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Improvement and Optimisation of Industrial Process Cleaning in the Brewing Industry

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Abstract

Heineken faces on-going business challenges due to the frequently increasing demand to attain more rigid production, sustainable, and financial targets. There are many factors which influence their ability to meet these targets within their production processes. One significant area which is often overlooked in industry is the limiting aspects within their cleaning in place (CIP) systems which includes; i) production down time, ii) cleaning costs, iii) effluent costs, and iv) quality control.

This thesis details the work done in three projects completed by the research engineer for the EngD with Newcastle University in collaboration with Heineken. The aims of the projects were to benchmark the CIP costs within Bulmers fermentation area, to optimise the detergent cleaning phase of the CIP process for fermentation vessels, and to develop a predictive model to determine the theoretical end point of a cleaning process. The thesis also details business benefits which have been seen from the EngD.

The research engineer has spent 3.5 years of the EngD working on site at Bulmers on the projects by i) collecting extensive data and site knowledge, ii) performing bench scale experiments, iii) analysis of results, and iv) on site verification of findings. The rest of the time was spent at Newcastle University for the taught section of the EngD, or performing pilot scale trials on the ZEAL pilot plant at Birmingham University.

Based on the outcomes of the projects, the work done may be implemented to optimise the detergent CIP step, reduce chemical and water consumption, reduce effluent costs and reduce production down time. The predictive model may also be further developed for implementation on site to provide cost benefits in the same aspects of site cleaning. The overall implementation is predicted to save more than £2,000,000 per annum for Bulmers with the opportunity to be extended and provide comparable savings for all Heineken sites.

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Declaration of Originality

I, Charlotte Atwell hereby declare that this thesis represents my original work and that I have used no other sources except as noted by citations. All data, tables, figures and text citations which have been reproduced from any other source, including the internet, have been explicitly acknowledged as such.

Signed:………………………………………………………….

Date:…………………………………..

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Nomenclature

A	Surface area (m^2)
ABV	Alcohol by volume
$_{aq}$	Aqueous solution
$CaCO_3$	Calcium carbonate
CBRT	Bright cider tank
CIP	Cleaning-in-place
CO_2	Carbon dioxide
CO_3^{2-}	Carbonate ion
C_2H_5OH	Ethanol
$C_6H_{12}O_6$	Glucose
$C_{20}H_{14}O_4$	Phenolphthalein
$C_{27}H_{28}Br_2O_5S$	Bromothymol Blue
c_b	Initial bulk removal parameter
c_P	Specific heat capacity (kJ/kg/K)
c_r	Residual clean parameter
DCF	Discounted cash flow
dT	Rate of change of temperature (K)
du	Rate of change of fluid velocity (m/s)
dy	Rate of change of height (m)
EngD	Engineering Doctorate
FTU	Formazin Turbidity Unit
FV611	Fermentation Vessel number 611
$_g$	gas
h_e	Specific enthalpy of evaporation (kJ)
H^+	Hydrogen ion
H_2CO_3	Carbonic acid
H_2O	Water
HCl	Hydrochloric acid
HCO_3^-	Bicarbonate ion
hl	hectolitre (100 litres)
K	Reaction rate constant (l/mol)
KOH	Potassium hydroxide

$_l$	liquid
mSPC	Multivariate statistical process control
$\dot{m}$	Mass flow rate (kg/s)
$\dot{m}_{\mathrm{steam}}$	Mass flow rate of steam (kg/s)
μ	Kinematic viscosity (N.s/m^2)
M	Molar concentration
M_{acid}	Molar concentration of acid
M_{base}	Molar concentration of base
NaCl	Sodium chloride
NaOH	Sodium hydroxide
Na_2CO_3	Sodium carbonate
$NaHCO_3$	Sodium bicarbonate
NIR	Near infrared
NPV	Net present value
OEE	Overall equipment effectiveness
OH^-	Hydroxide ion
Opco	Operational cost
OSA-N	Diversey brand of nitric acid
PLS	Partial least squares
PMT	Particle Measuring Technologies (company)
PPE	Personal protective equipment
p_f	value of fitted curve at time t
p_{avg}	average cumulative particle count over last 10 data points
q_{CIP}	Energy required to heat CIP stream (kJ/kg)
q_{steam}	Energy required from steam to heat CIP stream (kJ/kg)
R	Residual
RMSECV	Root mean square error of cross validation
ROI	Return on investment
S	Standard deviation
SAP	Business Software database package used by Heineken
SCADA	Supervisory Control and Data Acquisition
SOP	Standard Operating Procedure
SRES	Standard residual
T	Temperature (K)

t	time (s)
τ	Shear force of fluid (Pa)
T23	Tank 23
u	Fluid velocity (m/s)
V_{acid}	Volume of acid
V_{base}	Volume of base
v	Volumetric flow rate (m^3/s)
WACC	Weight average cost of capital
w/v	weight for volume
w/w	weight for weight
y	Height against tank wall (m)
ZEAL	Zero Emissions through Advanced Cleaning
ZnS	Zinc sulphide

Introduction

Project Outline

The overall aim of the EngD project is to improve and optimise the current cleaning-in-place systems which are in use at the Heineken sites. There are three key stages to the EngD project, which has been segmented into a portfolio of smaller, more manageable projects. These projects are as follows:

1. Benchmarking the Process
2. Optimisation of Cleaning Detergents
3. Cleaning Predictive Model

This will involve performing a review of the current systems in place and their effectiveness in order to benchmark the process capabilities, which will later enable future improvements and benefits to be quantified.

The optimisation of the cleaning detergents involves a detailed study of the use of cleaning detergents within Heineken's CIP systems with respect to their effectiveness of cleaning and the science behind their degradation. Based on the knowledge gained from this, it will be possible to optimise the detergent cleaning steps and provide Heineken with a cost saving and improved cleaning confidence.

The cleaning predictive model involves developing an advanced control system for CIP sets to predict the end point of cleans. This will be done by considering various online data measurements to determine which methods give the best information as to how clean the equipment is. Control algorithms using data fusion techniques will be developed to combine multiple sources of online data to give the optimum prediction of 'clean'. Confidence intervals of these predictions will be analysed and tested via industrial case studies to ensure a sufficient sensitivity of assessment. The next steps will involve investigating the transferability between different process types (e.g. cider, beer, etc.) to ensure that the system is useful for all Heineken sites.

Ultimately a business plan and design for the implementation of these projects to be utilised on site in conjunction with their current software and process systems should be developed in order to provide Heineken with greater benefits.

The outcome of the successful EngD will be to reduce clean times, ensure that every clean is sufficiently completed, reduce water, chemical and energy consumption, effluent volumes, and reduce production down time. Overall this will substantially reduce the CIP costs for Heineken on a global scale.

To progress the experimental phases of the project, an experimental road map was required. Due to industrial supervision availability, the site location was predetermined as Bulmers, however, the objective of all the projects was to benefit all of the factories. Bulmers is a cider only site, but occasional access is also available for John Smith’s; an ale and lager site.

Worst case scenario fouling was required for each project, to ensure that the minimum required cleaning is achieved through experimentation. Optimisation for soils with lower cleaning requirements can be attained after transferability between sites and soils is verified.

Beer bottoms from John Smith’s and lees from Bulmers were the worst case scenario soils from their respective sites. Please see the compositions of each in appendices 1 and 2. Cider lees (pH 2) was more acidic than beer bottoms (pH 4), which made it easier to clean. Due to this, beer bottoms were the fouling of choice for all projects. See table 1 which represents the road map of the three projects, the fouling used on each, the trial equipment, area, and application locations for each.

Table 1: Project Road Map for Fouling, Equipment, Trial Area, and Application of Results Areas

Project Road Map			
Projects	Benchmarking	Carbonate in Caustic	Predictive Model
Trial Equipment	Software tool	Bench Scale Rig	Pilot Plant
Trial Area	Bulmers Fermentation Area	Bulmers	Birmingham University
Application Area	Bulmers Wider Site and other Heineken Sites	Bulmers Fermentation Area	All Heineken Sites (once effectiveness and transferability verified)

Background Setting

Heineken are international, market leaders in brewing, and own many famous and successful brands of lagers, ales, and ciders e.g. Heineken, Fosters, Bulmers, John Smith's, Newcastle Brown Ale, Strongbow, and many more.

The majority of this project was based in Bulmers in Hereford, so knowledge of the cider production process is of great importance. However, knowledge of brewing ales and lagers was also important in order to understand the transferability of the project work between different sites, products, and countries. For the scope of this thesis, the production process of cider making has been detailed, along with an overview of a comparison to lager/ale brewing processes which are relevant to the content of these projects. This allows transferability opportunities to be assessed.

Cider Production

The overall cider production key process steps are summarised in figure 1.

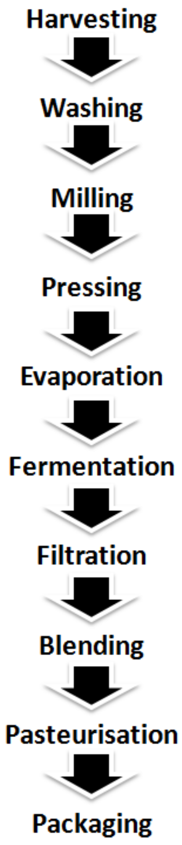


Figure 1: Cider Making Process Steps

Cider is made from apples (dependent on Heineken product, a specified mix of bittersweet and culinary apples), or pear cider can be made using fresh pears. Bulmers have their own bittersweet apple orchards which are used in their cider production.

Apples are unloaded from tankers into pits, and the buoyancy of the apples is utilised by passing water through the pits to transport the apples to a conveyor belt. The apples are observed by operators, and any large foreign objects are removed to prevent later problems with blocking of plant equipment, prior to washing the apples using automated sprays on the conveyor belt.

The apples are milled, where they are ground into a pulp. Enzymes are added to the pulp to depectinise the mixture. These enzymes hydrolyse pectins present within the pulp, to smaller, more soluble compounds. This produces a fruit mash of a lower viscosity which reduces the difficulty of extracting fruit juice from the mash.

The next stage is to pass the mash through the Bucher process which mechanically presses the resulting mash, to squeeze out a cloudy fruit juice of approximately 10 to 15 brix (brix is a measure of sucrose present in an aqueous solution). Remaining solids and pomace are also separated from the mix at this stage.

The fruit juice is then evaporated under a partial vacuum (to reduce energy requirements for heating) at 70^{o}C to 80^{o}C. This reduces the water content of the fruit juice which will then increase the overall sugar content to 70 to 72 brix. The resulting juice is very viscous and consists of sucrose, fructose, glucose and maltose. It also contains tannins which gives it a dark brown appearance. The apple concentrate then goes to storage until it is required for cider production, or sold and transported via tanker. Due to the high sugar content of the apple concentrate, it can be stored at ambient conditions for long periods of time (up to five years) without affecting the quality.

To produce cider, the sugars present within the apple concentrate must be fermented with yeast to yield ethanol, and carbon dioxide as a by-product. See equation 1.

$C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2$

Equation 1: Conversion of Glucose to Alcohol

The concentrate sugars alone will only ferment to approximately 6-6.5 % ABV (alcohol content), so glucose is added in addition to the concentrate to produce an alcohol content of approximately 12-13 % ABV. In addition to that, nutrients are added to promote yeast growth throughout the fermentation process. Fermentation takes place over a period of around two weeks (time varies depending on product requirements) until the specific gravity reaches a minimum, and so the alcohol content reaches a maximum. See figure 2.

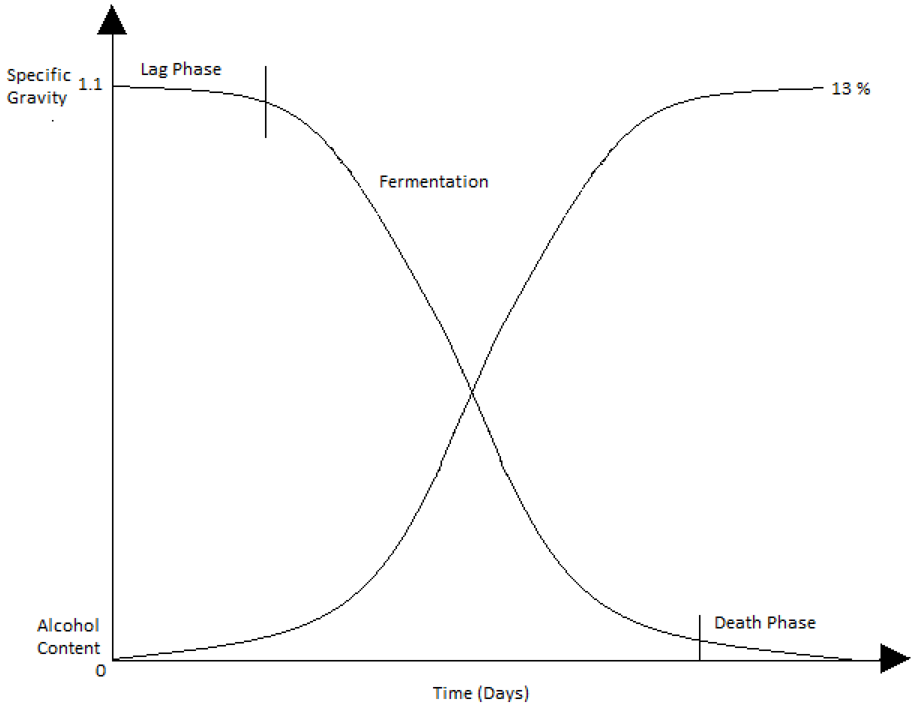


Figure 2: Graph of Specific Gravity and Alcohol Content vs. Time

During the first twenty four hours, fermentation is aerobic, where oxygen is used to facilitate the growth of yeast. After that, there is no remaining oxygen, so the yeast respires anaerobically. The yeast eventually dies when there is no remaining sugar, or the alcohol content is too high, or a combination of the two. After fermentation is completed, the cider is left to mature for approximately four days to ensure that the yeast is dead. The yeast is mostly removed when the fermentation vessel is drained due to the angle at the bottom of the vessel. The alcohol yield is usually around 93 %. The remaining 7 % is accounted for by yeast growth and glycerol which is another potential by-product of the process.

The cider produced is of a very cloudy appearance. To achieve the desired quality of Heineken cider, the next stage is to filter the cider to ensure that it is clear. Initially filtration is performed using cross flow filters at ambient temperature, where the cider is pumped through the filters at right angles to the filter to reduce the initial turbidity. The cider is then chilled to 0 to 2^{o}C as turbidity tends to increase as temperature is decreased. This is due to the solubility of the particulates (usually tannins) decreasing, and so crystallising on chilling. The chilled cider then flows through Orion sheet filters to remove the remaining haze. The product is now bright cider.

Bright cider contains a very small quantity of sugar (as the majority is fermented to alcohol), has a high alcohol content (for cider), and is very bitter in taste at this stage. The next stage of the process is to blend the cider by adding water, flavourings, and colour to meet the Heineken specification of the relevant cider product. Additives include sulphur dioxide (to preserve the cider), malic acid (to regulate the pH level), fructose, caramel, saccharin, and apple concentrate (to provide sweet flavours), aromas and essence (to provide colours and perfumes), de-aerated water (to reduce the alcohol content), and carbon dioxide (to add effervescence to the product).

At this stage, the product is tested offline to ensure that all measurable specifications are met in the analytical laboratory, and also in the sensory laboratory where a team of trained, experienced tasters ensure that the product tastes, smells, and appears as it should. If specifications are not met, then adjustments are made to rectify this. Once the product is satisfactory it is pasteurised (to ensure that no microbial growth will occur), packaged, and ready for consumption.

Brewing begins with a completely different process of converting malt to wort to be fermented. This process varies depending on the type of beer/lager and characteristics of the drink that you are trying to achieve. The fermentation process onwards is very similar to the cider making process, including maturation, filtration, blending if required, pasteurisation and packaging. The content of the EngD projects are mostly based around fermentation vessels and generic cleaning of multiple types of equipment and types of brewery/cider fouling. This means that the scope of this EngD will be relevant to both brewing and cider making.

The production processes of all of Heineken's drinks are very important to the final product quality and running of the business. Production efficiency has a direct impact on bottom line costs, which will influence the overall profit margins and affect all aspects of the business. The quality of the production processes also directly affects the quality of the final product. Without this quality, sales volumes and market segments will not be reached which will also have a significant impact on business profits and operation.

Through ensuring that production efficiency is optimised and quality standards are adhered to, a strategy to increase production unit profitability will be possible. This can directly improve overall business growth, by increasing profit margins and enabling capacity increase to aid meeting the increasing capacity which Heineken requires. This is due to their new range of innovative flavoured beers and ciders which are being developed and produced. A factor which has an extensive impact on both product quality and production efficiency is cleaning in place (CIP).

CIP

Product quality is extremely important to Heineken. An important feature which is often overlooked by members of the public is the cleaning of the production process. Producing a product which is consumable by humans poses a risk as microbial organisms within the food may infiltrate microbial growth, leading to product contamination, which may result in food poisoning for the consumer. It is essential to ensure that all equipment used in production is sufficiently clean before and after every batch is produced. If not, microbial growth may begin to occur which will require increased cleaning efforts. In addition it may go unnoticed and contaminate products, resulting in a major cost impact on the company, in addition to the impact on sales volumes due to negative publicity.

The current CIP systems have high costs which are associated with lengthy cleaning times (resulting in production down time and further costs), more CIP effluent requiring treatment than is necessary, and high quantities of water and electricity consumption. Sometimes cleaning requirements can vary despite being on the same cleaning setting and equipment; e.g. there may be product changeover and one product requires a more vigorous clean than the other. Where the cleaning parameters may meet the requirements of one batch, it may not be sufficient to clean another batch. In such a scenario it is unknown if the clean was not sufficient until after the clean has finished, so cleans may require repetition, resulting in further cost implications. Environmental targets from the Government are also continuously increasing in rigidity; so effluent, water, and electricity consumption should be reduced wherever possible.

Some direct problems arising with CIP include; i) the level of cleanliness of the equipment is unknown before or during the clean, as conductivity is not an indicative measurement of this, ii) formation of sodium carbonate in the caustic solution, which reduces the cleaning power and is very difficult to detect using the online conductivity meter. This problem causes excessive dumping of the chemicals if a cautious policy is adopted, which is expensive. This can sometimes result in chemical cleans which are not within specification being performed, if the caustic levels have fallen below the control limits. iii) There is uncertainty about the effectiveness of the cleans provided by different types of spray heads in vessels, iv) tanks, filters, and heat exchangers are more complex than ordinary pipe work is to clean.

ZEAL

Zero Emissions and Cleaning (ZEAL) is a consortium of companies that formed in 2005 with a mutual interest in the benefits of the optimisation and reduction of common challenges with CIP. It was funded by the UK Technology Strategy Board, and the partners involved included; Heineken UK, Newcastle University, Imperial College London, Bruker Optics, University of Birmingham, Unilever, GSK, Ecolab, Cadbury, Alfa Laval, and GEA. Further detail of the work carried out by the consortium is discussed in the literature review. Some of the physical outcomes of ZEAL included: a pilot scale cleaning plant, a bench scale cleaning rig, a benchmarking tool, identification of the potential of using particle density/count/size online for cleaning measurements as well as various other tools and activities. Each of the mentioned outcomes is used within this EngD project. Further details of how they were implemented are given in the relevant materials and methods section in each chapter of this thesis.

ZEAL is now finished, however, it is anticipated that a ZEAL 2 group will be continuing to develop the work done on CIP, and this EngD will be a key contribution to ZEAL 2 by considering various aspects of tank cleaning, chemical action on soils, and cleaning data integration and modelling.

Original Contributions to Literature

Throughout this EngD project, my key scientific deliverables are:

1. Taking a benchmarking strategy, modifying and adapting it to be applicable in industrial brewing, and to consider the transferability between breweries and varying product types.
2. Developing scientific understanding of the cleaning process, and the impact of cleaning detergents and their degradation in order to quantify the cleaning efficacy.
3. Identifying a measurement system for monitoring the cleaning process, and a method to use this set of different measurements to best effect.
4. Optimisation of the cleaning strategy within the industrial environment, balancing costs against benefits, and taking into account the industrial constraints.
5. Developing robust, experimental, lab scale cleaning trials that overcame operational challenges and produced repeatable and reproducible results.

Chapter 1. Business Benefits of the EngD

By utilising an EngD research engineer to perform their research, Heineken as a business have benefited with respect to cost and quality of work produced. Research engineers recruited are required to have a minimum of a 2:1 degree in a relevant discipline, along with being assessed on practical experience within their field. The research engineer is considered to be of a high graduate calibre, and can be taken on by the company at a cost of £8,000 per annum (over four years). A graduate salary for the 2014 intake at Heineken is £28,500. This, significantly lower annual cost (circa £20,500 less) also includes support from two academic experts in the field, access to state of the art research facilities, access to wider relevant research groups within academia and industry, and further funding or research from the government and academic institutions.

To estimate the financial benefits Heineken have received; the following assumptions have been made:

- Two Heineken supervisors at any one time for a total of five days per year each, at a cost to the company of £100 per hour per person. This equates to £8,000 per year.
- All assumed cost saving opportunities were implemented at the end of the four year EngD research period.

Costs are as follows:

£8,000 per year on EngD student

£8,000 per year on supervision

Over 4 years, £64,000 invested

Savings realised (excluding capital outlay):

£2,000,000 per annum from year 5

% ROI: 3,125%

%ROI from physical recommendations: 1500%

The EngD research engineer is able to benefit the company by being present on site for approximately 75% of the time, throughout the four years of the doctoral course. This enables the student to develop and utilise good process knowledge to benefit the overall project work. By completely submersing the research engineer within industry, they are also able to build networks throughout the company and with suppliers to further benefit the project and exchange useful information. All of this provides an ability to work more effectively to maximise the output from the research engineer, and so maximise return on investment.

The research engineer is able to focus around 90% of their research time to working on the specific research problems on a macro level within the scope of the EngD project. This would be an unrealistic time to expect a graduate or employee of the company to invest in these areas, which makes developing solutions and improvements difficult without sufficient time and people resources invested into the problem. By enabling these areas to be investigated in such detail, outcomes that would not otherwise have been achieved for the company have been reached without any additional manpower investment costs.

In addition to the research involved within the EngD, there are alternative activities which the research engineer is able to participate in for no additional cost to the business, but will add value to the business activities. The cost of time spent on additional business activities is approximately 28% of the cost of time spent by a current graduate (or an even smaller percentage of the cost spent by a current experienced employee). If the total time spent on these activities were to be recorded, there would be a significant, visible saving to the company. A list of activities which the research engineer has been involved with/completed is shown below:

- Report of faulty carbon dioxide mains valve found
- Attending weekly micro meetings and providing input to solutions

- Bench scale experiment performed to recommend deep clean used on fermentation vessels for tannins
- Report of leaking pump found
- Report of failed gasket found
- Input on project for minimising micro issues in fermentation vessels
- Recommendations to minimise carbonate formation in the caustic for fermentation vessels
- Calculation of specific utility costs for CIP
- Providing flow rates and information on water consumption
- Identified faulty pressure gauge
- Input with general problem solving
- Assisted with modification of the tank CIP cleaning routes to minimise the impact of interrupted cleaning head spray patterns and ensure full cleaning coverage of tank and mains associated.
- Assisted with the identification of the CIP routes for the CBRT process tanks.
- Assisted with the identification of an incorrectly installed valve on a heat exchanger (showing open when closed)
- Identification of low flow rates and pressures on the fermentation vessel CIPs which would result in lower cleaning capabilities due to the Toftejorg cleaning head requirements.
- Established how the Corning machine worked to identify dissolved carbon dioxide to assist with an investigation on the lines as to why the levels were too low on a shift. It was discovered that an operator had not been testing the levels and tried to blame the machine for needing calibration.
- Completed four tasting exams with marks between 80 and 100%. Now sit on the tasting panel weekly to assist with quality testing.
- Spoke at the 125th Anniversary for Bulmers
- Fixed a conductivity probe in the lab.
- Provided flow information to assist with the installation of the centrifuge and the pump.

- Acquired software for obtaining data from the flow meter to be used by everyone on site.
- Calculations to determine why the fermentation cleans were gaining heat energy. It was due to the exothermic reaction between carbon dioxide and sodium hydroxide.
- Calculations of expected pressure drops within pipes from the CIP supply to the tops of the fermentation vessels.
- Verification of the pH calculations used by Diversey to identify the quantities of sodium hydroxide and sodium carbonate.
- Assisted team members with the use of Microsoft Office to use various functions to complete their work more efficiently.

Chapter 2. Literature Review

2.1 Introduction

The objectives of this chapter are to review the literature around relevant subject areas in relation to the scope of this EngD project. Subject areas of interest are:

- Food and drink cleaning classifications
- Process benchmarking
- Equipment cleaning
- Cleaning chemicals
- Online monitoring and predictive models of cleaning

The literature review intends to summarise key literature throughout the research phases of the EngD, and highlight the research path moving forward.

The initial stages of this project came from previous work which was completed by the ZEAL consortium; a research group which specialises in food, drink and consumer product cleaning-in-place. Some of the literature which was developed by the ZEAL consortium has been discussed throughout this review.

The ZEAL consortium have performed extensive work on CIP cleaning theory, optimisation, modelling of deposit removal, correlations between process variables and cleaning, data fusion of online measurements, predictive modelling of clean end point, product recovery, and soil characterisation. The overall objective of this work was to develop new/improved technologies and methodologies to reduce CIP costs, chemical usage, effluent and product waste, utility consumption, and cleaning times.

Heineken and Newcastle University were both contributing members to the literature developed by ZEAL. Several improvement opportunities and further research questions were exposed. A collaborative EngD project between Newcastle University and Heineken was formed as a result of this.

2.2 Cleaning and Fouling Classification

Palabiyak et al (2014) emphasised that production efficiency and sustainability are both important necessities in a modern, manufacturing, multiproduct plant. CIP plays a large part in both production efficiency and sustainability. Wilson (2005) and Fryer and Asteridaou (2009) all highlight that ineffective CIP can have a detrimental effect on product quality, which is a threat to any food and drink business due to the impact of quality on company reputation and sales as a result. It is essential to ensure that the CIP set is achieving the cleaning level which is required for maximum product quality and production efficiency.

Burfoot and Middleton (2009) have discussed that CIP sets can have high environmental impacts and demands on water, energy, and waste treatment. Hien et al (2008) estimate that the brewing sector alone was using approximately 3.9-6.3m^3 of water for every m^3 of beer processed in 2008. Palmowski et al (2005) suggested that approximately 50% of sodium found in food and drink trade waste originates from CIP processes. Thus optimisation of CIP processes to minimise environmental demands is necessary for the future sustainability of the food and drink industry.

2.2.1 Classification of Fouling and Cleaning Requirements

It is important to have an understanding the types of fouling which needs to be cleaned, how their properties vary, and how to ensure that the correct cleaning mechanisms and chemicals will be applied. Changani et al (1997) identified that the fouling conditions in a process will dictate the required cleaning conditions.

Fryer and Asteriadou (2009) investigated soils which had previously been researched as part of ZEAL and classified them as soil types with their respective cleaning mechanisms. This encompassed information around their cleaning fluid flow rate, dissolution rates, and rheology. A cleaning map was developed to illustrate how the different soils’ cleaning mechanisms vary in a soil complexity vs. cleaning mechanism diagram. See figure 3.

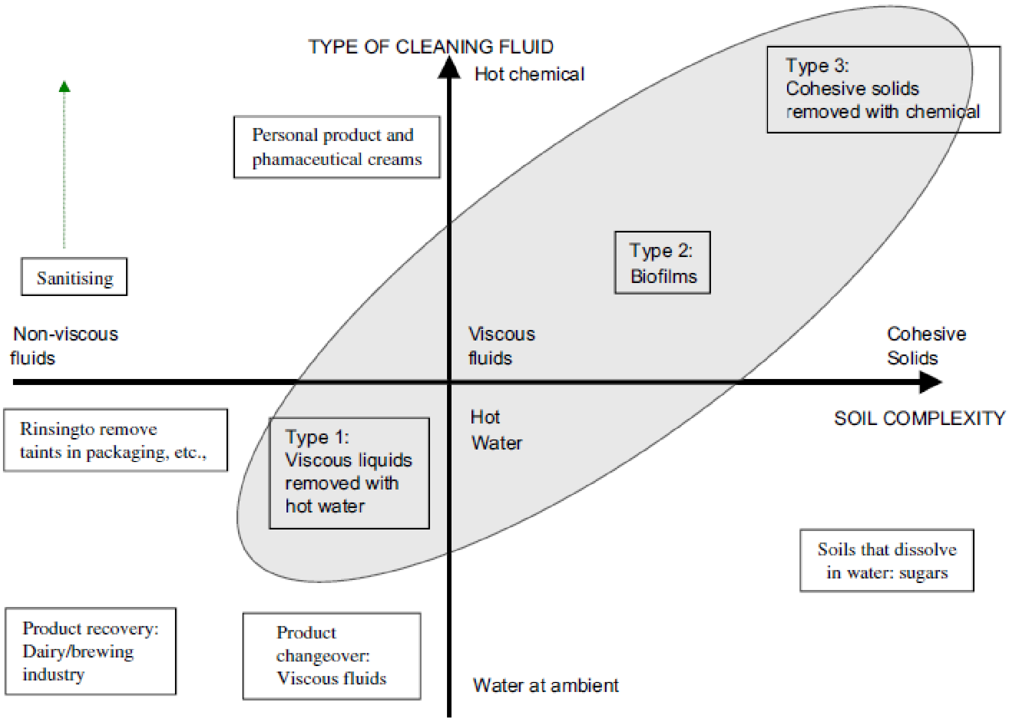


Figure 3: Soil Complexity vs. Type of Cleaning Fluid (Fryer and Asteriadou 2009)

The intension of Fryer and Asteriadou’s work was to benefit the wider industry in developing solutions to cleaning challenges. The concept of the cleaning map was a development from Epstein’s 5 x 5 fouling mechanism matrix (1981). The cleaning map shows a graph of problem clusters, where these clusters are typed and classified based on their cleaning requirements and mechanisms. The graph is intended to be qualitative and not quantitative for the purpose of simplicity, as scale up rules will vary for all different mechanisms.

This work has shown a distinct relationship between identified, grouped soil types and the cleaning mechanisms and chemicals which will be required. This will be useful for determining cleaning requirements throughout the project, as well as enabling transferability of the cleaning improvements and optimisation for other types of soil considered.

Goode et al (2010) performed investigations on the cleaning of brewery yeast slurry. It is a type 2 soil which can be cleaned mostly using a water rinse, but requires chemical cleaning to completely remove the soil. Goode et al also demonstrated that dried on brewery foam is a type 3 soil, which requires a hot chemical clean to completely remove the soil. Rao (1999) showed that type 2 soil cleaning is dependent on temperature and cleaning time. Christian (2004) and Aziz (2008) have both demonstrated that type 3 soils cannot be removed without chemical cleaning detergen, regardless of the cleaning flow rate which is used. Goode, Rao, Christian, and Aziz have all verified the information from Fryer and Asteriadou (2009) with regards to cleaning these soils.

This knowledge is useful around the cleaning of brewery soils, and will also assist with the selection of fouling type when experimental work will be performed and realistic brewery fouling representation will be required. The work also compliments the theory of the work of Fryer and Asteriadou (2009), as the findings can be fitted to the cleaning map which they developed.

2.2.2 Cleaning Theory

It is important to have an understanding of fundamental cleaning behaviours in a brewing or food and drink CIP system. This will enable process areas for investigation to be targeted more efficiently, and educated process changes to be made with an appreciation for the probable effects of those changes.

Goode et al (2010) investigated cleaning mechanisms of yeast slurry in CIP sets for Heineken. Goode's work consisted of optimisation of water rinse and chemical wash conditions by varying cleaning fluid flow rates and temperatures, as well as the classification of two of the key fouling types found with a Heineken brewery. The validation of clean was based on visual inspection. This showed that increasing flow rates reduces cleaning times, and increasing cleaning temperatures assists in the dissolution of soils, however, once the temperature exceeds 50^{o}C, the elasticity of the soil is increased which also increases the cleaning difficulty of the soil. The methodology used for simulating the yeast slurry cleaning in an industrial setting proved to be representative of theoretical industrial expectations.

Goode et al (2013) performed an extensive literature review on fouling and cleaning classification in the food and beverage industry. This work included discussions on; the effects of cleaning temperature, Reynolds number, cleaning fluid shear stress, and cleaning flow rate on cleaning time and effectiveness.

Goode et al demonstrated that increasing shear stress was shown to reduce cleaning time. Temperature and shear stress have varied effects on fouling removal. Increasing the shear stress and the cleaning temperature were shown to have more impact on cleaning time reduction than increasing the cleaning chemical concentration.

By increasing the cleaning temperature, it was possible to reduce the chemical concentrations required when cleaning type 2 and 3 deposits. This identified that by increasing the awareness of the cleaning process, it is possible to use cleaning monitoring methods for optimization purposes.

The knowledge from Goode et al's literature review will be useful for understanding cleaning theories. An interesting point which was discussed was the effect of cleaning shear stress on cleaning effectiveness and time. This should be investigated further.

Buchwald (1973) demonstrated that the energy required for the adhesion forces between the soil and the surface must be smaller than the energy required to transport the soil away, for suitable cleaning to occur. Liu et al (2002) demonstrated similarly, the pulling force required to remove the soil layer.

Liu et al (2006) went on to investigate types of industrial soils and their characteristics. Tomato paste (a soil with cohesive properties similar to those of yeast slurry) was modelled based on shear force of cleaning fluid to clean both baked on and unbaked fouling samples. Experiments were performed by fouling discs with the industrial soil, and implementing these discs in trials on a cleaning rig which was developed as part of ZEAL at the University of Birmingham. Liu demonstrated that increased surface roughness reduced the ability to clean the tomato paste from the surface. The variation in results between the baked and unbaked samples was also shown, with the baked on samples being more difficult to clean.

Guillemot et al (2006) backed up the theory of Lui et al (2006) by demonstrating the effects of shear force by rinsing saccharomyces cerevisiae cells from stainless steel surfaces with varied roughness. Increased shear stress and surface smoothness were both shown to reduce cleaning times.

These pieces of work identify the importance of cleaning parameter impact on the output quality of the clean, and also highlight the necessity to control these cleaning parameters at their optimal settings. There has also been much useful information discussed concerning brewery cleaning which will be useful to draw upon.

2.2.3 Key Factors to Consider for Cleaning

Palmowski et al (2005) performed a review of the current technology in the food and drink industry. A typical CIP cycle was demonstrated as being much the same as a Heineken CIP cycle, with acid, detergent, and water rinse steps. Goode et al (2013) discussed how cleaning regimes are usually kept confidential within plants, and are tailored to the individual plant requirements. CIP steps are often fixed steps which are determined semi-empirically within the industry. It was identified by Palmowski et al, that there are other chemicals available which may replace the traditional chemicals which are used in detergents (such as those mentioned in the Australian Standards (2001)). This should be considered when reviewing the cleaning chemicals used in place at Heineken.

The key factors identified to be considered for cleaning by Palmowski et al were: cleaning contact times, mass transfer and cleaning chemical transportation, cleaning temperature, turbulence, soil type, chemical type, and chemical concentration. All of these factors have been expanded upon throughout this literature review; however they should be investigated or at least accounted for throughout the EngD project portfolio.

