values increased. The discoloration was primarily at the top layer next to the top electrode (the cathode) only. Blending the concentrated juice after treatment reduced this discoloration as the majority of concentrated juice did not change in the colour as shown in *Figure 4.57*.

Comparison between the colour of the blended concentrated juice under the application of three different voltages (0, 15 and 30V) showed that the colour of the concentrate got darker as the voltage was increased *(Figure 4.58)*. In addition, the colour of samples decreased around 5%, 10% and 16% under 0V, 15V and 30V respectively on the light–dark spectrum which means that the concentrate got darker under the influence of EKEF and this discoloration increases under higher voltages *(Figure 4.59)*; and the browning index of the concentrate increased by 9%, 10% and 13% under 0V, 15V and 30V respectively compared with the fresh OJ browning index *(Figure 4.60)*.

Filtrates showed a neutral colour (very light) for the control samples and early stages of the 15V and 30V samples. Colour started to appear in the catholyte after 3hours of the experiment and 1.5 hours under 15V and 30V respectively. While in the anolyte, colour appeared clearly only in the 30V catholyte after 4hours as shown in *Figure 4.61*.

Figure 4.67 shows that the ethyl acetate phase turned into yellow in both fresh and concentrated samples while it remained blanc in the anolyte and catholyte samples which means that there is no carotenoids in the anolyte and catholyte samples and proves that the colour, which disappears from the control filtrate and shows later, is not related to carotenoids or any other juice compounds.

As mentioned in *Section 2.2.3*, the browning reactions in foods could be divided into: an enzymatic reaction (phenolase browning) and non-enzymatic reactions (Maillard, caramelisation and ascorbic acid oxidation) *(Nursten, 2005)*. The maximum temperature of the electrodes due to the application of EKEF was around 25-35ºC, which excludes the impact of Maillard reaction (90ºC) and caramelisation reaction (120ºC). Comparing the discolouration times in *Figure 4.62* with *Figures 4.51* to *4.56*, one could see a relationship between vitamin C degradation and discolouration as colour started to appear in the catholyte after 3hours and 1.5 hours of the experiment under 15V and 30V respectively. While in the anolyte, colour appeared clearly only in the 30V catholyte after 4hours (*Figure 4.62*); and these are the time when vitamin C content was almost lost totally (*Figures 4.51* to *4.56*). Going back to *Section 2.2.3* the formation of dehydroascorbic acid and diketogluconic acids from ascorbic acid oxidisation reaction (*Figure 5.6*) is thought to occur during final stages of the drying process and is capable of interacting with the free amino acids, non-enzymatically, producing the red-to-brown discoloration.

The difference of colour between the anolyte and catholyte is a dilution effect and is due to the differing flow rate out of each outlet which affects the vitamin C degradation; in this case using bigger-pores filters, if possible, could help speeding up the whole process and in turns increase the discoloration time as shown in *Figures 4.63* to *4.666*.

5.4.4. Dry matter content Changes

As the dewatering process progresses, the dry matter content of the concentrated juice keeps increasing due to the decrease of the water content. Different voltage values have different impacts on the dewatering process efficiency and in turn they have differing impacts on the dry matter content.

Figures 4.68 and *4.69* show that applying EKEF helped to increase dry matter content by 15.5% and 19% for OJ concentrate and by 12% and 14% for ME concentrate after 5.5hrs under 15V and 30V respectively while the maximum increase in dry matter content was about 14.75% and 9.63% under the control orientation after the same time for OJ concentrate and ME concentrate respectively. However, voltage values did not have a significant impact on dry matter content of the total filtrate.

Under the same condition of voltage and pressure, the main factor that impacted on filtrate dry matter content was the filter pore size, the larger the pore more of the dry matter passes through them.

All previous ME experiments were set up as the reversed orientation (the cathode at the bottom); to check whether dry matter contents were influenced by the electrode charge (anode or cathode) or the electrode position (top or bottom), the two orientations (normal and reversed) results under 15V, 4bar and through 1µ filter were compared as shown in *Figure 4.75*.

This figure shows that the lowering in dry matter content was in the normal catholyte (the top outlet stream) and the reversed Anolyte (the top outlet stream); which means that the effective factor is the position of the electrode. This result as well as the fact that the difference was significant between the anolyte and catholyte dry matter content under 0V and 15V but insignificant under 30V indicates that this impact could be related to the suspension separation into two phases as most of the ME bits deposit in the bottom and not due to the impact of EKEF.

To conclude, EKEF application has no significant impact on the filtrate dry matter content, while has an impact on the concentration dry matter by speeding up the filtration process and in turn decreasing the concentrate water content.

5.5. Power Consumption

Power consumption is one of the most important criteria to evaluate any food manufacturing process as it plays very important role in estimating the final product cost as well as the process impact on the environment. Decreasing power consumption is a very tempting area for researchers interested in new applications and methods to save energy.

The application of electrokinetics has been studied before by several researchers in terms of its power requirement. *Lo et al. (1991)* reported that using electrokinetics to strengthen soft and sensitive clays was considered economically impractical (primarily because of the high operating costs). This note came to support *Bjerrum et al.(1967)*, who used electrokinetics on clay as well and found that the cost of electricity was 25% of the total project cost, which is quite high.

At this level of cost, electrokinetics would most certainly only be used as a last resort when there is no alternative choice *(Fourie et al., 2007)*.

Although most of the better known examples of the application of electrokinetics are in traditional civil engineering applications such as applications to mining operations and slope stabilization, one new study investigated power consumption of the application of electrokinetics in dewatering food products. *Ng et al. (2011)*, investigated the impact of electrokinetics on dewatering food waste (Brewer’s spent grain, Orange peel, Melon peel, Mango peel and Cauliflower trimmings) by applying two voltages (15V and 30V) and compared it to a control (pressure impact with no voltage). In addition this study compared the power consumption of the application under (0V, 15V and 30V) with the power consumption needed to dewater the same volume of water using conventional thermal methods.

For all experiments, the increase in the voltage supply resulted in a power increase in consumption per unit water removed, and the theoretical calculated energies required for thermal removal were significantly higher than the energy required by electrokinetic dewatering to remove equal amounts of water. Brewer’s spent grain showed the highest differences (up to

60 times more energy saving compared with thermal processing, 0.001KWh under 15V/0.06KWh in thermal) while OJ showed the smallest ratios (up to 18 times more energy saving compared with thermal processing, 0.004KWh under 15V/0.07KWh in thermal) across the range of voltage applied *(Ng et al., 2011)*.

The present study followed the same method to compare the power consumption required using EKEF to dewater OJ and ME with the power consumption of applying the pressure only. *Tables 4.30* and *4.31* show that as expected EKEF application under 4bar pressure and two voltage values (15 and 30V) resulted in a large additional power consumption compared with applying the pressure only. Dewatering efficiency was less with no electrokinetic application, however, the required additional energy needed to achieve the same increase in dewatering efficiency by increasing the pressure only without EK is relatively small; 9.47×10^{-8} and 4.78×10^{-7} KWh for the equivalent of 15V and 30V, respectively for OJ, and 5.66×10^{-7} and 9.66×10^{-7} KWh for the equivalent of 15V and 30V, respectively for ME. This illustrates the power cost of the EKEF process, which is considerable compared with pressure alone. However, the additional energy expenditure is small compared with the energy needed to dehydrate foods under more conventional thermal processes suggesting that this method if scaled up could have industrial application. In addition, increasing pressure further to increase dewatering might not be realistic under commercial operation as this would require additional high-pressure equipment and increases the possibility of blocking in-line filters.

Tables 4.33 and *4.34* show that the electrical power consumed in applying pressure is very small and almost negligible compared with the electrical power consumed by the applied voltage, and the difference between the highest voltage and lowest voltage in terms of power consumption is very significant *(Figures 4.78* and *4.79)*.

Despite the fact that EKEF add more power consumption to the filtration process, this application still shows high power saving in comparison to the thermal methods; a comparison between the energy consumption of different methods was carried out. The application of EKEF saves energy compared with the power consumption of the thermal method required to evaporate an equal amount of water. This saving was up to 18.35 times and 47.34 times in OJ and ME dewatering respectively *(Figure 4.80)*.

It should also be taken into account that during the process, the electrical resistance increased gradually as the amount of water in each sample was reduced and subsequently the current across the sample is also reduced. This means that a higher voltage was required during the later stages. For more efficiency and energy saving it is advisable to increase the voltage gradually which over time will save energy and help to avoid the increase of electrical resistance *(Lockhart, 1983)*.

Chapter 6. Conclusion

6.1. Introduction

This thesis focuses on the design of an experimental rig and system to investigate the efficiency of the application of EKEF to concentrate different food materials. This efficiency was evaluated under different conditions to investigate their impacts on the process and the food products.

The main factors investigated in the experiments were: sample conductivity, applied voltage, applied pressure, filters pore size, initial sample thickness, as well as the sample chemical properties of pH and LCP values.

Investigating the impact of these factors led to some theoretical guidelines which help to choose the best application setup and to predict the process progress according to the food material properties.

Results also showed that using EKEF has some impacts on the product quality compounds such as pH, colour, dry matter content and moisture content; these impacts were measured and compared with the impacts on such factors by the use of thermal methods.

In addition, the WPD between two electrodes, the main filtration outlet and how to decide its position were thoroughly investigated; and the power consumption of this application of EKEF was also calculated and compared with the thermal methods power consumption.

6.2. Review of Chapters

In addition to the aims and objectives of this study which are stated in **Chapter 1**, the need to use non-thermal methods in the food processing area is discussed in **Chapter 2** (the literature review). Furthermore, this chapter focuses mainly on the scientific principles driving the electrokinetic process and its results and applications in previous studies. The factors with potential impact on the electrokinetic process are also reported, as well as the potential impact of this application on food materials to justify the experimental system used in this study.

Chapter 3 shows the experimental rig which is designed for this study to help applying EKEF and investigate the impact of certain factors on the process and the application’s impact on the foods studied. In this chapter each part of this device is shown and some of these parts are discussed in detail including their material and type such as electrodes and filters. The other part of this chapter shows the experimental samples properties (orange juice OJ and malt extract ME), and the experimental design. In addition, analytical equipment methods and protocols were introduced in this chapter.

Chapters 4 and **5** show the results and their discussion, including the efficiency of the application of EKEF to concentrate OJ and ME samples under different conditions and the impact of these conditions on the process and the food products; and present some theoretical guidelines which help to choose the best application setup and to predict the process progress according to the food material properties.

In addition, these two chapters shows the impacts of EKEG application on the product quality compounds such as pH, colour, dry matter content and moisture content; these impacts were measured and compared with the impacts on such factors by the use of thermal methods. Furthermore, the power consumption of this application of EKEF was also calculated and compared with the thermal methods power consumption.

6.3. Objectives and conclusions

Going back to the objective mentioned in Chapter1, we can conclude the following:

- Designing an experimental rig:

  The rig designed for this study was successful to control the effective factors that have an impact on the EKEF process such as sample conductivity, applied voltage, applied pressure, filter pore size, initial sample thickness (the distance between the two electrodes); and helped to accurately collect multiple filtrate samples over the time from the anolyte and catholyte separately, and to record several type of data such as the total filtrate weight every 5 second (with ability to control the recoding duration and the frequency), the voltage and the current intensity (in Amps).

  However, there was some limitations such as:

  - the impossibility of taking samples from the concentrate during the process due to the difficulty of adding an outlet to the cell wall which reduce the sealing and increase the potential damage under high pressures, therefore the concentrate samples were only taken at the beginning and the end of each experiment.
  - Temperatures were measured at the beginning and the end of each experiment only for the same reason.
  - There was no way to measure the temperature and take concentrate samples from different juice layers over the distance between electrodes which would help to get clearer idea about the impact of EKEF on juice before blending.

- Investigating the efficiency of the application of EKEF for concentrating different food materials (orange juice and malt extract):

  Applying EKEF improved the filtration process and achieved net dewatering efficiencies up to 7.43% and 4.86% for OJ and 10.68% and 6.26% for ME after 5.5hrs of processing under 30V and 15V respectively.

- Investigating the impacts of the main factors those control the EKEF process progress and efficiency:

  - The performance of a process utilising EKEF was found to be dependent upon several parameters such as the conductivity, applied voltage, applied pressure, filter pores' size (filtration type), and the sample initial thickness or volume (the distance between the two electrodes). Each of these previous parameters has an impact on the performance of the EKEF process efficiency by influencing the filtrate discharge volume in total and in turn the dewatering efficiency.

  - The sample initial conductivity value plays a significantly positive part in increasing the filtration efficiency due to the increase in the electric current intensity, ($P<0.001$). Process conductivity mainly depends on the electrode conductivity which is relatively stable due to the stability of electrode material (Titanium), and the sample conductivity; this conductivity drops over the process due the decrease of the moisture content and fluctuates due to the gas generation, this fluctuation is higher under higher voltages.

  - Increasing the voltage has also a significant positive impact ($P<0.001$) by increasing the electric current intensity under the same conductivity. However 15V showed better impact than 30V up to the VCP point, when 30V had improved the process efficiency. Monitoring this point can be used to apply a lower voltage at the beginning and then increase the voltage which improves the efficiency, reduces the negative impact on the food quality compounds and helps to save energy.

  - Pressure and filter pore size also had a significantly positive impact ($P<0.001$) on the total process efficiency as increasing pressure and pore size reduces the caking impact and speeds up the flow rate through the filters.

  - Sample thickness (the distance between the two electrodes), in the other hand, has a negative impact on EKEF efficiency by reducing the capacitance.

- Investigating the changes that could be resulted from applying EKEF to foodstuffs by analysing some quality compounds, and compare these results of this application with the results of published conventional methods:

  - The impact of EKEF on the chemical properties and the quality compounds of the foodstuffs is because that the EKEF phenomenon happens due to the ions movements which creates an electric current, some heat and gas.