Palmowski et al identified several methods of lowering CIP costs to a business through optimisation and process developments. These methods included: optimisation between the use of CIP recycling systems and single use systems, option cost evaluation based on process parameters and their associated costs, and engagement and training for process operators to improve operational efficiency. Palmowski et al’s key identification was that there was no single answer to optimising the process, and that each plant has different requirements and priorities for process variables.

This will be important to take into consideration when considering optimisation in the EngD project investigations.

2.3 Process Benchmarking

The first stage of the project will be to benchmark the process in order to gain baseline data on consumable consumption and business costs. This will also enable opportunities of high levels of consumption and costs to be identified and prioritised for improvement and optimisation within the scope of the EngD project.

Ahmad and Benson (1999) have carried out substantial work on benchmarking in the process industries. Their work looks into the opportunities of improving process performance and competitiveness of plant by benchmarking against best practice methods throughout the world. The book draws upon their experience of over 200 plants, along with tried and tested measurements and standards which can be utilised. The authors have claimed that their methods will increase production output, reduce stock, and reduce both fixed and variable process costs, with minimal capital expenditure. The book content includes information around:

- How to measure process performance and gather benchmarking data on customer service and warranty, company assets (e.g. overall equipment effectiveness (OEE), production rate, quality rate, technical availability), operational excellence, and people management (e.g. absenteeism, safety).
- How to calculate improvement opportunities from the benchmarked data, by quantifying areas of “hidden plant” where operation is lost through inefficient use of capacity or processing time, managing fixed and variable costs, managing stock turnaround, and calculating performance gaps from these improvement opportunities.

- Measurement of performance by metrics and quality models such as the industrial ISO standards.
- Methods of statistical review of the performance indicators and performance gaps.

Goode (2010) made use of a benchmarking tool which was developed as part of ZEAL. In addition to this, Goode discussed use of knowledge gained from the work of Benson and McCabe (2004), in order to produce a cost benefit analysis of opportunities for process improvement at the Tadcaster Brewery (a Heineken brewery which Goode has performed research on process developments).

The work Goode et al and the ZEAL tool will be used and enhanced for the project. The learning from this can be benchmarked against the results of the Bulmers factory benchmarking due to the similarities in processes. The differences between cider making and brewing using Heineken methods may also be highlighted at this stage. The knowledge from the work of Ahmed and Benson will be useful in enhancing the benchmarking tool to fit the Bulmers process, without losing sight of the benchmarking fundamentals and industrial standards.

2.4 Equipment cleaning

Goode et al (2013) discussed that fouling in the food and drink industry is unavoidable due to the nature of industrial processing. A key area of consideration for CIP in brewing is the challenges faced with the equipment cleaning itself. This can include; the complexity of the geometries of the equipment to be cleaned, the types of cleaning heads which are used when cleaning tanks, and the properties of the cleaning fluid which is used to clean the equipment.

Prosek et al (2005) investigated cleaning a sample of soiled pipes containing bends and valves using ethanol as a detergent. Prosek constructed various designs of stainless steel pipe and filled them with model solution representing Dermal ‘Product A’. The product was rinsed with ethanol and rinsing solutions, then analysed by gas chromatography. It was shown that cleaning difficulty increases with complexity of the pipe, where valves are approximately four times more difficult to clean than pipe bends. The contribution of the impact on cleaning by the valves and bends was shown to be cumulative. A model to predict cleaning around bends and valves was developed as a result of this work, by implementing the use of novel bend and valve factors. Prosek stated that a new model would be required to be developed and tailored for use on any other plant.

Prosek et al also discussed the difficulty of measuring cleaning on an industrial scale due to the nature of plant equipment and that it is not easy to disassemble.

Jensen et al (2007) investigated flow variation in different equipment geometries. Their results indicated that greater flow rates give a greater removal rate. They also demonstrated that there are some areas within a T piece which are not contacted by turbulent flow due to a dead leg effect. These areas are more difficult to clean, and the difficult points within the dead leg are not affected by increasing the flow rate.

Both Prosek and Jensen et al have demonstrated how cleaning can vary with the complexity of equipment geometries. Their work will be important in defining the way the experimental phases of the project are considered, and how these results are scaled from a smaller, less complex arrangement of pipework to a complex industrial scale scenario.

Kohler (2014) investigated the cleaning performance of moving, impinging jets when cleaning Xantham gum, by considering cleaning rate, efficiency, and cost. Kohler demonstrated that increasing jet speed, decreased the width of the path of the cleaning jet, and so the total cleaning surface area per unit of time. This has also been shown by Morrison and Thorpe (2002). The best hydraulic energy performance was shown to have a small nozzle, a low pressure, but a high jet speed. The fastest cleaning time has a high jet speed and nozzle diameter. Kohler discussed that there is no simple optimisation strategy, and that optimisation must be based on priority being given to the preferred performance indicators for the cleaning scenario. The overall outcome showed that some sacrifice must be made for either cleaning time or cost.

Benezech et al (2002) and Tamine (2008) have demonstrated the importance of cleaning with the correct pressures and flow patterns when using dynamic cleaning heads to achieve effective results. Mabrouki et al (2000), Burfoot and Middleton (2009), and Burfoot et al (2009) have all demonstrated that high pressure jets are effective for cleaning biofilms and stubborn soils, however the removal of these soils also has a necessity for detergent cleaning.

Demilly et al (2006) investigated the effects of shear stress for the cleaning removal of yeast cells from stainless steel surfaces. Etched steel was quicker to clean than polished. Demilly et al showed that there was a threshold shear stress, which when exceeded, removed all yeast cells.

This work has shown the importance of the set-up of dynamic cleaning heads and their suitability to stubborn brewery soils and biofilms. It has also demonstrated how cleaning heads are important when developing a cleaning optimisation strategy. This knowledge should be considered when investigating the industrial cleaning equipment on site and developing tailored cleaning optimisation recommendations for Heineken. This will also be required to take into consideration when developing bench scale experimental rigs with results to be used for industrial scale up.

2.5 Cleaning Chemicals

A key area which was identified as an area with significant costs within the CIP benchmarking was the use of cleaning chemicals. In particular, the use of sodium hydroxide as a detergent and the excessive quantities which are degraded and require replacement due to residual carbon dioxide which is present in the fermentation vessels.

Previous work has been performed on the optimisation of both acid and alkali rinses. Bremer et al (2006) has investigated the optimisation of alkali concentration along with flow velocity and cleaning chemical temperature which has demonstrated the importance of the detergent quality on the cleaning end result.

Goode et al (2010) has investigated the removal of yeast cells in a brewing environment with sodium hydroxide as a detergent. Goode et al showed that increasing temperature and cleaning flow rate decreased the cleaning times, as well as ensuring that sufficient detergent was present.

Hikita et al (1976) performed extensive research into the absorption of carbon dioxide into aqueous sodium hydroxide and sodium carbonate and bicarbonate solutions. Hikita's experiments were analysed based on the penetration theory for gas absorption. Hikita showed that there is a two-step reaction which occurs when CO_2 is added to NaOH (shown below).

$$2NaOH_{(aq)} + CO_{2\,(g)} \rightarrow Na_2CO_{3\,(aq)} + H_2O_{(l)}$$

Equation 2: Reaction between sodium hydroxide and carbon dioxide

$$CO_2 + OH^- \leftrightarrow HCO_3^- \qquad (K_1=3.2\times10^7\ l.mol^{-1})$$

Equation 3: Reaction between carbon dioxide and hydroxide ion

$$HCO_3^- + OH^- \leftrightarrow CO_3^{2-} + H_2O \qquad (K_2=3.5\times10^3\ l.mol^{-1})$$

Equation 4: Reaction between bicarbonate and hydroxide ions

Note that equations 3 and 4 are both equilibrium reactions and form part of step 2. Hikita proved that when CO_2 is absorbed into strong NaOH solutions, the chemistry will exhibit the behaviour of an irreversible second order reaction and only equation 2 will be required to model this behaviour. Hikita's experiments which demonstrated this involved NaOH solutions with concentrations between 0.5%w/v and 2.0%w/v.

Mahmoudkhani (2009) researched into the capturing of CO_2 from air by the use of NaOH or KOH. This paper was written from an environmental perspective with a view to develop carbon capture and storage techniques. Mahmoudkhani developed a novel method to remove CO_3^{2-} from OH^- solutions. This was through the investigation of scrubbing techniques. Mahmoudkhani showed practically, that caustisation of Na_2CO_3 to NaOH and $CaCO_3$ was possible to generate lime mud and regenerate NaOH after it had been used to absorb CO_2.

Zeman (2007) investigated capturing CO_2 from the environment by separating it from atmospheric emissions through the scrubbing of ambient air. Zeman looked in further detail at the thermodynamic feasibility of the air capturing process as well as into long term CO_2 storage with $CaCO_3$. This work demonstrated the absorption rate and that it is limited by the available quantity of CO_2 within the system. This work was verified using the previous work done by Hikita et al (1976). An energy balance was performed to estimate the energy consumption in this process and the chemistry can be seen in equations 5 and 6.

$$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O \qquad \Delta H^\circ = -109.4 kJ/mol$$

Equation 5: Reaction between sodium hydroxide and carbon dioxide

$$Na_2CO_3 + Ca(OH)_2 \rightarrow 2NaOH + CaCO_3 \qquad \Delta H^\circ = -5.3 kJ/mol$$

Equation 6: Reaction between sodium carbonate and calcium hydroxide

This literature shows that there is scope for process improvement and optimisation of fermentation vessel CIP in a brewing environment. This will require investigation of the true impact of chemical degradation with sodium bicarbonate as well as the impact of other process variable factors such as temperature and cleaning flow rate. The theory demonstrated by Hikita et al (1976) will be a useful information source in understanding the chemical interactions throughout the CIP process. Mahmoudkhani and Zemen also demonstrated how carbon dioxide can be easily captured from the atmosphere by NaOH. They both also suggest potential methods of recovering the NaOH from the carbonate solutions, which would make an interesting point for future work to uncover further potential savings within a brewery.

2.6 Process Monitoring and Control

Some of the key findings from the ZEAL project work were that measuring and modeling of cleaning could be a valuable technique for process optimisation. Due to this, a review of work around measurements, modeling, and predictive cleaning was performed.

2.6.1 Measuring

Prosek et al (2005) discussed traditional cleaning measurements which typically involved equipment swabbing and taking rinse solution samples, and how this is not necessarily representative of the whole process, only providing offline data of the cleaning results after the clean has taken place.

Responses to this data can only be reactive, and does not enable proactive improvements to be made.

With regards to measuring cleaning, Schluber (1970) and Bode et al (2007) both discussed measuring a continuous cleaning rate of mass per unit area. There are various techniques which can be used for measuring real-time cleaning, but as Ali et al (2014) discussed; a purpose built measurement device is required.

Van Asselt et al (2002) performed a study on the online monitoring of CIP in a dairy evaporator. Van Asselt et al also developed a device for monitoring cleaning efficiency, instead of using potentially, unnecessarily long, fixed cleaning times whilst monitoring the cleaning offline. The investigation was carried out on an industrial dairy process, using conductivity, turbidity, and calcium to analyse the fouling level. Van Asselt et al showed that real-time monitoring of a cleaning process is possible, however; further investigation around the robustness and detection of the fouling is required to ensure system reliability. The device considered offline analysis of fouling removal against online, cleaning parameter measurement.

Results showed that conductivity has proven useful for identifying cleaning phase separation, and providing an indication of chemical concentration. Protein removal by the use of detergent cleaning and mineral removal by the use of acid cleaning was discussed, as well as the impact of temperature, chemical concentration, flow rate, and cleaning time on cleaning efficiency.

Monitoring of fouling removal was shown to provide more relevant information on cleaning efficiency, of which turbidity and calcium were good measurement indicators. Turbidity exhibited sensitivity to foam which impacted on some of the online turbidity readings.

Fickak et al (2011) and Van Asselt et al (2002) have both presented the uses of turbidity and conductivity for monitoring CIP. Van Asselt et al (2002) also demonstrated that non-product such as cleaning detergent and air bubbles may give rise to misleading data.

Van Asselt et al’s work has shown a difference in the benefits between offline and online measurements. This work has also verified that turbidity and conductivity can be suitable measures for real-time CIP monitoring, however care will need to be taken to ensure that misleading data due to non-product is accounted for.

Pereira et al (2008) used controlled nanovibrations to detect clean end point under varying cleaning conditions for shampoo. A mechatronic surface sensor was used to measure the mass of residual shampoo and provide real time cleaning curves. A new monitoring method was developed as a result of this work, based on the effects attachment and detachment of deposits has on the vibration properties of the monitored surface. This system requires that cells are zeroed before use. The experiments were also performed on a small scale.

Pereira’s work has shown another method in which cleaning monitoring can be used for the method of directly predicting cleaning end point. The same principles can be used from Pereira’s work to develop cleaning curves and produce predictive models from other methods of CIP monitoring. This work also emphasises the importance of equipment calibration when working with real-time monitoring equipment.

Wilson (2005) investigated cleaning optimisation based on monitoring and validation of the clean. Wilson developed a model of the factors to be considered when establishing plant cleaning methods, and the importance of linking cleaning effectiveness to the nature of the fouling to develop optimal CIP operating conditions was discussed. Cleaning design requirements and the practicality of achieving these in reality was also highlighted in this work, as well as creating better designs by improving physical understanding of cleaning.

Wilson performed a case study investigation on cleaning whey protein and developed a mechanistic cleaning model of this. Challenges associated with monitoring of cleaning were discussed, such as the uncertainty of the resulting cleanliness of the equipment and how good a representation of the clean the monitoring actually is. Finally, Wilson completed a review of cleaning measurement techniques along with their respective benefits and issues.

Wilson has demonstrated how CIP monitoring may be used for process optimisation, and also verifies the work of Changani et al (1997) by discussing the importance of linking cleaning to fouling conditions.

This work highlights the risk in model predictive cleaning where there is a remaining uncertainty of the final cleaning result, as well as of the modelling representation of the overall clean. This information must all be taken into account when investigating model predictive cleaning throughout the project.

Discussions throughout the ZEAL consortium identified the possibility of using particle count as a method of monitoring clean. A company called “Particle Measuring Technologies” (PMT) became involved and were able to provide access to a state of the art, online particle counter. Xu (2013) and Verhaverbeke and Pagliaro (2000) have both demonstrated how particle count has been a useful measuring technology for air cleaning and cleaning in the semiconductor device manufacturing respectively. Hendricks (2006) has shown how particle count has previously been used to measure the quantity of clay particles present in water in the US offline for quality purposes.

This knowledge, combined with the state of the art equipment from (PMT) gives rise to an exciting opportunity to monitor real-time particle count for CIP measurement purposes.

Clauberg and Marciniak (2009) performed work on offline particle count, and have shown that different size particles scatter light differently to give variation in readings from the measuring device. This is important to take into account due to the variable nature of the fouling removal which will be encountered in a CIP process.

2.6.2 Modelling

Leilievre et al (2002) modelled the removal kinetics of Bacillus spores in CIP. This was carried out to establish the criteria which affect cleaning performances of unheated parts of a plant with bacteria soiling. A small scale pilot plant was used with test pipe dimensions of 15cm length and 2.3cm internal diameter. The test pipes were cleaned in series. Sodium hydroxide was shown to induce an adhesion strength decrease in spores. Data modelling of this investigation was not possible due to variability of the results and uncertainty that the equipment was clean once the CIP was completed.

Leilievre has demonstrated that modelling can be used to visualise removal kinetics of CIP on a pilot plant using varying concentrations of sodium hydroxide detergent concentrations. This proves that a CIP model can be produced, but emphasises the uncertainty which can arise in the results without effective measurement.

Cole et al (2010) experimented in detail with toothpaste on the cleaning rig at Birmingham University, and on the pilot scale cleaning plant at the University. Both the rig and the plant were outcomes of the ZEAL project. Cole showed that turbidity and conductivity were suitable online measurements to provide information about the level of cleanliness of the plant. Cole also demonstrated that toothpaste cleaning happened in two stages; core removal of the soil, and film removal from the surface of the pipes. Variation of the length of fouled pipe did not show any significant variation in cleaning time which illustrated that pipe length had no impact on the cleaning capabilities of the system as there was uniform fouling removal throughout. Shear stress was identified to have a strong correlation with cleaning, as did Reynolds number of the cleaning fluid with a derived, dimensionless, cleaning time.

Cole et al's work was part of the ZEAL consortium, and was completed on the same pilot plant which will be utilized for this EngD project. This makes Cole's experimental techniques extremely valuable for the development of this project. Cole also uses turbidity and conductivity to model cleaning, as does Fickak et al (2011) and Van Asselt et al (2002). The demonstration of the phases of cleaning, and the effects of varying pipe length will be helpful in understanding the cleaning process involved in this project. The work on relating cleaning shear stress and cleaning time verifies the theories of Guillemot et al (2006), Liu et al (2006), and Demilly et al (2006). Shear stress is an important investigation factor to consider in this project.

2.6.3 Predicting

Yang et al (2008) underlined the importance of the use of forecasting models for CIP optimisation, and that data captured throughout real-time monitoring could be utilised to predict the end point of cleaning processes, without over or under cleaning the equipment.

This is a key statement which justifies the ZEAL guided direction of an EngD project down the predictive cleaning model route.

Yu et al (2010) developed a data fusion algorithm for the tracking of an industrial fermentation process using process control variables and NIR spectroscopic data to predict offline glucose concentration measurements as part of ZEAL. Yu considered two methods, using sequential modelling and weighted multivariate calibration. Partial least squares (PLS) calibration models were produced for the spectroscopy data and process data. Optimisation based on weightings of models and weightings of individual variables were considered for minimising the root mean square error of cross validation (RMSECV). The sequential model provided a 50% improvement from the spectroscopic process data, whilst the weighting of variables to optimise and develop the algorithm provided further improvement again.

Yu’s work has demonstrated the possibility of a real-time prediction model on an industrial bio-process to predict off-line measurements of product. This shows that the statistical techniques used by Yu can be applied to a process and manipulated to benefit and optimise a process effectively. This will be important to consider techniques such as this when considering the predictive model development for the EngD.

Durr (2002) developed a mathematical model of cleaning kinetics and performed an appraisal of a modeling approach on the cleaning kinetics of a dairy production heat exchanger by Durr and Grabhoff (1999). Durr discussed a life time distribution of soil removal against the model, including the different phases of model removal steps, and demonstrated how it can be fitted to the model. Durr's work provides a mathematical reference for studying, analysing, and improving a CIP process.

The application of statistical analysis to a specific CIP process shows that other statistical modeling work which has been reviewed has the potential to be transferred to a CIP process. This is a positive indication of the development of the EngD project work.

Wilson (2014) investigated the cleaning of food layers of dried Xanthan gum with ZnS particles. A mathematical model was developed for cleaning, by moving vertical jets of cleaning fluid. This was complimentary to the work of Kohler et al (2014) on cleaning the same soil, and gave the same results. These are discussed in more detail in the equipment cleaning subsection of the literature review, based on the information provided on cleaning with dynamic heads. Predictions of observed cleaning were able to be made based upon the model. This also enabled the effects of the process parameters to be captured with quantitative descriptions of the behaviours observed on the moving jets.

Wilson's work demonstrates the possibility of using a predictive model on a more complex CIP scenario, using dynamic cleaning heads. Heineken also uses dynamic cleaning heads throughout the business. This work will provide further useful information for the EngD project, and further understanding has been provided on relevant CIP process behaviours.

2.7 Conclusions

The following conclusions and implications for the scope of the research within this EngD have been drawn from this literature review:

- Cleaning and Fouling Classification
    - CIP optimisation is essential to reduce business costs, and for the improvement of product quality, production efficiency, environmental impact and sustainability of the industry

    - Fryer and Asteriadou (2009)’s cleaning map will be useful for understanding cleaning requirements for Heineken and the transferability of cleaning across different soils
    - Goode has provided much brewery cleaning knowledge which will be useful for the understanding the cleaning theory for the project development, and for representative fouling selection for the experimental stages of the project.
    - Rough surfaces are more difficult to clean than smooth surfaces
    - Fouling which has been dried or baked on is more difficult to clean
    - Increasing cleaning shear stress, temperature (up to 50^{o}C) reduces cleaning time
    - Cleaning contact times, mass transfer and cleaning chemical transportation, cleaning temperature, turbulence, soil type, chemical type, and chemical concentration should all be considered as parameters which could impact upon the overall cleaning effectiveness throughout the project
- Process Benchmarking
    - Benchmarking techniques have successfully been utilised and implemented within Heineken and other closely related industries
    - A benchmarking tool was developed as a result of the ZEAL consortium work
    - These techniques and tool can be applied and developed to enhance them for use within this EngD project
- Equipment Cleaning
    - Higher cleaning flow rates can reduce cleaning time
    - Turbulent flow is necessary for effective cleaning, but can be inconsistent throughout complex geometries such as t pieces
    - Scaling modelling from small scale to industrial scale will be a challenge
    - Dynamic jet cleaning optimisation is not simple. Factors to be considered include; flow pressure, flow speed, nozzle diameter

    - Jet cleaning optimisation will affect cleaning jet impact surface area and cleaning time
    - A sacrifice for cleaning time or cost will have to be made throughout jet cleaning optimisation
    - Replication of cleaning head behaviours will be required for completion of project experimental work
- Cleaning Chemicals
    - Alternative cleaning detergents can be considered from sodium hydroxide
    - Detergent quality is important for the end cleaning result
    - Sufficient detergent concentration is essential for ensuring effective cleaning of brewery soils
    - The two step theory on carbonate formation is important to understand NaOH degradation.
    - Further understanding of NaOH degradation will be required through investigation
    - There is a potential to investigate recovery methods of NaOH after degradation with CO_2.
- Process Monitoring and Control
    - Offline measurements enable an operator to react to poor cleaning, but not be proactive and avoid a completely ineffective clean from occurring.
    - Benefits of real-time online monitoring of CIP have been shown
    - Turbidity and conductivity have been previously used as real-time measurement systems for CIP.
    - Care should be taken when monitoring a process to ensure that misleading data is not affecting the results, and that measuring devices are working effectively.
    - Predictive model development is possible from alternative real-time measurement devices with the correct application of statistical modelling theory
    - Calibration of instruments for real-time monitoring is essential

- There is a risk of uncertainty in model predictive cleaning due to the inability to visualise the cleaning until it is completed
- Particle counting is a potentially suitable real-time cleaning measurement system
- Particle count measurement is size dependant and particle sizes in CIP will be variable which may impact on reliability
- Mathematical modelling is possible to view cleaning kinetics
- Soil removal stages in CIP have been identified
- The ZEAL pilot plant and experimental techniques from Cole will be useful for the project
- Linear pipe length variation shows no impact on cleaning consistency throughout
- Transferability and application of statistical modelling techniques is possible for developing predictive models in different types of industry

Chapter 3. Benchmarking

3.1 Problem Statement

Plant cleaning costs are not typically monitored in any great detail with respect to specific equipment and process areas. Benchmarking of a process in detail may provide additional information about individual clean types and their associated costs. This may then give rise to the identification of potential areas where modifications could be put in place to optimise the process and provide further cost benefits. The main focus of this chapter is to discuss and determine the CIP costs associated with the fermentation area of Bulmers, where the majority of the project has been completed.

3.2 The Benchmarking Tool

The benchmarking tool was an outcome from the ZEAL consortium. The tool was developed for the purpose of calculating base CIP costs for a variety of bioprocessing companies. The tool takes into account many aspects of the running and cleaning of a plant, including; costs, flow rates, utility consumption, effluent production, operational procedures, frequencies of cleans, and management views and techniques. This makes the tool ideal for use within this project for considering the base CIP costs and also comparing them to estimated cost benefits from later proposals as outcomes of the project. The tool has been used previously on the John Smith's brewery at Tadcaster by Goode (2010) as part of the ZEAL project work.

The results from this project and the tool have been used throughout other projects within this EngD portfolio, for the estimation of potential cost benefits on implementation.

3.3 Plant Cleaning Description

The Bulmers site is split into two main segments; cider production and packaging. Cider production is also split into two sections; fermentation and process. Due to the size of the site and the areas of investigation of the project, the benchmarking work will be focussed on the fermentation area of the cider production side.

The fermentation area is cleaned with one CIP set. This set consists of a water tank, a dilute sodium hydroxide tank, and a dilute nitric acid tank. The three tanks are all connected to a CIP supply pump which pumps the cleaning fluid through a steam heat exchanger, to the equipment which is being cleaned, and is then returned and recirculated through its respective dilute tank (except in the case of the water, which all goes directly to drain). This is shown in figure 4.

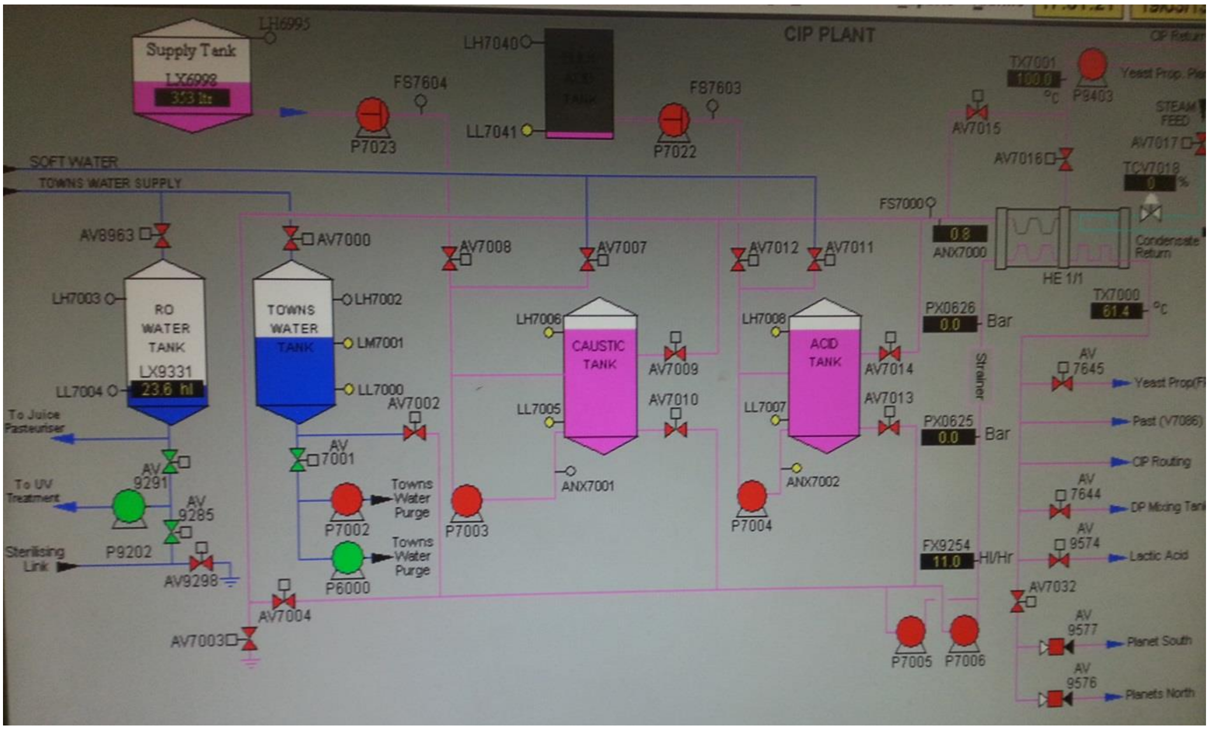


Figure 4: Print Out of CIP SCADA Mimic

The equipment within the fermentation area of the site consists of large fermentation and maturation vessels, mains pipes of various lengths and diameters, a pasteuriser, and some smaller vessels (e.g. yeast pitching vessel, juice buffer tank, etc.).

3.4 Project Deliverables

Based on the plant cleaning description and knowledge of its operation, the key deliverables of this project were identified:

- Identify and source effective measurement procedures to retrieve detailed CIP process information.
- Provide costs of CIP within the fermentation and process areas of Bulmers.
- Provide detailed cost breakdowns of CIP for individual equipment types within these areas.
- Identify CIP areas of non-value added time for potential savings proposals.
- Use benchmarked costs to provide estimated cost benefits for proposals for projects completed to be throughout the EngD.

3.5 Methodology

The benchmarking tool has been developed as a Microsoft Excel spreadsheet. For the fermentation area, there are challenges involved in obtaining the necessary data and information to populate the tool.

There are no fixed flow meters on site, so a non-intrusive flow meter was required to obtain flow data for the different types of cleans. Data on the parameters and frequencies of clean types was required to be extracted from the SCADA control system and SAP database on site as well as additional local knowledge and hand written records from the operators. It was essential to monitor cleans carefully, to understand the process well and take note of changes and intermediate steps which may not be recorded e.g. when the process is being recycled or drained.

It was necessary to liaise with many people within and outside the company to gather information on management views, business operations, and external resources such as chemical costs.

There are no fixed methods of measuring steam or electricity being used, so the kilowatt ratings of the pumps have been collected, and the electricity used was calculated based upon measured operating times of the equipment per clean. Steam calculations were required as there is no direct measure of steam usage on the CIP set. These were performed based upon the heat transfer requirements to increase the temperature of the cleaning liquid by a known quantity over a known time. The methodology used for these calculations can be seen in appendix 3.

3.6 Results and Discussion

The flow rates for the cleaning of the different equipment types, times, thermal energy requirements and electrical energy requirements were determined. Flow diagrams for the main types of cleans can be seen in appendix 4. The cleaning times were put directly into the benchmarking tool along with the electrical energy requirements. The table for thermal energy requirements may be seen in appendix 5. The following flow diagram (figure 5) is an example of a large fermentation vessel clean which can be used to describe what is happening in the clean. The diagram may be seen on a larger scale in appendix 4.

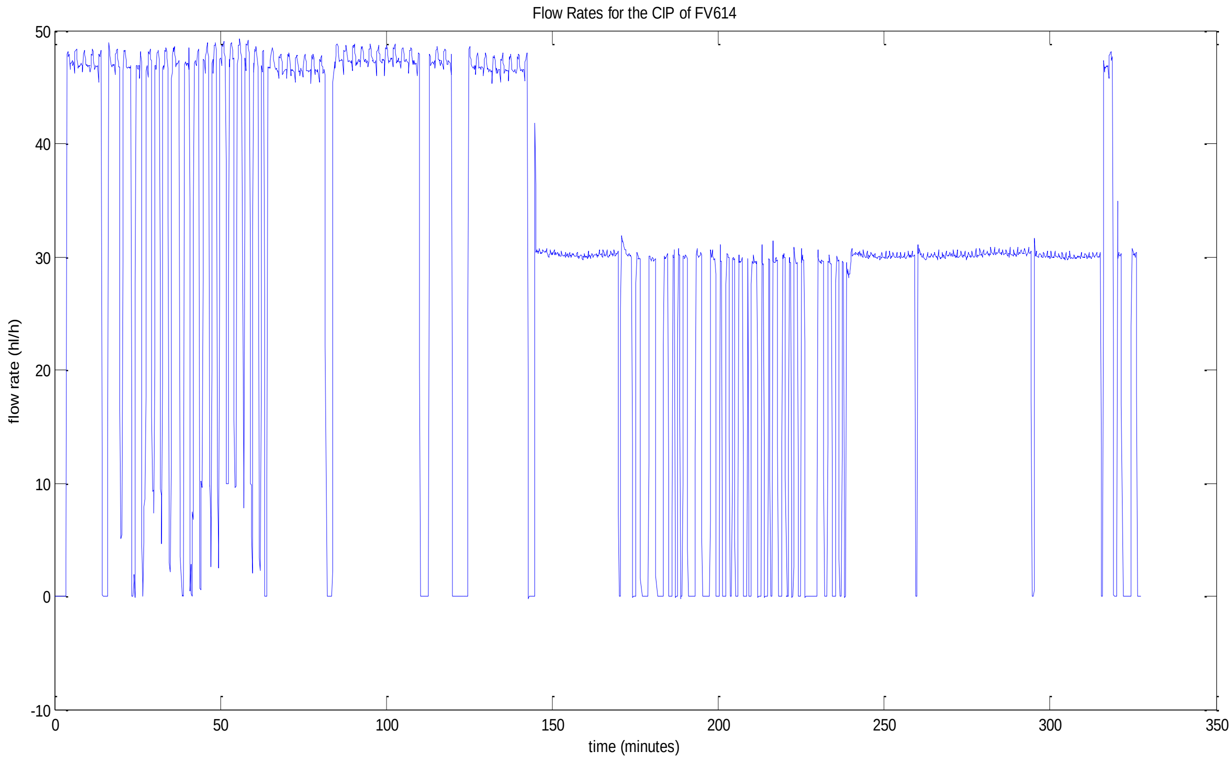


Figure 5: Flow Diagram for CIP of a Large Fermentation Vessel

On commencing a clean from the SCADA control interface, there are several minutes of no flow. This is due to the dilute caustic and acid tanks being made up to the required conductivity specifications to clean effectively. The fermentation vessel clean is then divided into two main sections; the first cleaning the pipework around the side racking and cooling loop of the vessel, and the second being the vessel itself. See figure 6.

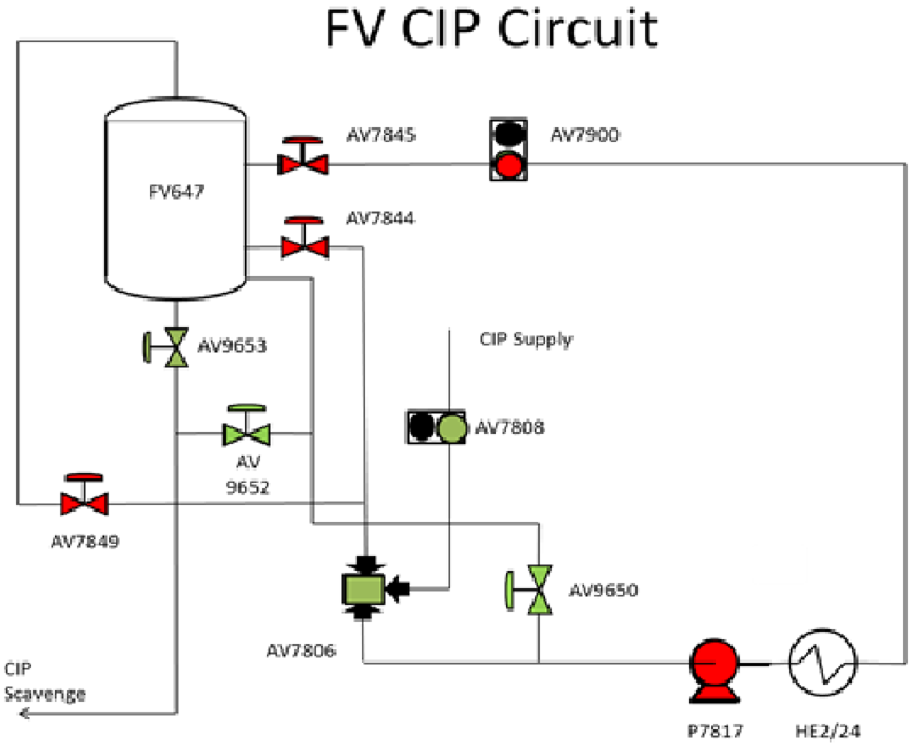


Figure 6: Schematic of FV647 Cleaning Routing

The flow rate of the pipework is much higher than that of the vessel due to the pressure drop in pumping the fluid up to a height of 20m when cleaning the tank itself, and also from the back pressure of cleaning through the cleaning head.