  - The impact on the filtrate pH occurs when the electric field is applied, water electrolysis, started at the anode and the cathode, produces cations ($H3O^+$) at the anode and anions (OH^-) at the cathode; this ions production results in increasing the anolyte acidity (decreasing pH value) and catholyte alkalinity (increasing pH value), this change is related to the electrode charge regardless of the position (top or bottom). However, change in the pH value of the concentrate under different voltages was insignificant compared with the fresh sample pH as the produced ions, which cause the pH change, move to the opposite electrode to exit the cell with one of the flows (either the anolyte or the catholyte depending on the type of the ions). However, the same reason causes an extensive change in the pH value of the total filtrate especially under 30V.

  - Regarding the impact on the heat-sensitive food compounds represented by vitamin C, applying EKEF for 5.5hours reduced vitamin C retention of the concentrated juice to 41% under 15V and 7.14% under 30V. The retention of the total filtrate was also influenced by the impact of the EKEF, where it dropped to 47.8% under 15V and 35.45% under 30V.

    Vitamin C content decreased to 50% after 2:50hr under 15V and 1:15hr under 30V. The maximum temperature of the electrodes due to the EKEF application was around 25-35ºC, which means that vitamin C content dropped more rapidly at a specific range of temperatures under the impact of EKEF compared with the same temperature impacts without EKEF. This negative impact could be due to the combination of heat and oxidization resulting from the gas generation under EKEF, this gas generation contributes to vitamin C degradation due to the high sensitivity of vitamin C to the oxygen which oxidises ascorbic acid to dehydroascorbic acid.

However vitamin C retention was improved by utilising the EKEF process compared with its value under more conventional thermal methods, as it needed about 10-15min at 50-75ºC and 3min at 90ºC to drop to 50% or less *(Vikram et al., 2005)*.

The main degradation of vitamin C content occurred in the catholyte which lost its content very rapidly in comparison with the anolyte. This difference between the anolyte and the catholyte could have resulted from the slow flow rate of the catholyte which results in a thinner juice layer and longer contact time between juice and the electrode which increased the negative impact of the heat on the juice. However, the main outlet when EKEF is applied in OJ experiments was the anolyte which means that the main volume of the total filtrate exits with the anolyte, and this decreased the total loss of the vitamin C content in the final total filtrate when re-combined.

- EKEF also had an impact on OJ colour which was greater next to the catholyte (the secondary outlet) in comparison with the anolyte (the main outlet), due to the formation of dehydroascorbic acid and diketogluconic acids from ascorbic acid oxidisation reaction. Later on, when most of the juice exits and both filtrate rates slowdown, colour starts to appear in the anolyte in the highest voltage conditions.

  Concentrated juice colour was also influenced by the application of EKEF; the discoloration was clearly obvious at the top layer next to the top electrode (the cathode) only, while it decreased by blending concentrated juice at the end of the process.

- The impact of the EKEF was influenced by the processing time and the temperature of the electrodes which increases with higher voltages; this negative impact increased with a longer processing time and higher temperature (higher voltage values generate higher temperature next to the electrodes).

- Applying EKEF helped to increase dry matter content from 14% to 33.09% and 29.52% for OJ and from 20% to 34.2% and 32.14% for ME after 5.5hrs under 30V and 15V respectively while the maximum dry matter content was 28.75% and 29.63% under the control orientation after the same time for OJ and ME respectively. The Brix degree (and the dry matter content) of the industrial concentrate orange juice, produced by the thermal methods, is 40-45°Bx and could reach 65°Bx in some products *(Clark et al., 2014)*.

However, changing the applied voltage value did not have a significant impact on dry matter content of the total filtrate, as the main factor that has impact on dry matter contents is the filter pore size.

Results showed that the filter with the bigger pore size (1µ) allowed more particles to pass through which in turn resulted in significantly higher dry matter content compared with the small pore filter (0.2µ) ($P<0.001$).

Regarding the impact of the electrode type and position on dry matter content, results showed that the fall in dry matter content was in the normal catholyte (the top outlet stream) and the reversed Anolyte (the top outlet stream); which means that the effective factor is the position of the electrode; this impact could be related to the suspension separation into two phases as most of the larger fragments of pulp in the juice deposit at the bottom by gravity and not due to the EKEF impact.

- To measure the power consumption of this application under different running conditions:

  EKEF processes resulted in a large additional power consumption compared with applying the pressure only; and the required additional energy needed to achieve the same increase in dewatering efficiency by increasing the pressure only without EK is relatively small. On the other hand, EKEF showed a good efficiency in energy saving compared to the power consumption required in more conventional thermal methods used to evaporate an equal amount of water. This saving was up to 18.35 times and 47.34 times in OJ and ME dewatering respectively which indicates that at a commercial scale use of EKEF could have a major impact on energy expenditure if it were to replace conventional thermal drying techniques. Using EKEF would also allow the use of lower pressure vessels than would have to be used under high pressure dewatering methods.

- To suggest some ways to control conditions affecting the EKEF process to increase the outcome (total filtrate), decrease the negative impacts on foodstuff quality compounds and reduce the power consumption:

  - Adjusting pH to increase the difference between pH value and LCP which increases the impact of EKEF and the total improvement.
  - Using a larger filter pore size reduces the negative impact of this method on vitamin C and colour.
  - Applying gradual voltage increase also decreases the negative impact on the food quality compounds and reduces the power consumption.
- Finally, to setup theoretical guidelines depending on the relationship between the chemical properties of samples and the direction of the water profile after applying the electric field which helps in the choice of the optimal application specification and to predict the process progress according to the food material properties:

  One of the main findings of this study was that the chemical properties of the food sample (pH and LCP values) determine the WPD, the main outlet and the optimal orientation; in this case analysing the sample before running the experiment is essential and then it is possible to follow one of these specifications:

  - When pH<LCP: WPD is toward the anode (the main outlet is the anolyte) which must be set as the bottom electrode (normal orientation).
  - When pH>LCP: WPD is toward the cathode (the main outlet is the catholyte) which must be set as the bottom electrode (reversed orientation).
  - When pH≈ LCP: the suspension is neutral and EKEF has no significant impact on the filtration flow rate, in this case adjusting pH value to increase the difference between pH and LCP values will improve the process and increase the impact possible from EKEF.

  In addition to the main water movement to the main outlet (WPD), there is also a movement toward the secondary outlet and the ratio of these two movements depends on the difference between pH and LCP values and presented by A/C ratio. This result means that neither the anolyte nor the catholyte should be ignored but at the same time the main outlet position must be chosen in a way that makes the water movement toward it supported by the sum of the affecting forces.

6.4. Wider Commercial Implications

As mentioned in the conclusion, applying EKEF helped to increase dry matter content from 14% to 33.09% and 29.52% for OJ and from 20% to 34.2% and 32.14% for ME after 5.5hrs under 30V and 15V respectively which is lower than the Brix degree of thermally produced concentrate orange juice *(Clark et al., 2014)*.

Furthermore, EKEF processes showed a good efficiency in energy saving compared to the power consumption required in more conventional thermal methods used to evaporate equal amount of water. This saving was up to 18.35 times and 47.34 times in OJ and ME dewatering respectively.

The theoretical calculated energy required for thermal removal of water is 43kJ.mol^{-1}, as quoted by *Al-Asheh et al. (2004)*, and this means that power consumption increases in thermal process, as well as the negative impact of heat on the product quality compounds, as the amount of water required to evaporate increases, therefore, using electrokinetics as a first stage will help to decrease the negative impacts and the power consumption of the thermal process which may be used in later stages for achieving higher dry matter content.

This present study showed that the main reason behind the negative impacts of EKEF on the food quality compounds is the heat produced at the electrodes and the slow flow rate of the secondary filtrate which is in a direct contact with the hot electrode. Therefore, using mixers or finding a way to cool down the temperatures of the electrodes will help avoiding the negative impact of EKEF in a commercial scale process.

Also it should be taken into account that during the process, the electrical resistance increases gradually as the amount of water in each sample is reduced and subsequently the current across the sample is also reduced, which means that a higher voltage is required at the later stages of the process. For more efficiency and saving energy it is advisable to increase the voltage gradually which over time will save energy and help to avoid the increase of electrical resistance.

Chapter 7. Limitation and Further Work

7.1. Study limitations

This study set out to investigate the factors that could have a potential impact on the use of EKEF to dewater foodstuffs, as well as examining the potential impact of EKEF on the food quality compounds.

The main limitation of this study was the time available; a large part of this study was spent designing and manufacturing the experimental rig.

In addition, each experiment was run for 5.5h for OJ and 3.5h for ME; the large number of experiments needed to cover all study objectives and the duration of each experiment as well as the analysis time increased the difficulty of doing a large number of replications. Therefore, the replications were done for the main experiments only.

Due to the high pressure used and the need for perfect sealing, the cell was designed in a way that does not allow taking concentrate samples over time, therefore all concentrate samples were the samples collected at the end of the experiments unlike the filtrate which was sampled each half hour over the experiment period.

The other limitation is the scarcity of information about applying electrokinetics to foodstuffs; only a few researchers have examined the efficiency, feasibility and impacts of this process application and all of them were in the area of civil engineering. The only one study done on food was in food waste area to investigate the impact of electrokinetics on dewatering food waste and to compare the power consumption of the application with the power consumption needed to dewater the same volume of water using conventional thermal methods.

In addition, there was difficulty in measuring the IEP value of OJ and ME; as a foodstuff is a large combination of different compounds and particles which have different IEPs. Most of the devices that are usually used to measure the IEP are calibrated for single specific compounds such as protein, lipid, polymer, etc.

However, using the definition of IEP, another point was measured to show the potential pH where the net charge of the material (the combination of liquid and pulp) becomes almost neutral before turning to the opposite charge; analysis showed that at a specific pH value, conductivity shows the lowest value; this pH value is called in this study LCP (the lowest conductivity point).

In case of having enough repetitions, it is the author believe that time series analysis may reveal further answers in the data when comparing different results under different experimental conditions.

7.2. Future Research Work

This thesis focuses on the factors affecting the application of electrokinetics and the implications for future research, all these factors could be investigated separately in more details, and as a combination taking into account the interaction between these factors to find the best combination to run the application sufficiently.

More research needed to prove which process is behind the discolouration and the reason of changes, and to measure whether there is an ions migration from electrodes.

Also the shape of the cell could be modified to allow measuring the change in the concentrate properties over time and the cell length (sample layers).

More repetitions are needed to show the reliability of these results and to do so each part could be studied separately as the required experiments and analytical procedures are highly-time consuming.

In addition, using EKEF process on a larger scale or even to a commercial scale is an important area to be covered in the future research.

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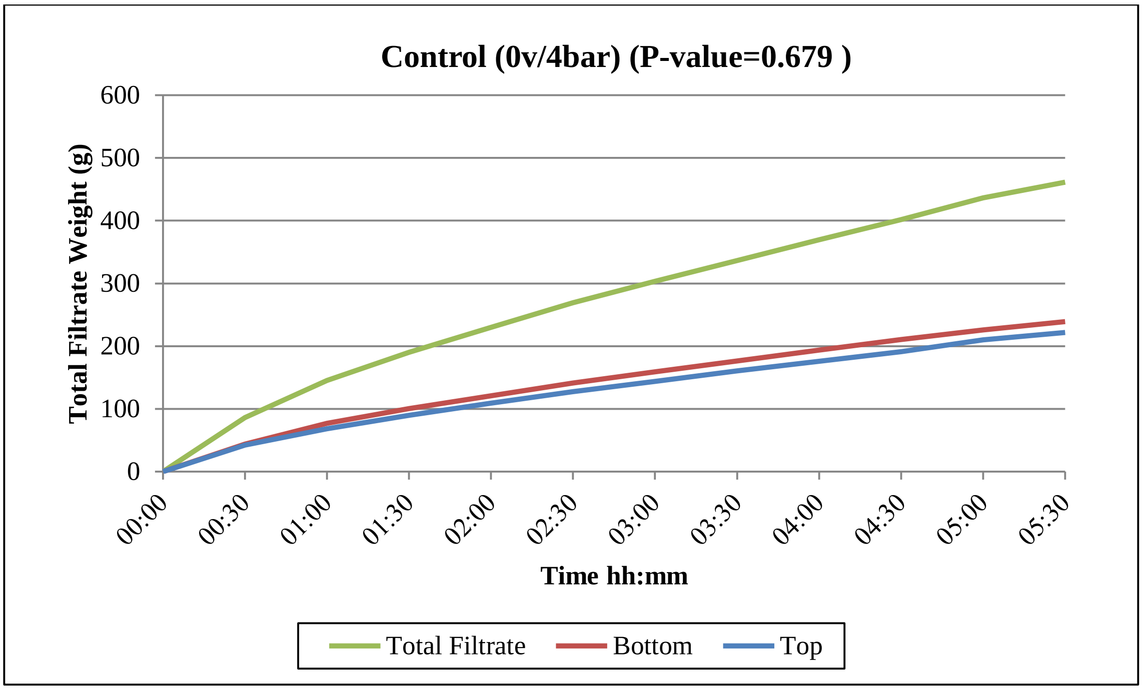
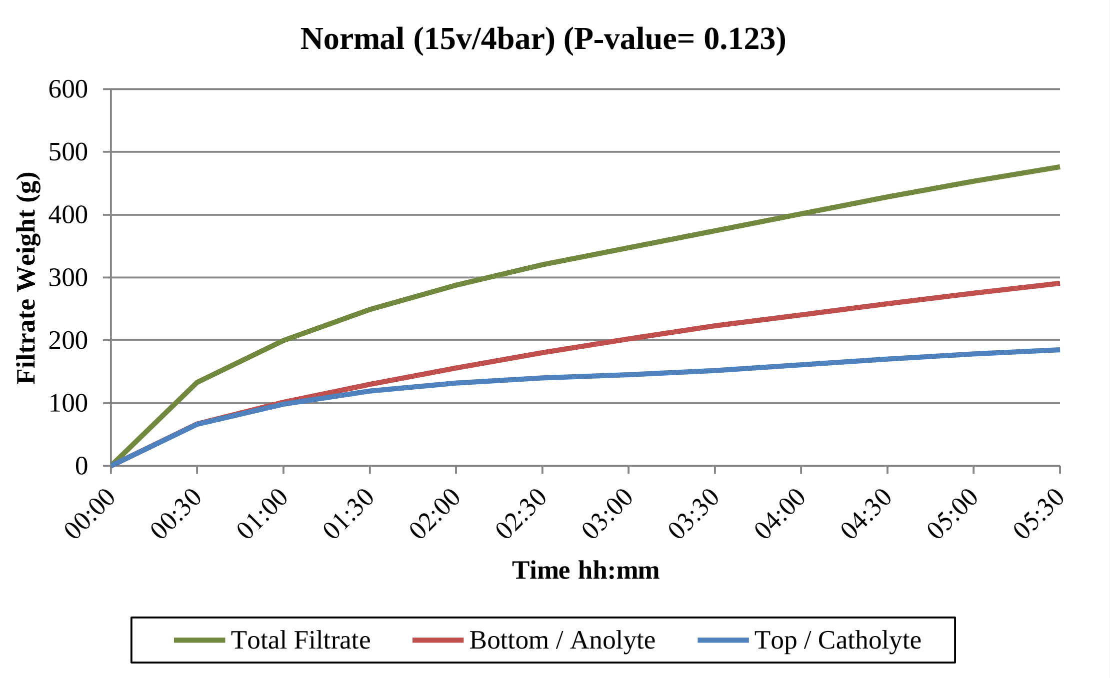
Appendices

Appendices 1

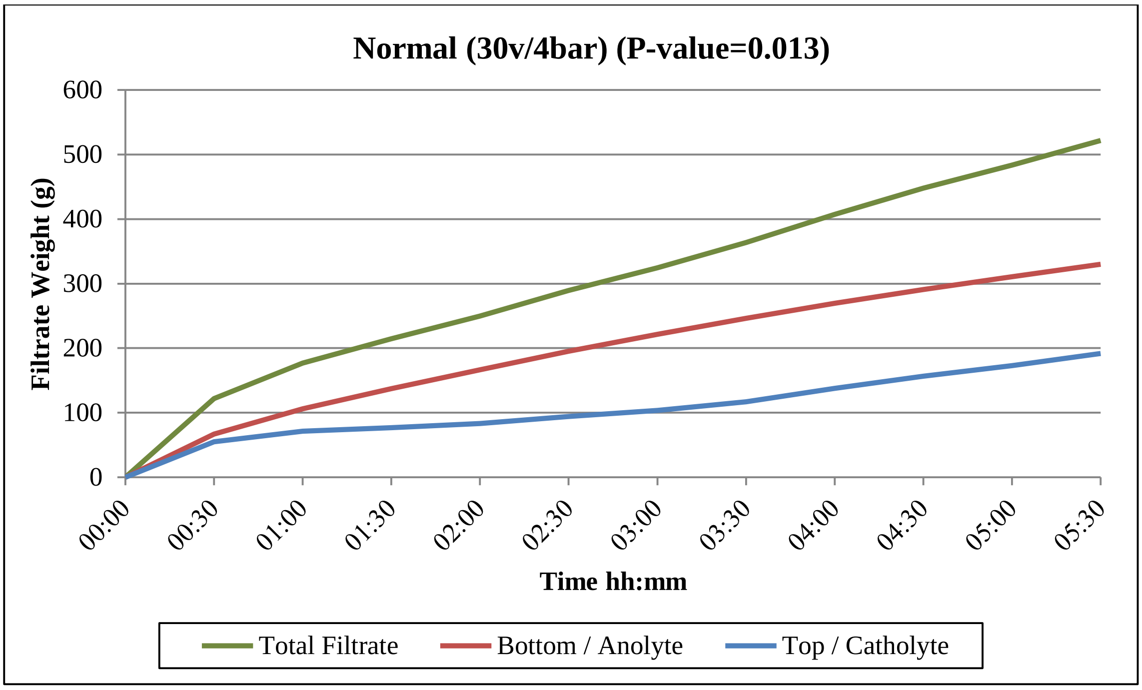
The change in the weight of OJ bottom, top and total filtrates over time under three voltages (0, 15& 30V):



Appendix 1.1. Change in the weight of OJ bottom, top and total filtrates over time under the control orientation (0.2µ filter, 0V, 4bar, 5.5 hours)



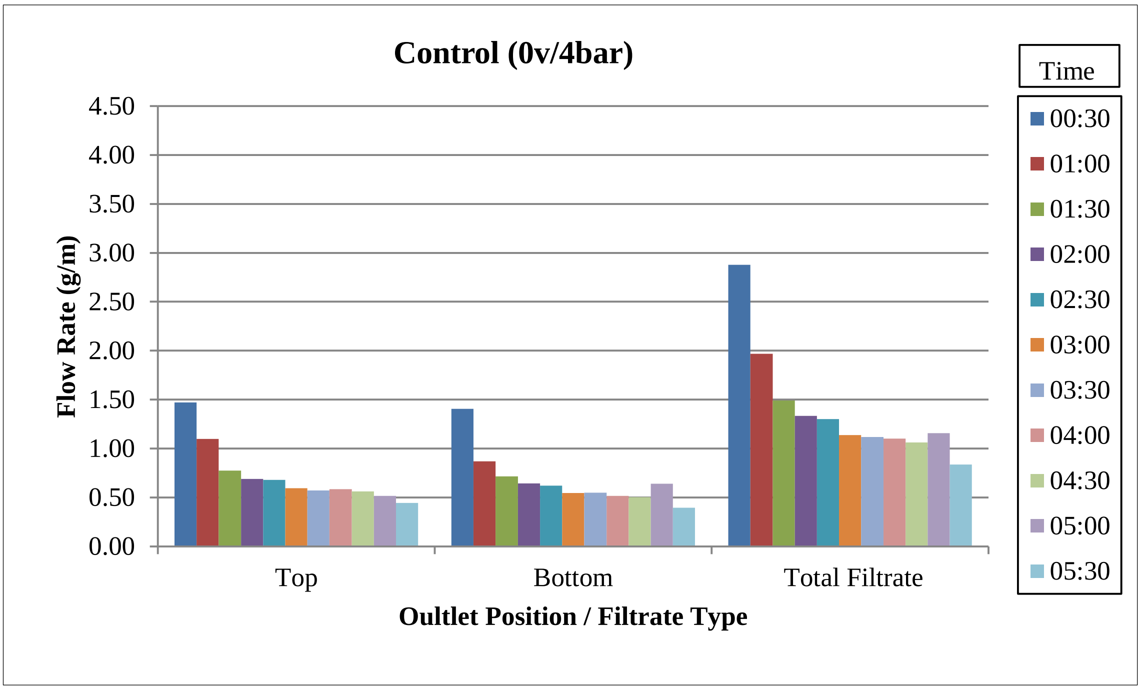
Appendix 1.2. Change in the weight of OJ anolyte, catholyte and total filtrate the time under the normal orientation (0.2µ filter, 15V, 4bar, 5.5 hours)



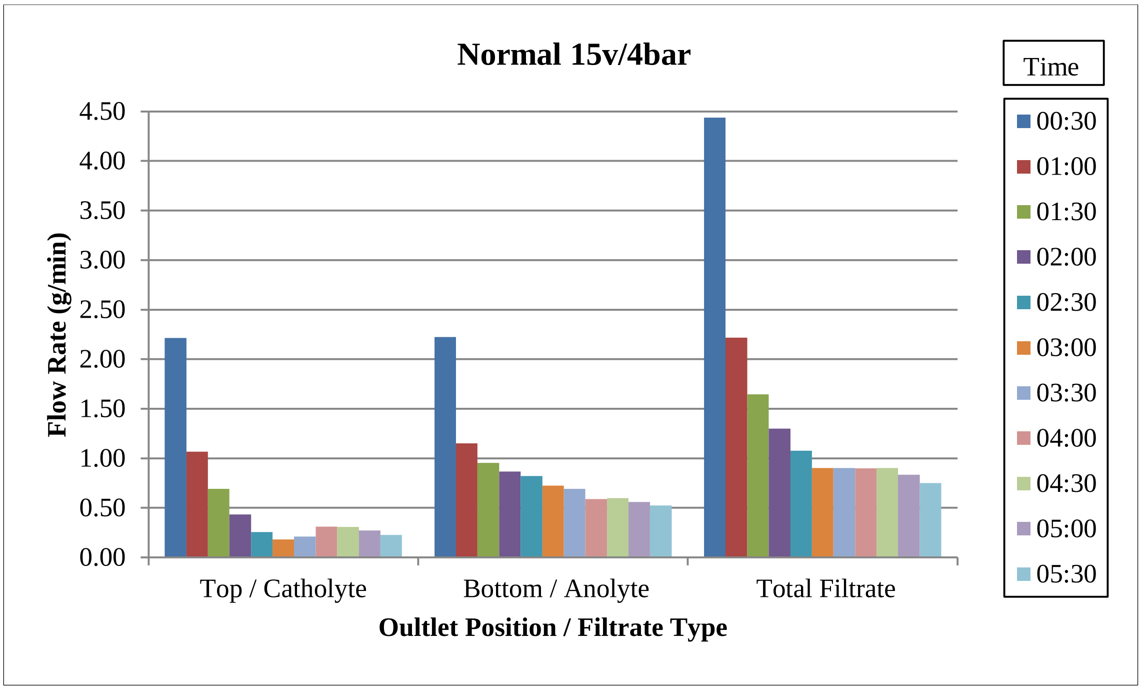
Appendix 1.3. Change in the weight of OJ anolyte, catholyte and total filtrate over time under the normal orientation (0.2µ filter, 30V, 4bar, 5.5 hours)

Appendices 2

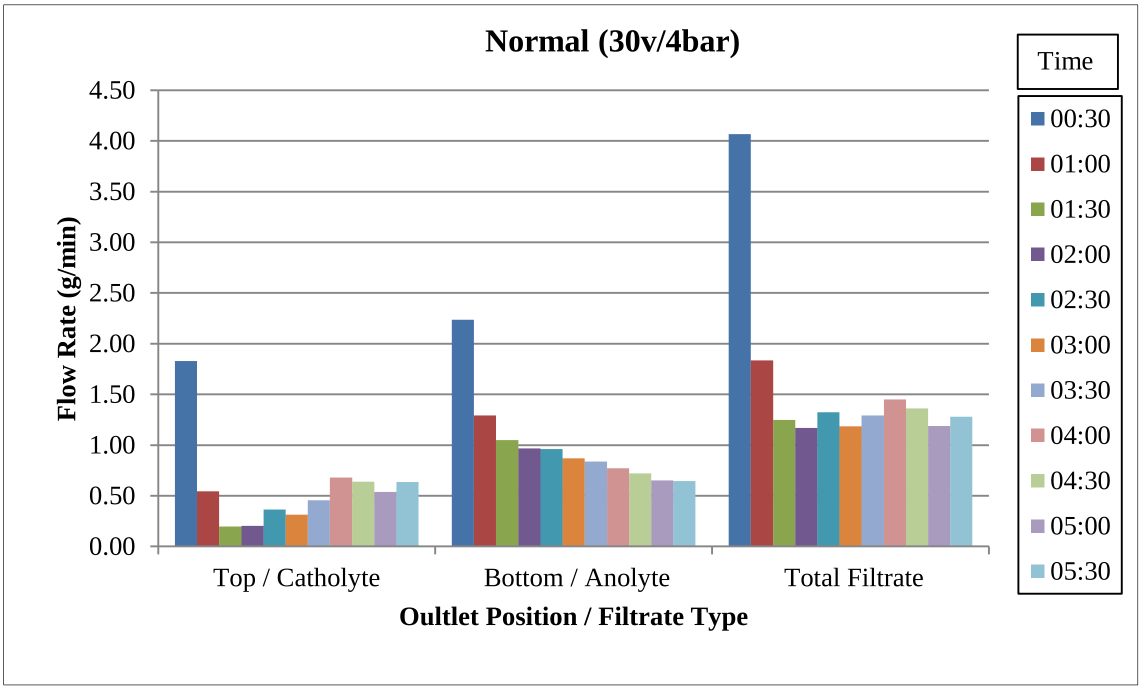
The change in the flow rate of OJ bottom, top and total filtrates over time under three voltages (0, 15& 30V, 0.2µ filter, 4bar, 5.5 hours):



Appendix 2.1. Change in the flow rate of OJ bottom, top and total filtrates over time under the control orientation (0.2µ filter, 0V, 4bar, 5.5 hours)



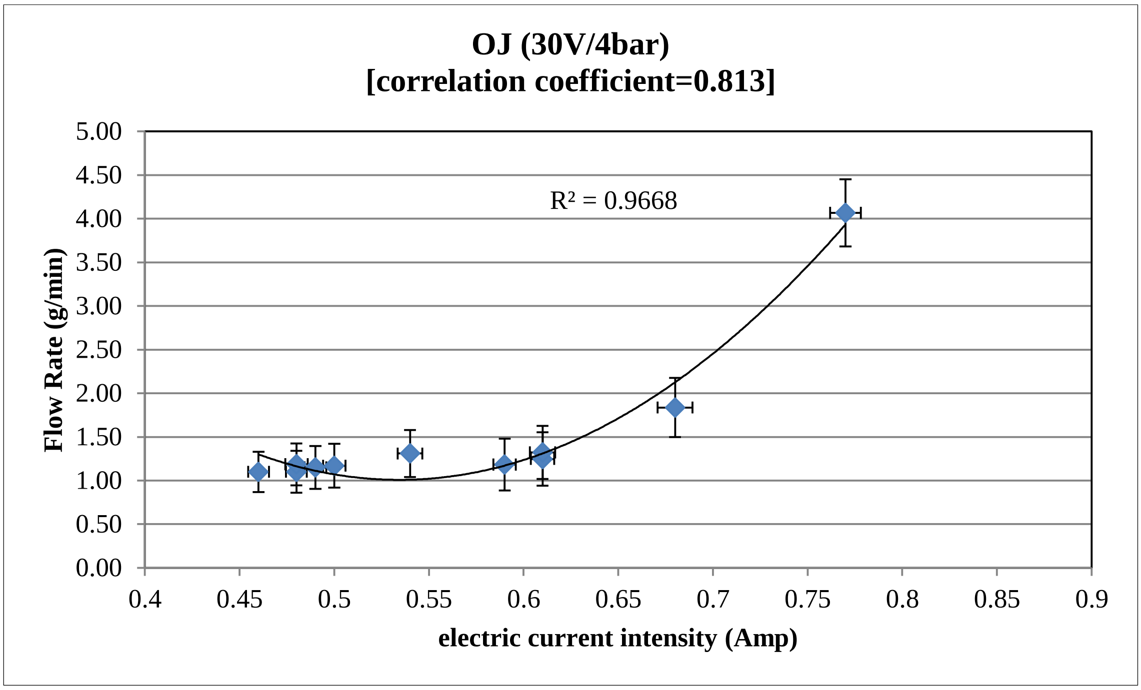
Appendix 2.2. Change the flow rate of OJ anolyte, catholyte and total filtrate over time under the normal orientation (0.2μ filter, 15V, 4bar, 5.5 hours)