The clean begins with a 10 minute water rinse being sent directly to drain via the vessel mains only. This is followed by a 30 minute caustic rinse by the mains only, which is recirculated via the dilute caustic tank. Throughout this step, the clean goes into hold multiple times (due to conductivity levels dropping below specification). These hold times may be seen on the cleaning diagram in figure 5 at each point the flow rate drops to (or near) 0 $hl.h^{-1}$

between times of 10 minutes and 60 minutes. The total time spent in hold per clean may be totalled and shown as a percentage of cleaning down time.

The vessel mains pipework cleans then follows with another water rinse, an acid rinse then a final water rinse before going on to the same cleaning sequence but for the vessel clean itself. See figure 5 at approximately 145 minutes, where the cleaning flow rate changes from approximately $47m^3.h^{-1}$ to $30m^3.h^{-1}$. Once this vessel sequence is completed, there is a final 3 minute water rinse for both the mains and the vessel respectively. Each water rinse step goes completely to drain and the acid/caustic steps are all recirculated via their respective tanks. Changeover times between the cleaning steps may be seen at the points where the flow rate has dropped to zero.

The benchmarking tool spread sheet with populated information and results may be seen in electronic appendix 1. With respect to the chemical consumption, the chemicals have been divided into the bulk acid, bulk caustic, and softened well water which is used to make up the dilute solutions. The quantity used per clean has then been calculated as the total cost to make up a tank, divided by the average number of cleans which has been used per tank. A titration is performed every morning on samples from the caustic and acid tanks to ensure that it is on specification and aligns with the information provided by the conductivity probe. The dilute caustic tank is typically emptied and refilled daily. From further experimentation in chapter 4 it has been shown that after one fermentation vessel clean, the dilute caustic will be off specification. The benchmarking costs have been based on the dilute tank being dropped daily on the CIP system and procedures in place at the time of the benchmarking project (Autumn 2012).

The results show that the fermentation area typically has approximately 53 cleans per week, and 2760 per year. Downtime costs have not been included at this stage, as the plant is not currently running at full capacity. The purpose of this investigation was to observe the plant cleaning in the fermentation area along with its associated costs.

Table 2 shows the weekly and annual costs spent on utilities for the whole fermentation area.

Table 2: Utility Costs for Fermentation Area

	Weekly (£)	Annual (£)
Water	750	39,000
Electricity	54	2,800
Thermal Energy	33	1,740
Total	840	43,700

The water costs are based solely on towns water. The softened well water costs are included in the chemical costs as it is only used for making up of the dilute chemical tanks. The electricity and thermal energy costs are low as the two supply pumps are low kW ratings. There has been a new boiler house implemented on site which is running at 90% efficiency, resulting in improved thermal energy costs. Thermal energy costs could be reduced further by lowering the cleaning temperatures of the heated cleans and lagging the outdoor pipework, particularly through the winter months where heat losses would be greater due to the lower external temperatures.

Water rinses are all sent directly to drain. Final rinse water could be collected and used as pre rinse water for the next clean. This would save approximately one third of the water costs (£13,000 per annum). If the fermentation area building capacity will allow, then this would be a recommendation for implementation.

The net present value (NPV) over a ten year period has been calculated to show the cost benefit of making the change. The calculation is based on the assumption that the tank will cost £48,400 including installation, see appendix 6 for the tank cost calculation. Once the tank has been installed, it is also assumed that there will be no operational costs (other than those covered by general site maintenance). The NPV is £34,000 with a payback time of approximately 5 years. See appendix 7 for the NPV calculation. The quota used by Heineken typically requires that the payback time will be less than one year. As this is greater than one year, it will not be recommended to be put forward with immediate effect; however it will be a recommendation to be included when a new CIP set is installed.

The total cleaning chemical costs for the fermentation area are £3,200 per week and £166,500 per annum. Table 3 shows how these costs are divided per chemical.

Table 3: Chemical Costs for Fermentation Area

Chemical	% total cost	Chemical Cost per Week (£)	Chemical Cost per Annum (£)
Bulk Caustic	63.4	2,020	104,900
Bulk Acid	33.8	1,080	56,300
Soft Water	2.3	74	3,830

Softened water is a small percentage of the total chemical costs, with bulk caustic being the greatest at around £105,000 per year. Chapter 4 discusses an investigation which has been completed to reduce this bulk caustic cost by approximately 56% which will be a saving of around £59,000 per year. This investigation was performed as a direct outcome from the results of the benchmarking work, due to the identified costs and down times associated with the caustic cleaning steps of fermentation vessels.

Effluent treatment works out to be approximately £3 per week, and £157 per annum. This is a small cost, but would be reduced by reducing the water and chemical consumption. If the recommendation to recycle the final rinse water were put in place, this would reduce the volume of water to effluent by 33%.

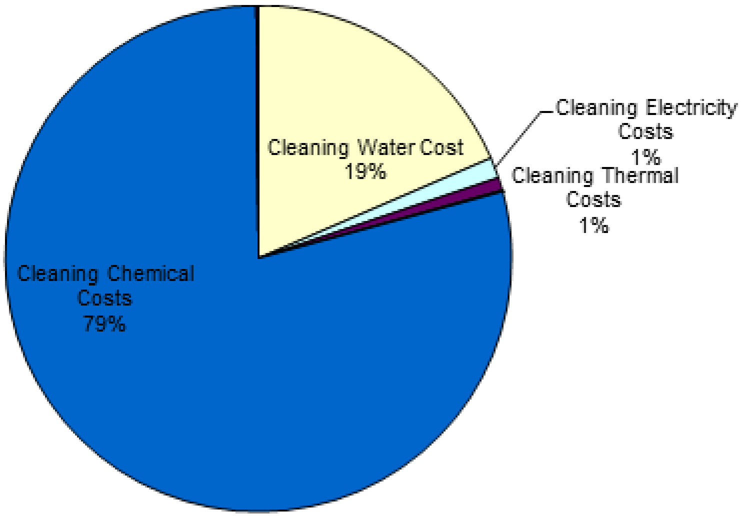


Figure 7: Pie Chart of Cost Distribution for Fermentation Area

Figure 7 has been taken from the results of the tool in electronic appendix 1. From this, it can be seen that 79% of cleaning costs are based on chemical consumption and 19% from water consumption based on an average clean. Individual breakdowns of cleans per equipment type may be seen directly in the tool (electronic appendix 1). These are the two key areas which cost reduction should be focussed on to give the greatest impact. The total cleaning cost per week is £4050 and £210,500 per annum, based on utilities, water and chemical consumption alone. If all of the recommendations mentioned were implemented (recycling of water and reduction of caustic usage), then this cost could be reduced by around £72,000 per year. Fermentation vessels are the greatest cost per unit clean at approximately £100 each. This is based on the assumptions given in this investigation (where the caustic tank is replaced with fresh caustic daily and used for all of the cleans in a given day). These results emphasise that cost reductions of the fermentation vessel cleans and the use of cleaning chemicals should be a key area for investigation.

3.7 Conclusions

The following conclusions have been drawn from this investigation:

- Fermentation vessel cleaning hold times typically add 25% of non-value added time on to total cleaning times.
- The fermentation area has approximately 2760 cleans per year.
- Water consumption is the greatest factor of the total utility consumption.
- Water consumption and effluent costs could be reduced by collecting the final rinse water and using that for the pre rinse water of the next clean. This is a key recommendation to be included in the implementation of a new CIP set on site.
- Bulk caustic accounts for approximately 63% of cleaning chemical costs.
- Water and chemical costs account for 98% of the total cleaning costs and should be key areas for investigation/improvement.
- Fermentation vessels are the most expensive clean types in the fermentation area at Bulmers, costing £100 per clean and should be a key area for investigation.
- The recommendations as a result of this investigation will result in an estimated saving of £72,000 per year, subject to implementation.

3.8 Recommendations

The following recommendations have been drawn from this investigation:

- Complete benchmarking around the rest of the Bulmers site and other Heineken sites to identify key areas for investigation and cost reduction for the scope of future sight project work.
- Collect the final rinse water to use as the pre rinse water of the following clean when installing a new CIP set.
- Implement the suggested method of bulk caustic use reduction from chapter 4 to save money on chemical cleaning and fermentation vessel cleaning which are the largest identified costs from this project.
- Investigate further methods of reducing non value added cleaning time throughout the whole process to improve production efficiency. These

will be useful in the implementation of a new CIP set to improve the overall CIP process efficiency. Some examples of reducing this time are to use larger pipework for draining of vessels to enable this to be completed more quickly without the use of further resources and costs.

Chapter 4. Optimisation of Carbonate in Caustic

4.1 Problem Statement

Effective equipment cleaning in the brewing industry is essential to achieve the correct end product quality, with respect to taste, appearance, and health and safety legislation. Factors which are important when considering cleaning are production efficiency, cost, environmental impact and cleaning efficacy.

Bulmers uses sodium hydroxide as a cleaning detergent throughout their CIP process. Sodium hydroxide is known to effectively clean brewery/cider soils, however; sodium hydroxide reacts readily with carbon dioxide to form sodium carbonate which results in chemical degradation and reduced cleaning efficacy. Fermentation vessels are typically full of carbon dioxide and the quantitative impact on the overall cleaning due to this is uncertain.

The level of sodium hydroxide and sodium carbonate is measured online in the process by using a conductivity probe to estimate the concentrations present and is used to determine whether immediate dosing with more sodium hydroxide is required. It is uncertain how accurate this method is at predicting the state of the detergent and just how close to the pre-requisite specifications the detergent concentration actually is.

The business drivers behind this study relate to operational aspects of the CIP policy and its consequences. The current CIP set up and pre-requisite specifications results in frequent replacementof the detergent and multiple occurrences of the process being held due to sodium hydroxide dosing. This costs large amounts of money through chemical usage and down time, and as a consequence, affects production efficiency. Excessive chemical waste increases the environmental impact of chemicals going to drain, as well as increasing water and utility consumption due to increased clean times. Vessels are potentially not being cleaned properly due to ineffective cleaning detergent use, which will also give rise to possible microbial contamination and vessel staining. This is undesirable for a consumable product.

The intention of this project is to determine the impact of sodium carbonate formation on the cleaning efficacy of sodium hydroxide and to investigate if the pre-requisite specifications are suitable. Online indications of the detergent quality will be investigated and any potential optimisation of the current CIP set up determined. Costs benefits of all proposals will also be included.

4.2 Background Knowledge

4.2.1 Chemical Reactions

When glucose is fermented to form alcohol, carbon dioxide is produced as a by-product.

$$C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2$$

Equation 7: Conversion of Glucose to Alcohol and Carbon Dioxide

Once the fermentation vessel has been emptied, residual carbon dioxide which filled the head space of the vessel throughout the fermentation is left in the bottom of the vessel. During the detergent cleaning phase, aqueous sodium hydroxide reacts with this carbon dioxide to form sodium carbonate and water. See equation 8.

$$2NaOH_{(aq)} + CO_{2\,(g)} \rightarrow Na_2CO_{3\,(aq)} + H_2O_{(l)}$$

Equation 8: Absorption of Carbon Dioxide into Sodium Hydroxide

Based on the work of Hikita et al (1976), it is a reasonable assumption to base the experimentation on equation 8.

As carbonate ions are formed, so the quantity of hydroxide ions is reduced, thus reducing the cleaning efficacy. The level of cleaning efficacy reduction is unknown.

4.2.2 Conductivity Measurements

Currently, the concentration of sodium hydroxide and sodium carbonate present in the CIP cleaning detergent is estimated by the use of an online conductivity probe on the CIP return main which delivers cleaning chemicals in recirculation back to the dilute storage tank. When the conductivity drops below 60 mS, the clean is put on hold so that more sodium hydroxide may be added and the conductivity increased to above 60 mS. Sodium carbonate is also conductive, so with high levels of sodium carbonate the conductivity reading may give a “false reading” as to how much sodium hydroxide is present as the system bases the estimation on sodium hydroxide alone. This potentially means that cleans may be continued with under specification levels of sodium hydroxide and over specification levels of sodium carbonate.

4.2.3 Titrations

The operators check the chemical composition of the detergent every 24 hours. The check is performed by titrating a sample from the dilute sodium hydroxide tank against hydrochloric acid and monitoring the volumes of HCl required to change the colour indicators of phenolphthalein and bromothymol blue from purple to colourless and blue to yellow respectively. See appendix 8.

The set method provided by the cleaning company; Johnson Diversey (now Sealed Air) for the operators to follow has been verified as part of this project. This was to ensure that the method was appropriate for the use of testing the concentrations of the samples prepared as part of this investigation as well as for the samples taken on site. See appendices 8 and 9 for the methodology and verification.

4.3 Current CIP Set Up

The fermentation vessels are cleaned with one CIP set. This set consists of a water tank, a dilute sodium hydroxide tank, and a dilute nitric acid tank. The three tanks are all connected to a single CIP supply pump, which pumps the cleaning fluid through a steam heat exchanger to the equipment being cleaned, and then returns and recirculates back through its respective dilute tank (except in the case of the water, which all goes directly to drain). See figure 8.

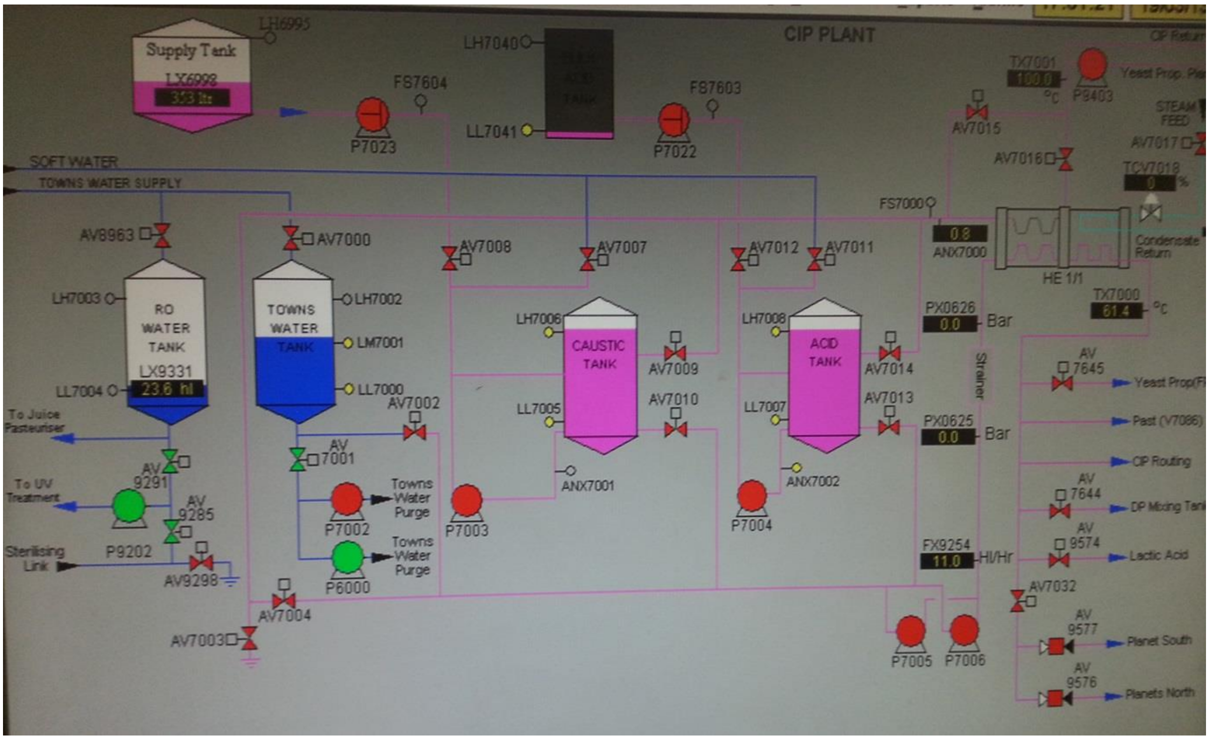


Figure 8: CIP SCADA Mimic

On the CIP return there is a conductivity probe which is used to monitor the detergent/acid concentrations. If the conductivity goes out of the expected range throughout either of these cleaning steps, then the clean will go into hold (the supply pump and measure of time is paused) and the dilute tank will be dosed with concentrated detergent/acid until the conductivity switch on that respective tank shows that the correct conductivity is present. This means that cleaning times are often greater than the specified parameters, due to the additional cumulative amount of time when the CIP system is in hold. The dilute sodium hydroxide tank is emptied and refilled approximately every day, and the dilute nitric acid tank is emptied and refilled approximately once a fortnight.

4.4 Project Objectives

The objectives of the project are:

1. Investigate the maximum quantity of sodium carbonate which may be formed in the sodium hydroxide solution used for cleaning-in-place before the cleaning efficacy is reduced sufficiently that it can no longer clean effectively.
2. Verify these findings using plant data.
3. Compare the actual carbonate formation rate with the theoretical carbonate formation rate using plant data, and also quantify the benefits (if any) of using a fan for carbon dioxide removal during cleaning.
4. Consider offline measurements for the potential application of a predictive model for determining the end point of the use of the sodium hydroxide cleaning detergent based on the determined specifications.
5. To calculate baseline cleaning-in-place costs and estimate the potential savings of the proposed solutions.
6. To recommend the optimal cleaning method for cider and beer fermentation vessels based on cleaning times, chemical consumption, environmental impact and cost.

4.5 Methodology

4.5.1 Fouling

In order to investigate cleaning parameters, it was necessary to replicate realistic conditions of fouled brewery equipment. When attempting fouling methods, it was difficult to replicate cider fouling which required no more than a cold water clean to become visually clean. This would not have been appropriate for the purpose of this investigation. Beer bottoms from John Smith’s brewery were used to provide a worst case scenario (See composition in appendix 2). Cold water alone would not clean beer bottoms, but a sodium hydroxide solution of the current site cleaning pre-requisite concentration was sufficient. Beer bottoms consists of the bulk of the proteins and some viable yeast slurry from beer fermentations, with filter powder (protein complex) to aid in the filtration process before the beer product goes on to the blending stage. The purpose of this investigation is solely to investigate the carbonate formation and measurement of the cleaning chemicals. The point of the worst case scenario fouling selection will ensure that as long as the caustic can clean the brewery soils, then the cleaning can be optimised for the caustic usage.

Stainless steel 316 coupons of 5cm squares, cut from the same sheet of metal, were fouled by placing 5g of post filtered beer bottoms onto the coupons and spreading evenly across the surface area. The beer bottoms were left to completely dry on naturally over a period of at least two days. See figure 9.

Figure 9: 5cmx5cm Fouled Coupon for Use on Bench Scale Cleaning Rig to Replicate Brewery Cleaning Scenario

4.5.2 Equipment

In addition to fouled equipment, it was also essential to develop bench top experimental equipment to perform trials in a safe and controlled environment before scaling up the experiments to an industrial scale.

A bench scale cleaning rig was designed to perform the experiments for this investigation on a realistic simulation of the actual fermentation CIP process. The rig consisted of a cuboidal, plastic tank with a capacity of approximately 20l. Within the tank was a stainless steel hook, approximately halfway up one of the tank walls. A 4l pool of cleaning solution (made up manually of NaOH, Na_2CO_3, and towns water) sits in the bottom of the tank, and a continuous sample is drawn from the bottom of the tank and recirculated back into the top of the tank through peristaltic tubing and a hose nozzle which directed the solution towards the hook in the tank. The manually made up cleaning solutions may later be compared with the supplier made caustic when performing the site verification for any cleaning differences. Fouled coupons may be suspended on this hook for each experimental run to be cleaned by the varied solutions. The recirculation loop consists of a small centrifugal pump which pumps the cleaning solution through a three way valve and another line of peristaltic tubing which is directed back into the pool of cleaning solution in the bottom of the tank. This valve acts as a by-pass valve to enable control of the flow rate of the cleaning fluid through the hose nozzle. See figure 10.

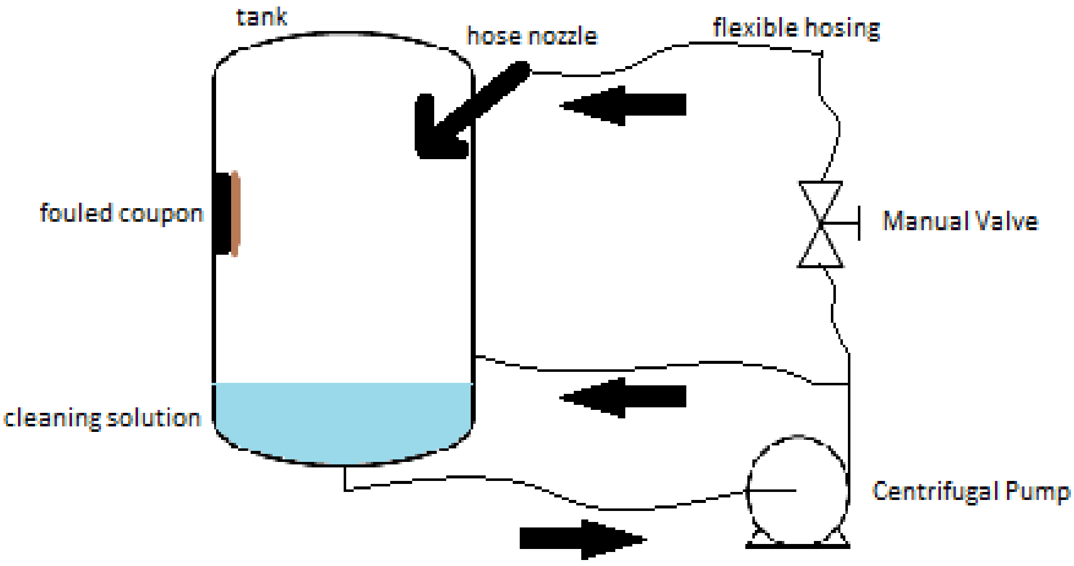


Figure 10: Bench Scale Cleaning Rig

The hose nozzle is sprayed directly onto the top of the fouled coupon to form a waterfall type effect over the coupon with a shear force of at least 3 mPa (the same as that in a large scale fermenter on site (Jensen, 2012)). The design calculations for the mini rig to ensure that this specification could be met can be seen in appendix 10. The materials of the rig are Perspex, peristaltic tubing, copper, and stainless steel. All of which are sufficient for the use of dilute caustic (UK Copper Board, 2012).There is a bypass valve which enables the flow to the nozzle to be varied. The top of the tank is made of clear Perspex, which enables the operator to see the coupon at all times and monitor the stage at which the coupon becomes visually clean.

4.5.3 Experimental Design

Solutions of sodium hydroxide and sodium carbonate at different strengths and mixtures were made up and used to clean consistently fouled beer bottom stained coupons. The sodium hydroxide strengths ranged between 0 and 2% w/v as the site currently uses 1.5%. Sodium carbonate strengths vary between 0 and 12% w/v (Current site limit is 4%). There were a total of 90 experimental runs; consisting of triplicates of each of the combinations seen in table 4. This makes up a full factorial experimental design.

Table 4: Experimental Design for Carbonate in Caustic Work

Flow Rate ($ml.s^{-1}$)	NaOH (% w/v)	Na_2CO_3 (% w/v)
50	0	0
50	0	2
50	0	4
50	0	8
50	0	12
50	1	0
50	1	2
50	1	4
50	1	8
50	1	12
50	2	0
50	2	2
50	2	4
50	2	8
50	2	12
100	0	0
100	0	2
100	0	4
100	0	8
100	0	12
100	1	0
100	1	2
100	1	4
100	1	8
100	1	12
100	2	0
100	2	2
100	2	4
100	2	8
100	2	12

4.5.4 Experimental Procedures

The standard operating procedure for the bench scale cleaning rig may be seen in appendix 11. After each experimental run the fouled coupon and cleaning solution is replaced, and the equipment rinsed through with towns water before adding the next cleaning solution. The clean must be monitored at all times throughout each experimental run using the Perspex lid at the top of the rig. The cleaning times may then be recorded using a stopwatch to measure the point that the coupon appears to be visually clean. Visually clean has been selected as the acceptable clean level as in a real CIP on site, there will be a sanitation step after the detergent step to ensure that the equipment is cleaned for microbial growth to an acceptable standard and water rinse stages after these steps.

For each experimental run, samples of the cleaning solution were taken before and after the experiment. This was to perform titrations to verify that the correct cleaning solution had been made up and to take offline measurements of each sample of the pH, conductivity and turbidity for the online measurements section of this investigation.

4.5.5 Health and Safety

The risk assessment for the experiments and use of the mini rig may be seen in appendix 12. When performing the experiments, it was mandatory to wear the correct PPE; this included safety goggles, gloves, and a dust mask when weighing out the solid forms of sodium carbonate and sodium hydroxide.

4.5.6 Stakeholder Overview

If benefits from this study were sufficient, on completion of the project it would be desirable to arrange for implementation of the outcomes and have the greatest impact. In order to do this a stakeholder overview was developed to identify the key stakeholders within Heineken UK and Heineken NV for the global outreach. Once completed, the outcomes were be presented and distributed to these key stakeholders. See figure 11.

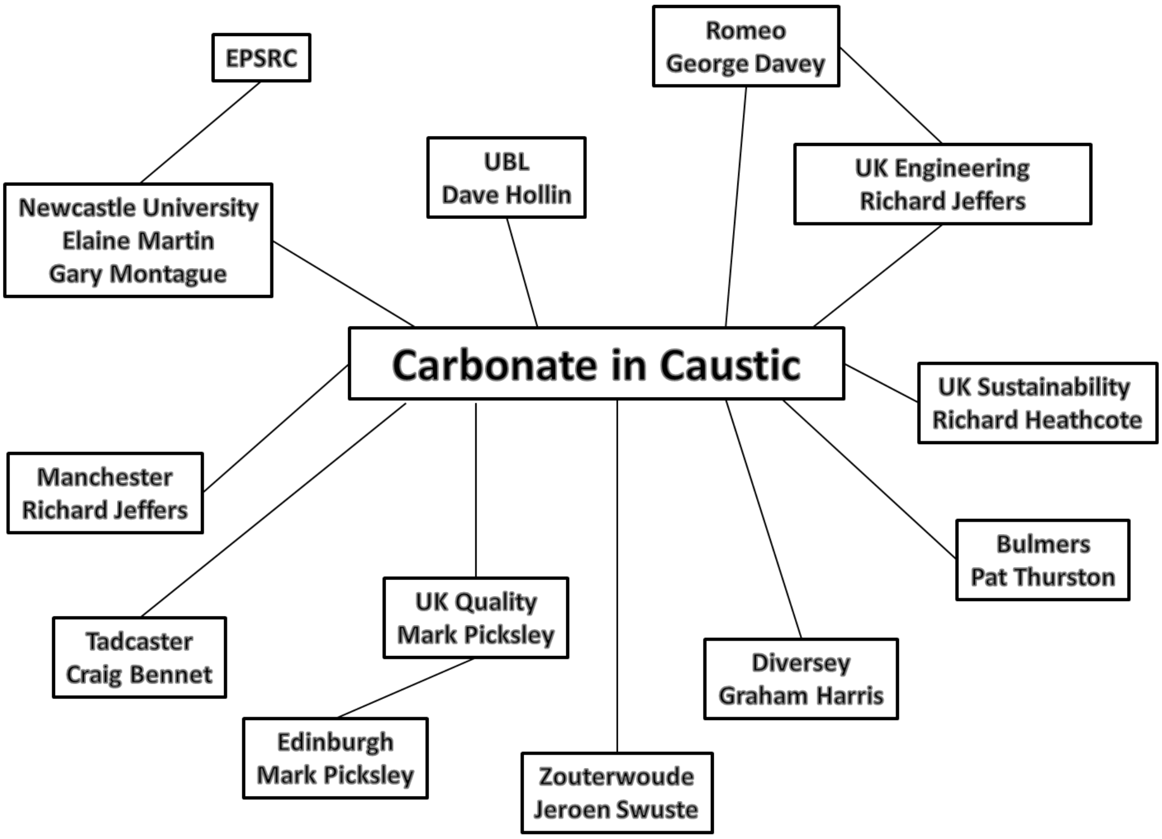


Figure 11: Stakeholder Overview for Carbonate in Caustic Work

4.6 Results and Discussion

4.6.1 Maximum Carbonate Value

The overall experimental results can be seen in the spread sheet in electronic appendix 2. To determine the maximum carbonate value which could be used it was necessary to consider the concentrations of sodium hydroxide and sodium carbonate, the cleaning flow rate, and the time taken for the coupon to reach a visually clean standard. No coupon was cleaned for longer than 10 minutes, as no area within the fermentation vessel would receive direct spray coverage for that period within a clean. Therefore, if the coupon did not appear clean within 10 minutes, it was assumed that this solution and flow rate did not clean.

As there were a large quantity of different combinations with the three input variables, a main effects plot of the individual variables and how cleaning responds to their variation individually is shown in figure 12.

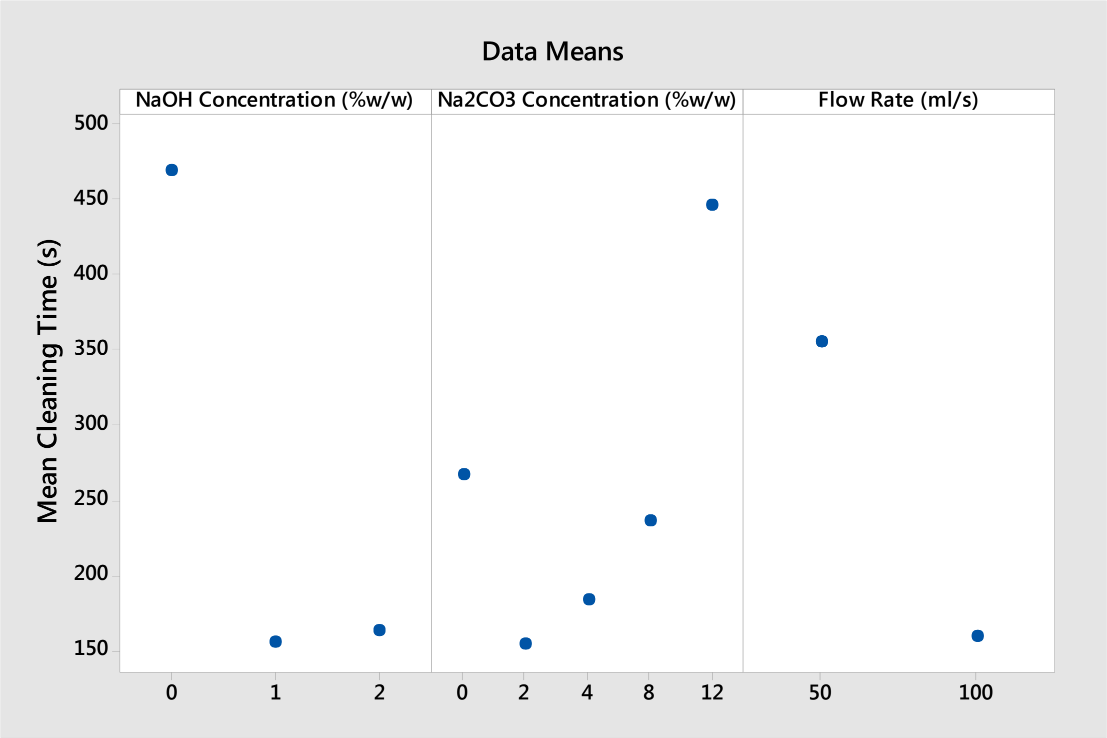


Figure 12: Main Effects Plot for Caustic Results Showing the Individual Effects of Flow, NaOH Concentration, and Na_2CO_3 Concentration on Mean Cleaning Time

It is possible to see from figure 12 that cleaning time is reduced with a higher flow rate, sodium hydroxide up to 1% w/v reduces cleaning time, but after this does not reduce time any further, and sodium carbonate appears to improve cleaning up to 2% w/v, but gradually increases cleaning time up to 8%, and the increases cleaning time rapidly between 8 and 12 %.

Increasing flow rate improved cleaning as would be expected due to the increased shear effect of a greater force being applied to the fouling by the liquid. Higher sodium hydroxide concentration improved cleaning also as would be expected, due to the increasing of chemical strength initially. This does not make an improvement with concentrations of greater than 1% w/v. This is verified with results from cleaning trials performed by Johnson Diversey (Edwards, 2011) on the detergency and effectiveness of sodium hydroxide with varying concentration. The graph of their result on this may be seen in figure 63 in appendix 13. This is due to the pH influence of sodium hydroxide with logarithmic behaviours, meaning there is an initial rapid improvement followed by a gradual improvement in cleaning above concentrations of 1% w/v. The sodium carbonate results are the most interesting results given the lack of information in previous literature. The data appears to suggest that sodium carbonate assists in cleaning at around 2 to 4% w/v and begins to inhibit the cleaning process after this. When cleaning with sodium carbonate without sodium hydroxide, the solutions were only effective up to 4% with a high flow rate. Physical appearances of the coupon, with high levels of sodium carbonate that did not clean sufficiently, with a thin film across the coupon may be seen in figure 13.

Figure 13: Photo of 5cmx5cm Coupon with Thin Film Remaining on Surface after Clean

This film could be easily rinsed off under a cold tap (implying a final water rinse would remove it in a plant cleaning scenario), and if left to dry, appeared as a thin white powder cake across the coupons surface (see figure 14).

Figure 14: Photo of 5cmx5cm Coupons with Dried on White Powder Cake

This suggests that the cleaning is inhibited with high concentrations of sodium carbonate due to levels of saturation of the solution. The underlying reasons are outside the scope of this study, but it is a strong recommendation for future work.

To analyse the data, the software package Minitab was used to develop a general linear model based on the randomised design of experiments which was input into the software. The initial input variables were sodium hydroxide concentration, sodium carbonate concentration, and flow rate with the response variable of time. Below are the results of the model:

General Linear Model: time versus naoh, na2co3, flow

```
Factor  Type   Levels  Values
naoh    fixed       3  0, 1, 2
na2co3  fixed       5  0, 2, 4, 8, 12
flow    fixed       2  50, 100

Analysis of Variance for time, using Adjusted SS for Tests

Source            DF   Seq SS   Adj SS  Adj MS      F      P
naoh               2  1684330  1710291  855146  99.70  0.000
na2co3             4   819759   757397  189349  22.08  0.000
flow               1   639387   559023  559023  65.18  0.000
naoh*na2co3        8   376732   403764   50471   5.88  0.000
naoh*flow          2     6190    10239    5119   0.60  0.554
na2co3*flow        4   245942   260036   65009   7.58  0.000
naoh*na2co3*flow   8   382499   382499   47812   5.57  0.000
Error             51   437417   437417    8577
Total             80  4592255

S = 92.6110   R-Sq = 90.47%   R-Sq(adj) = 85.06%

Unusual Observations for time

Obs     time      Fit  SE Fit  Residual  St Resid
  7  150.000  450.000  53.469  -300.000     -3.97 R
 13  156.000  452.000  53.469  -296.000     -3.91 R
 38  420.000  212.667  53.469   207.333      2.74 R
 40   60.000  212.667  53.469  -152.667     -2.02 R
 66  420.000  247.333  53.469   172.667      2.28 R

R denotes an observation with a large standardized residual.
```

Based on the P values of this model, the sodium hydroxide concentrations and flow rate variables do not interact with each other to impact on the final result. This is not as would be expected based upon the initial screening of the data when considering the main interactions. The overall model has a good R^2 value, which would imply that it is a good fit to the data, and a reasonably low standard deviation, given the nature of the variability of the results. Before considering the unusual observations and the removal of the variables which did not interact it was necessary to review the residuals plots of the data to this model.