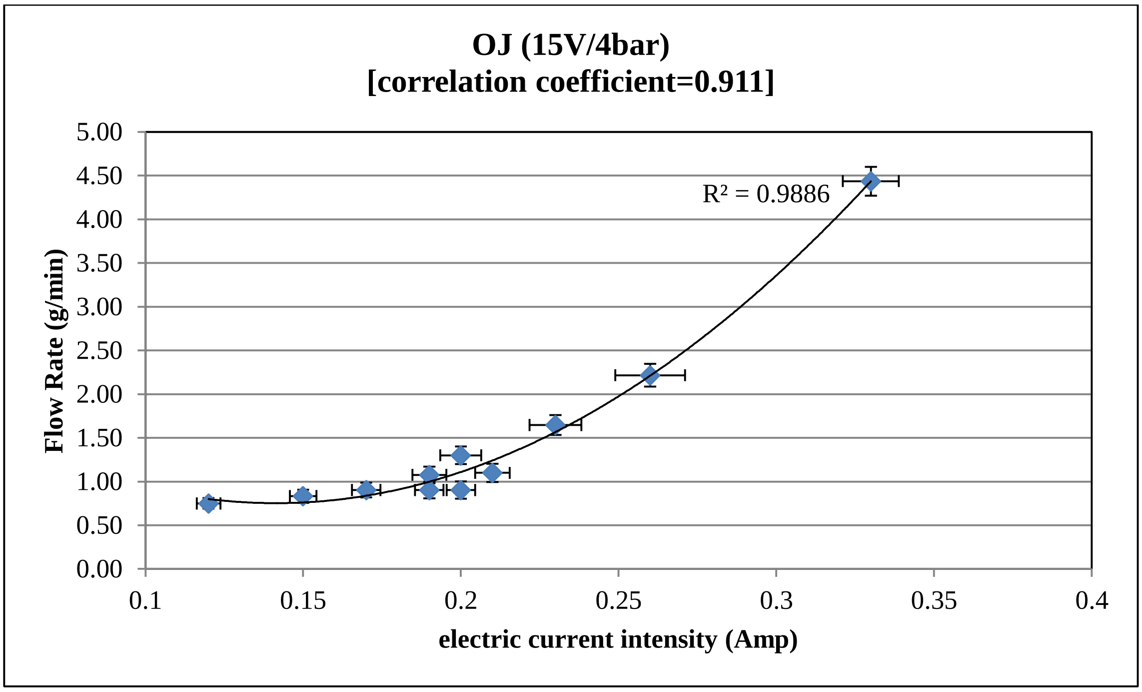
Appendix 2.3. Change in the flow rate of OJ anolyte, catholyte and total filtrate over time under the normal orientation (0.2μ filter, 30V, 4bar, 5.5 hours)

Appendices 3

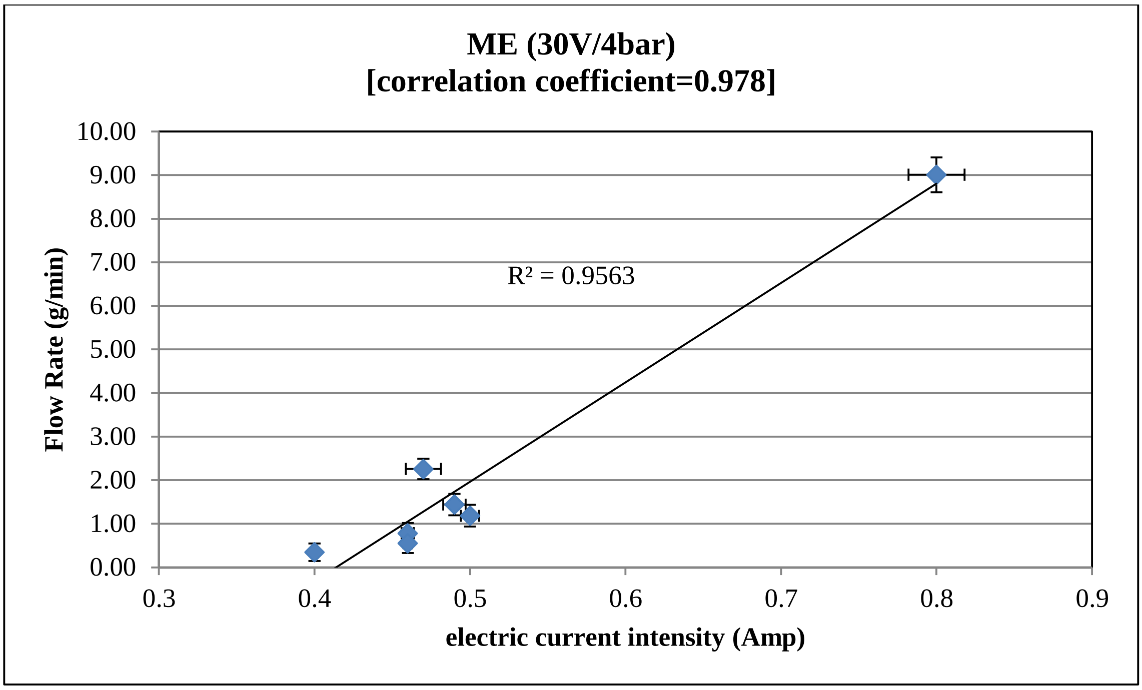
The correlation between the OJ and ME filtrate flow rate and electric current intensity over experiment time under different conditions:



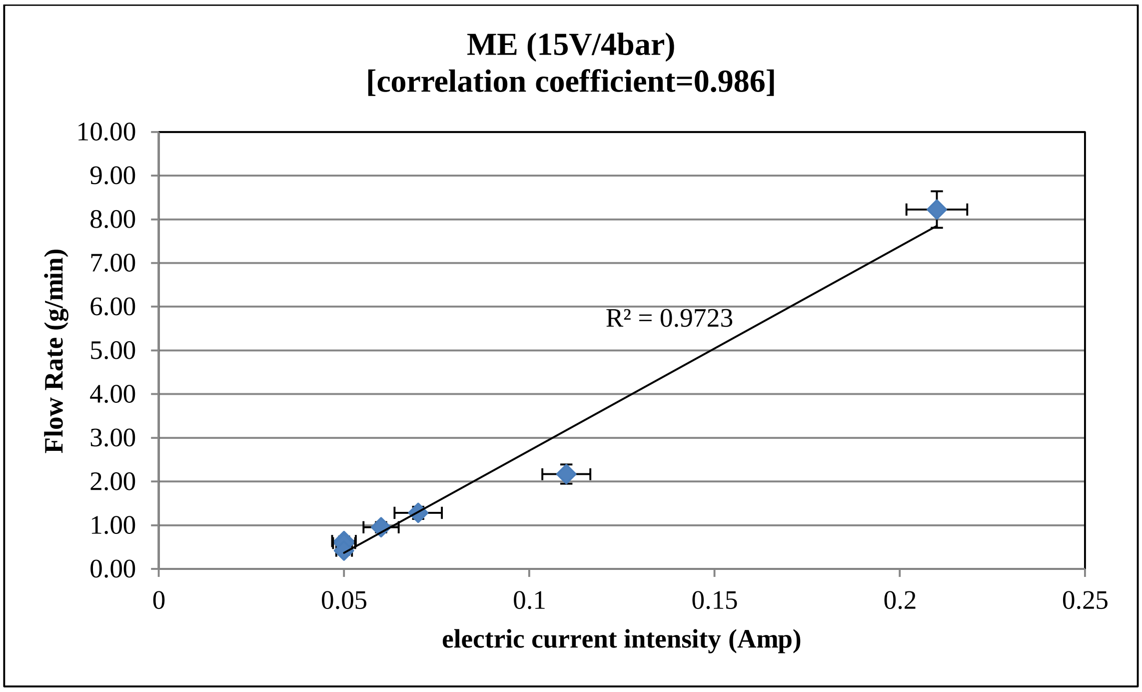
Appendix 3.1. The correlation between OJ filtrate flow rate and electric current intensity over experiment time under 30V, a pressure of 4bar, 0.2µ filter and for 5.5 hours. Each value is expressed as mean ± standard deviation (n=3)



Appendix 3.2. The correlation between OJ filtrate flow rate and electric current intensity over the experiment time under 15V, a pressure of 4bar, 0.2µ filter and for 5.5 hours. Each value is expressed as mean ± standard deviation (n=3)



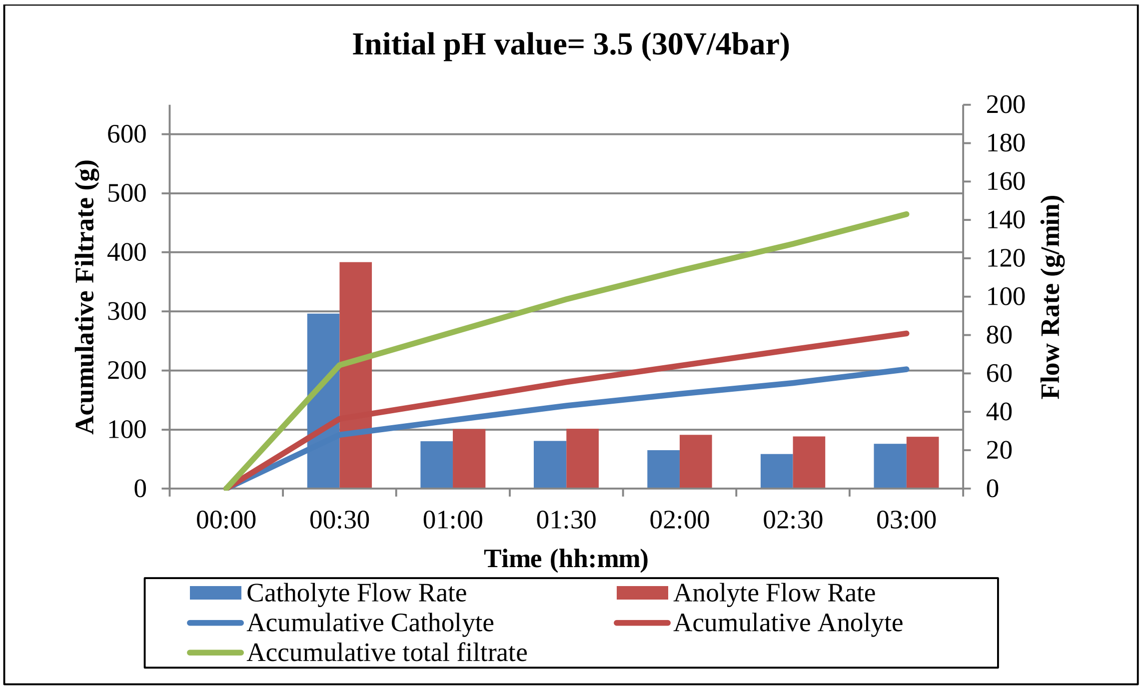
Appendix 3.3. The correlation between ME filtrate flow rate and electric current intensity over the experiment time under 30V, a pressure of 4bar, 1µ filter and for 3.5 hours. Each value is expressed as mean ± standard deviation (n=3)



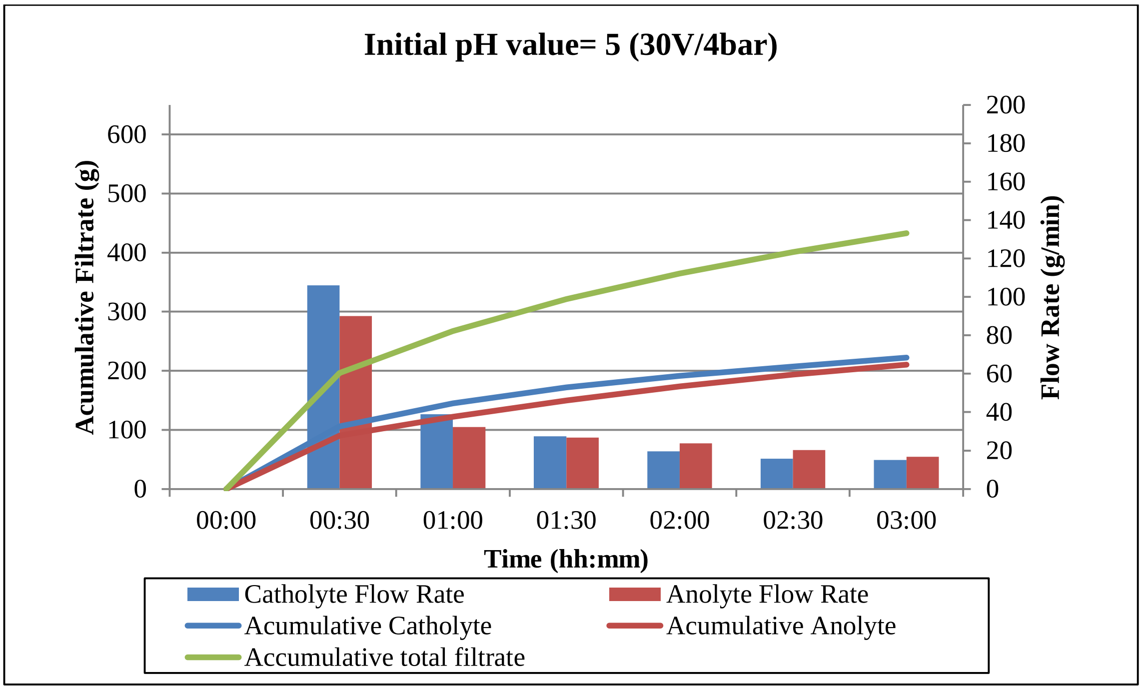
Appendix 3.4. The correlation between ME filtrate flow rate and electric current intensity over the experiment time under 15V, a pressure of 4bar, 0.2µ filter and for 3.5 hours. Each value is expressed as mean ± standard deviation (n=3)