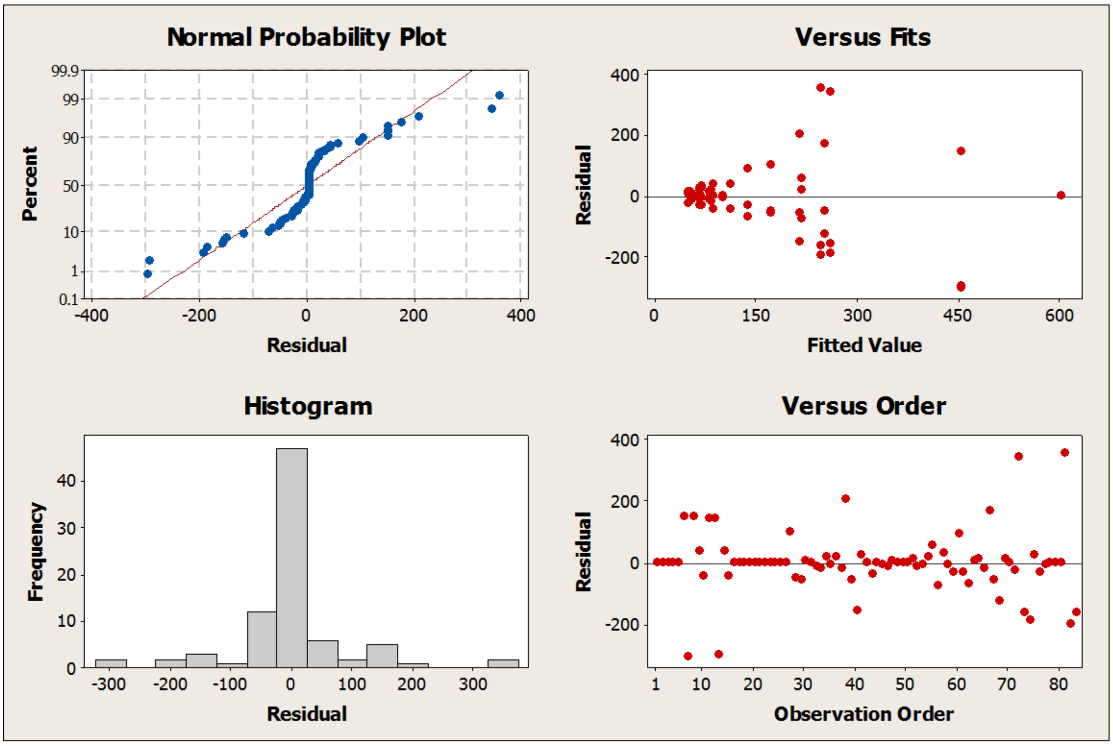


Figure 15: Residuals Plots to show the Normality, Distribution, and Residuals for Fitted Values and Observation Order for Cleaning Time

It can be seen in figure 15 that the model fit could be improved. The data does not demonstrate a good fit to the normal probability plot and the versus fits and versus order graphs show that the variability is not good due to the large cluster of points with a zero residual around similar observation points as well as variability appearing to increase with time (with the exception of a few unusual observations). The model was recreated, but this time taking logs of time as the response variable. This was in an attempt to transform the data to provide a more linear normal probability plot. Logs were used due to the logarithmic nature of pH which is expected to influence the results of the model. The results of this model are shown below:

General Linear Model: logtime versus naoh, na2co3, flow

```
Factor   Type   Levels  Values
naoh     fixed       3  0, 1, 2
na2co3   fixed       5  0, 2, 4, 8, 12
flow     fixed       2  50, 100

Analysis of Variance for logtime, using Adjusted SS for Tests

Source             DF    Seq SS   Adj SS   Adj MS      F      P
naoh                2   5.37452  5.46621  2.73310  71.24  0.000
na2co3              4   2.44629  2.22187  0.55547  14.48  0.000
flow                1   3.71272  3.25156  3.25156  84.75  0.000
naoh*na2co3         8   0.89648  0.97867  0.12233   3.19  0.005
naoh*flow           2   0.25837  0.28359  0.14179   3.70  0.032
na2co3*flow         4   0.63988  0.65237  0.16309   4.25  0.005
naoh*na2co3*flow    8   0.96100  0.96100  0.12012   3.13  0.006
Error              51   1.95668  1.95668  0.03837
Total              80  16.24594

S = 0.195873   R-Sq = 87.96%   R-Sq(adj) = 81.11%

Unusual Observations for logtime

Obs  logtime      Fit   SE Fit  Residual  St Resid
  7  2.17609  2.57746  0.11309  -0.40137     -2.51 R
 13  2.19312  2.58314  0.11309  -0.39002     -2.44 R
 38  2.62325  2.20002  0.11309   0.42323      2.65 R
 40  1.77815  2.20002  0.11309  -0.42187     -2.64 R

R denotes an observation with a large standardized residual.
```

Taking logs of time results in an improvement in the model. All of the input variables interact and effect the final cleaning result (as would be expected), and the R^2 value is still high with a low standard deviation compared to the size of unusual observations.

The unusual observations were cross checked against the results in the spread sheet and were shown to be points where the solution used to clean had varied slightly from the intended solution. When draining the equipment after use using a gravitational syphoning technique, 500 ml of residual solution would remain in the pipes of the system. Between each fresh run, the whole system would be flushed with water and drained again, but there was the potential for trace diluted amounts of the previous solution to remain in the system and mix with the solution for the next run. Due to this, some runs may be seen where the cleaning is not as expected, but on titrating the solution after the investigation there were slightly higher levels of one or both of the chemical concentrations which would affect the results. Figure 16 shows the residual plots of the model using the logs of time.

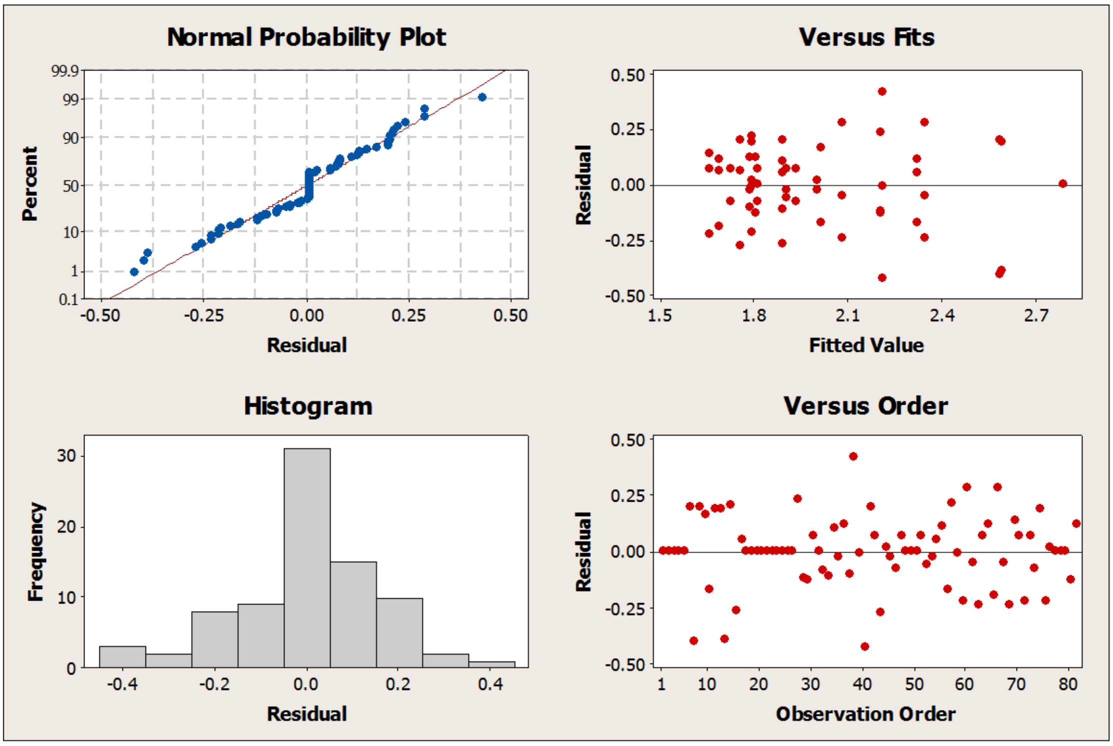


Figure 16: Residuals Plots to show the Normality, Distribution, and Residuals for Fitted Values and Observation Order for the Log of Cleaning Time

Figure 16 shows that the data has a better fit to the model based on the normal probability plot. The versus fits plot and the histogram also show much better variability and frequency distribution.

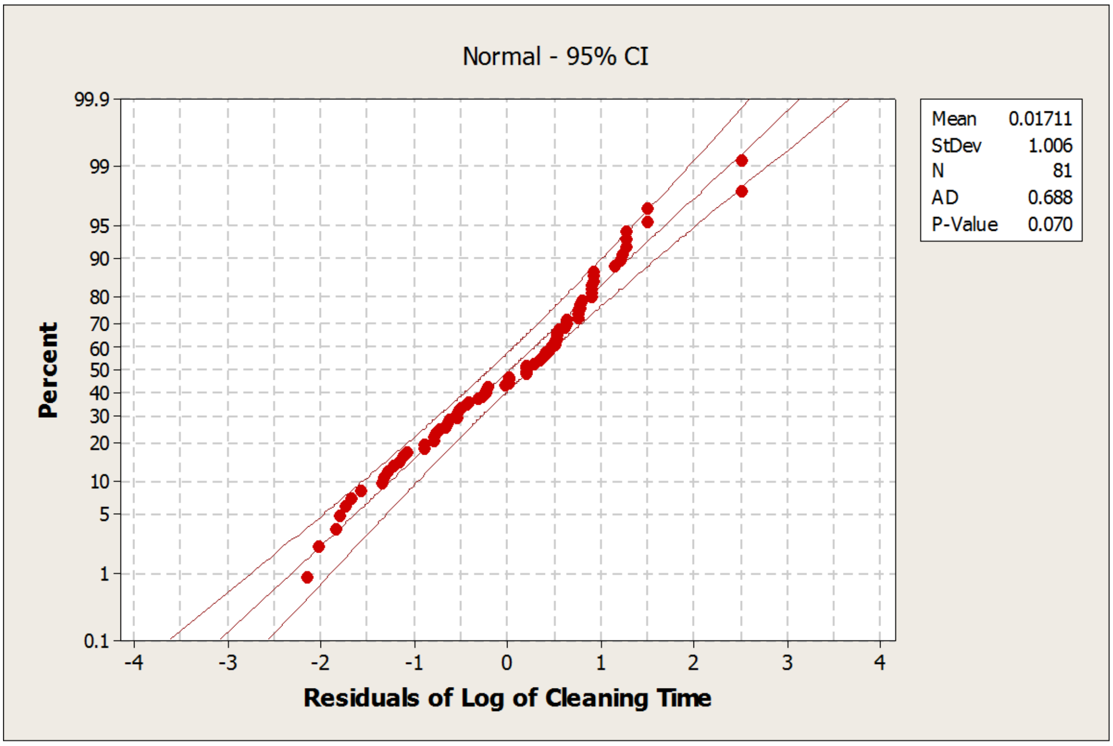


Figure 17: Probability Plot for the Residuals of the Log of Cleaning Time with 95% Confidence Intervals

Figure 17 shows the probability plot for the model and the fit of the data. This also shows that the model is a good fit, and that there are no outliers of concern within the results. Some points sit just on the edge of the95% confidence intervals, but these are the same points that were identified as unusual observations. These points will be left in the dataset to ensure that the model is not over fitted.

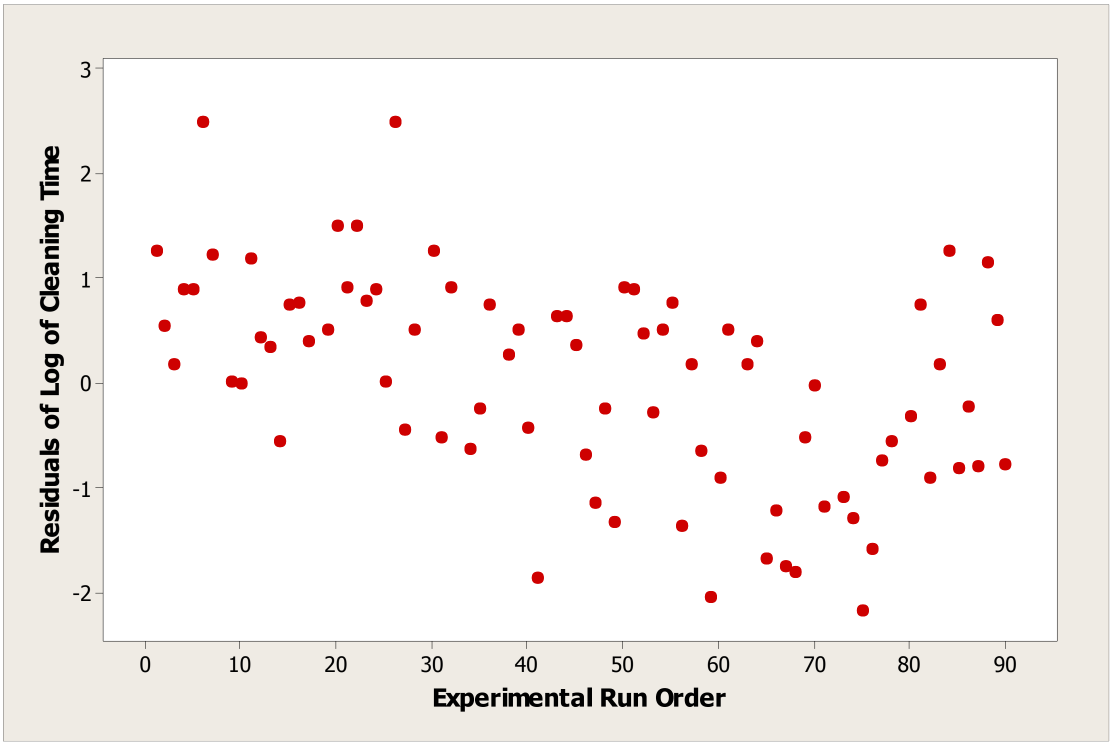


Figure 18: Scatterplot of the Residuals of the Log of Cleaning Time against Experimental Run Order

Figure 18 shows the residuals for the log of time plotted against the randomised experimental run order. Compared to the versus order residuals plot in figure 16, it can be seen that the variability with run is better than with observation order. This is due to the way that the data has been entered into Minitab. The observational orders entered are shown in increasing numbers of concentrations and flow rates, so poor variability would be expected. In terms of the experimental run order versus residuals, there are a few areas where variability is not as great, but on cross checking these points with the results spread sheet it could be seen that these are points with two very similar/same consecutive runs.

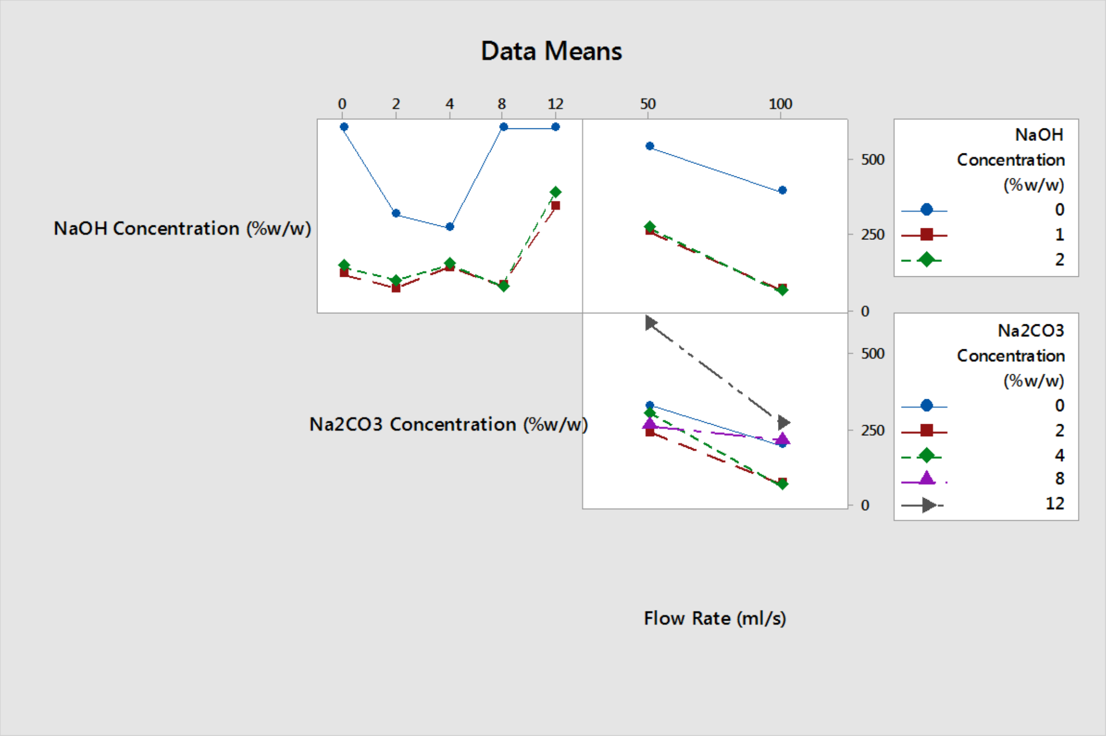


Figure 19: Interaction Plot to Show how Data Means for Cleaning Time Varies with Simultaneous Variations in Flow Rate, NaOH Concentration, and Na_2CO_3 Concentration

Figure 19 shows the overall interactions between the input variables with each other and their results on the cleaning time. It can be seen from these results that no sodium hydroxide present generally will not clean, but does clean slowly with 2 – 4% sodium carbonate. 1 -2% sodium hydroxide will clean well unless the sodium carbonate level is greater than 9%. However, sodium carbonate levels present at 12% will still clean with a sufficiently high flow rate. The results also show that there is a strong dependency of cleaning ability with the flow rate. From this it can be determined that sodium hydroxide should be at least 1% w/v and sodium carbonate should not be greater than 8% carbonate to ensure that a sufficient clean will occur. Figures 20 and 21 reiterate this point.

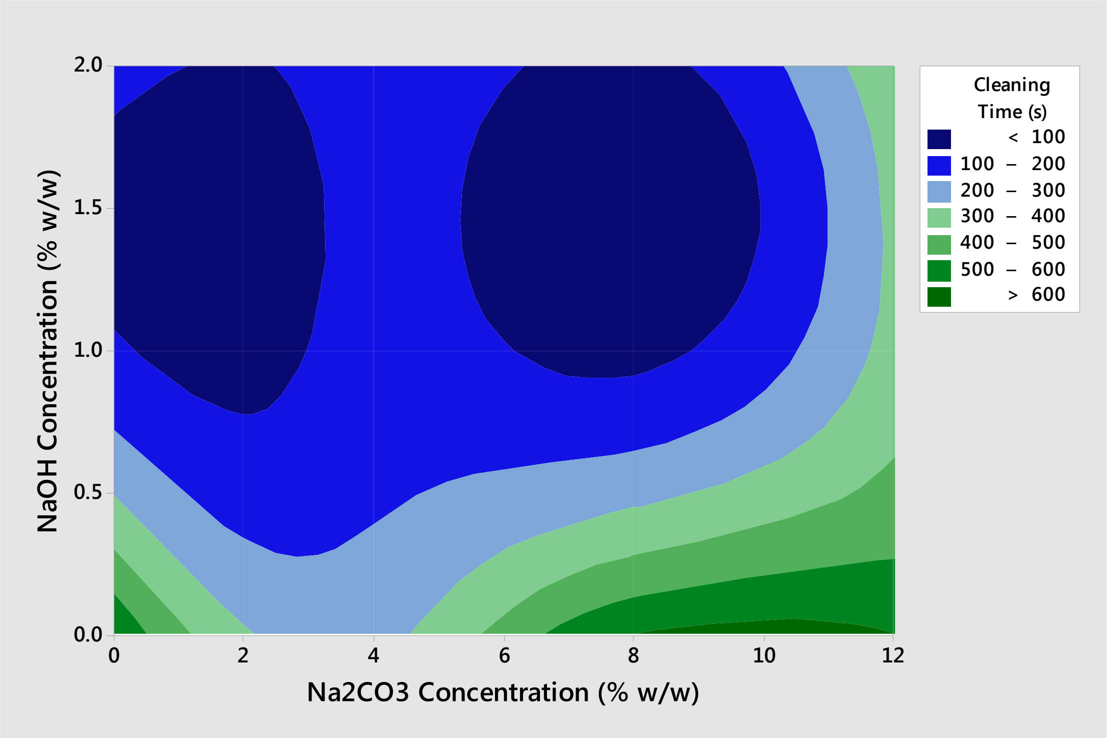


Figure 20: Contour Plot Showing Effects of Varying Concentrations of NaOH and Na_2CO_3 on Cleaning Time

Figure 20 shows the contour plot for sodium hydroxide and sodium carbonate based on time. The blue areas are the areas which cleaned well. The limits of 1% sodium hydroxide and 9% sodium carbonate are clearly defined within these limits. The section between 2 and 4% sodium carbonate also shows that slight cleaning power of sodium carbonate alone. This section is in a lighter shade of blue which shows that although it does clean at this strength without sodium hydroxide presence, it will not be sufficient to clean the fermentation vessel effectively as the overall time per unit area of vessel required to clean will be longer.

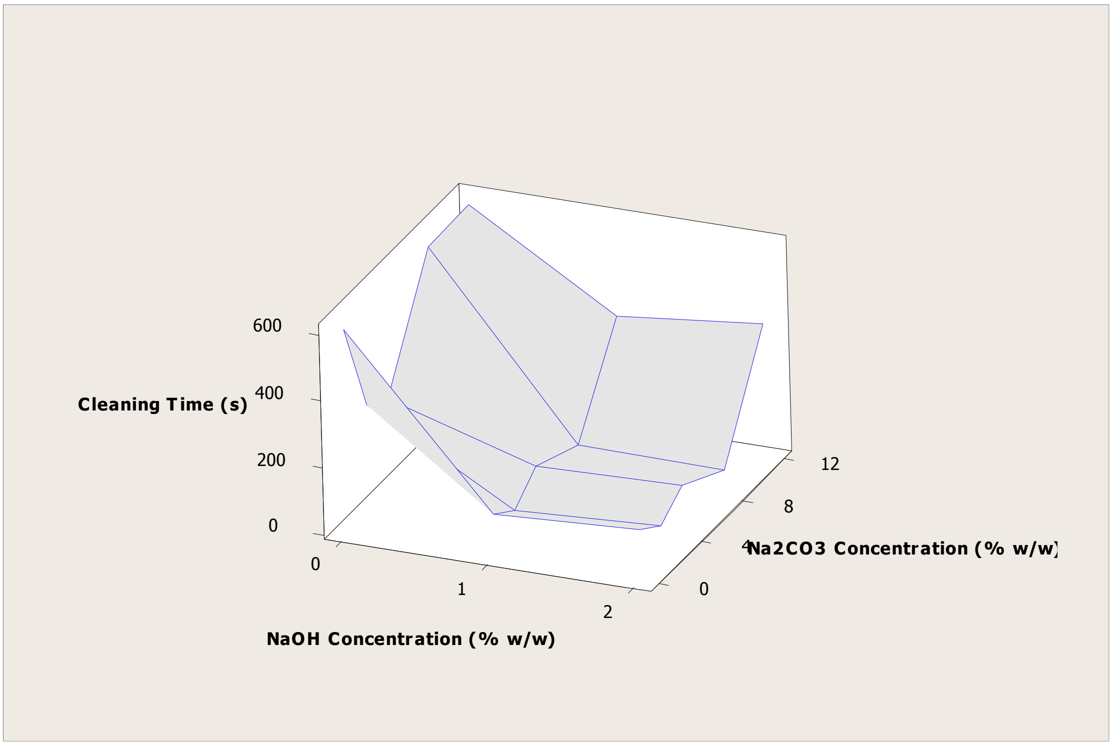


Figure 21: Surface Plot Showing Effects of Varying NaOH and Na_2CO_3 Concentrations on Cleaning Time

Figure 21 shows the surface plot of the sodium hydroxide and sodium carbonate strengths against time and again illustrates the limit of 1% sodium hydroxide and 9% sodium carbonate before cleaning times are significantly increased.

4.6.2 Offline Measurements

Offline detergent measurements were considered to investigate further potential methods to develop a new online measurement and control system to improve and optimise the current CIP detergent cleaning step.

When considering the offline measurements, pH and conductivity both appeared to give much information, but the turbidity results were unclear (see electronic appendix 2). Due to the type of turbidity analytical device used to analyse the turbidity offline, the results were inconsistent based upon the time taken for the liquid to completely settle in its sample bottle before taking the measurement. Due to the nature of the work in the Bulmers analytical laboratory, it was not feasible to spend long periods of time waiting for 90 samples to settle out (with only one bottle) as the equipment was also needed for the day to day work. Based on this, the results are not useful for further analysis.

The pH and conductivity results however have different natures to each other based upon the technologies involved. This resulted in the most suitable form of analysis being to develop two separate models to analyse both responses and draw together the results once both analyses have been performed.

4.6.3 Conductivity

The conductivity results may be seen in the spread sheet in electronic appendix 2. The conductivity results were analysed in Minitab using a general linear model. This time, sodium hydroxide and sodium carbonate were the input variables and conductivity was the response variable. The variables were put in as part of a design of experiments for analysis. The results of this model are shown below:

General Linear Model: Con versus NaOH, Na2CO3

```
Factor   Type   Levels  Values
NaOH     fixed       3  0, 1, 2
Na2CO3   fixed       5  0, 2, 4, 8, 12

Analysis of Variance for Con, using Adjusted SS for Tests


Source       DF    Seq SS   Adj SS   Adj MS      F      P
NaOH          2   4203.23  3992.61  1996.31  77.34  0.000
Na2CO3        4   3257.09  3306.38   826.59  32.03  0.000
NaOH*Na2CO3   8   1466.77  1466.77   183.35   7.10  0.000
Error        74   1909.97  1909.97    25.81
Total        88  10837.06

S = 5.08040   R-Sq = 82.38%   R-Sq(adj) = 79.04%

Unusual Observations for Con

Obs      Con      Fit  SE Fit  Residual  St Resid
 54  48.1000  38.0200  2.0741   10.0800      2.17 R
 60   2.1000  31.5333  2.0741  -29.4333     -6.35 R
 61  41.5000  31.5333  2.0741    9.9667      2.15 R

R denotes an observation with a large standardized residual.
```

The results show that all of the variables interact to affect the final results (as would be expected based on the theory that site currently bases their online conductivity measurements on). The R^2 value is high, implying a good fit to the model, and there are three unusual observations with residuals 2 to 6 times as large as the standard deviation which is relatively small. Figure 22 shows the residual plots for the model.

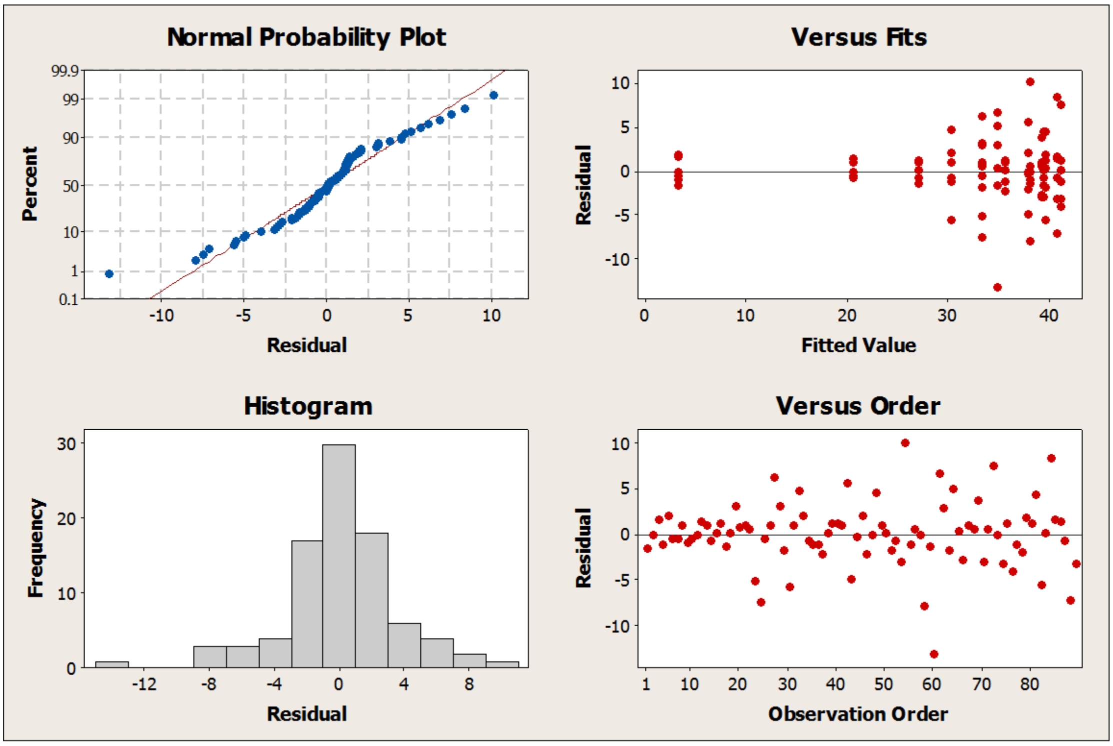


Figure 22: Residuals Plots to show the Normality, Distribution, and Residuals for Fitted Values and Observation Order for Conductivity

The results show that the data is a reasonably good fit to the model, with the exception of one outlier. The versus fits and order graphs show a large level of heteroskedastic behaviour. A number of other models were attempted, taking logs of the conductivity and 10 to the power of, but these models did not fit as well as the standard conductivity model. By cross checking the small clusters of data points on the versus fits graph with the results spread sheet, it was possible to see, that they are clusters of solution group types. E.g. the first small cluster is water, the second is water with 2% sodium carbonate. It would appear that the heteroskedastic variation is due to the nature of the measurements of conductivity and the groupings of the solution types used in the experiments.

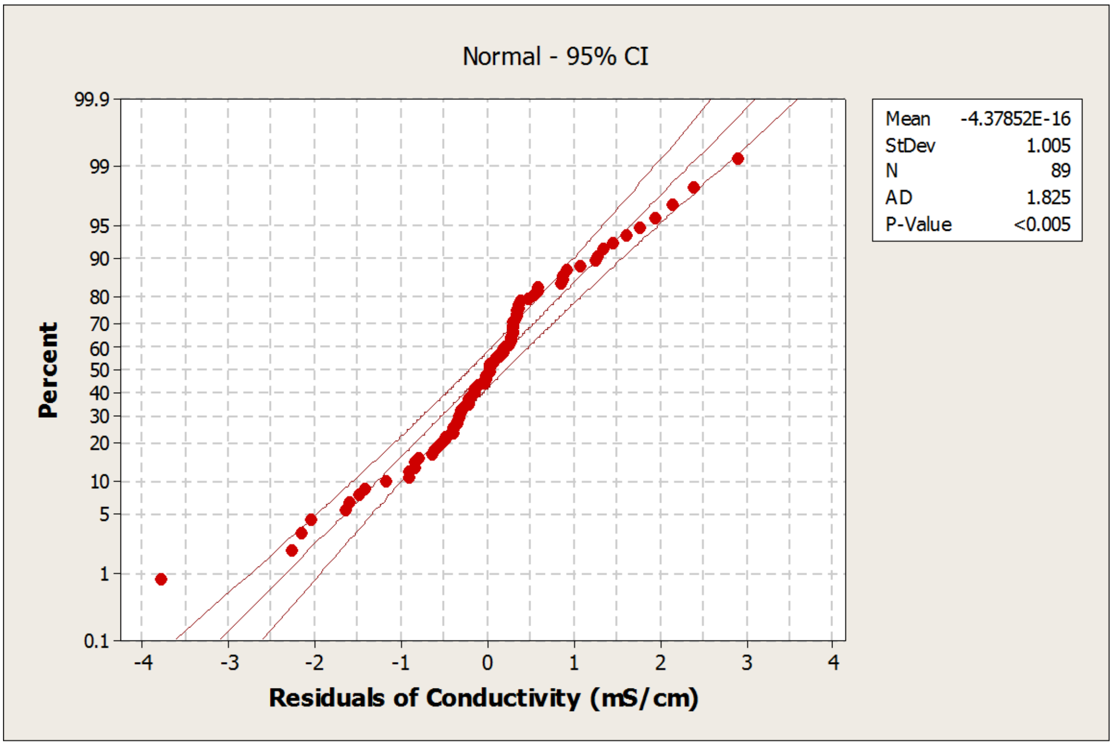


Figure 23: Probability Plot for the Residuals of the Log of Cleaning Time with 95% Confidence Intervals

Continuing with the analysis of the conductivity model, figure 23 shows the probability plots for the standard residuals. The model does appear to fit well within the boundaries with the exception of one major outlier and some smaller ones. The major outlier was crosschecked against the spread sheet in electronic appendix 2 (run 1), and the conductivity reading for this run was different to most of the readings. The conductivity probe had not been used for some time before this investigation, so it is likely that there was some residue on the plates within the probe that may have impacted upon this result. The probe was cleaned and calibrated after this.

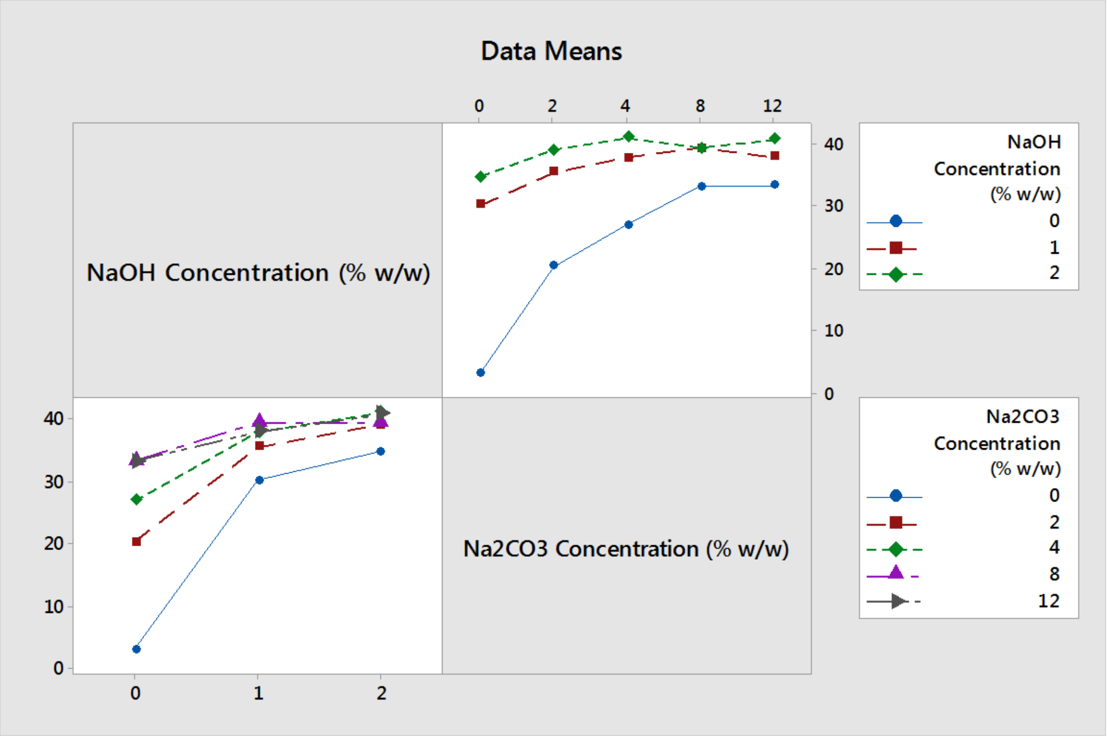


Figure 24: Interaction Plot to Show how Data Means for Conductivity Varies with Variations in NaOH and Na_2CO_3 Concentrations

Figure 24 shows the interaction plot for sodium hydroxide, sodium carbonate and conductivity. It can be seen that 1% sodium hydroxide gives a conductivity reading which is the same as that of approximately 5% sodium carbonate. Due to this, it is possible that readings from the conductivity probe will give a false sense of security with cleaning chemical specifications based on the current cleaning system set up. Readings of both under specification sodium hydroxide values, and over specification sodium carbonate values would be recognised as sufficient based on the current conductivity monitoring system (e.g. 0% NaOH and 12% Na_2CO_2). Based on the conclusions of section 4.6.1 on the cleaning chemical limits required, the conductivity should be at least 60mS for sodium hydroxide alone, and not greater than 80 for sodium hydroxide and sodium carbonate. There could however be a reading of 80mS with 12% carbonate and also a reading of above 60mS with no sodium hydroxide and more than 9% sodium carbonate. It is known from this investigation that neither of these solution types will clean effectively so site has potentially had extended periods of time where their fermentation vessels have not been cleaned effectively.

4.6.4 pH

The results for the pH may be seen in the electronic appendix 2. The results were analysed in Minitab using a general linear model. Sodium hydroxide and sodium carbonate were the input variables and pH was the response variable. The variables were put in as part of a design of experiments for analysis. The results of this model are shown below:

```
General Linear Model: pH versus NaOH, Na2CO3
Factor  Type   Levels  Values
NaOH    fixed       3  0, 1, 2
Na2CO3  fixed       5  0, 2, 4, 8, 12

Analysis of Variance for pH, using Adjusted SS for Tests

Source       DF    Seq SS   Adj SS   Adj MS      F      P
NaOH          2   74.4073  72.8419  36.4210  94.28  0.000
Na2CO3        4   10.6120  10.7238   2.6809   6.94  0.000
NaOH*Na2CO3   8   15.3647  15.3647   1.9206   4.97  0.000
Error        74   28.5852  28.5852   0.3863
Total        88  128.9692

S = 0.621520   R-Sq = 77.84%   R-Sq(adj) = 73.64%

Unusual Observations for pH

Obs       pH     Fit  SE Fit  Residual  St Resid
  2   6.9300  9.7150  0.2537   -2.7850     -4.91 R
  3  12.2300  9.7150  0.2537    2.5150      4.43 R
  4   6.9500  9.7150  0.2537   -2.7650     -4.87 R
  6  11.8000  9.7150  0.2537    2.0850      3.67 R

R denotes an observation with a large standardized residual.
```

The results show that all of the variables interact to affect the pH results. This is as expected due to the basic properties of both chemicals in solution. The R^2 value is high and the standard deviation is low with four unusual observations. The residual plots for the model can be seen in figure 25.

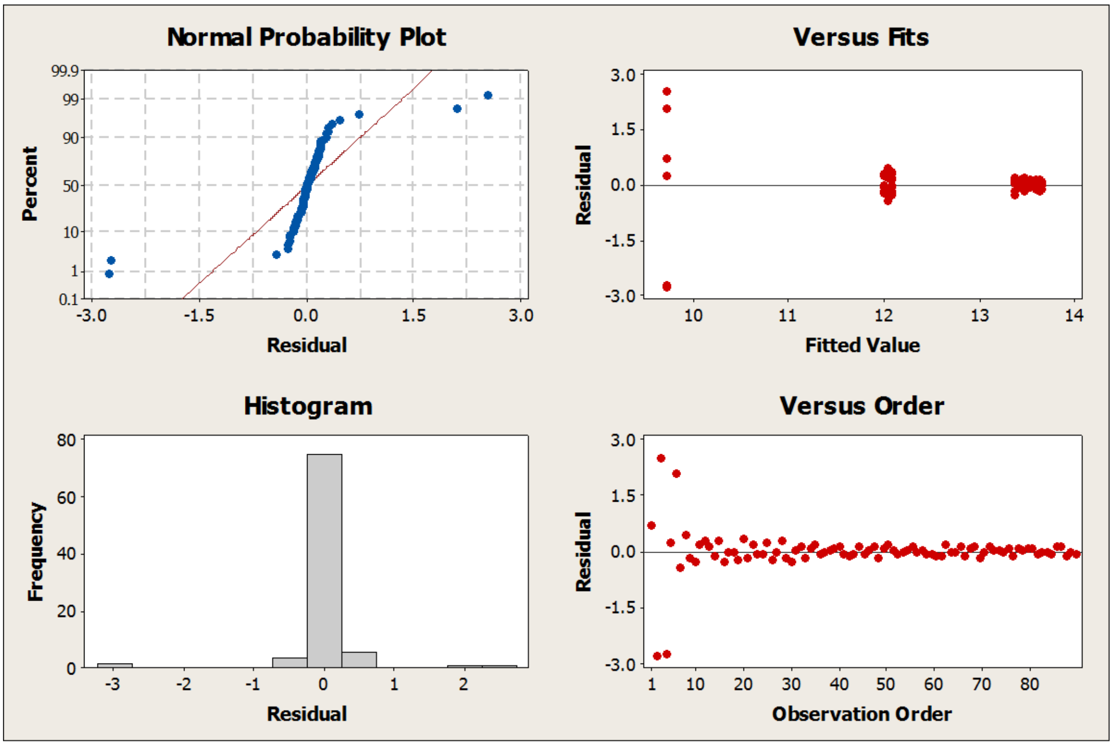
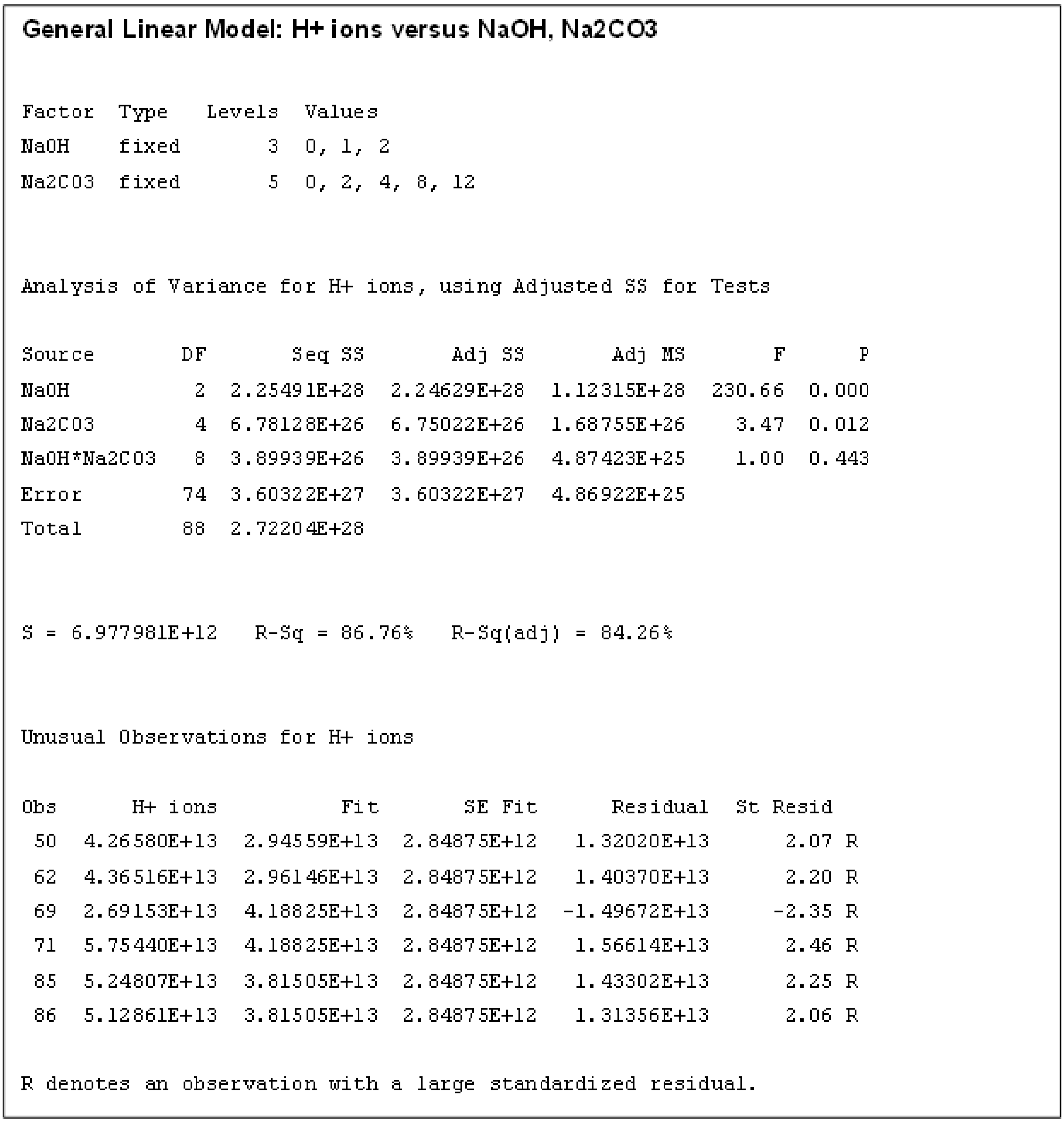


Figure 25: Residuals Plots to show the Normality, Distribution, and Residuals for Fitted Values and Observation Order for pH

Figure 25 shows that the data does not fit to the model well at all. The data barely touches the normal probability plot, and the variability decreased almost exponentially with time. Looking at the shape of the graphs and considering the knowledge of the relationship between hydrogen ions concentration and pH, a model of the H^+ ions was investigated. This was done by taking 10 to the power of the pH. The results from this model are shown below:



```
General Linear Model: H+ ions versus NaOH, Na2CO3


Factor   Type   Levels  Values
NaOH     fixed       3  0, 1, 2
Na2CO3   fixed       5  0, 2, 4, 8, 12



Analysis of Variance for H+ ions, using Adjusted SS for Tests


Source        DF       Seq SS       Adj SS       Adj MS       F      P
NaOH           2  2.25491E+28  2.24629E+28  1.12315E+28  230.66  0.000
Na2CO3         4  6.78128E+26  6.75022E+26  1.68755E+26    3.47  0.012
NaOH*Na2CO3    8  3.89939E+26  3.89939E+26  4.87423E+25    1.00  0.443
Error         74  3.60322E+27  3.60322E+27  4.86922E+25
Total         88  2.72204E+28



S = 6.977981E+12   R-Sq = 86.76%   R-Sq(adj) = 84.26%



Unusual Observations for H+ ions


Obs      H+ ions          Fit       SE Fit      Residual  St Resid
 50  4.26580E+13  2.94559E+13  2.84875E+12   1.32020E+13      2.07 R
 62  4.36516E+13  2.96146E+13  2.84875E+12   1.40370E+13      2.20 R
 69  2.69153E+13  4.18825E+13  2.84875E+12  -1.49672E+13     -2.35 R
 71  5.75440E+13  4.18825E+13  2.84875E+12   1.56614E+13      2.46 R
 85  5.24807E+13  3.81505E+13  2.84875E+12   1.43302E+13      2.25 R
 86  5.12861E+13  3.81505E+13  2.84875E+12   1.31356E+13      2.06 R

R denotes an observation with a large standardized residual.
```

The results show that initially, the P value for sodium hydroxide and sodium carbonate combined should not be included as it is too high. On removal of this factor, the results were as follows:

General Linear Model: H+ ions versus NaOH, Na2CO3

```
Factor  Type   Levels  Values
NaOH    fixed       3  0, 1, 2
Na2CO3  fixed       5  0, 2, 4, 8, 12

Analysis of Variance for H+ ions, using Adjusted SS for Tests

Source  DF       Seq SS       Adj SS       Adj MS       F      P
NaOH     2  2.25491E+28  2.24665E+28  1.12332E+28  230.68  0.000
Na2CO3   4  6.78128E+26  6.78128E+26  1.69532E+26    3.48  0.011
Error   82  3.99316E+27  3.99316E+27  4.86971E+25
Total   88  2.72204E+28

S = 6.978331E+12   R-Sq = 85.33%   R-Sq(adj) = 84.26%

Unusual Observations for H+ ions

Obs      H+ ions          Fit       SE Fit      Residual  St Resid
 69  2.69153E+13  4.15610E+13  1.94784E+12  -1.46457E+13     -2.19 R
 71  5.75440E+13  4.15610E+13  1.94784E+12   1.59830E+13      2.39 R
 85  5.24807E+13  3.79466E+13  1.94784E+12   1.45341E+13      2.17 R

R denotes an observation with a large standardized residual.
```

These results show that both sodium hydroxide and sodium carbonate individually are important, as well as the R^2 value being good with a low standard deviation relative to the size of the results. The residual plots for this model can be seen in figure 26.

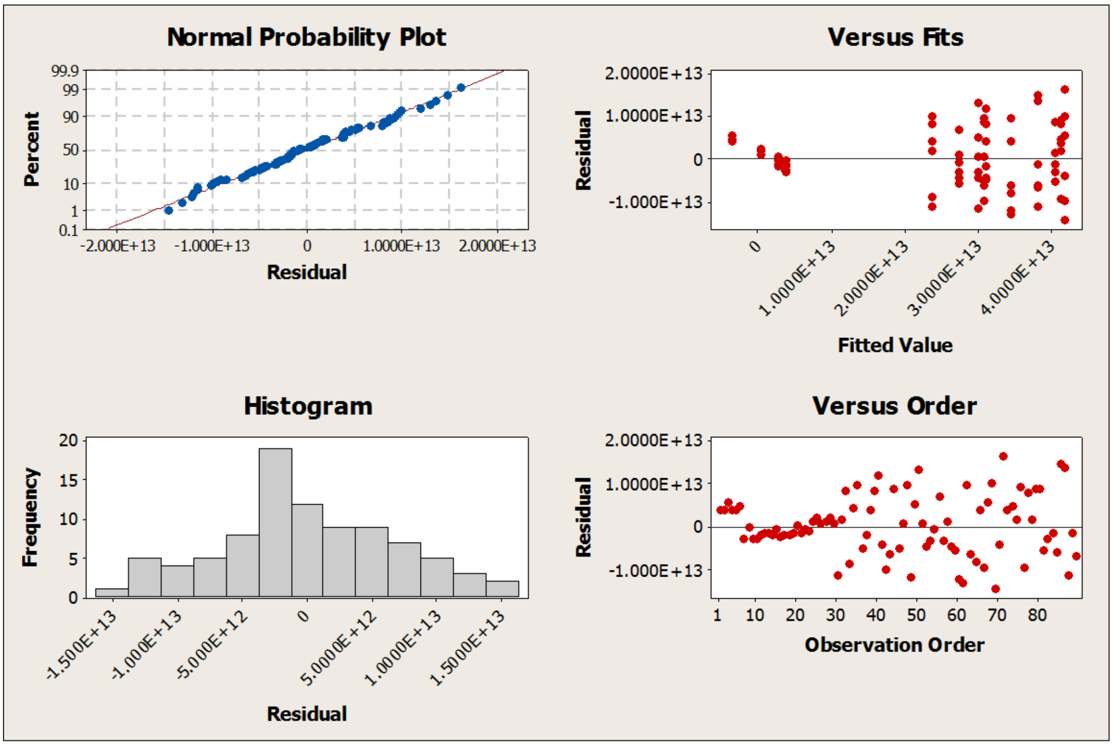


Figure 26: Residuals Plots to show the Normality, Distribution, and Residuals for Fitted Values and Observation Order for H^+ Ions

The results show that the model appears to be a good fit based on the normal probability plot and the data fit on there, and the frequency distribution on the histogram. The versus fits and versus order need to be considered in more depth.

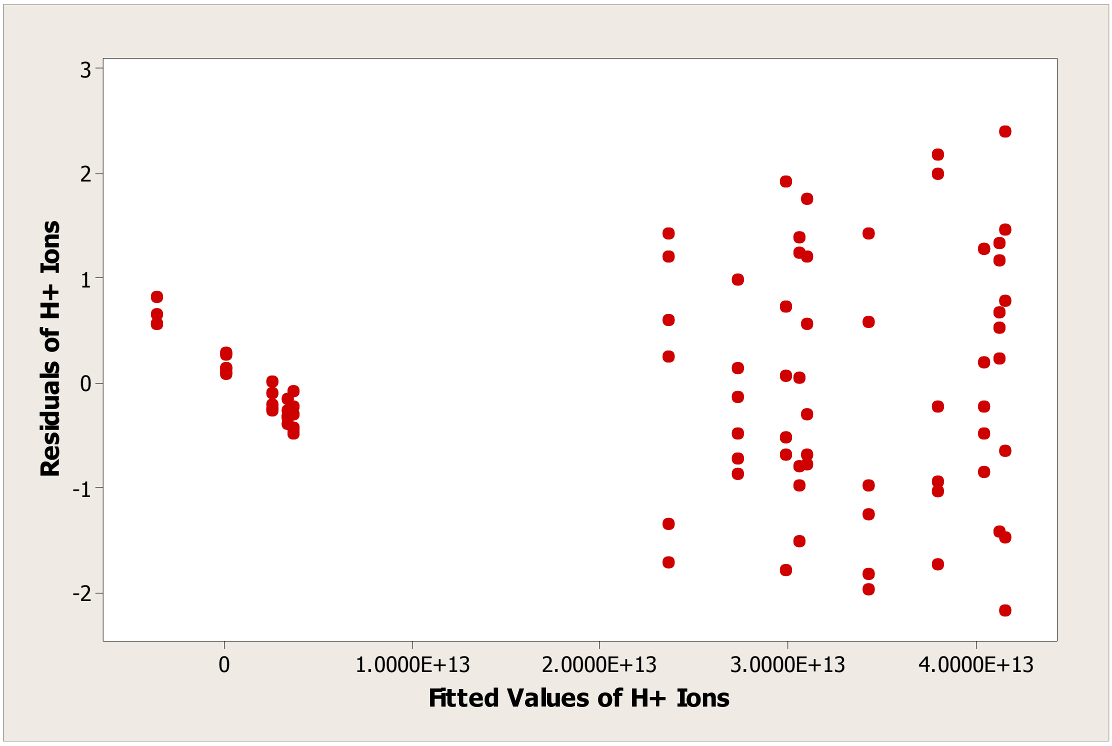


Figure 27: Scatterplot to Show Variability BetweenResiduals of H^+ Ions and Fitted Values of H^+ Ions

Figure 27 shows that the variability on the standard residuals versus fits graph is good on the right hand side of the graph. When cross checking the data points on the left hand side which do not quite fit with the rest of the data against the results spread sheet, it is possible to see that these are water samples and low sodium carbonate samples with no sodium hydroxide in. Due to this the concentration of H^+ ions will be significantly lower than all of the other solutions containing sodium hydroxide and high levels of sodium carbonate. This behaviour of the results is believed to be due to the logarithmic nature of pH measurements.

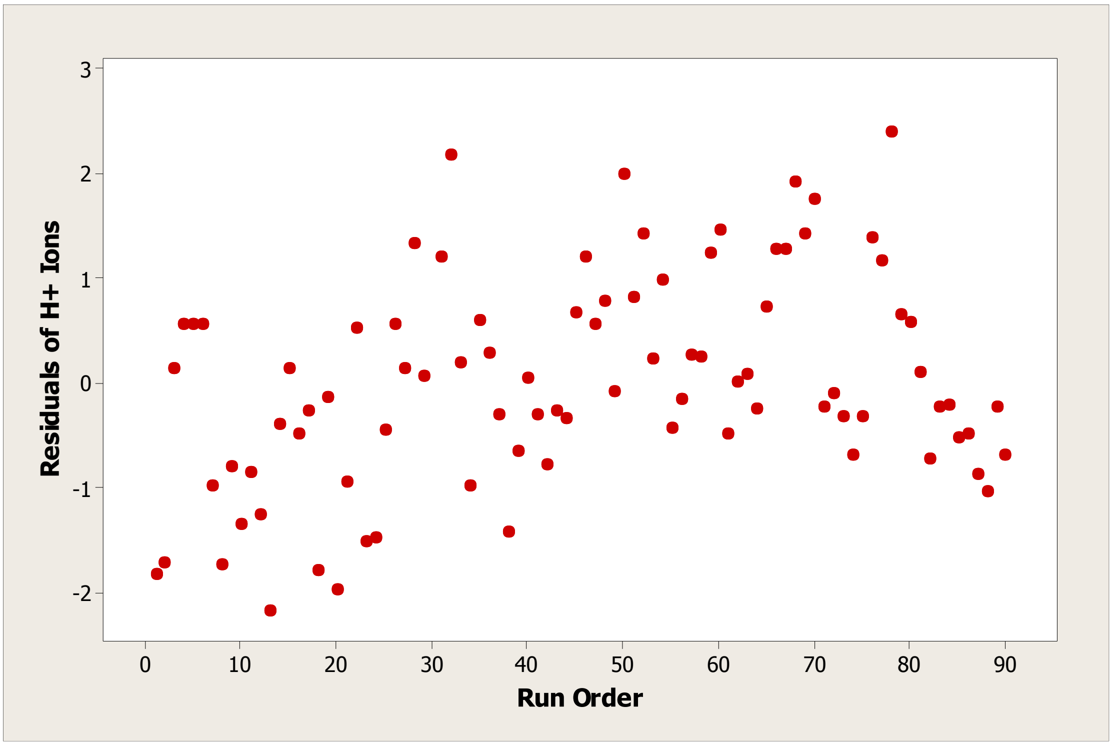


Figure 28: Scatterplot to Show Variability of Standard Residuals of H+ Ions against Experimental Run Order

Figure 28 shows the variability of residuals against experimental run order. It can be seen that the variability of the model overall is good, with some exceptions. E.g. runs 4, 5 and 6 do not show good variability, but when checking them with the results spread sheet, they are all water solutions which happened to be run consecutively by chance. This also explains some of the behaviour exhibited by the data points in figure 27.

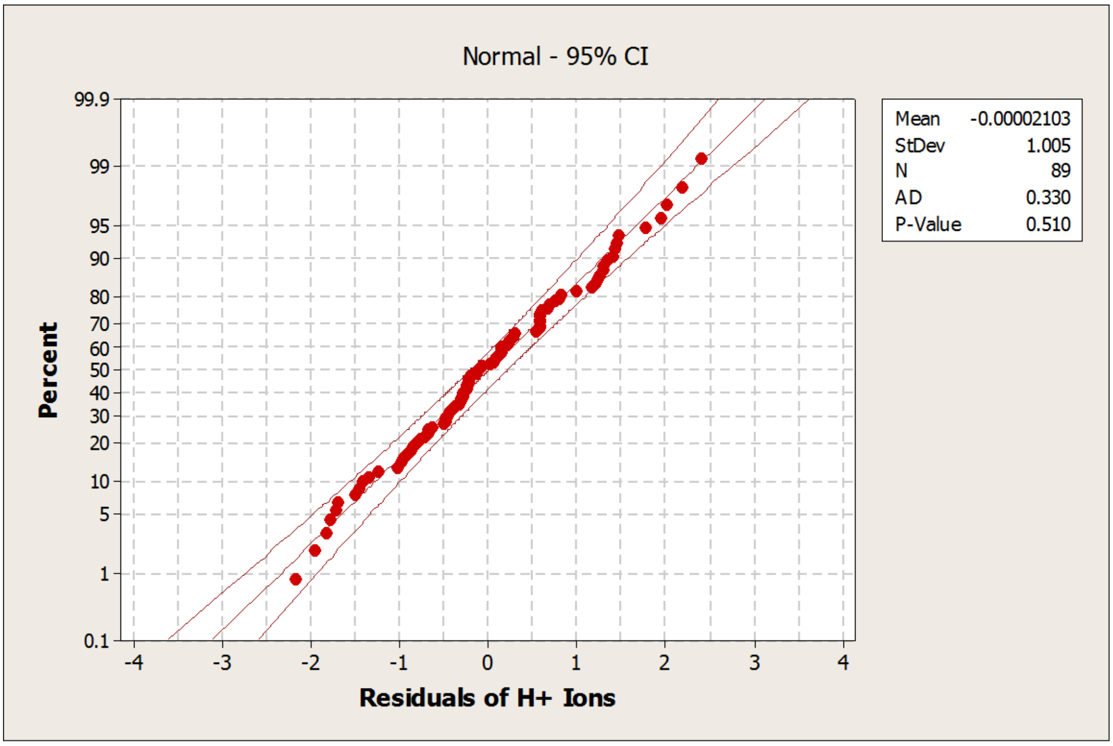


Figure 29: Probability Plot for the Residuals of H+ Ions with 95% Confidence Intervals

Figure 29 shows the probability plot for the residuals of the H^+ ions. The graph shows that the data fits well within the 95% confidence intervals for the model with no outliers.

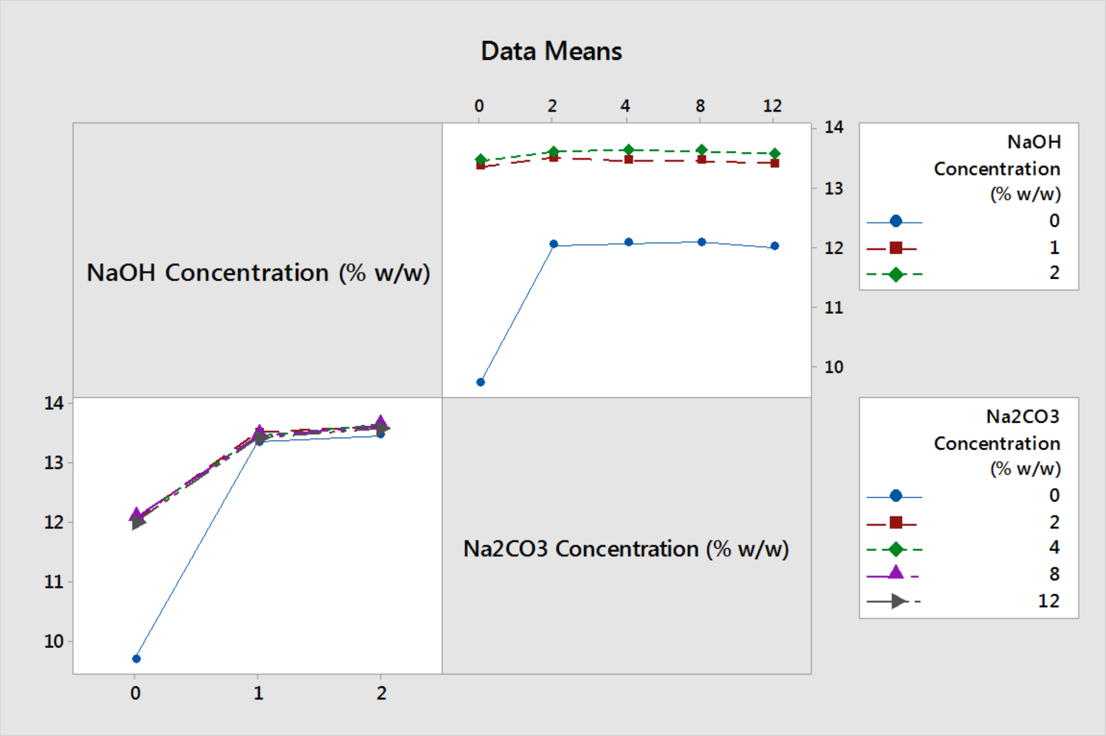


Figure 30: Interaction Plot to Show how Data Means for pH Varies with Variations in NaOH and Na_2CO_3 Concentrations

Figure 30 shows the interaction plots for sodium hydroxide and sodium carbonate and their results on pH. It can be seen that samples of only water will have a pH of less than 10. Some have pH values of as high as 10 due to residual solutions in the reactor. Solutions of sodium carbonate alone will have a pH of approximately 12, regardless of concentration. Likewise with sodium hydroxide, it will have a pH of around 13.5. pH is slightly higher as sodium hydroxide concentration is increased, but not by more than 0.2. When combined solutions of sodium hydroxide and sodium carbonate are present with more than 1% sodium hydroxide, sodium hydroxide appears to dominate the overall pH, resulting in a pH of around 13.5.

Based on the results for the chemical composition requirements found in section 4.6.1, the pH value should be greater than 13. This will be useful information, as when sodium hydroxide levels are low, but sodium carbonate levels are high, the pH probe will be able to identify this as the pH will drop to around 12, whilst the conductivity readings are still within their limits. When the conductivity rises to greater than 80 mS, it will then be apparent that although the sodium hydroxide may be within the correct limits, the sodium carbonate is now too high to enable the solution to clean effectively.

4.6.5 pH and Conductivity

To conclude from sections 4.6.3.1 and 4.6.4.2, the conductivity value should be greater than 60 and less than 80 mS, and the pH should be greater than 13.

Samples were taken from a fermentation vessel clean (FV611) at five minute intervals to verify this. A table of the results from this clean may be seen in table 10 in appendix 14. There are two samples taken at 30 minutes as one is from the end of the mains clean, and the other is the initial sample from the vessel clean where some making up of the dilute tank would have occurred in between those times. Figures 31, 32 and 33 show the chemical concentrations, the conductivities, and pH values respectively.

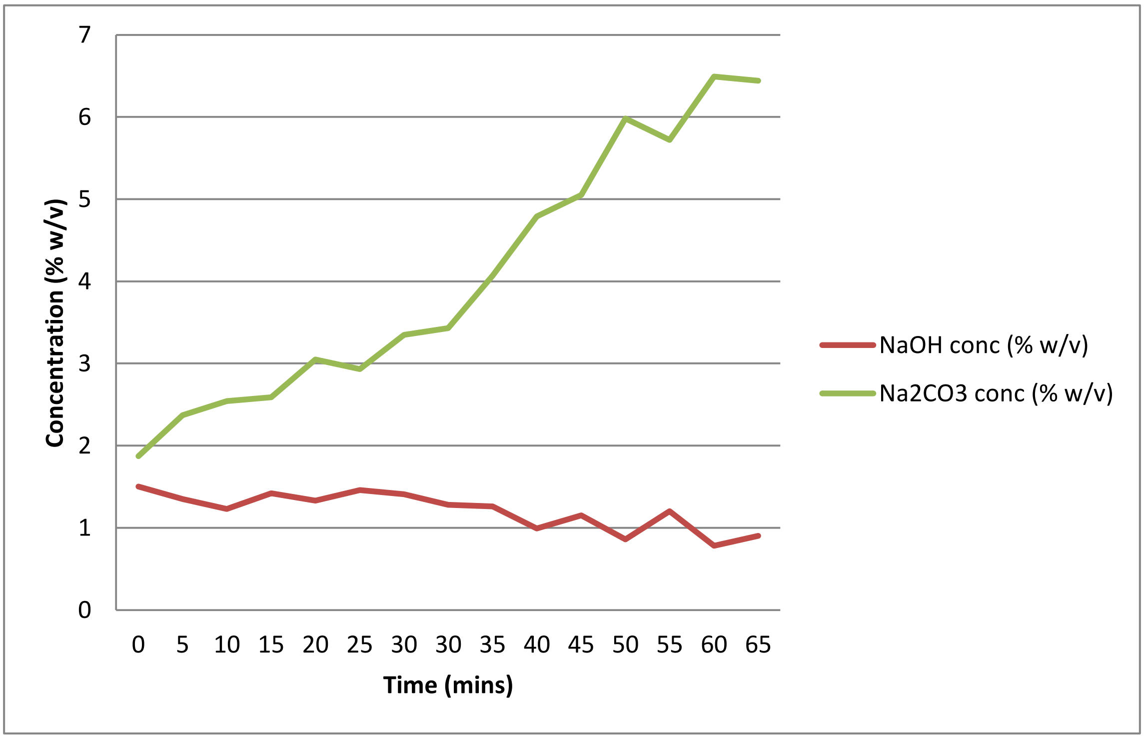


Figure 31: Graph of Caustic and Carbonate Concentrations during Fermentation Vessel 611 on Current CIP Setup

Figure 31 shows that based on current site limits, the sodium hydroxide was off specification of 1.3% after 35 minutes, resulting in 30 minutes of cleaning without sufficient sodium hydroxide. The sodium carbonate exceeded the 4% limit after 35 minutes, again resulting in 30 minutes of off specification cleaning. If the new limits were in place the sodium carbonate would have been within the limits of 9% w/v throughout the entire clean. Based on current limits the dilute tank should have been replaced half way through, but based on new limits the tank could have been dosed by small amounts throughout the last 30 minutes and would have been acceptable for at least one more equipment clean afterwards, provided that it is not a fermentation vessel or any other equipment with a high level of residual carbon dioxide present.

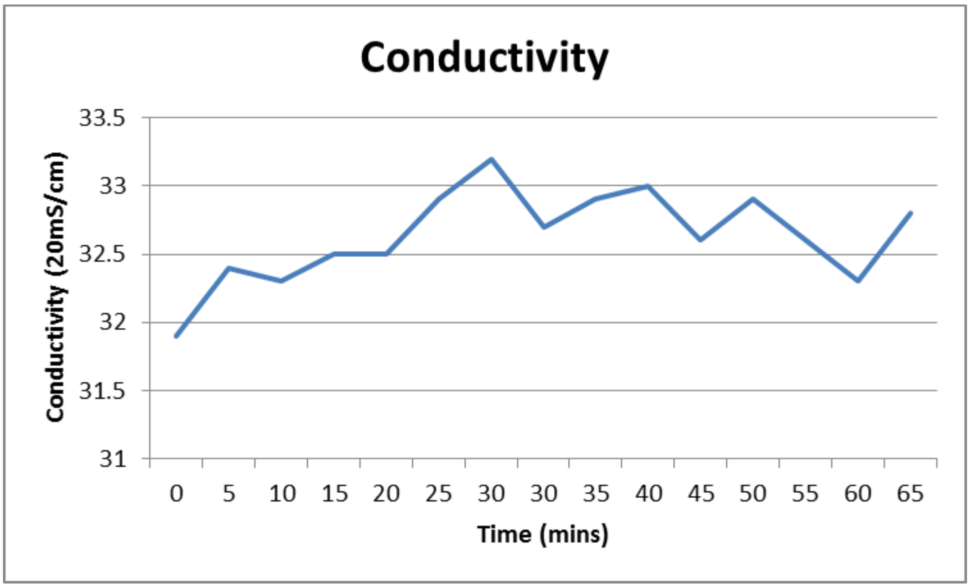


Figure 32: Conductivity Values of Of Fermentation Vessel 611 CIP Samples (Measured Offline) against Cleaning Time

Figure 32 shows that the conductivity remained in specification (greater than 60mS) throughout the whole clean. It can be seen from figure 31 that this was not the case. This confirms the claim that conductivity alone is not a suitable measure of the chemical state under these high carbon dioxide conditions, as it has not identified that the cleaning is not sufficient. It must be noted that the conductivity probe in line has slightly different readings to the offline measurements as it will be calibrated differently and taken at different places within the recirculation loop, but overall both measurements fundamentally show the same information. However, the in line measurements would have been crossing the 60mS limit, for the system dosing to occur as the measurement drops below 60mS.