Appendices 4

The change in the accumulative filtrates and filtrate flow rate of OJ samples with different pH values over time:



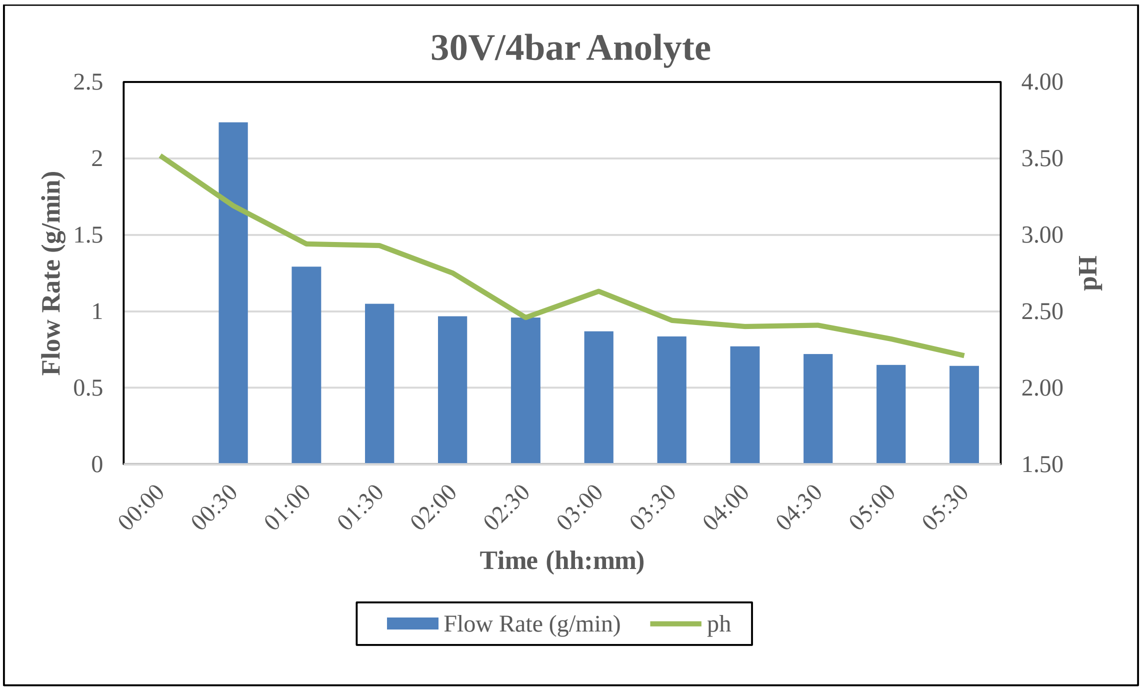
Appendix 4.1. Change in OJ accumulative filtrates and filtrate flow rate over time under the application of EKEF with initial pH value=3.6 (normal orientation, 30V, 4bar, 1µ filter and 3hours duration)



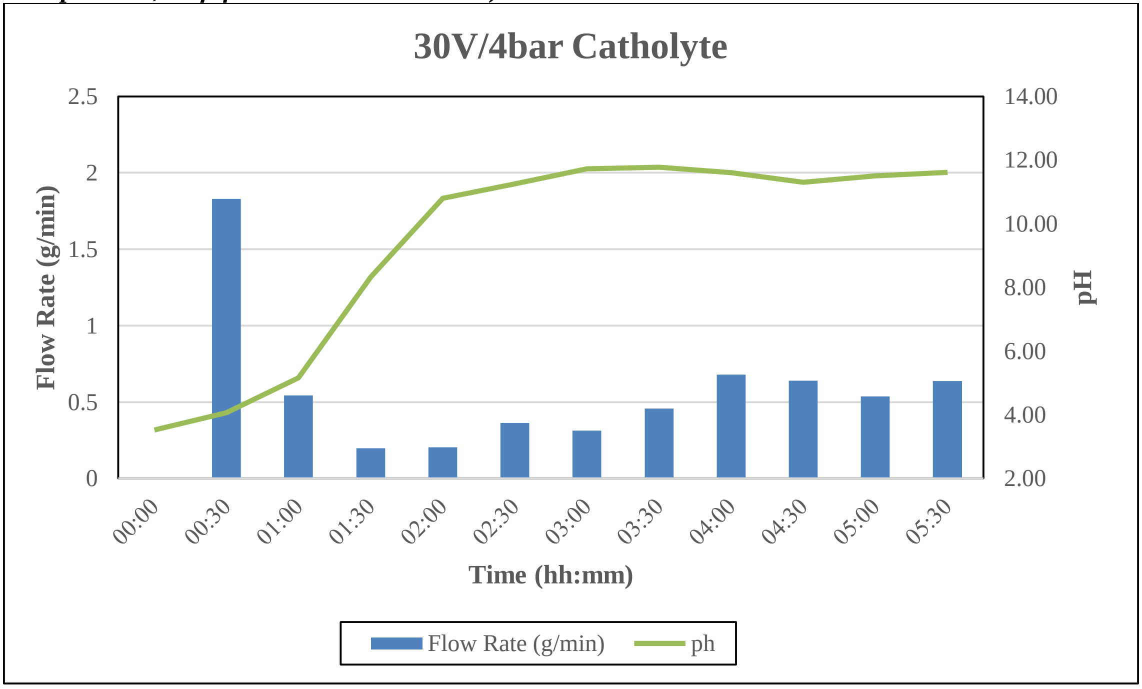
Appendix 4.2. Change in OJ accumulative filtrates and filtrate flow rate over time under the application of EKEF with initial pH value=5.0 (normal orientation, 30V, 4bar, 1µ filter and 3hoursduration)

Appendices 5

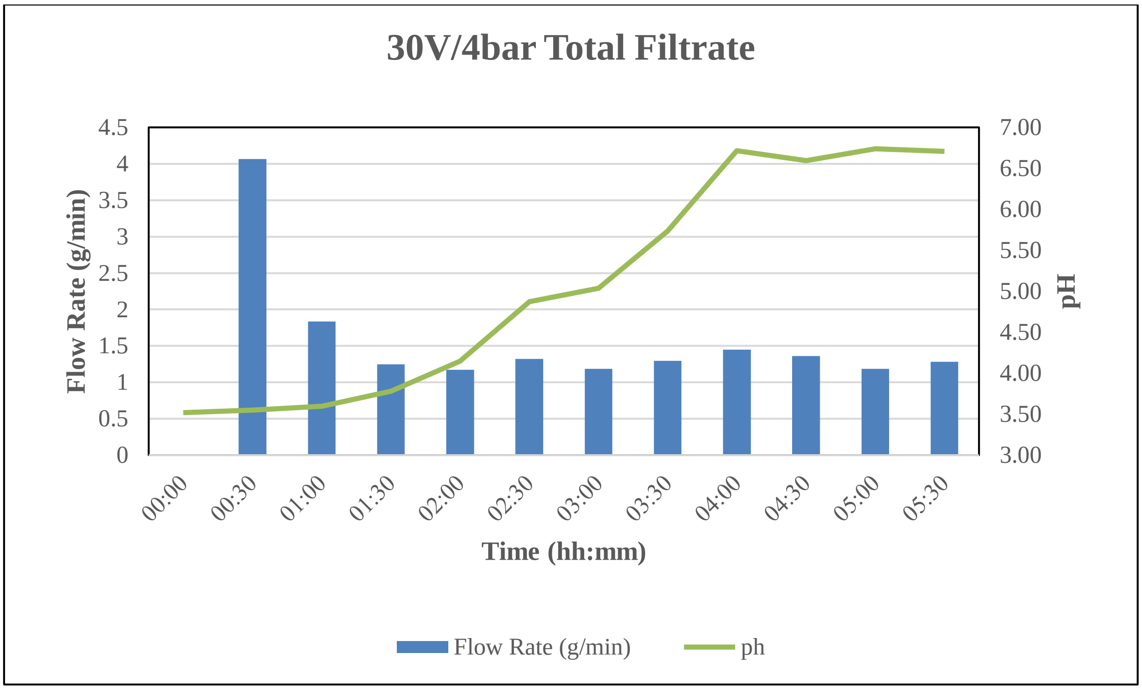
The correlation between the change of the OJ anolyte, catholyte and total filtrate pH value and the flow rate over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration):



Appendix 5.1. The change of the pH and the flow rate of OJ anolyte over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)



Appendix 5.2. The change of the pH and the flow rate of OJ catholyte over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)



Appendix 5.3. The change of the pH and the flow rate of OJ total filtrate over time under (normal orientation, 30V, 4bar pressure, 0.2µ filter and 5.5hr duration)

Appendices 6

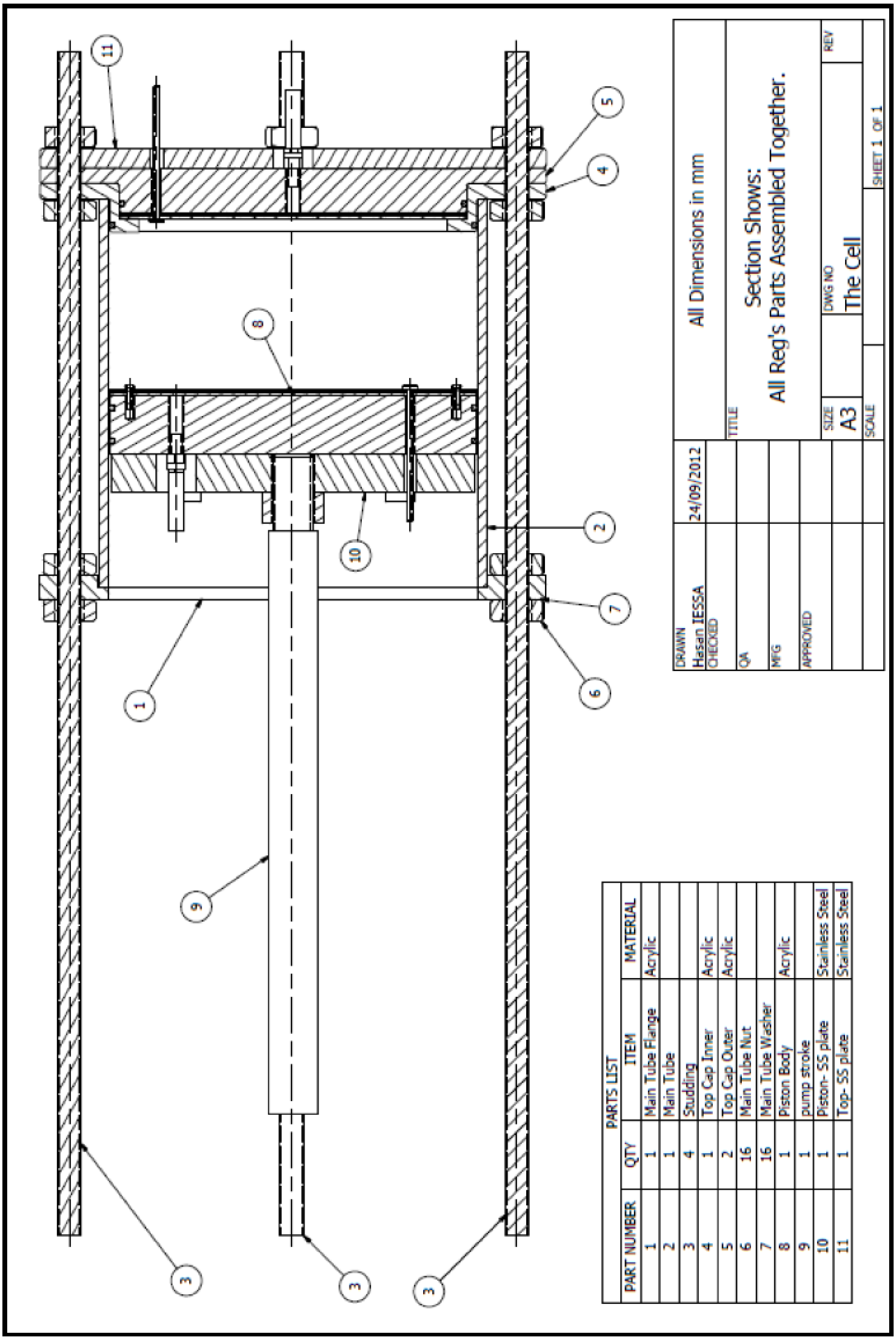
The running conditions of experiments done for each test:

Test	Experiments	Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	Time (Hour)	Filter (μ)
The voltage and pressure impact on WPD and A/C ratio	Exp 1x3	OJ	Control	1	0	2	5.5	0.2
	Exp 2 x3	OJ	Normal	1	15	2	5.5	0.2
	Exp 3 x3	OJ	Normal	1	30	2	5.5	0.2
	Exp 4 x3	OJ	Control	1	0	3	5.5	0.2
	Exp 5 x3	OJ	Normal	1	15	3	5.5	0.2
	Exp 6 x3	OJ	Normal	1	30	3	5.5	0.2
	Exp 7 x3	OJ	Control	1	0	4	5.5	0.2
	Exp 8 x3	OJ	Normal	1	15	4	5.5	0.2
	Exp 9 x3	OJ	Normal	1	30	4	5.5	0.2
	Exp 10 x3	ME	Reversed	1	0	4	3.5	1
	Exp 11 x3	ME	Reversed	1	15	4	3.5	1
	Exp 12 x3	ME	Reversed	1	30	4	3.5	1
The conductivity impact on dewatering efficiency					Conductivity (S)			
	Exp 13 x3	OJ	Normal	1	0.22	4	5.5	0.2
	Exp 14 x3	OJ	Normal	1	0.33	4	5.5	0.2
	Exp 15 x3	OJ	Normal	1	0.5	4	5.5	0.2
The sample initial pH Value impact on the dewatering efficiency	Pressure 4bar in all					pH		
	Exp 16 x3	OJ	Control	1	0	3.6	3	1
	Exp 17 x3	OJ	Normal	1	30	1.5	3	1
	Exp 18 x3	OJ	Normal	1	30	3.6	3	1
	Exp 19 x3	OJ	Normal	1	30	5	3	1
	Exp 20 x3	OJ	Normal	1	30	6.5	3	1
The filter pore size on the dewatering efficiency	Exp 1 x3	OJ	Control	1	0	4	5.5	0.2
	Exp 2 x3	OJ	Normal	1	15	4	5.5	0.2
	Exp 3 x3	OJ	Normal	1	30	4	5.5	0.2
	Exp 21 x3	OJ	Control	1	0	4	5.5	1
	Exp 22 x3	OJ	Normal	1	15	4	5.5	1
	Exp 23 x3	OJ	Normal	1	30	4	5.5	1
	Exp 24 x3	ME	Control	1	0	4	2.5	0.2
	Exp 25 x3	ME	Reversed	1	30	4	2.5	0.2
	Exp 10 x3	ME	Control	1	0	4	2.5	1
	Exp 12 x3	ME	Reversed	1	30	4	2.5	1
The sample initial thickness on the dewatering efficiency	Exp 26 x3	ME	Control	0.5	0	4	2	0.2
	Exp 27 x3	ME	Reversed	0.5	30	4	2	0.2
	Exp 1 x3	ME	Control	1	0	4	2	0.2
	Exp 3 x3	ME	Reversed	1	30	4	2	0.2
	Exp 28 x3	ME	Control	1.5	0	4	2	0.2
	Exp 29 x3	ME	Reversed	1.5	30	4	2	0.2

Test	Experiments	Material	Orientation	Volume (L)	Voltage (V)	Pressure (Bar)	Time (Hour)	Filter (μ)
pH change under EKEF application	Exp 1 x3	OJ	Control	1	0	4	5.5	0.2
	Exp 2 x3	OJ	Normal	1	15	4	5.5	0.2
	Exp 3 x3	OJ	Normal	1	30	4	5.5	0.2
	Exp 10 x3	ME	Control	1	0	4	3.5	1
	Exp 11 x3	ME	Reversed	1	15	4	3.5	1
	Exp 12 x3	ME	Reversed	1	30	4	3.5	1
Vitamin C retention change under EKEF application	Exp 1 x3	OJ	Control	1	0	4	5.5	0.2
	Exp 2 x3	OJ	Normal	1	15	4	5.5	0.2
	Exp 3 x3	OJ	Normal	1	30	4	5.5	0.2
The dry matter content change under EKEF application	Exp 1 x3	OJ	Control	1	0	4	5.5	0.2
	Exp 2 x3	OJ	Normal	1	15	4	5.5	0.2
	Exp 3 x3	OJ	Normal	1	30	4	5.5	0.2
	Exp 30 x3	ME	Control	1	0	4	3	0.2
	Exp 31 x3	ME	Reversed	1	15	4	3	0.2
	Exp 32 x3	ME	Reversed	1	30	4	3	0.2

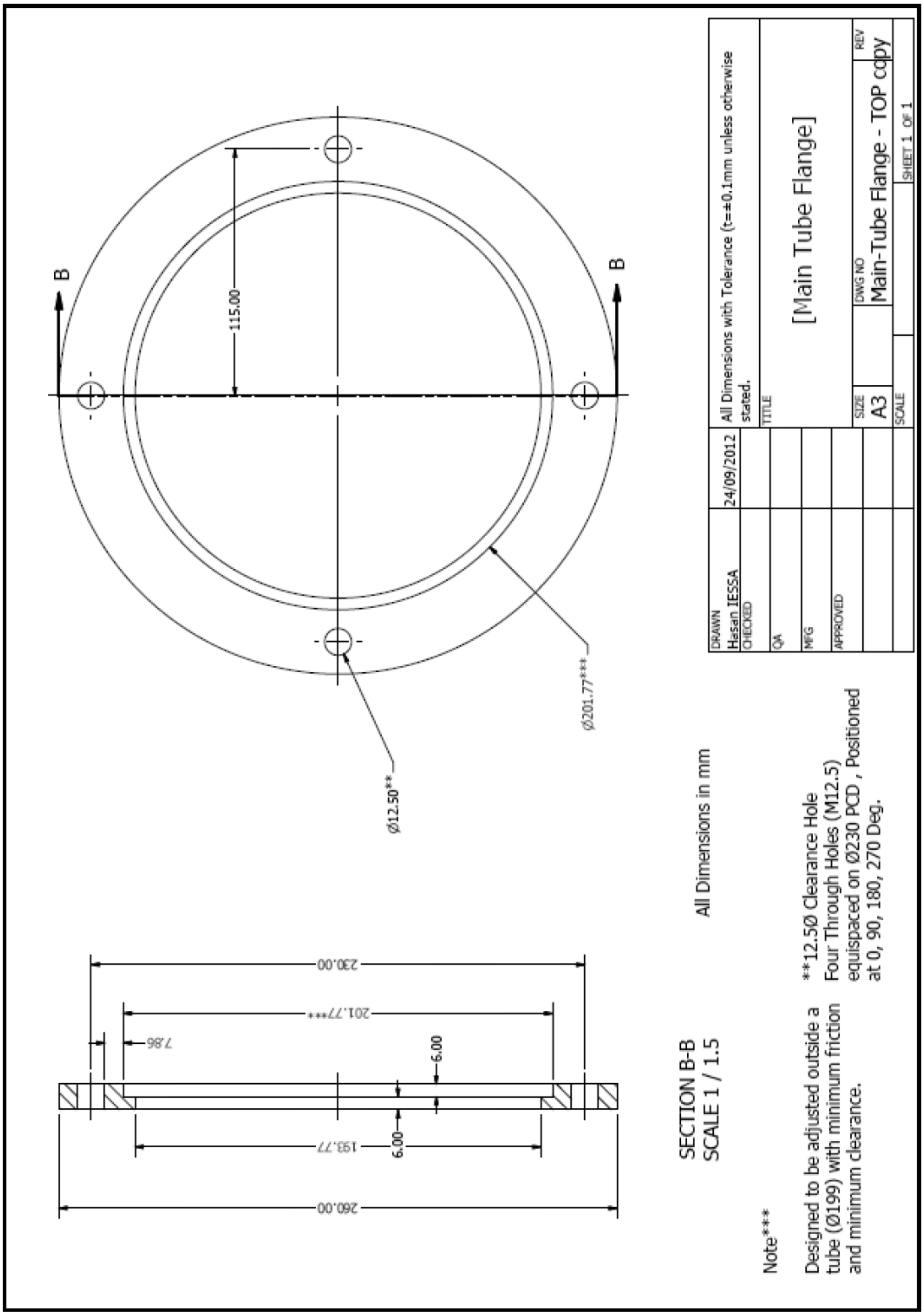
Appendices 7

The experimental rig's parts:

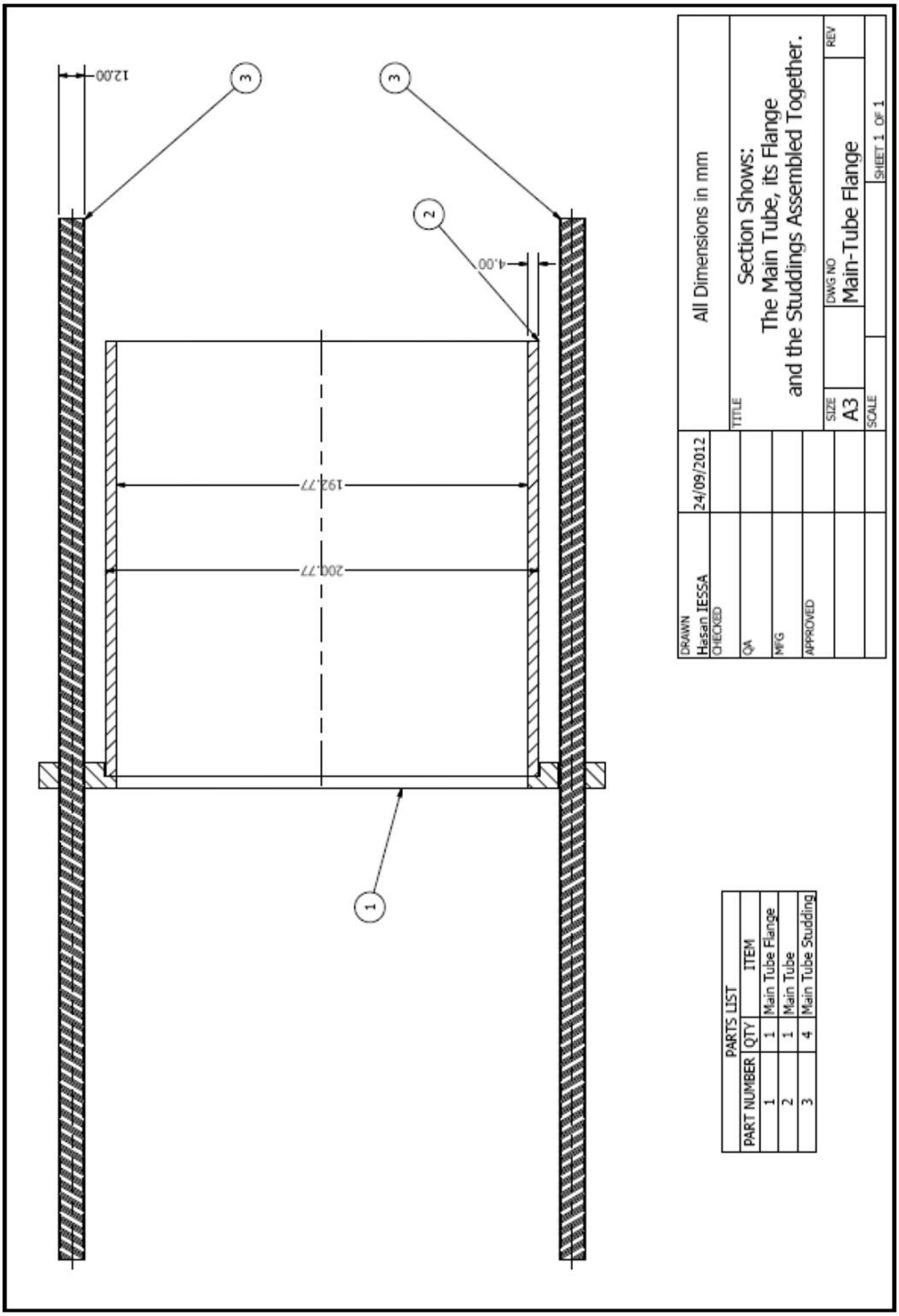


PARTS LIST			
PART NUMBER	QTY	ITEM	MATERIAL
1	1	Main Tube Flange	Acrylic
2	1	Main Tube	
3	4	Studding	
4	1	Top Cap Inner	Acrylic
5	2	Top Cap Outer	Acrylic
6	16	Main Tube Nut	
7	16	Main Tube Washer	
8	1	Piston Body	Acrylic
9	1	pump stroke	
10	1	Piston- SS plate	Stainless Steel
11	1	Top- SS plate	Stainless Steel

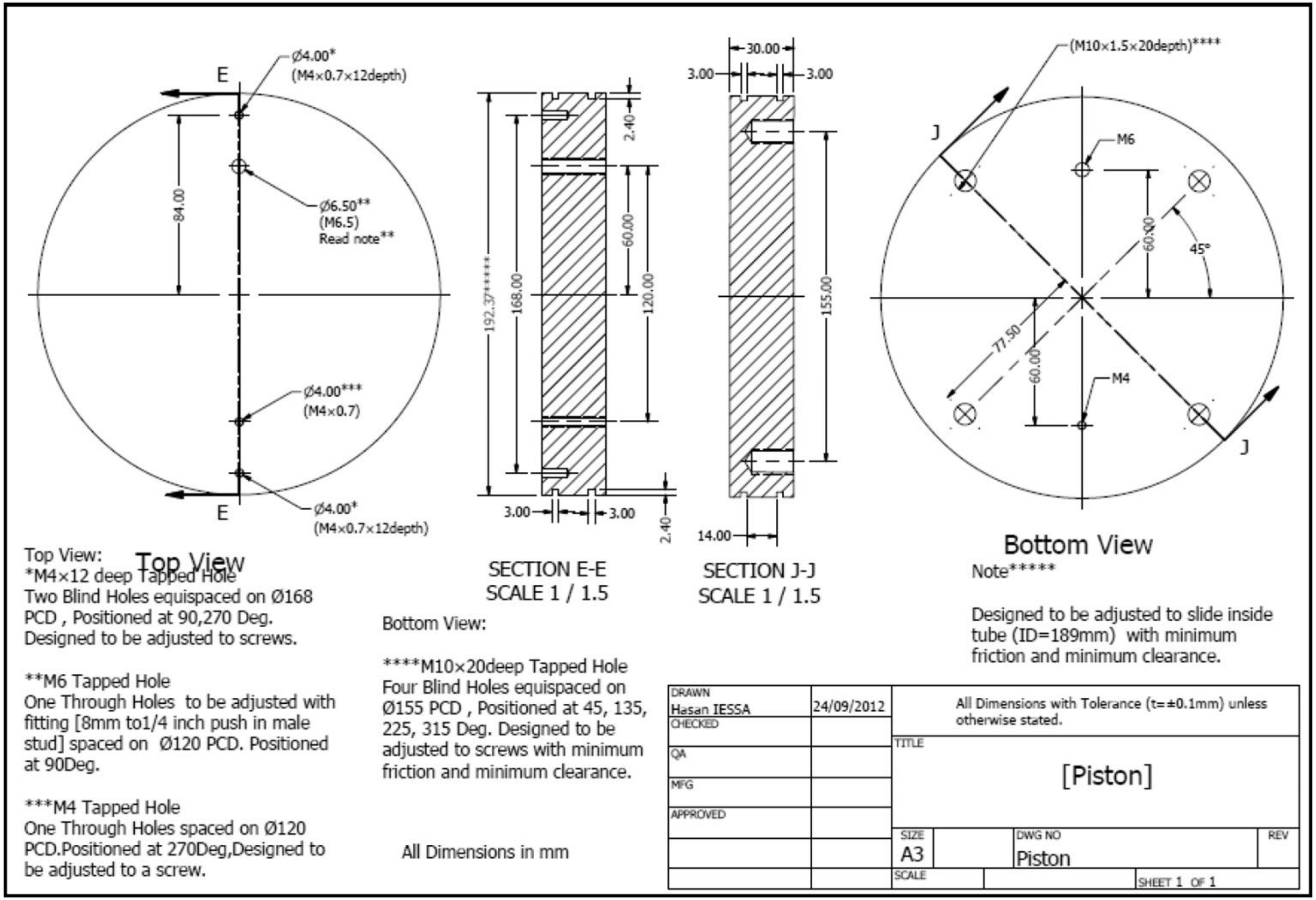
Appendix 7.1. The Cell (all parts)



Appendix 7.2. The Main Tube Flange



Appendix 7.3. The main Tube with the flange and (Studding, Nuts and Washers)



Note*****

Designed to be adjusted to slide inside tube (ID=189mm) with minimum friction and minimum clearance.