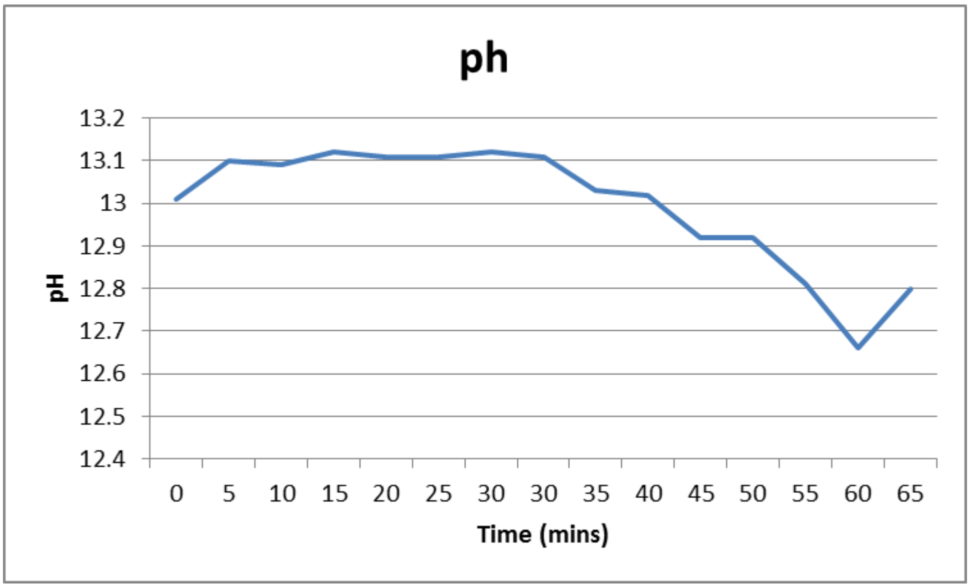


Figure 33: pH Values of Fermentation Vessel 611 CIP Samples (Measured Offline) against Cleaning Time

Figure 33 shows that the pH did not go off specification until 35 minutes. At this point the concentration of sodium hydroxide was 1.26% and the sodium carbonate was 4.07% based on a hand titration. After this point the sodium hydroxide continued to exhibit a generally falling trend and increase in small amounts (with dosing) until the end of the clean where it reached 0.9%. It did not exceed 1.26% at any point after 35 minutes.

These three figures have illustrated a real industrial cleaning example from Bulmers, and how the pH and conductivity may be used to gain a clear confirmation that the cleaning detergent in use is on specification and cleaning the vessel effectively.

The behaviour of the cleaning with caustic from the supplier in an industrial scenario demonstrates that there is no significant variation in results from the manually made up solutions. This is because the additives in the supplier provided caustic are sequestrants, which are solely present to prevent calcium carbonate scale build up on the tank walls as a result of hard water

(Edwards, 2016). The thin film built up on the experimental coupons may have been scale build up as a result of using towns water without sequestrants in the caustic solution.

This on site verification also verifies that using beer bottoms to foul for the experiments has had no impact on the investigation of the carbonate rate formation and cleaning detergent behaviours in this aspect, as they have been replicated on cider soils.

4.6.6 Titration Accuracy

When performing titrations, the method stated in appendix 8 is used. The method includes a dilution factor, which may impact on the overall results of the titration. The standard method involves a sample of 2.5ml, but the method used for titration of the 90 experimental samples in a reasonable time frame used samples of 0.25ml. This increases titration error by tenfold from this alone and this is why the titrations used to verify the experimental solutions are only used as a guide in analysis, and are not relied upon for the results.

The titration itself also involves an element of human error based on perception of the colour indicators and reaction time to close the burette tap once the colour change has occurred. There is a ±0.05ml error on the reading of the burette scale.

Based on the titration theory used in appendix 9, the colour change points for both indicators are over a large pH range, this also provides scope for a large error in titration result, particularly if the reaction time to close the burette was slow, so the acid present for the following indicator is already in excess which may give a lower sodium carbonate value than is present.

4.6.7 Cost Analysis

Benchmarking the CIP costs of the overall fermentation area at Bulmers has been discussed in chapter 3, including a breakdown of the chemical costs. To calculate the cost benefits of this project, it has been necessary to look into the benchmarking for a fermentation vessel in more detail and the fermentation vessel specific benchmarking spread sheet can be found in electronic appendix 3 and the improved version with the optimisation included in electronic appendix 4.

It has been identified that the typical hold time based on sodium hydroxide cleaning steps is 50 minutes. By optimising the cleaning set up, it will be possible to completely remove this hold time, so this has been incorporated into the calculation by considering capacity and down time costs (assuming the plant was running at full capacity).

Thermal costs have been excluded from this section as there are no heated vessel cleans. Acid costs have also been excluded as the modifications suggested in this project will not affect the acid step times or consumptions.

Based on all of the fermentation vessel cleans monitored, the sodium carbonate value always increases by more than 4%. If the dilute caustic were to be titrated by an operator immediately after a fermentation vessel clean, the procedure should be to make up a fresh dilute caustic tank. It has been assumed that each fermentation vessel clean uses one dilute caustic tank, as if these changes are not put in place, the monitoring of the sodium carbonate should be increased so as to avoid completing more cleans off specification.

Based on these assumptions, and the monitoring of large fermentation vessel cleans, the following information has been deduced:

There are typically 5 large fermentation vessel cleans per week, so 260 in a year. Based on downtime costs of £68.61/min (Orton, 2012).

Utility costs work out to be around £290 per week, and chemical costs are approximately £2735 per week, with an effluent cost of £2 per week. This results in a total weekly cost of £3000 (not including down time) and £600 per clean. The weekly lost production capacity due to cleaning is around £122,500 or £6,370,000 per annum. See figure 34 where down time accounts for 98% of chemical costs, and cleaning chemicals account for 2% (not including acid costs).

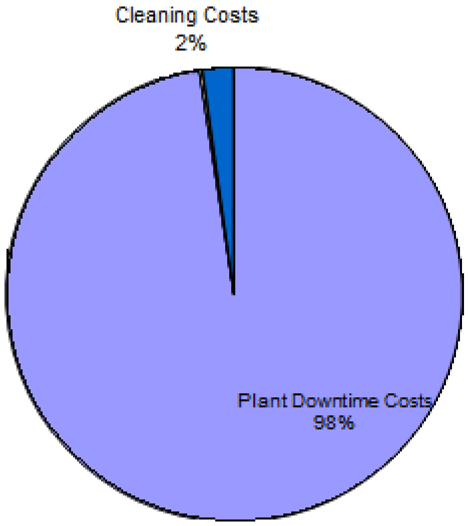


Figure 34: Pie Chart of Cost Distribution for Fermentation Vessel Cleans

If 50 minutes of downtime were removed per clean (based on optimisation of the CIP detergent steps and removal of the average hold time of 50 minutes), that would save 250 minutes per week and 13,000 minutes per year (more than 9 days). This would save £17,000 of downtime per week or £900,000 per annum. This makes no change to the percentage distribution between chemical costs and down time, due to the high volume of down time costs.

This only accounts for the removal of the downtime. In Chapter 3 it was shown that the whole of the fermentation area was spending approximately £105,000 on cleaning with bulk caustic. If the new limits of this investigation are put in place, then that sodium hydroxide will effectively last twice as long (assuming specifications are currently being adhered to). This would result in a cost saving on chemicals of approximately £59,000 per annum.

The project recommendations will free up approximately 250 minutes per week of fermentation time (assuming full plant capacity). Based on this, the capital cost which would be involved in purchasing and installing a tank which could be used for the same volume of production would be £330,000. See appendix 15 for calculation.

Overall, by removing downtime, increasing plant capacity, and modifying the chemical limits (excluding capital expenditure), the plant will save around £959,000 per annum plus a capital saving of £330,000 (15% of total large FV annual CIP costs).

Effluent will also be reduced with the new strategy, reducing the volume of dilute caustic which is sent to drain by 56%. This will also reduce the COD levels within the effluent.

In addition to these quantified savings, there will also be an added level of confidence that the equipment is being cleaned by effective detergent at all. The cost implications of failed micro batches and reworking costs would show a greater cost benefit, but this is not a straightforward calculation as there are many other factors which could also affect the micro scores.

The net present value (NPV) over a ten year period has been calculated to show the cost benefit of implementing these project recommendations. The calculation is based on the assumption that the new instrumentation, pipework, automation and installation will cost £30,000, see appendix 16 for the cost breakdown. Annual operational costs are assumed to be £1000, mainly to maintain the instrumentation. The NPV is £6,480,000 with a payback time of approximately 2 weeks. See appendix 17 for the NPV calculation. The quota used by Heineken typically requires that the payback time will be less than one year, therefore this project is highly recommended for implementation.

The NPV calculation includes costs based on down time and assuming the plant is running on full capacity. If these costs were to be excluded, and only the direct savings on chemicals to be considered, then the NPV is £365,000 with a payback time of approximately 7 months. See appendix 18. Although this value is significantly smaller, it still meets Heineken’s standard to be recommended for implementation.

4.6.8 *CIP Structure Observations*

4.6.8.1 *General CIP Structure*

The CIP for the detergent step works by opening the dilute tank, and pumping it off to the equipment then back to the CIP return. The fluid is sent directly to drain until the conductivity probe on the CIP return shows that there is sodium hydroxide present. At this point, the sodium hydroxide is recirculated through the dilute caustic tank and the drain closed. As shown in figure 35.

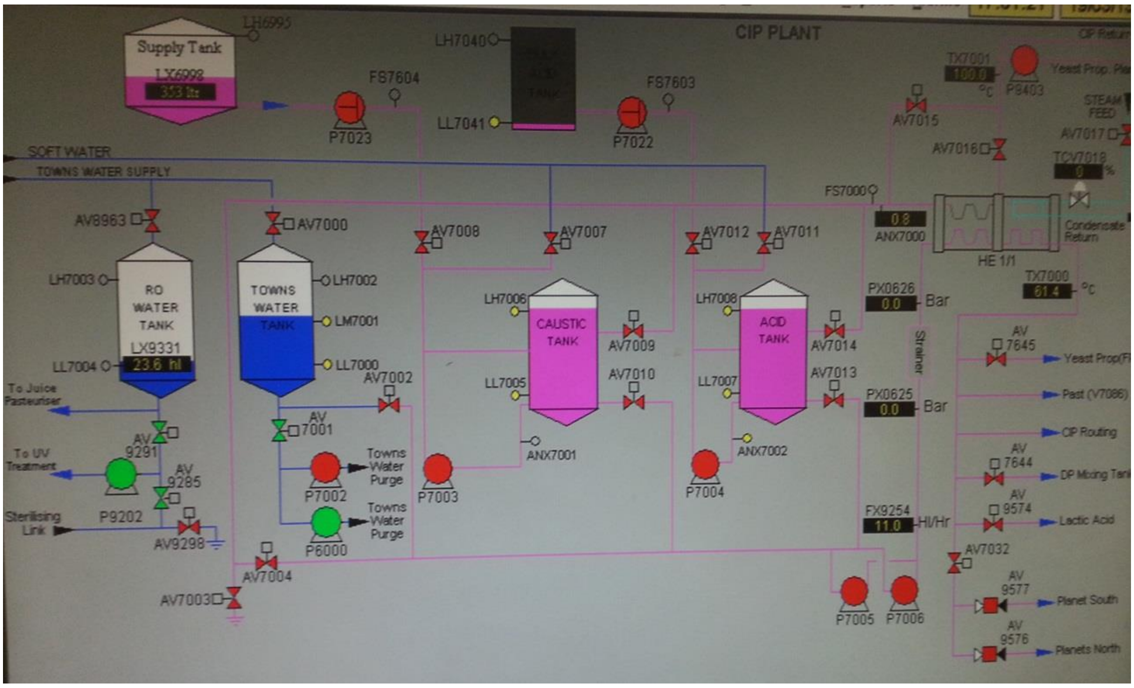


Figure 35: Print Out of CIP SCADA Mimic

The detergent step runs for 30 minutes recirculating via the mains on the side of a fermentation vessel (see figure 36), then later for 30 minutes via the vessel itself.

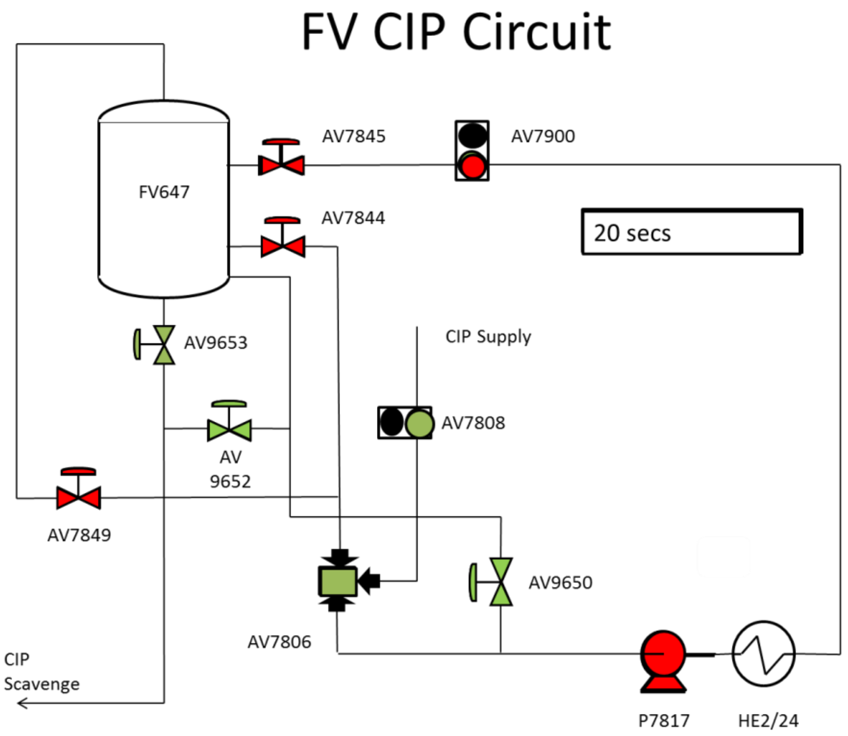


Figure 36: Diagram of Fermentation Vessel and Mains Set Up

If the conductivity on the CIP return line drops below 60 mS, the process goes into hold. This means that the CIP supply pump is turned off and the concentrated sodium hydroxide from the bulk tank is pumped into the dilute tank until the conductivity shown on the dilute tank is up to specification. On completion of each detergent step, the remaining detergent is pumped back into the dilute tank until the conductivity probe reads that water is in the line and not sodium hydroxide.

4.6.8.2 Carbonate Formation Rate

Appendix 19 shows the calculation of the carbonate formation rate based on samples taken from a fermentation vessel at fixed intervals throughout the clean. One of the potential outcomes of this project was to optimise the amount of time which is spent sending the sodium hydroxide to drain before recirculating via the dilute caustic tank with the objective of “mopping up” residual carbon dioxide from the vessel. The calculations have shown that it would take at least 28 full vessel cleans to remove all of the carbon dioxide from a large vessel. This would not be cost effective or feasible based on time and resources.

Based on the stoichiometry of the chemistry of the formation of sodium carbonate, if dosing did not happen throughout a clean (i.e. fresh sodium hydroxide was not added to replenish the detergent after the formation of sodium carbonate), then no more than 2.25% w/v of sodium carbonate would be formed and sodium hydroxide would drop rapidly to 0% and cleaning would not be effective. A fermentation vessel clean was monitored with a system override to stop any additional dosing which confirms this theory. See figure 37.

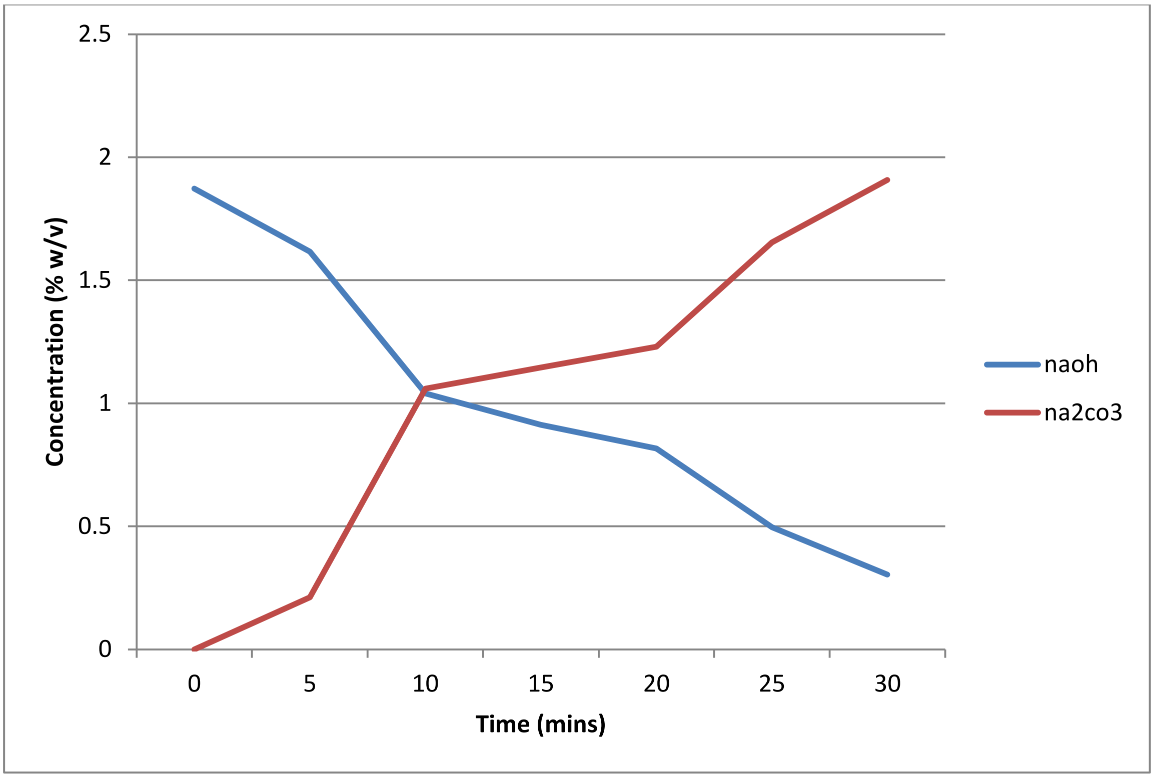


Figure 37: Graph of Concentrations of NaOH and Na_2CO_3 with Cleaning Time throughout Fermentation Vessel Clean Without Additional NaOH Dosing

In order to achieve the suggested chemical limits, the tank should be made up to 1.1% as it currently is, and controlled by monitoring pH and conductivity to ensure that the caustic does not drop below 1.1% w/v (1.1 instead of 1.0 to allow for error and system delays, but ensuring that sufficient caustic is present), and the sodium carbonate does not exceed 9% w/v.

4.6.8.3 Draining

When cleaning, water is consistently sent to the drain and not collected at any point. This was discussed in the benchmarking project, and has scope for the potential to save some money but is outside the remit of this particular project.

When the sodium hydroxide is dosed throughout a clean; if the dilute tank was already full, then there will be too much dilute sodium hydroxide to be returned to the tank at the end of the clean. Due to this, the system has been observed draining sodium hydroxide once the tank is full at the end of a detergent cleaning step. This drained sodium hydroxide will potentially be on specification and drain times have been observed as taking as long as 5 minutes to remove the residual sodium hydroxide at a flow rate of 470hl/h which results in a total of approximately 40 hl going to drain. Based on the figures calculated in the Benchmarking project (£0.01 per litre of made up solution, this provides an additional cost of around £40 which is equivalent to half a full dilute caustic tank (at 80hl working volume). This has not been included in the overall cost analysis as it was only observed on some cleans. The project recommendations will avoid the requirement to unnecessarily drain dilute caustic which is in within specification.

4.6.8.4 Hold Times

It has been noted that when the pipes connected to the fermentation vessels are being cleaned, the detergent step goes into hold every 3 minutes. The main vessel cleans typically go into hold every 2 minutes. Overall this results in an average hold time of 50 minutes per clean. This alone accounts for approximately 20% of non-value added cleaning time of fermentation vessels, which also limits processing capacity significantly over a year and indicates inefficient production.

4.6.8.5 Spray Pattern Interruptions

Each of the fermentation vessels contains a Toftejorg rotary spray jet cleaning head. These types of cleaning head are heavily dependent on continuous flow at sufficient flow rate and pressure to ensure that full coverage of the spray pattern is achieved and so should ensure that the vessel is sufficiently cleaned all over.

An experiment was performed by sitting on top a fermentation vessel whilst it was being cleaned and monitoring the movements of the cleaning head. Each time the clean went into hold, the spray head was paused, but some movement continued with what would have had insufficient pressure and flow to clean the full surface area of the tank covered by that movement. It is uncertain how much of the spray pattern coverage would be missed and what the overall impact of this would be, but it is likely that it will have a negative effect on the vessel cleaning and goes against the cleaning head manufacturer recommendations.

A recommendation for a future research project is to investigate what the impact of the spray pattern interruptions is. Removing hold times will prevent these interruptions from occurring in the meantime, and will also reduce the impact of wear and tear on the CIP supply pump from continuously stopping and starting with a large flow rate.

4.7 Conclusions

The following conclusions have been formed as a result of this investigation:

- Sodium hydroxide cleans effectively at values from 1% w/v but cleaning effectiveness does not increase at values greater than this.
- Manually made up sodium hydroxide and supplier sodium hydroxide containing sequestrants of gluconates and phosphonates showed no significant variation in cleaning results.
- Sodium carbonate cleans slightly at values of 2-4% w/v but not sufficiently to be used to cleaning brewery soils alone.
- Sodium carbonate appears to assist sodium hydroxide with cleaning between 2 and 4% w/v but at values greater than that it appears to have an inhibiting effect on cleaning abilities.
- Sodium carbonate appears to prevent effective cleaning at values greater than 9% w/v.
- The limits to clean effectively should be >1.1% w/v sodium hydroxide and <9%w/v sodium carbonate.

- Conductivity alone is not a good measure of the level of sodium hydroxide and sodium carbonate present and false readings may occur from the current set up.
- Conductivity and pH together provide a good source of information on the levels of sodium hydroxide and sodium carbonate present, as well as increased confidence that sufficient cleans are being performed.
- The pH and conductivity readings for the new chemical limits should be >pH 13 and >60mS but <80mS respectively.
- Site data has verified the pH and conductivity theory developed from this investigation.
- Implementation of the results of this project will save around £959,000 per annum in operational costs, and £330,000 in capital.
- Improvements to the system can be made by removing hold times to reduce impact on the pump and cleaning head spray pattern interruptions.
- There is too much residual carbon dioxide to remove with a sacrificial sodium hydroxide step per clean.
- Dosing is required throughout a clean to reach levels of more than 2.25% sodium carbonate.
- The cheapest solution would be to completely eliminate carbon dioxide from the equation or use an alternative detergent which is not degraded by carbon dioxide.

4.8 Recommendations

4.8.6 Changes to Chemical Limits

The sodium carbonate limit should be increased to 9% w/v and it can be ensured that effective cleans are maintained and a saving of £59,000 per annum would be made.

4.8.7 Set Up Modifications and Control

To optimise the set up so that hold times can be reduced, a pH probe should be put online near to the conductivity probe on the CIP return. On changeover from the pre-rinse step to the detergent step, the caustic should be able to flow around the equipment and back through the return to drain (as it currently does), until the conductivity and pH show that it is on specification. Then the drain may be closed and also the dilute tank should be closed at the top, so that the detergent is recirculating through the loop around the tank. See figure 38.

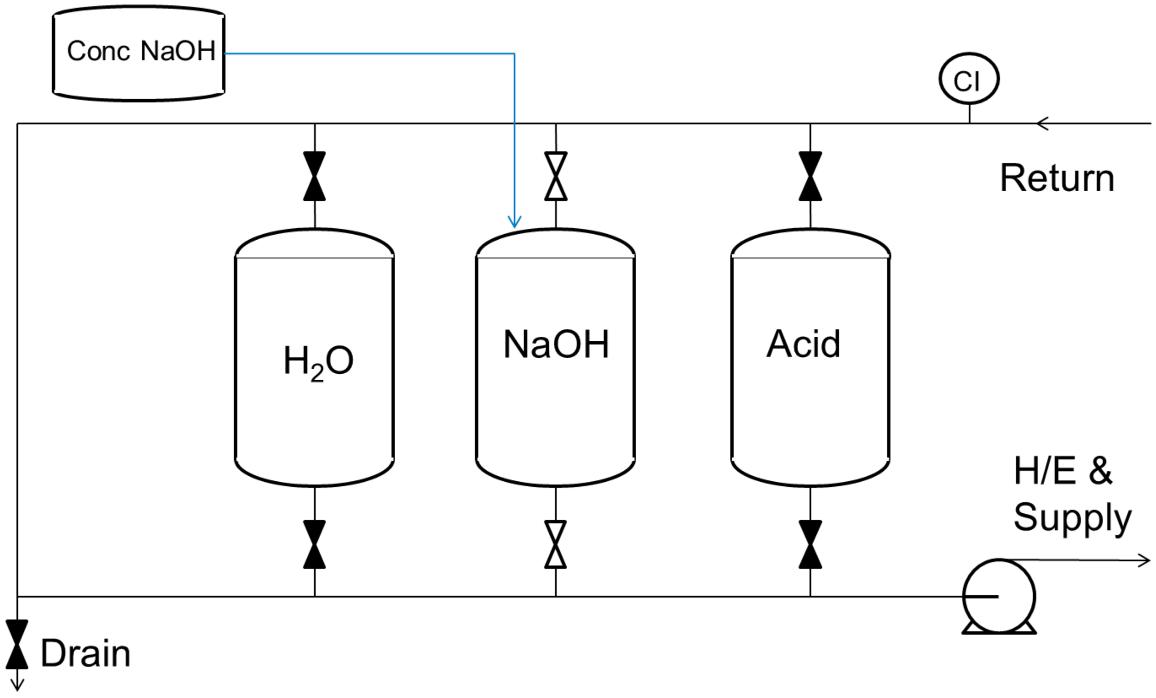


Figure 38: Schematic of Optimised CIP set up

This cleaning procedure should be controlled by the use of the conductivity and pH probes to ensure that the detergent remains within the specifications which have been determined based on the work completed throughout this chapter. This can be done by the use of continuous online control via the SCADA, ensuring that the caustic is maintained at 1.1% w/v and the sodium carbonate does not exceed 9% w/v. An additional line from the bulk caustic tank to the mains pipework which recirculates around the dilute tanks will be required. When caustic is required to be dosed to maintain the caustic concentration, the bulk caustic can be added directly to this line (whilst monitoring the flow so that the volume added to the line is known).

The concentration of sodium carbonate present may be monitored by the use of an algorithm which calculates the quantity of sodium carbonate which has been formed, based on the initial concentrations of sodium carbonate and caustic present at the start of the cleaning step, the volume of concentrated caustic which has been added throughout the step, and the stoichiometry of the chemical reaction which is taking place.

Once the recirculating detergent has reached the maximum of 9% sodium carbonate, then the drain valve should be opened and the recirculation loop refilled with fresh dilute caustic from the dilute caustic tank. The valve at the bottom of the dilute caustic tank should remain open throughout the entire detergent cycle, so that once the drain has opened; fresh dilute caustic will be drawn immediately to replace the missing fluid. Equally if there is an imbalance (which is highly likely) between the CIP supply pump and the scavenge pump at the bottom of the fermentation vessel, then there will not be an issue with the CIP supply pump running dry as the dilute tank will effectively behave like a large dead leg.

This set up will result in the removal of hold times, avoid contamination of fresh dilute sodium hydroxide with sodium carbonate, reduce cleaning head spray pattern interruptions, reduce fresh caustic going to drain at the end of a detergent cycle, reduce the load on the CIP supply pump from stopping and starting continuously, and reduce the dependency on offline titrations.

All other fermentation area cleans should be performed as normal (with the current CIP equipment and control set up), but with the increased sodium carbonate limit.

4.8.8 Future Work

The following key areas are recommended for future work as an outcome of this investigation:

- Future work for this project involves implementation of the outcomes on site to provide savings to Heineken. This can be done by using the stakeholder overview to achieve the most efficient impact. The work may potentially be implemented on other Heineken sites to provide further savings to the company.
- Saturation points of sodium hydroxide and sodium carbonate combined will be an interesting recommendation for future work to determine if

there is a relationship between this and cleaning efficacy of the detergent.

- Investigate the impact of Toftejorg spray head interruptions throughout cleaning procedures to quantify the inhibition to the ability of cleaning the complete surface area with the standard that has been set out by the cleaning head manufacturers.
- Investigate alternative methods of elimination of carbon dioxide or alternative cleaning detergents which will not react with carbon dioxide for a long term cost effective solution. One option for this includes purging the fermentation vessels with nitrogen to remove residual carbon dioxide. On the installation of a brand new CIP set, these would be the more cost effective options by removing the root cause of the carbonation formation.
- Investigate the possibility of capturing regenerating the sodium hydroxide for recycling, and the carbon dioxide to be recycled in the bottling plant.

Chapter 5. Cleaning Predictive Model

5.1 Problem Statement

CIP in industry often results in high levels of operational costs for food and drink companies. Associated with these costs are long cleaning times; which results in further costs associated with down time, further effluent treatment, increased water and energy consumption, and additional cleaning chemicals. In addition to this is an uncertainty as to how effectively the equipment has been cleaned once the CIP is completed, based on cleans being performed with fixed time, flow rate, temperature and chemical concentration. Fixing these parameters does not allow account to be taken of any process or product variability which will occur on a process of this nature.

Online monitoring is often neglected where CIP processes are concerned, as it is often seen as not adding value to a manufacturing process. Due to this, essential information and data is often not available. Throughout the CIP processes, exact cleaning strength, flow rates, and pressures are mostly unknown, which increases the uncertainty of the effectiveness of the clean as there is no evidence that the specifications for the fixed cleaning parameters have been adhered to. Due to this uncertainty, companies often over compensate with their cleaning parameter specifications to ensure that the equipment can be assumed to be clean. This results in additional expense to the company and it is highly likely that the equipment is being over cleaned. This represents an inefficient process.

5.2 Background knowledge

The justification for the critical research path of the EngD project was an outcome of the work of the ZEAL consortium. The key objectives of the Consortium were to improve and optimise CIP systems for the partner companies involved. The ZEAL pilot plant will be used for the majority of data collection for the predictive model work, and it was also used for much data collection on soils from products of other companies within the consortium.

Much work was done by the ZEAL partners on predictive modelling of the end point of clean, which proved to be successful. This has prompted the development of a predictive model control for clean end point as part of this EngD project for Heineken, with a view to; minimise production down time, reduce chemical, water and energy consumption, and also to reduce effluent production, given that generic procedures are employed. The outcome of this project could potentially benefit other industries as well as brewing within the food and drink, and consumer products industries.

The work done by ZEAL includes the study of various food and drink process soils and quantifications of the level of cleaning required to sufficiently clean these soils to an acceptable level (Fryer and Asteridaou, 2009). This work includes investigations of cleaning brewery soils during studies on smaller scale experiments (Goode et al, 2010), which will provide an indication of the results which can theoretically be expected when cleaning these soils on a larger scale.

The previous ZEAL work has provided useful information from the pilot plant with the instrumentation in place. This instrumentation includes turbidity, conductivity, flow rate, NIR, and temperature. This will also be useful to provide an indication of results which can be expected. Investigations were also performed where cleaning progression was monitored offline using particle counts. This proved to be a useful study which provided much additional information about the cleaning process. Due to this, an online particle counter has been acquired to investigate the benefits of incorporating this to develop a cleaning predictive model.

The control system will implement predictive modelling to data taken from the various online measurements of clean. The methodologies incorporate a combination of data fusion and data mining techniques with multivariate statistical methods to provide improved knowledge of the process and to develop a predictive model.

5.3 Current Operations

Bulmers' CIP currently consists of a towns water pre-rinse, a sodium hydroxide detergent rinse, another towns water rinse, a nitric acid sanitation step, and a final towns water rinse before commencing with production. Depending on the equipment being cleaned, the water and detergents can be heated to a maximum of 50^{o}C. Cold cleans are used wherever possible to minimise cleaning costs and environmental impact. Acid stages are all performed cold due to health and safety requirements. All tanks are cleaned cold due to undersized anti-vac valves. Without these valves, transferring from a hot water clean to a cold acid clean would result in a vacuum effect within the vessel which would result in physical damage and deformation to the tanks.

Cleans are run over empirically determined fixed time parameters for each stage dependent on the equipment to be cleaned. Assumptions are made that the cleaning time for each step is sufficient to complete the clean properly. Initial trial and error experimentation was performed to determine the length of the cleaning time parameters. The equipment is checked regularly for microbial contamination to ensure that there is no microbial growth accumulating.

The process is controlled using a SCADA interface. Monitoring is performed with the use of flow switches (to ensure flow is present), thermocouples (for temperature monitoring), conductivity probes (to monitor the chemicals and concentrations present whilst in CIP), and level alarms (to ensure that there is no pooling in the bottoms of the tanks). Automated process control is used for the pump flow rates, the opening and closing of automated valves, temperature of cleaning fluid, and chemical dosing due to conductivity requirements.

Cleaning confidence is based on empirical results and weekly micro scores based on routine testing. Microbial growth often does not show up in the weekly scored until the growth has built up sufficiently that it has caused a contamination issue throughout the equipment. Due to this and the inability to physically and visually monitor the results of each CIP process, there is always a level of uncertainty as to whether each clean has thoroughly cleaned the equipment. The level of process monitoring also restricts the ability to measure specific cleaning costs as there is no direct measure of flow rate, steam usage, energy usage, and chemical waste production.

5.4 Project Objectives

The objectives of the project were to:

- Replicate a realistic cleaning scenario with soils representative of those which are commonly found in a brewery.
- Develop an effective SOP and experimental design to maximise the output of the investigation.
- Determine which online measurements are useful and suitable for the online monitoring of CIP.
- Collect sufficient experimental data to develop a predictive model based on least squares methods with the optimal online sensors.
- Validate the model and determine the transferability between products and equipment types.
- Trial a risk based optimisation strategy prototype with scale up of the design to be implemented on site
- Develop a control interface with current Heineken process control software
- Perform final testing of the developed protocols

5.5 Methodology

5.5.1 Fouling

Achieving consistency of fouling was a time constraint to the project. The fouling used was required to represent the fouling which can be found on Heineken sites after production has been completed. This fouling does not come off easily with cold water, and beer soils would be considered to be a type two soil complexity (Goode et al, 2010). Beer bottoms were used from the John Smith's Brewery in Tadcaster. The beer bottoms are a result of finings being added to a maturation vessel after fermentation has been completed. This causes all of the proteins and yeast to accumulate and sink to the bottom of the vessel. This resulting slurry is then taken and pressed to extract any remaining alcohol from it for re-working into the process to minimise waste. The remaining pressed bottoms then goes to a waste slurry tank to await removal for recycling into yeast extract food products as another waste minimisation process. The composition of these beer bottoms can be seen in appendix 2. It is this remaining pressed bottoms slurry which has proven to give suitable fouling under worst case scenario cleaning conditions. The worst case scenario soil has been selected to get an idea of the most complex cleaning scenario. Once a model has been developed for predicting these clean types, further work can be performed to ensure that the model can be developed to predict cleaning end point for other Heineken soil types.

Stainless steel 316l pipes of 2 inch diameter and 1m length were required to be fouled consistently and throughout the entire internal surface area of the pipes. This was to ensure repeatability of results and comparability when cleaning conditions are changed. To enable this, a rotational device was developed to ensure that the inner surface of the pipe could be completely and evenly fouled with a fixed quantity of beer bottoms. Multiple pipes were fouled in advance of cleaning trials to ensure that the pipes were dried sufficiently before performing the cleaning experiments.

A domestic fan was placed at the end of the open ended pipes to blow ambient air across the inner surface area of the pipe and enable the water to evaporate at an increased rate. A standard operating procedure for the use of the rotational device has been developed to ensure the consistent fouling of each pipe, and can be found in appendix 21.

5.5.2 Equipment

Experiments were performed on a pilot plant at Birmingham University for improving cleaning in place research and associated processes. The plant is designed to be flexible to suit a variety of companies within the ZEAL consortium to perform research on various soils, instruments, configurations of pipework, and equipment.

For the Heineken predictive model experiments, a 1m long, fouled, stainless steel pipe of 2 inch diameter was used in the test section area. Chemical solutions were made up from Diversey supplied sodium hydroxide solutions (containing sequestrants in addition to sodium hydroxide. See MSDS in appendix 20) in one of the water tanks shown (also referred to as tank 23 throughout this section of the thesis), and the plant configuration was set to recirculate the fluid via T23, rather than going to drain. A diagram of the pilot plant can be seen in figure 39 taken from Martin et al (2013).

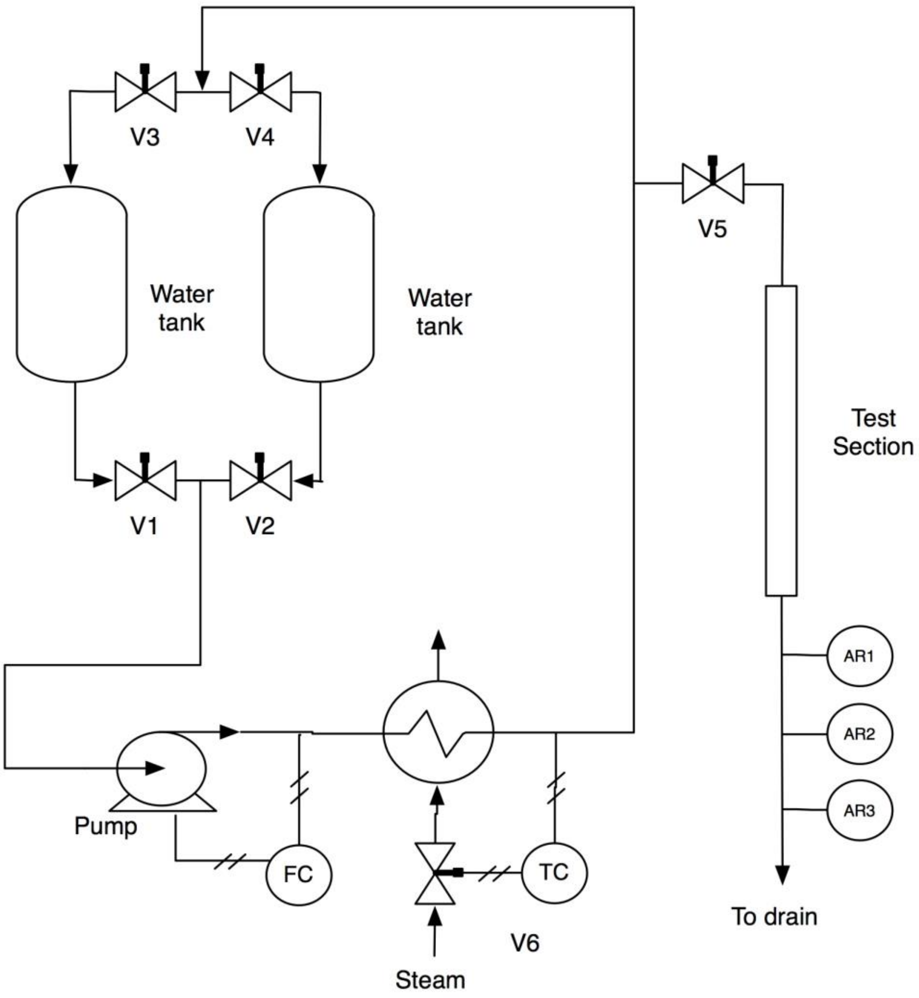


Figure 39: Birmingham University Pilot Plant (Martin et al, 2013)

Note that the configuration is set up for the trials in Martin et al's investigation in the diagram, and is for illustrative purposes of the idea of the plant only, and not the experimental configuration of the predictive cleaning model investigation.

The process pumps were capable of delivering flows in the range of 0 - 3.5 $m.s^{-1}$ in 2 inch diameter pipe. Cleaning could be carried out at temperatures from ambient up to 80°C. The two water storage tanks have volumes of $0.75m^3$

The heat exchanger in place is for recirculating cleaning solutions from T23 until they are heated to the desired cleaning temperature, before recirculating around the test section pipework for fixed time periods, and the test section monitored for cleanliness after.

There are 21 test section pipes which will be pre-fouled in advance. These pipes will be connected into the pilot plant using tri-clover clamps and o rings, and cleaned with varied parameters as specified in the given experimental designs. Data is then recorded from the instrumentation in place onto the laptop used to control the pilot plant whilst each pipe is cleaned. This data is saved and taken with a USB stick at the end of the experimental trials.

The instrumentation to be used includes; turbidity probes, conductivity probes, temperature probes, flow meters, and a particle counter. Due to there being less available test section pipes than experimental runs per visit to the pilot plant, and the cost implications involved with purchasing more pipes, necessitated pipes which are used during the early stages of the trials to be re-fouled during the rest of the trials.

5.5.3 Experimental Design

Experimental designs include variations of cleaning temperature, chemical concentration and flow rates to be investigated. On the first attempt of obtaining experimental data from the pilot plant, there were issues with; reaching the turbidity probe saturation point, foaming of caustic on recirculation, and air bubbles in the system which affected the cleaning flow rate. The operating procedures had to be trialled, and modifications made where necessary to tackle these challenges. The detailed, improved standard operating procedures for each of the variables to be investigated can be seen in appendices 22, 23 and 24. Further explanations of selections and modifications to the procedures are discussed in section 5.6 with the first sets of experimental results obtained.

The experimental design for the following set of trials included two different levels of fouling, four different flow rates, and four different levels of chemical concentration. The design may be seen in appendix 28.

5.5.4 Experimental Procedures

The standard operating procedure (SOP) for the rotational fouling device may be seen in appendix 21. This is consistent for every pipe that is fouled, although the volume of fouling inserted into the pipe may vary depending on the required volume within the experimental design.

The SOPs for the pilot plant experimental runs may be seen in appendices 22, 23, and 24. There are three separate SOPs depending on the requirements of the runs to be performed and the experimental design. The SOPs cover a run at ambient temperature with no chemicals, a heated run, and a run which requires chemical cleaning solution to be made up. These runs are repeated as required until the experiments are complete, and the shutdown procedures on the SOPs are followed.

After each experimental run the pipes were visually inspected for visible levels of cleanliness. Visually clean has been selected as the acceptable clean level as in a real CIP on site, as there will be a sanitation step after the detergent step to ensure that the equipment is cleaned for microbial growth to an acceptable standard and water rinse stages after these steps. If there was a light residue or obviously loose particles remaining, the pipe was assumed to be clean based on the fact that there would always be a water flush after the detergent step which would remove any of these loose particles. Photographs were taken after each run to provide evidence for the level of cleanliness achieved for monitoring and analysis of the results.

Throughout each experimental run, samples of the cleaning solution were taken before and after. This was to perform titrations to verify that the correct strength of cleaning solution had been made up and to take offline measurements of the particle counts to verify the online particle count readings.

5.5.5 Health and Safety

The risk assessment for the fouling device and for the experiments performed on the pilot plant, along with student assistant safety briefs may be seen in appendices 25, 26 and 27 respectively .When performing the experiments, it was mandatory to wear the correct PPE; this included safety goggles, gloves, and a lab coat at all times when they plant was in operation. When handling concentrated caustic to make up the cleaning solutions, a visor and protective apron was required, in addition to the standard PPE.

5.5.6 Stakeholder Overview

It is essential to identify key stakeholders early on to ensure maximum awareness and support of the project, which speeds up the process of completion, and benefits the project by having a greater network of help available when challenges arise. On completion of the final project it is desirable to arrange for implementation of the outcomes and achieve the greatest impact with minimum effort required. The stakeholder overview enables the outcomes to be presented and distributed effectively. See figure 40.

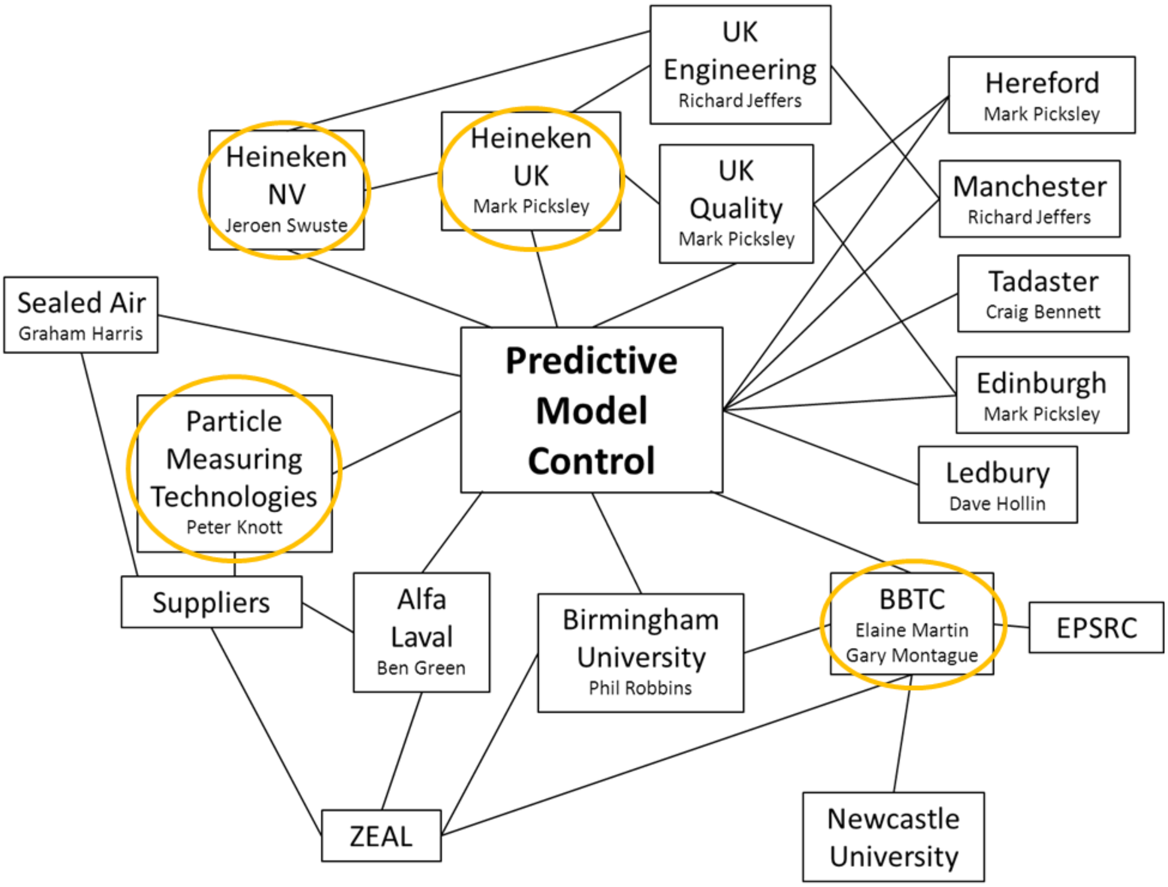


Figure 40: Stakeholder Overview for Cleaning Predictive Model Investigation

The overview shows that contacts in all UK sites for Heineken as well UK quality and global headquarters have a vested interest in utilising the outcomes of the project. There are also several suppliers who are involved to benefit themselves with the knowledge gained from the investigation, as well as providing us with the necessary materials and equipment to carry out the trials. In addition to this, there are two academic institutions, a research council, and a special interest research group involved. Together, all of these stakeholders are essential to the success of this project and the future implementation to achieve cost benefits.

5.6 Preliminary Experimental Data Analysis

The trials investigated the removal of thick (300 ml) beer bottoms fouling with cleaning solutions of different sodium hydroxide concentrations and flow rates. These experiments were critical to the success of the project, to perform the preliminary data analysis and gain an improved understanding of the process and interactions between process variables. These experiments were also important for developing the experimental and standard operating procedures to enable more effective trials whilst progressing through the project.

Four days were spent gathering experimental data at the pilot plant. The instrumentation used provided data on turbidity, conductivity, temperature, flow and time.

The initial experiments were performed over days one and two, and were carried out using route 5A which is a recirculation loop which is pumped around the test section only, not via any of the tanks or to drain. The system was filled by sending the contents of tank 23 to drain via the test section with a clean pipe in the test section. Then stopping, draining the system enough that the test section pipe was empty, and replacing the pipe with a fouled one.

The first set of results and observations are shown in table 5 below. Flow rates were varied between 10, 12, and 14 m^3/h, and caustic solution strengths were varied between 0, 0.75, and 1.5% w/w. “Max Turb Time” refers to the time taken for the turbidity to reach the maximum value for that experimental run.

Table 5: Set 1 of Preliminary Trial Results

Day	Run	Temp (^oC)	NaOH Conc (% w/w)	Flow Rate (m^3/h)	Repeat	Max Turbidity (FTU)	Max Turb Time (s)	Total Time (s)	Comments
1	1	20	0	14	1	520	200	363	Not clean but incorrect flow rate. Thick deposit trail remaining.
1	2	20	0	14	2	540	250	337	Clean.
1	3	20	0	14	3	530	45	187	Not clean. Thick deposit trail remaining. System primed from this run on.
1	4	20	0.75	14	1	550	45	135	Clean with suds remaining. Clean after manual rinse.
1	5	20	0.75	12	1	545	30	132	Clean.
1	6	20	0.75	10	1	550	40	121	Not clean. Fouling layer remaining on half of pipe.
1	7	20	1.5	10	1	550	40	79	Almost clean with some residue.
1	8	20	1.5	12	1	550	25	120	Still very dirty.
1	9	20	1.5	14	1	535	35	129	Clean. Heavy fouling to begin with.
2	10	20	1.5	14	2	550	35	121	Still very dirty.
2	11	20	1.5	12	2	550	35	124	Still very dirty.
2	12	20	1.5	14	3	550	55	309	Still dirty, even with longer cleaning time.
2	13	20	0	14	4	550	65	335	Clean.
2	14	20	1.5	14	3	190	?	300	Clean. Recirculation through tank 23 from this run.
2	15	20	1.5	14	4	190	?	308	Clean. Tank mixed whilst cleaning which increased foaming.

The turbidity initially appeared to increase and decrease in a plug type fashion and then eventually level out at a maximum turbidity. This could also be seen through the clear section of the pilot plant where large lumps of fouling would appear to pass until eventually the water all looked consistently dirty. This is as would be expected when the liquid was being recycled around the dirty test section piece until it was mixed with nowhere to drain. See figure 41 which shows the turbidity readings of run 1 and illustrates this point.

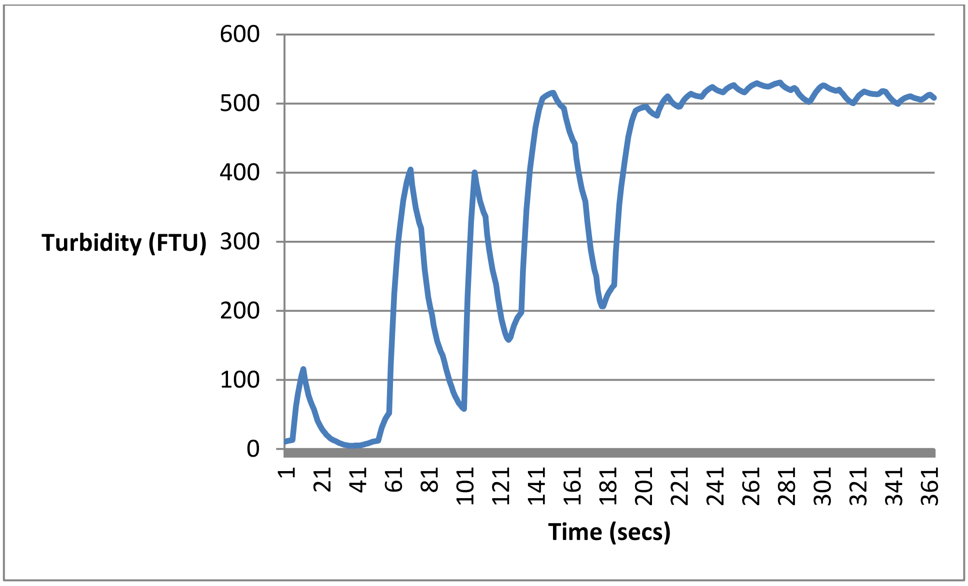


Figure 41: Turbidity v Time for run 1 of Experimental Set 1 of Results on the Pilot Plant

The first run did not clean the test section pipe, and after the second run it became apparent that the flow rate was not reaching the specified 14 m^3/h until very late on in the clean. See figure 42 which shows the flow profile of run 2.

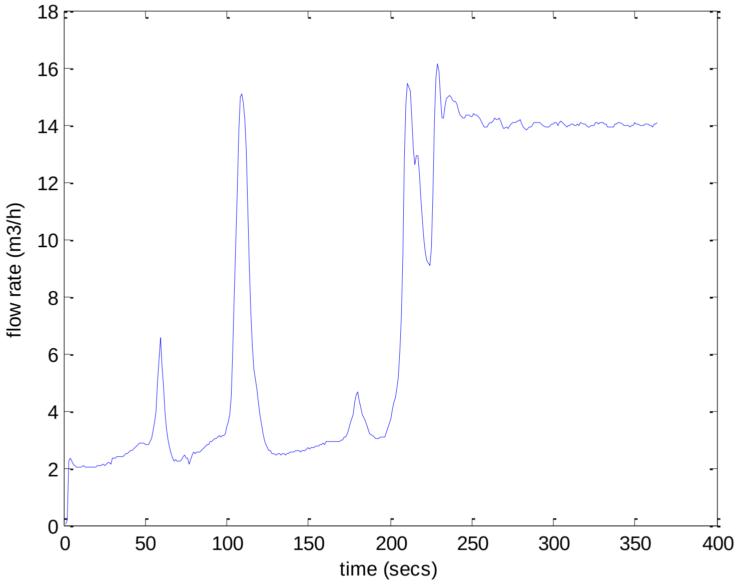


Figure 42: Flow Rates for Run 1 of Experimental Set 1 of Results on the Pilot Plant

During the first two runs, the pump was operating very loudly, so it is believed that there was sufficient air within the system to cause cavitation in the pump and not enough liquid for the correct flow rate to be achieved. From run 3 onwards, the system was then primed by topping up the pipework with the fouled test section piece in place, up to the top until no air bubbles were present. This was necessary to reduce potential damage to the pump by cavitation as well as to achieve the correct flow rates for the experiments. Figure 43 shows the flow profile for run 3, and that priming the test section was successful in achieving the correct flow rate within a shorter time.

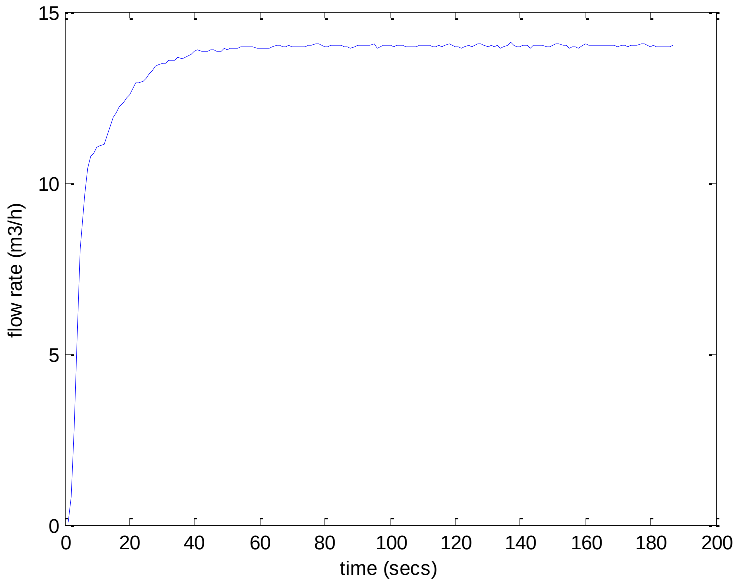


Figure 43: Flow Rates for Run 3 of Experimental Set 1 of Results on the Pilot Plant

With the air removed from the system, the turbidity appeared to level out much faster with less of a plug effect, other than in the initial few seconds of the clean. See figure 44 of the turbidities of run 3.

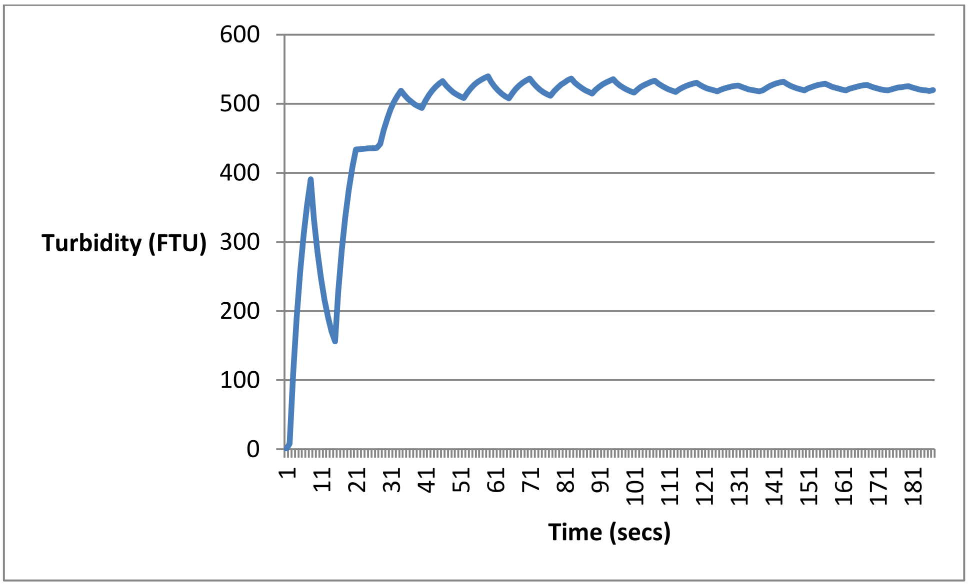


Figure 44: Turbidity for Run 3 of Experimental Set 1 of Results on the Pilot Plant

The next run used caustic at 0.75 % w/w. The test section at the end was full of caustic suds, but by performing a manual rinse on the pipe at the sink using a measuring jug, the suds easily washed away. A typical plant cleaning scenario would end in a final water rinse which would be drained. At this point it was decided that the best representation of the cleaning would be to lightly rinse through the pipe after each experimental run which will also account for any caustic suds or residue remaining due to the recycling of dirty water rather than a fouled pipe.

Figure 45 shows the turbidity for run 4 using the caustic. It shows that the turbidity values rise much more quickly, and no plug effect could be seen. This could be due to the fact that the saturation point of the turbidity probe is at 550 FTU.

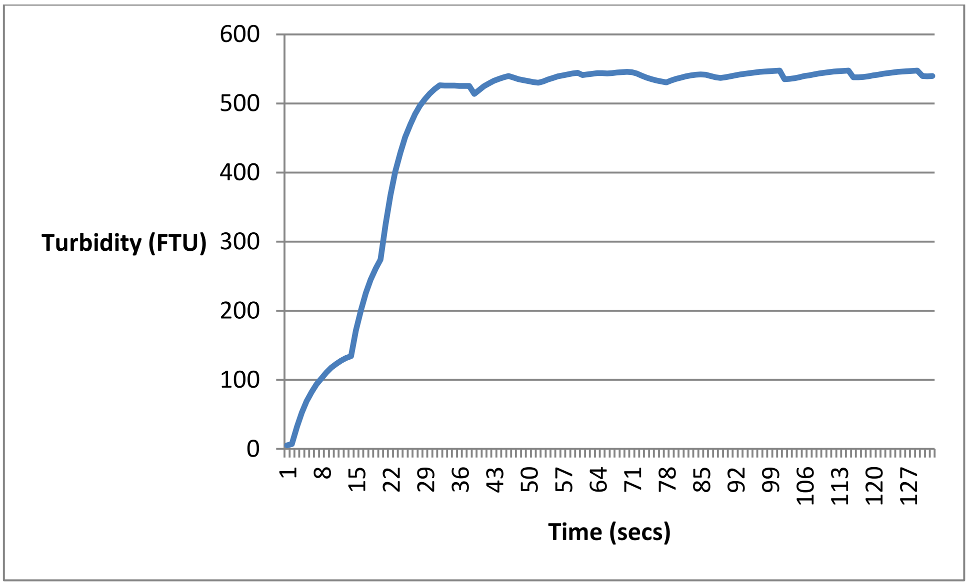


Figure 45: Turbidity for Run 4 of Experimental Set 1 of Results on the Pilot Plant

All runs with caustic showed the same turbidity behaviour as run 4 for the remainder of day 1.

On day two, additional pipes were required to be re-fouled to perform more experiments as there were only 21 pipes fouled in advance. A fan was used overnight to increase the drying speed and ensure that all fouled pipes were dry. This does imply that pipes fouled from day 2 onwards were potentially fouled more heavily than those which were fouled in advance. The pipes also appeared to be more difficult to clean based on the comments for day 2 runs than from the first set of experiments, which would back up the theory of heavier fouling due to the fan. This will cause variation in the results, although the clean times were not consistent for each experimental run, which will also have impacted upon the results.

The turbidities of the cleans on day 2 behaved in the same way as those on day 1, but looking at the results, it became apparent at this stage that the probe saturation point was at approximately 550 FTU. This also appeared to be a point at which information about what was happening with the turbidity during the clean would be most helpful. A method to avoid this happening was required, so the last two runs of day two were done using route 9 which recirculates liquid from tank 23, via the test section, and back into tank 23. The idea was that having a larger body of fluid to recirculate would reduce the concentration of fouling per unit volume, and so increase the overall turbidity at a slower rate. Priming of the test section was no longer required for this cleaning method as the pressure from the head of the tank would ensure that air bubbles in the system would be minimised.

Figure 46 of the turbidities of run 14 shows that the maximum turbidity reached throughout the clean was much lower at approximately 195 FTU. This provided more information about what was happening with the turbidity of the clean, however, the turbidity appeared to behave very differently to the previous experimental runs. It would be expected that the turbidity should continue to level out as the fouling becomes mixed into the overall body of fluid. A potential explanation of the increased turbidity spikes is due to foaming of the caustic as it is recirculated into the tank and bubbles as it drops from the pipe to the liquid level of the tank.

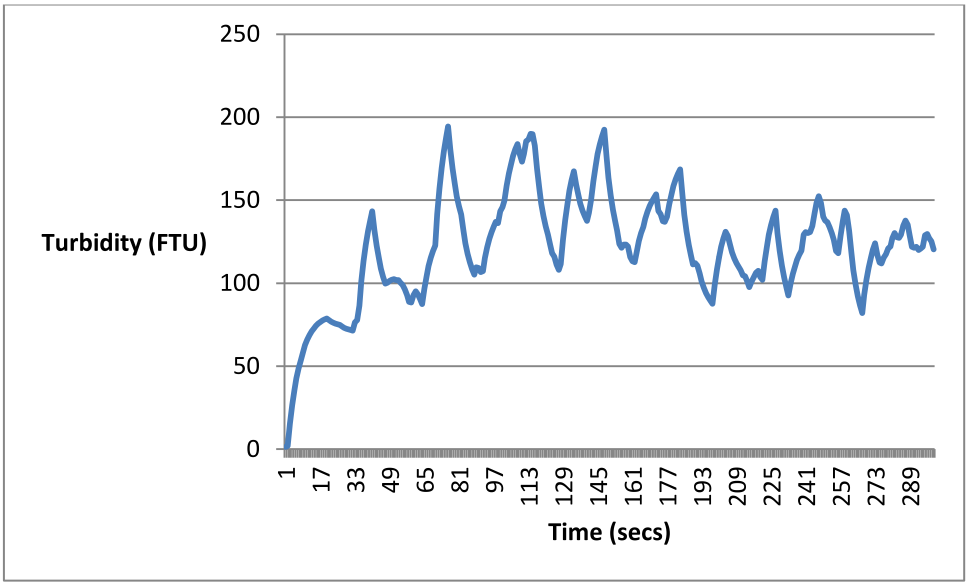


Figure 46: Turbidity for Run 14 of Experimental Set 1 of Results on the Pilot Plant

Run 15 behaved in the same way as run 14, with mixing of the tank performed in an attempt to minimise the caustic foaming, but this had the opposite effect.

After the modifications to improve the standard operating procedure were made, it was decided to develop a new experimental plan with a further three sets of results. The first set were completed over the remainder of day two, and varied flow rates between 10, 12 and 14 m^3/h, with no caustic and at ambient temperature. Each flow rate was done in triplicate and over a consistent time of five minutes to improve the accuracy of results. The turbidity at the start of each experimental run was then recorded so that the change in turbidity after each run could be monitored. Table 6 shows the observations made for the second set of experiments.

Table 6: Set 2 of Preliminary Experimental Results

Day	Run	Temp (^{o}C)	NaOH Conc (% w/w)	Flow Rate (m^3/h)	Repeat	Max Turb (FTU)	Start Turb (FTU)	Level Time (s)	Total Time (s)	Comments
2	1	20	0	10	1	35	0	75	315	Clean. All runs cleaned for 5 minutes after this run.
2	2	20	0	12	1	52	9.8	?	333	Clean.
2	3	20	0	14	1	84	23.2	100	301	Clean.
2	4	20	0	10	2	100	38.5	95	300	Not clean. Heavily fouled to begin.
2	5	20	0	12	2	120	51.7	70	301	Clean.
2	6	20	0	14	2	213	64.9	80	348	Clean after manual rinse. Heavily fouled to begin.
2	7	20	0	10	3	350	105.0	110	300	Not clean. Large fouling deposits remaining.
2	8	20	0	12	3	250	133.9	60	300	Clean.
2	9	20	0	14	3	350	151.5	165	301	Clean. Final turbidity 214 FTU.

One batch of a known volume of liquid in tank 23 was used for each experimental set, which consisted of 9 runs. The liquid level in tank 23 was also filled to be higher than the inlet pipe on the recirculation loop in an attempt to minimise foaming during experiments.

The turbidity of run 1 of the second set of experiments may be seen in figure 47. The turbidity now appears to increase rapidly to a peak, then rapidly drop and level out at a higher value than the starting turbidity. It is believed that the peak is the initial plug where the bulk of the fouling is removed in the first minute of the experiment, and the drop and levelling out due to the mixing of the fouling in the tank with the larger body of fluid.

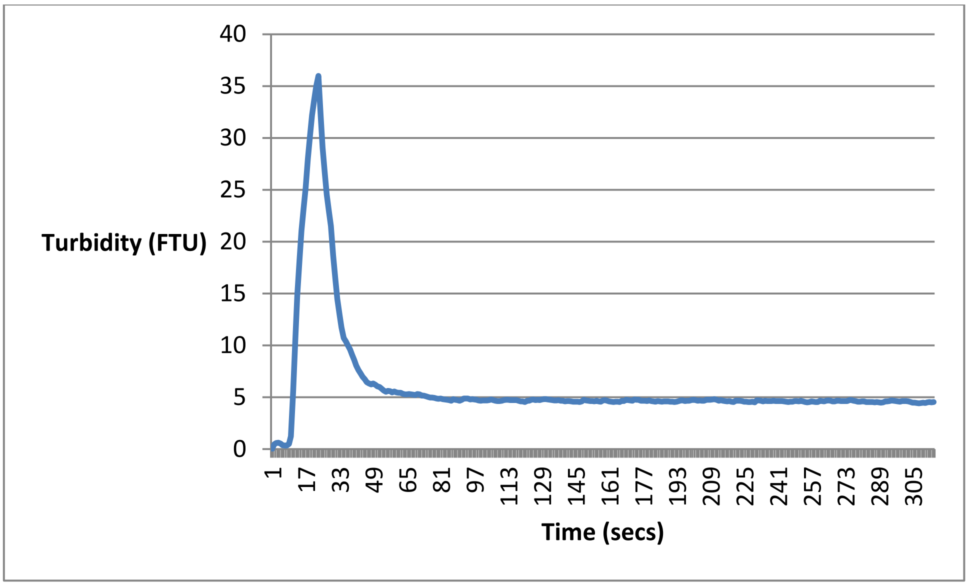


Figure 47: Turbidity for Run 1 of Experimental Set 2 of Results on the Pilot Plant

All experimental runs from the second set of experiments exhibited the same behaviour which implies that this method has improved repeatability, except for that of run 2 which can be seen in figure 48 below:

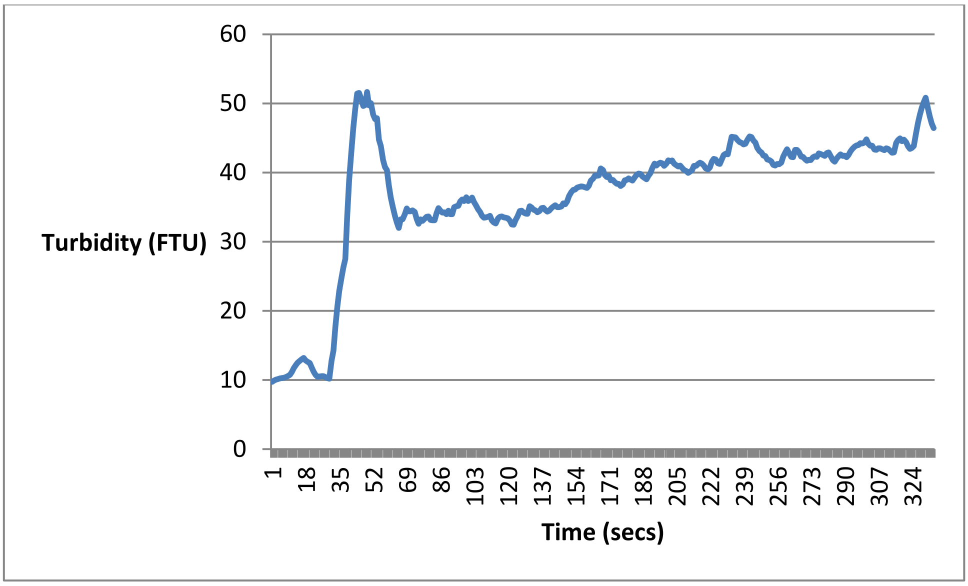


Figure 48: Turbidity for Run 2 of Experimental Set 2 of Results on the Pilot Plant

The initial plug part of the behaviour was still observed, however, the turbidity did not level out within the time of the experiment. The pipe cleaned on this run was noted as being more heavily fouled than others at the start, so potentially it took longer to clean, but the pipe was clean at the end of the run.