Top View:
*M4×12 deep Tapped Hole
Two Blind Holes equispaced on Ø168 PCD , Positioned at 90,270 Deg. Designed to be adjusted to screws.

**M6 Tapped Hole
One Through Holes to be adjusted with fitting [8mm to1/4 inch push in male stud] spaced on Ø120 PCD. Positioned at 90Deg.

***M4 Tapped Hole
One Through Holes spaced on Ø120 PCD.Positioned at 270Deg,Designed to be adjusted to a screw.

Bottom View:

****M10×20deep Tapped Hole
Four Blind Holes equispaced on Ø155 PCD , Positioned at 45, 135, 225, 315 Deg. Designed to be adjusted to screws with minimum friction and minimum clearance.

All Dimensions in mm

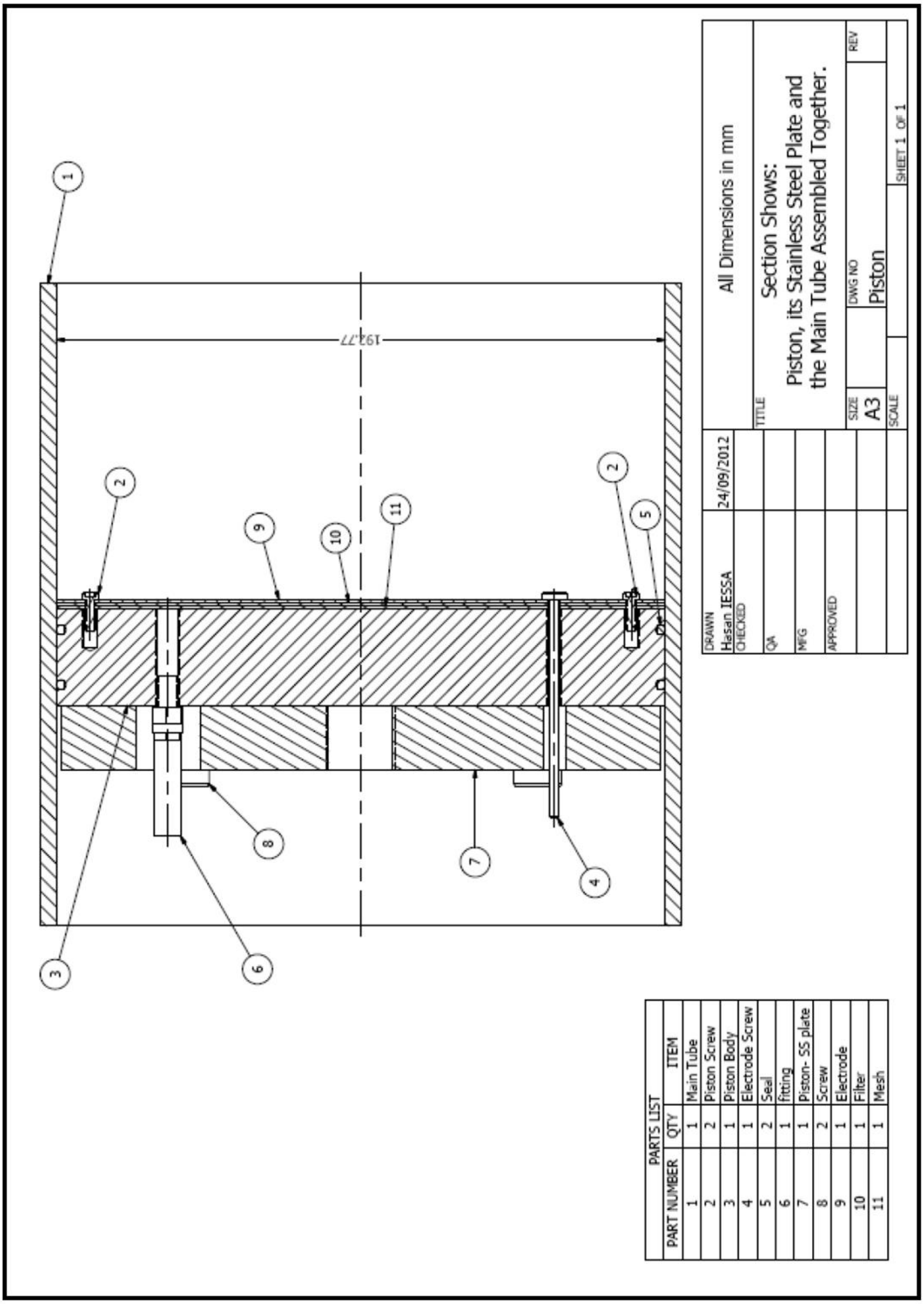
DRAWN Hasan IESSA 24/09/2012
CHECKED
QA
MFG
APPROVED

All Dimensions with Tolerance (t=±0.1mm) unless otherwise stated.

TITLE [Piston]

SIZE A3
DWG NO Piston
REV
SCALE
SHEET 1 OF 1

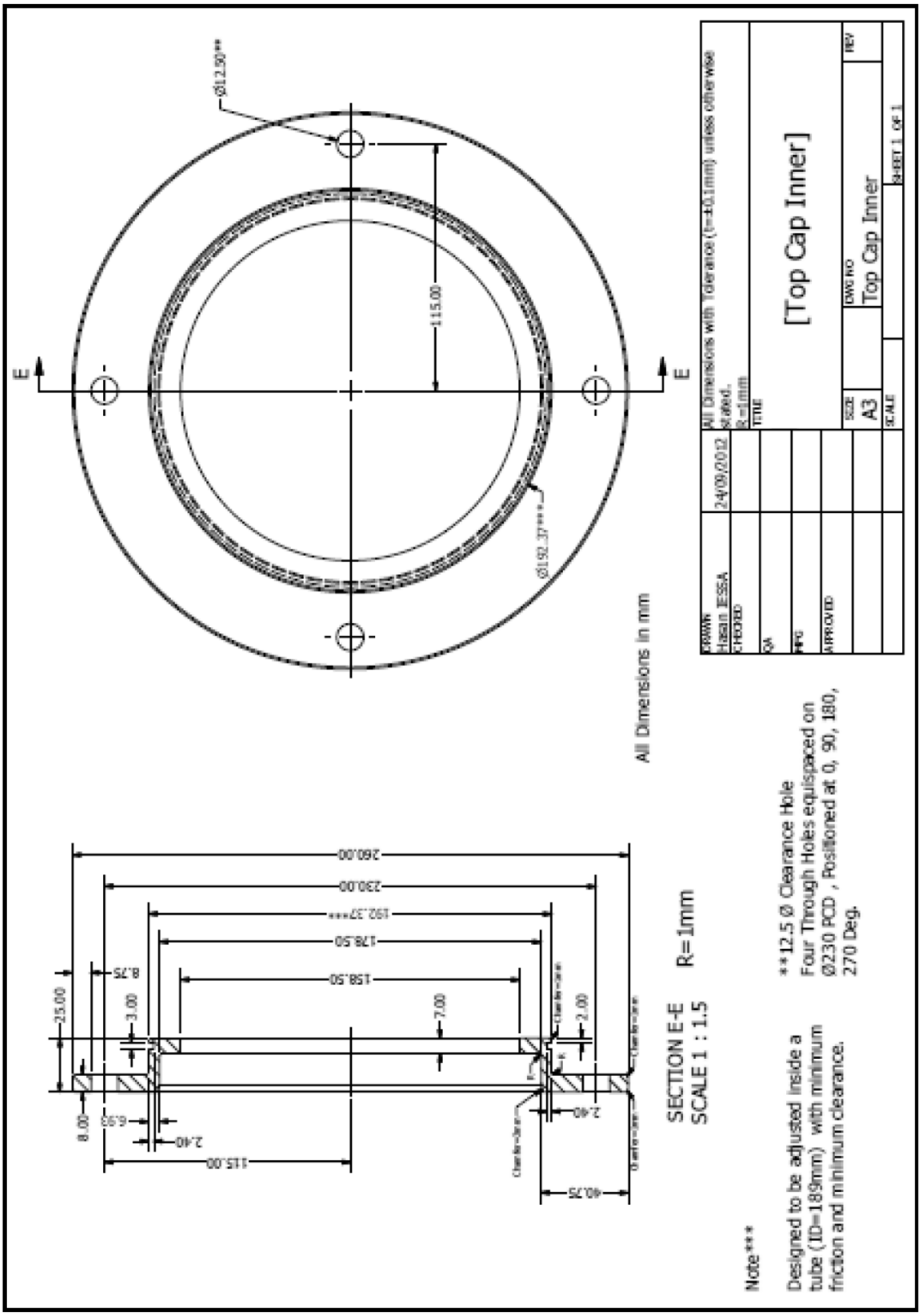
Appendix 7.4. The Piston



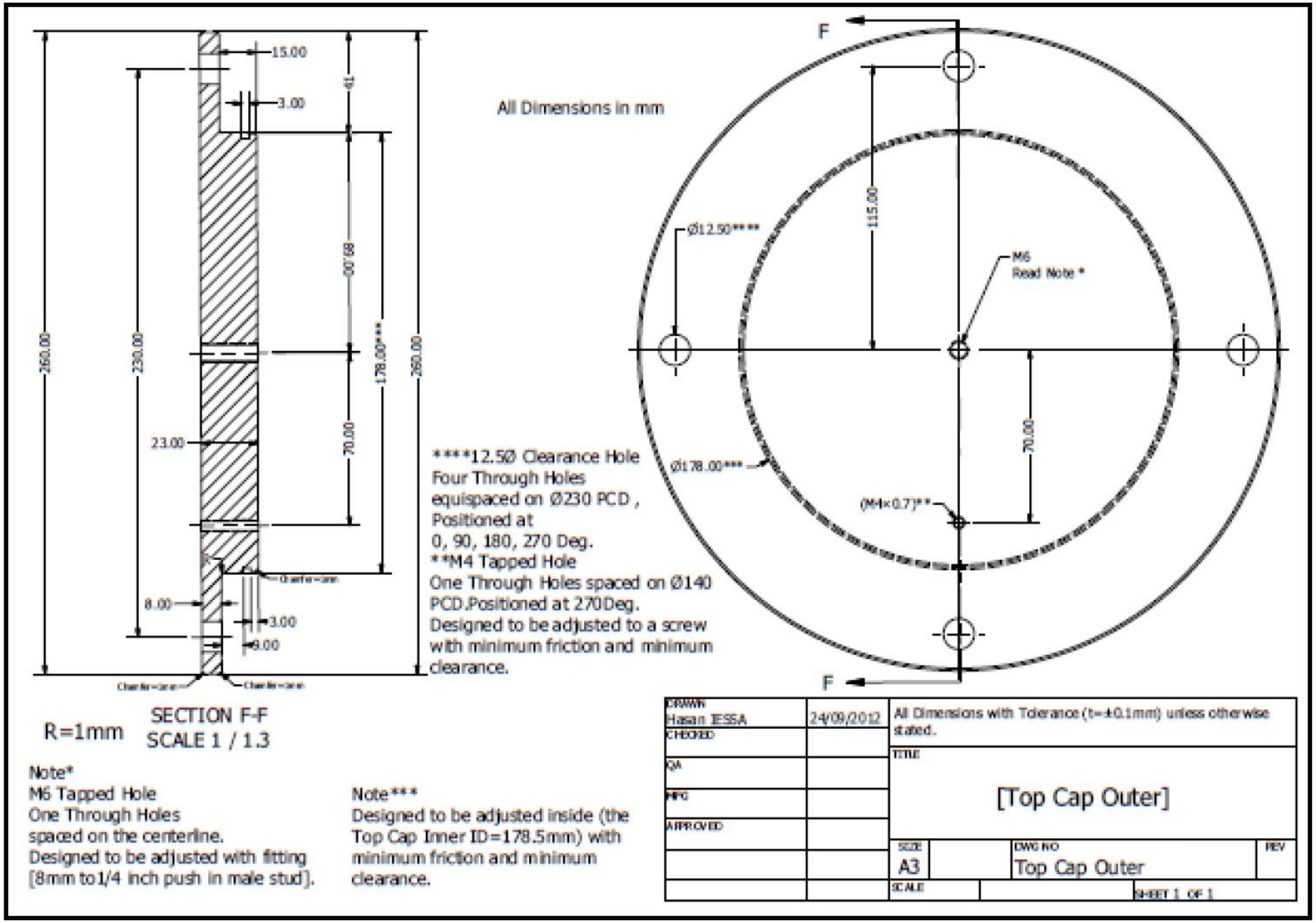
PARTS LIST

PART NUMBER	QTY	ITEM
1	1	Main Tube
2	2	Piston Screw
3	1	Piston Body
4	1	Electrode Screw
5	2	Seal
6	1	fitting
7	1	Piston- SS plate
8	2	Screw
9	1	Electrode
10	1	Filter
11	1	Mesh

Appendix 7.5. The Piston with the main Tube and the Actuator Stroke



Appendix 7.6. The Top Cap Inner



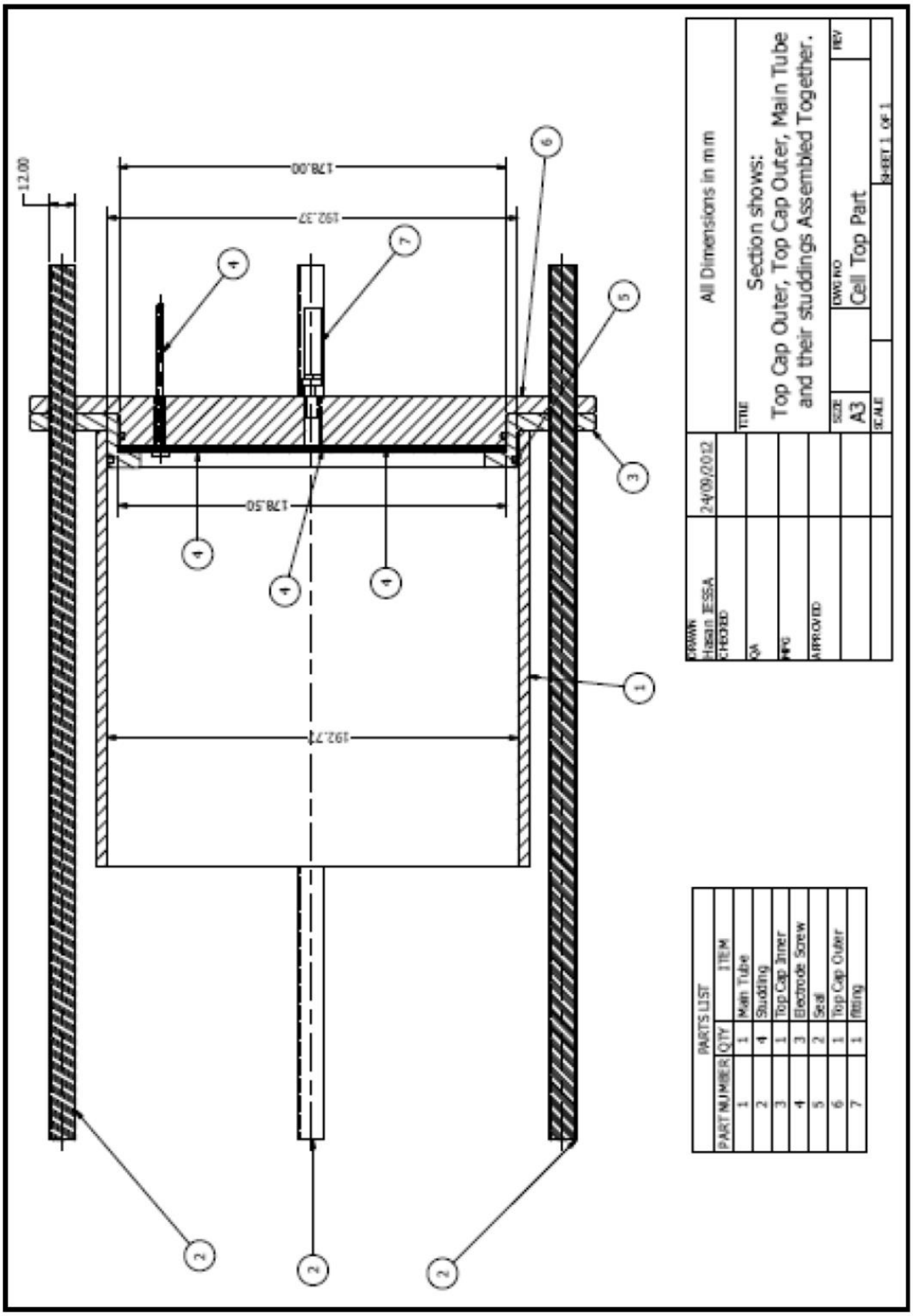
****12.5Ø Clearance Hole
Four Through Holes
equispaced on Ø230 PCD ,
Positioned at
0, 90, 180, 270 Deg.
**M4 Tapped Hole
One Through Holes spaced on Ø140
PCD.Positioned at 270Deg.
Designed to be adjusted to a screw
with minimum friction and minimum
clearance.

Note*
M6 Tapped Hole
One Through Holes
spaced on the centerline.
Designed to be adjusted with fitting
[8mm to1/4 inch push in male stud].

Note***
Designed to be adjusted inside (the
Top Cap Inner ID=178.5mm) with
minimum friction and minimum
clearance.

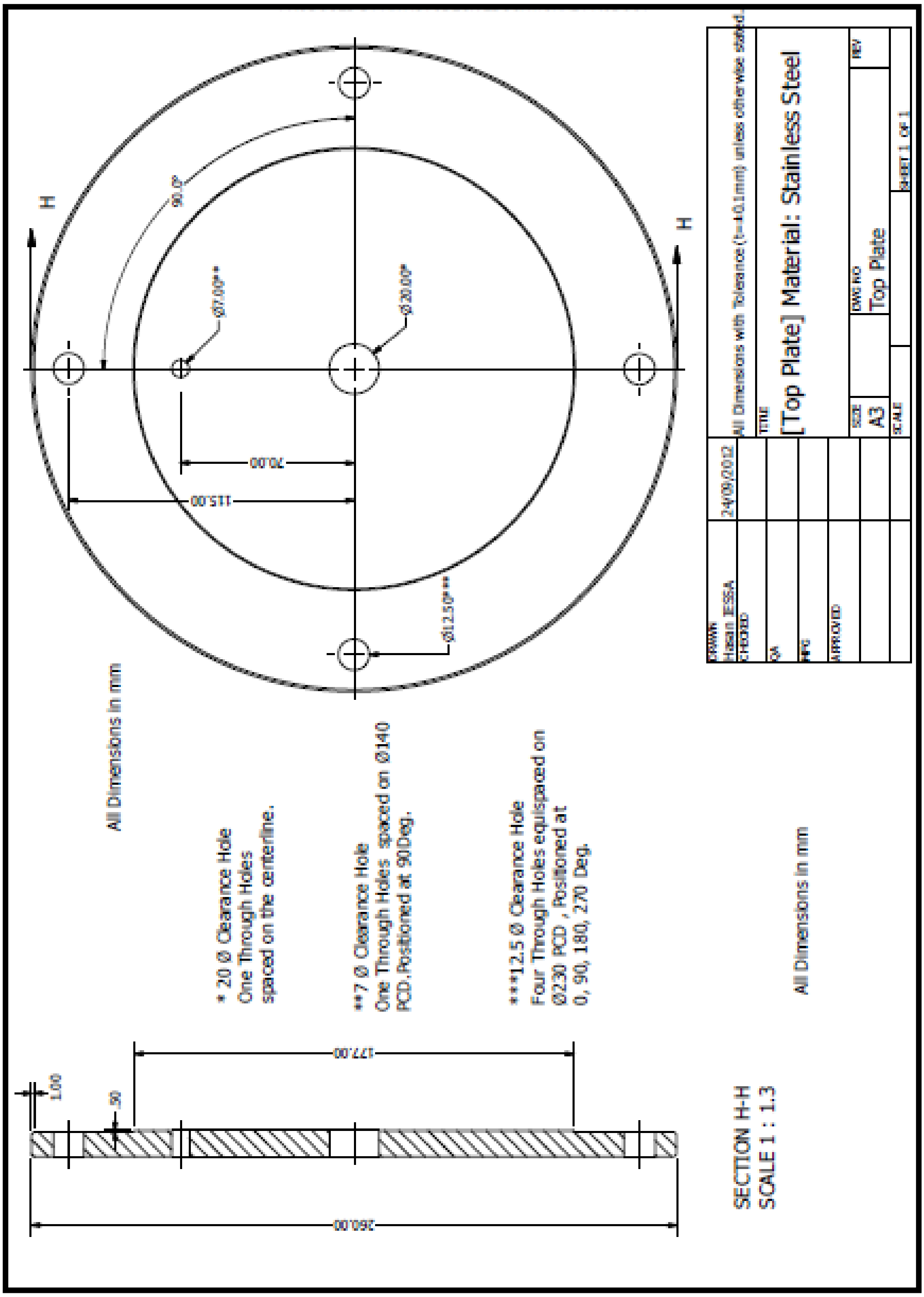
DRAWN
Hasan IESSA
24/09/2012
CHECKED
QA
MFG
APPROVED
All Dimensions with Tolerance (t=±0.1mm) unless otherwise stated.
TITLE
[Top Cap Outer]
SIZE
A3
DWG NO
Top Cap Outer
REV
SCALE
SHEET 1 OF 1

Appendix 7.7. The Top Cap outer

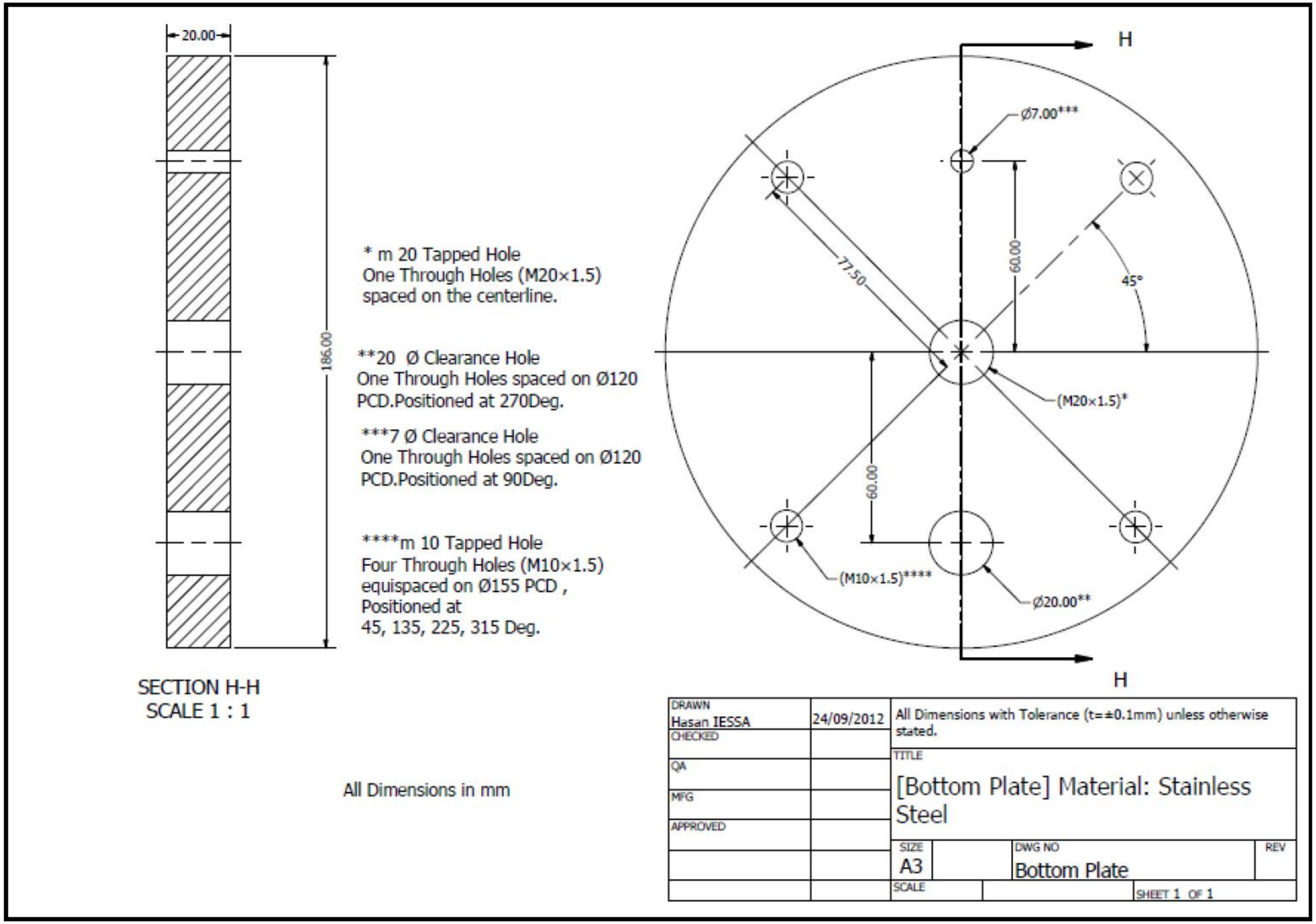


PARTS LIST		
PART NUMBER	QTY	ITEM
1	1	Main Tube
2	4	Studding
3	1	Top Cap Inner
4	3	Electrode Screw
5	2	Seal
6	1	Top Cap Outer
7	1	fitting

Appendix 7.8. The Cell Top Part: Top Cap Inner, Top Cap Outer Plus Studding



Appendix 7.9. Top stainless steel plate



* m 20 Tapped Hole
One Through Holes (M20×1.5)
spaced on the centerline.

**20 Ø Clearance Hole
One Through Holes spaced on Ø120
PCD.Positioned at 270Deg.

***7 Ø Clearance Hole
One Through Holes spaced on Ø120
PCD.Positioned at 90Deg.

****m 10 Tapped Hole
Four Through Holes (M10×1.5)
equispaced on Ø155 PCD ,
Positioned at
45, 135, 225, 315 Deg.

All Dimensions in mm

DRAWN Hasan IESSA 24/09/2012
CHECKED
QA
MFG
APPROVED

All Dimensions with Tolerance (t=±0.1mm) unless otherwise stated.

TITLE
[Bottom Plate] Material: Stainless Steel

SIZE A3
DWG NO Bottom Plate
REV
SCALE
SHEET 1 OF 1

Appendix 7.10. Bottom stainless steel plate