The pipes cleaned for runs 4 and 10 did not clean, but they were also noted as being heavily fouled, as was the pipe used for run 6 which did clean. Run 6 had a higher flow rate which would be expected to clean better. Run 7 was not heavily fouled and did not clean, but it was on the slowest flow rate which would also be expected not to clean as well. There does not appear to be an obvious relationship between the turbidity behaviour and the cleaning time at this stage, but as times are fixed, it is difficult to confirm this. An experimental plan with varied times over controlled parameters would be useful to further analyse these results.

It is unknown how the suspended fouling within the liquid affects its cleaning capabilities. Towards the end of an experimental set, as the starting turbidity increases, the cleaning fluid will have more fouling dissolved in it, which may impact upon how well it will perform at cleaning. Therefore, pipes cleaned later in a succession of experimental runs using the same batch of cleaning detergent, may be worse cleaned as a result of the increased quantity of suspended solids within the detergent. This needs to be monitored carefully in future experiments.

The third set of experiments was performed on day three, varying the same flow rates with no caustic at 30 ^{o}C. The observations made for experimental set 3 can be seen in table 7.

Table 7: Set 3 of Preliminary Experimental Results

Day	Run	Temp (^oC)	NaOH Conc (% w/w)	Flow Rate (m^3/h)	Repeat	Max Turb (FTU)	Start Turb (FTU)	Level Time (s)	Total Time (s)	Comments
3	1	30	0	10	1	37	0	120	306	Not clean. Thick residue remaining.
3	2	30	0	12	1	87	10.0	150	301	Not clean. More residue remaining than run 1.
3	3	30	0	14	1	120	31.8	120	301	Cleaner than run 1 and two with large deposits.
3	4	30	0	12	2	255	65.0	160	301	Some residual dirt. Heavy, inconsistent fouling to begin.
3	5	30	0	14	2	312	115.5	150	301	Almost clean after manual rinse.
3	6	30	0	10	2	364	140.9	190	301	Bottom half of pipe clean, residue remaining on top.
3	7	30	0	14	3	448	226.4	60	301	Bottom half of pipe clean, residue remaining on top.
3	8	30	0	10	3	445	342.6	110	304	Clean. Matlab error meant cleaning went on for longer.
3	9	30	0	12	3	500	380.2	180	301	Clean.

The turbidities began by showing similar behaviour as those for the second set of experiments. See figure 49 for run 1 of set 3. The only difference is that the drop from the peak of the maximum turbidity is more gradual for the turbidity to level out.

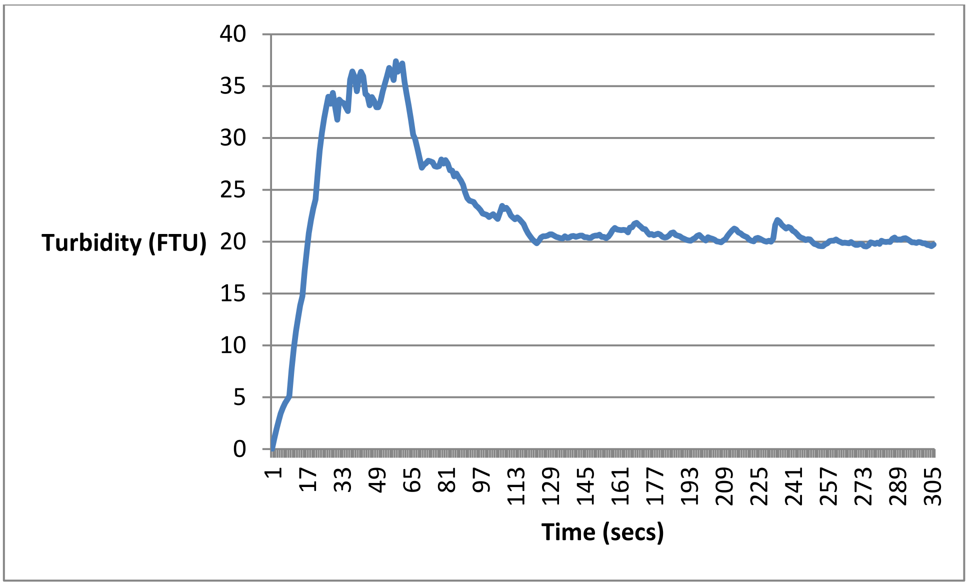


Figure 49: Turbidity for Run 1 of Experimental Set 3 of Results on the Pilot Plant

As the number of experimental runs increased, the maximum turbidity peak became less prominent, and the turbidity also did not appear to drop much below the maximum towards the end of the experimental run. See figure 50 for run 9 of set 3.

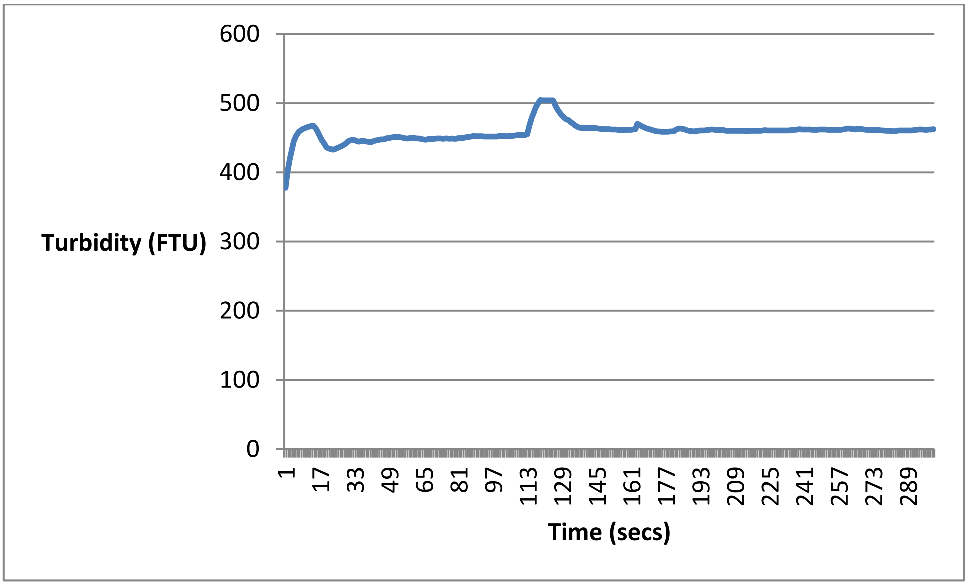


Figure 50: Turbidity for Run 9 of Experimental Set 3 of Results on the Pilot Plant

The maximum turbidities recorded for the third set of experiments were much higher than those recorded for the second set with ambient water. It is believed that this is due to the higher temperature of the water increasing the solubility of the fouling within, therefore resulting in a cloudier appearing liquid.

The pipes cleaned on day three were much more difficult to clean. This could be partly due to the pipes having a longer exposure time to the fan for drying, and potentially producing heavier fouling again, but also could be due to the cleaning water having more fouling dissolved within it, impacting on its ability to clean more pipes.

Due to the pipes not having a sufficient amount of time on the rotatory device to dry, but continuing to be fully dried with the fan once removed from the rotary device, much of the fouling is dried with a thicker layer at the bottom of the pipe. See figure 51.

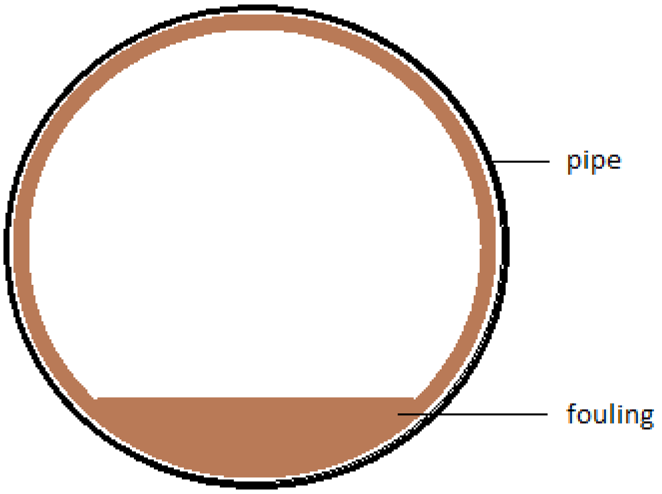


Figure 51: Illustration of Fouling Distribution in 2 inch Diameter Pipes without Rotational Device Used until Fouling Fully Dried

After observing that the fouling was forming in this way, care was taken to ensure that the line of worst fouling was placed at the bottom part of test section, and if any deposits remained it could be noted if they were from this thicker fouling as they would be in the same location of the pipe at the end of the clean.

Runs 6 and 10, were clean on the bottom half of the pipe, but not on the top (see figure 52) which is interesting as the worst fouling was placed at the bottom. This could imply that fully developed turbulent flow was not reached on these experimental runs, although the flow data does not appear to exhibit differences to the other runs. These runs were also of the highest flow rate, and no observations were made of cavitation being heard from the pump.

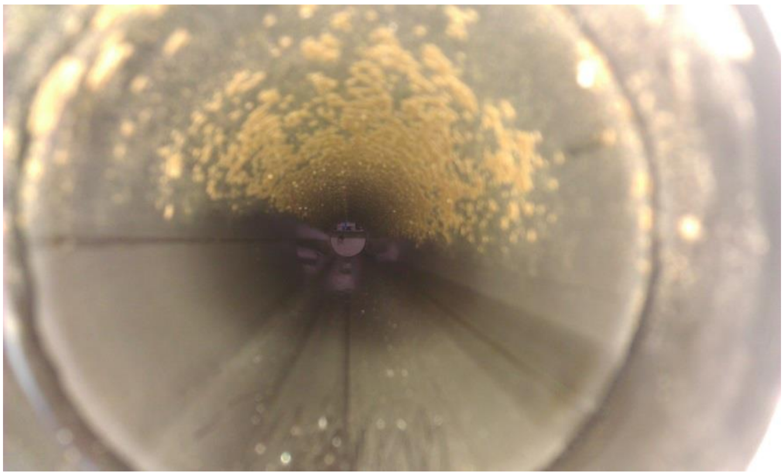


Figure 52: 2 inch Diameter Pipe with Top Half not cleaned after Experimental Cleaning Run

On run 8 there was a Java runtime error which caused Matlab to crash, so an emergency stop was required to switch off the pump, but some of the valves failed to close. This resulted in the pressure from the head of the tank continuing to push liquid through the test section to drain until the drain could be closed manually which meant that run would have been exposed to a longer cleaning time than the data recorded.

Overall, the third set of experiments generally showed that a higher flow rate cleans better, and that warmer water does not appear to clean as well as ambient water on a recirculation system.

The fourth set of experiments were performed on day four, with the same flow rates varied at ambient temperature with 1 % w/w caustic solution. The fouled pipes would also have had a longer time of exposure to the fan for drying, so cleaning was expected to be more difficult. The observations made can be seen in table 8.

Table 8: Set 4 of Preliminary Experimental Results

Day	Run	Temp (^oC)	NaOH Conc (% w/w)	Flow Rate (m^3/h)	Repeat	Max Turb (FTU)	Start Turb (FTU)	Level Time (s)	Total Time (s)	Comments
4	1	20	1	10	1	27	0	180	300	Not clean. Thick trail residue along bottom.
4	2	20	1	12	1	54	5.1	150	300	Not clean. Deposits remaining on bottom.
4	3	20	1	14	1	81	6.1	125	300	As run 2. Cleans run for 10 minutes after this run.
4	4	20	1	14	2	94	15.8	220	599	Clean after manual rinse.
4	5	20	1	10	2	80	25.5	160	606	Almost clean. Two small deposits remaining.
4	6	20	1	12	2	80	37.7	200	601	Clean with the exception of one deposit. Matlab error meant clean ran for longer.
4	7	20	1	12	3	172	49.5	125	601	Mostly clean with slight residue.
4	8	20	1	14	3	301	76.1	100	600	Clean after manual rinse.
4	9	20	1	10	3	260	108.9	130	600	Clean with some small deposits.

After first two experimental runs, the pipes were not completely clean, so it was decided to increase the cleaning times to 10 minutes for this final set of experiments to see how this effected the cleans.

Most of the pipes were cleaned, but with deposits of fouling remaining on bottom. This is believed to be more of an issue due to the inconsistent fouling trails which were left at the bottom of the pipes, as the rest of the pipes were clean. See figure 53.

Figure 53: 2 inch Diameter Pipe with Thick Deposit Trail Remaining after Clean

The turbidity behaviour appeared to be the same as that observed with experimental set 2, with a rapid maximum peak, then a rapid drop and the turbidity levelling out for the remainder of the clean. See figure 54 for run 9 of set 4.

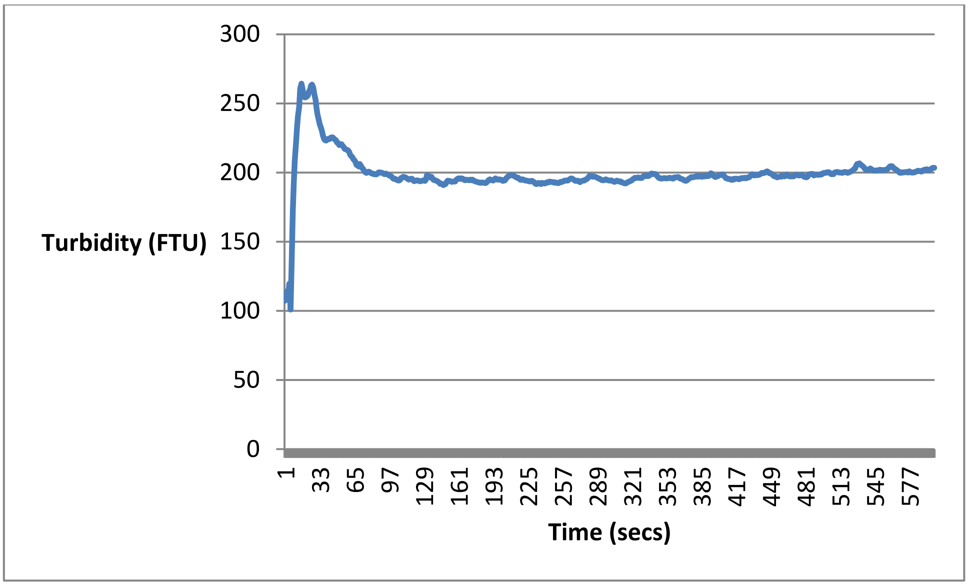


Figure 54: Turbidity for Run 9 of Experimental Set 4 of Results on the Pilot Plant

Experimental set four showed that caustic appears to clean better than water, which is as would be expected although the runs did have longer cleaning times. These results cannot be relied upon quantitatively, due to this variation in cleaning time (as it was supposed to be fixed the same as the other experimental runs), however; qualitatively, they do give an indication of what to expect in future. The maximum turbidity for caustic at the end of the set of experiments was lower than for hot and cold water which would be expected as it would create the least soluble solution of the three cleaning liquids.

By the end of the experimental trials, the standard operating procedure of the pilot plant for this soil had been improved, as well as the fouling methods to produce consistently fouled pipes. The final sets of experiments were also showing good repeatability.

5.7 Detailed Experimental Data Analysis

5.7.1 Further Trial Information

Following on from the trials completed for the preliminary data analysis, further trials were required to broaden the depth of information on the available process variable parameters and to increase the range of information with the use of an online particle counter. The same experimental method can be seen in the SOP which was developed as a result of the first trials, but with the additional use of the particle counter which is run alongside the equipment.

Three further sets of trials were performed over two separate periods. The first set was over two weeks in July 2013, with the first use and set of data from the online particle counter. The latter two sets of trials were performed in spring and summer 2014, however both sets of trials were unsuccessful so the data collected was not sufficient to be used within the detailed experimental analysis. The experimental design for this data may be seen in appendix 28.

The trials in spring 2014 were unsuccessful due to a technical issue with the particle counter. The self-help and supplier instructions were followed in an attempt to rectify the issues, but these attempts were unsuccessful. There were also issues with data storage on the particle counter using a flash card and card reader. Neither the particle counter nor data storage methods were sufficiently robust for continuous industrial use. The particle counter user interface is complex and awkward to use. This would not be suitable for use in an industrial application by a process operator. There is an opportunity for improvement to the device by the supplying company. Further details of the operational observations of the particle counter are discussed later in this chapter. The trials were stopped early, and the particle counter returned for repair.

The trials in summer 2014 were intended to capture information with variations in cleaning temperature. These trials were unsuccessful due to maintenance issues with the steam supply at Birmingham University. Due to this, there was no feasible, cost/time effective manner to heat the cleaning chemical tanks up to the required temperatures. These trials were also stopped early as a result of this. Unfortunately, there was no further experimental trial time remaining on the engineering doctorate at this point. However, there was sufficient data captured in the July 2013 trials to progress with the project.

5.7.2 Data Mining of Results

The analysis of the July 2013 trials was performed by Tait et al (2013). Tait investigated data mining methodologies and developed a method to make modelling predictions for the end point of a cleaning step based on multivariate statistical process control (mSPC). This method took into account the batch nature of data collection from the process. Tait's techniques have been incorporated into the report to develop the model predictive control, and the results of the data mining methodologies have been used to investigate further into the industrial application of the model, and to develop a base cost benefit analysis and novel conclusions, based on the use of a tool of this nature for cleaning in place monitoring. This section of the report will briefly outline the work which was done by Tait, to complete the description of the project investigation.

The data collected was used to characterise two key progressive stages of fouling removal during a cleaning process. These steps involved bulk removal of the initial fouling, followed by residual removal of the remaining fouling until the process equipment is cleaned.

Pre-screening and pre-treatment techniques were used to maximise the value of the output from the data. Techniques considered included methods to account for missing data, determination of outliers, and filtering of noise from the response variables.

The results of the trials in July 2013 showed that there is a bivariate relationship between the input variables. This relationship showed that NaOH is less effective with higher flow rates, than when the flow is slower, which enables greater residence time for the NaOH to have a cleaning effect on the fouled equipment. Data from the particle counter was shown to be more informative than other data alone based on the distinctive correlation between particle count within the cleaning solution and level of equipment fouling remaining. This is what was expected based on previous trials and information provided from previous work undertaken in the ZEAL consortium.

The definition of the bulk and residual soil characteristics were able to be incorporated into the data analysis by combining the information from recorded data with basic cleaning mechanism knowledge. A two parameter curved fitting model which defines the relationship between these two fouling removal characteristics may be seen in equation 9:

$$P_f = P_{avg}\left[1-\left(\frac{t}{T}\right)^{c_b}\right]\left[1-c_r\left(\frac{t}{T}\right)^{2}\right]$$

Equation 9: Two Parameter Curved Fitting Model

Where:

P_f is the value of fitted curve at time t

$c_b > 2$ is the initial bulk removal parameter

$0 < c_r < 1$ is the residual clean parameter

P_{avg} is the average cumulative particle count over the last 10 data points

t is cleaning time

T is cleaning temperature

An ideal clean would comprise of a large bulk removal parameter and a small residual cleaning parameter.

Based on the results of the trials, it would appear that increasing the flow rate has the greatest impact on the bulk fouling removal, whereas NaOH concentration had a lower impact on this. NaOH did however require a minimum of 0.5% w/w concentration to clean effectively. This justifies the claims made in chapter 4 for the determination of the minimum NaOH concentration required to clean. This also verifies that there is no significant variation in cleaning results when using supplier made up NaOH solution with sequestrants, and manually made up NaOH solutions.

The optimal conditions to provide the best results, when accounting for the available cleaning parameters, were to have a $c_b >> 2$ and a $c_r \rightarrow 0$. Cost implications to achieve these parameters must also be accounted for, so an optimisation is required to maximise cleaning, but reduce cleaning costs, without negatively effecting cleaning.

The particle counter data may be used to provide a strong indication of the cleaning end point. Based on the process being in recirculation, the total particle count will cumulatively increase until a fixed point has been reached. It is at this point that there are no further particles being removed from the equipment fouling and adding to the particles present in the cleaning solution. Observing the data, there was some variability and a slow gradual increase in particle count once the fixed point has been reached. This is believed to be due to the recirculation of the fluid and turbulence causing further breakup of the particles in the cleaning solution, which will then show as an increase in quantity of particles, despite not having an increase in mass or volume of particles present in the solution.

There is one potential issue with this method of monitoring, as there is no way of knowing if there are fouling particles remaining adhered to the equipment surface. The only way to improve confidence of using this method is to gather further data on trials on different equipment and sites, and to monitor how clean the equipment is after cleaning, and after varied times of cleaning once the maximum particle count of the cleaning solution has been reached. This is not particularly feasible in practice, although the theory behind the predictive cleaning is interesting and holds great potential for industrial benefit, if a more robust monitoring and measurement system were applied.

5.8 Industrial Application of Results

Application of the predictive model with a user friendly control interface will enable an effective process cost optimisation to be performed on a clean by clean basis. This continuous optimisation process will minimise the cleaning time of individual equipment, whilst ensuring that quality requirements and effective cleaning is maintained. As a result of this, costs for down time, cleaning time, chemical, water, energy, and effluent will be reduced. The environmental impact of the factory will also be reduced as a result of reducing energy consumption and chemicals going to effluent.

If this were to be developed into a marketable product, this would allow companies to clean their equipment with improved confidence in the quality of their cleaning. This will also remove the guesswork from empirical determination of cleaning process parameters. There is a high level of risk around these methods due to the uncertainty that the equipment is being cleaned effectively. It will not be apparent that cleaning parameters are insufficient until a microbial contamination issue arises; which results in further costs, product losses/reworks, and down time for maintenance/deep cleaning with stronger chemicals to resolve the issue.

To develop a product which is suitable for industrial requirements; there are some initial needs which require addressing. The particle counter itself is not sufficiently robust for industrial application. There were several issues which occurred within the relatively small sample of experimental trials. These issues were related to the internals of the equipment becoming blocked, and also saturation levels of the device being reached. An appropriate device cleaning/maintenance method requires developing to prevent reoccurrence of this. The device also needs to be appropriately sized for the size and quantity of particles which will be passing through it, in addition to ensuring that it is fit for purpose when considering the abrasive nature and concentrations of the chemicals which flow through it. The device is currently operated at 10ml.min^{-1} which is far too low when considering industrial flows at Bulmers of approximately 7hl/min^{-1}. The attachments to the equipment for sampling need to be adapted to account for the pressure within the system and instil confidence in the operator that they will remain securely attached throughout the process operation, as there were several times when the connection failed in the pilot scale trials. Flow control of the sample stream is essential to ensure accuracy of the readings; this is another item which needs more simple operation.

The user interface of the particle counter requires significant attention from the operator when the trial is running. This is not an effective way to operate a plant as it is not difficult to make a mistake, and will require a lot of time to operate, potentially resulting in an increased requirement of manpower. The manpower costs and increased process difficulty will decrease the overall benefits from the product. Development of an online control interface which is compatible with the current SCADA operating system will resolve this. Data storage should be achieved with a current, efficient storage method. Flash cards have a small capacity, and are susceptible to physical damage which will also result in inefficient processing.

Although the product will improve confidence of cleaning for companies when in operation, before this can be developed, it is essential to ensure that there is confidence that the predictions are correct. This can be realised by collecting significantly more data on this; performing further pilot trials which account for additional fouling and temperature variation, as well as full industrial scale trials over several different sites, producing multiple products.

Once the challenges have been addressed, and sufficient data and trials have been performed, a prototype product may be developed and tested rigorously through industrial scale site trials. A marketable product with potentially large scale cost benefits for the brewing industry (as well as the wider food and drink and consumer goods markets) may be created. The cost to bring this to market and implement throughout customer sites could be significantly less than the cost benefits made to the companies. There is a potential to take this product to market as a separate business venture with a significant return on investment, dependent on the use/sale of the intellectual property which has been produced.

Further technology developments once the product has been developed will be required in order to ensure compatibility with the wider markets. This could be built into a business plan for future investment as the business grows, so as to reduce the market risk. This is all pre-emptive planning based on the assumption that the product development will be successful, the challenges can be reduced significantly enough to minimise the impact upon the overall product, the target market will want the product, and that the financial benefits will be great enough to result in sustainable business growth.

5.9 Cost Benefit Analysis

It has not been possible to develop a detailed cost benefit analysis on this investigation due to the fact that there have been no site trials or prototype testing. Without these trials, it is not possible to quantify the magnitude of savings.

It is, however, necessary to quantify the benefits of this investigation before moving forward with further research, trials, and potential industrial application and product development. A theoretical example, based on site knowledge and experience has been developed to give an overview of the potential savings to be made. The example compares the costs, flow rates, and consumptions which were used throughout the benchmarking project in the early phases of the EngD. This will maintain consistency between results, although inflation and cost increases by suppliers may vary the predicted savings from this report.

The example includes a one minute time saving per cleaning step. Based on these results, an approximation of the benefits to be obtained may be made. Unfortunately, no further detail into these savings can be provided as the times to be saved are unpredictable without any site trials in place.

The initial benchmarking work that was done including downtime costs may be seen in detail in electronic appendix 1. The case study with one minute saving per cleaning step may be seen in detail in electronic appendix 5.

These may then be crosschecked against each other to quantify the theoretical benefits of the case study. It has been assumed that the relationships between thermal energy, water, chemicals, and electrical usage all individually have a linear relationship between their respective consumptions and cleaning time.

It can be seen that a reduction of just one minute per cleaning step on each piece of equipment will save a total of £18,400 per week or an average of £7 per clean. This equates to an approximate total of £1,000,000 per annum with a 4% saving on utilities, 11% on chemicals, and a 4% saving on down time.

The potential savings for this project are very high based on this small example case study. In addition to the cost savings, the reduced cleaning times will also result in an increased plant capacity which also has a cost benefit. This is also very difficult to calculate without further knowledge of the actual cleaning times to be saved.

These numbers represent the potential benefits which may be large numbers. Unfortunately, they cannot be used to estimate realistic savings without further site trials and prototype testing. However, they do indicate that each site could expect a great cost benefit, and that continuation to progress with this project is worthwhile.

5.10 Summary of Experimental Conclusions

The following conclusions have been formed as a result of this investigation:

With regards to the fouling procedures:

- The consistency of fouling for the completed experiments was poor, and makes the overall results unreliable. It is essential that fouling is as consistent as possible for future experiments. These experiments also need to be repeated with consistent fouling.
- A fan blowing ambient air though the pipe on the rotary device will speed up drying of the fouling, and enable fouling consistency to be improved.

With regards to the use of the equipment and the experimental procedures:

- Air in the cleaning system causes pump cavitation which effects the cleaning flow rate and cleaning abilities of the system. Cleaning by recirculating through the supply tank or priming the pilot plant by topping up the pipework to remove all air bubbles resolves this.
- Caustic suds and light residues of fouling may remain in the test section which can be rinsed away easily with a manual rinse after runs. This is representative of a final rinse on a real plant cleaning scenario.
- The Ecolab turbidity probe saturation level is at 550 FTU. This is the point of interest when the cleaning is recirculated via the test section alone, so a more suitable cleaning route is to recirculate the liquid via tank 23 with a larger overall body of fluid.
- The standardisation of pipe fouling and operating procedures improves the repeatability of results

With regards to the experimental results of the investigation:

- Turbidity increases rapidly as cleaning commences and the first plug of fouling and cleaning liquid passes the probe. The turbidity then rapidly drops, and levels out at a consistent rate. There is not yet enough evidence to link this behaviour to the state of cleanliness and time taken to clean the pipe.
- Longer cleaning times and stronger caustic solution strengths give better cleaning results.
- Higher temperatures improve solubility of fouling which increases the turbidity of the cleaning solution at a faster rate. More dissolved fouling potentially affects the cleaning abilities of the fluid.
- Using a predictive model to determine the end point of clean is possible with the use of multivariate curve fitting.
- There are two progressive cleaning stages of fouling removal which have been identified.

- Sodium hydroxide is less effective as a cleaning detergent when the cleaning flow rate is higher. This is believed to be due to the reduced residence time of cleaning detergent per unit surface area which gives the chemical less time to break down the residual fouling on the pipe surface.
- A two parameter curved fitting model may be used to define the relationship between the two identified fouling removal characteristics.
- An ideal cleaning scenario will consist of a large bulk removal parameter and a small residual cleaning parameter.
- Cleaning flow rate has the greatest impact on bulk fouling removal.
- The minimum NaOH concentration required to clean this soil type effectively is 0.5% w/w. This confirms the theory which was established in Chapter 4 around the investigation of the maximum carbonate and minimum carbonate strengths required.
- Sequestrants provide no additional cleaning benefit to the detergent effects of NaOH.
- Optimisation of individual cleaning processes is required to maximise the cleaning effects and efficiency, whilst remaining cost effective.

With regards to the use of the particle counter for the predictive model:

- Particle count is an effective monitoring system for determining is the removal rate of fouling. This could be combined with information from other measurements to determine how clean equipment is throughout the cleaning process
- The particle counter used for the experiments is currently not robust enough for industrial application
- The control interface for the particle counter needs to be more simple and user friendly for process operators
- The breaking up of fouling particles which have already removed and are in recirculation can impact upon the results of the particle counter readings, because it measures only count and does not

relate to total mass of particles in solution. This can be accounted for mathematically in the monitoring and control process.

- There is uncertainty within the system as to the quantity of particles which may remain adhered to the equipment surface after a fixed period with constant particles. This can be rectified by obtaining further empirical data and performing trials to improve the confidence in the system.
- Once the uncertainty around particles remaining in equipment has been resolved, this process will improve confidence in cleaning ability of equipment and reduce the likelihood of microbial contamination outbreak.

With regards to potential business developments as an outcome of the project:

- Improvement and development of a future marketable product could be possible
- The completion of this investigation and development into a site compatible product could reduce environmental impact for companies as well as associated cleaning costs by optimising the cleaning times.
- The successful development of this product on site could result in a marketable product which could be modified slightly for customers outside of the brewing industry in food and drink, consumer goods and potentially other industries.
- Further technological and market research would be required to develop the marketable product, and an agreement for the use of the intellectual property would need to be reached.
- An improved cost benefit analysis is required in further investigation, and to develop a business plan
- A simple investigation into the cost savings to be made showed that these savings could be substantial, however much further work is required to verify this.

5.11 Recommendations for Future Experimental Work

This project requires further investigation for determination of effectiveness and benefits to justify industrial application. Once sufficient justification has been developed, work should be done towards implementation to achieve the cleaning and cost benefits.

Further investigation points include:

- Collection of experimental data on the pilot plant to include heated cleans at varying temperatures ranging from ambient to 50^{o}C, which can then be incorporated into the predictive model to account for the effect of temperature on fouling removal
- Collection of experimental data on the pilot plant to include different brewery fouling types from across the Heineken product ranges to account for product and site variability, which can also be incorporated into the predictive model. This should include cider lees, lager brands, cider brands, and ale brands. This will allow the model to be transferred to different products, or additional product specific models to be developed
- Trials on the pilot plant to develop a method with increased confidence that the end result is clean. This will require investigation into additional measurement methods and verification steps. The methods and steps should consider practicality and scale for application in an industrial environment.
- Site trials for improved confidence in the scaled up site design, by installing the measurement devices on site and recording data for analysis and verification offline
- Development of a user interface to be applied on site for operators to control as part of the CIP process. This could be done using a graphical user interface on a programme such as Matlab. Ultimately this user interface will need to be compatible with SCADA
- Development into a useable product which could potentially have some commercial viability for other companies within the food and drink industry, and possibly into other industries which use CIP

Chapter 6. Thesis Conclusions

Product quality is of great importance to the business, and when not properly sustained, can have a major impact on the success of a food and drinks manufacturer. By utilising an engineering doctorate student for a relatively low cost to perform research, companies may gain a substantial return on investment with a short payback time. In doing this, the company also provides the research engineer with the opportunity to gain sufficient industrial experience. This will enable them to add value to the company as an employee immediately after graduating with the EngD, should they choose to recruit them into the business.

The use of the benchmarking tool after specific tailoring to meet process requirements of an individual site is a powerful tool. It will enable industrialists to define and measure their cleaning process inputs, allow analysis of their process, implement improvements with quantifiable benefits, and achieve enhanced process control. The tool fundamentally provides an opportunity to model key process cost and waste contributors so that improvements may be focussed on the areas which will offer the greatest benefits. The benchmarking which was completed within the scope of this EngD project identified a quick win saving of £13,000 per annum.

The investigation around degradation of sodium hydroxide as a cleaning detergent has resulted in the identification of some clearly defined cleaning requirements. These specifications can be utilised practically with immediate effect to ensure that effective detergent cleaning is taking place at all times with an improved level of confidence. This work also demonstrated that the current detergent cleaning control and measurement system on site is not adequate in the existing conditions, and offers a cost effective alternative. Implementation of these findings on one site will provide cost savings for the company of £959,000 per annum in operational costs and £330,000 in capital due to increased capacity from freed up production time.

The project for the development of the cleaning predictive model saw an innovative method for replicating brewery fouling developed which enabled consistency, as well as reproducibility and repeatability of experimental conditions. This is extremely important when developing a mathematical model from results which are influenced by variability of this nature. An experimental procedure was developed to ensure repeatability of a variable natured set-up of process equipment and also to remove inconstant conditions such as air in the system, reaching measurement probe saturation points, and cleaning of the residual fouling within the recirculation loop.

The experimental results verified the cleaning limits which were provided by the chemical degradation investigation. This is in addition to showing other relationships between the cleaning parameters and fouling removal steps which confirm that a predictive model for the cleaning end point is possible to develop. A cleaning model can also be used with a cost optimisation to provide the optimal cleaning effectiveness for the lowest operational costs.

The use of a particle counter for monitoring a cleaning process proved to be effective; however, the limited robustness and availability of online particle counters conclude that this is currently not appropriate for industrial use. With some improvement work on this equipment, this would be a key factor in the development of an industrial functional predictive model.

There is an element of risk involved in the predictive model at present, as there is a lack in confidence that the cleaning end point has been reached. This requires further experimental work which is discussed in the future work section to reduce this risk and improve the effectiveness of the model.

The development of a low risk, operative, predictive model would yield a potentially globally marketable product which could provide cost, production, quality, and environmental benefits for companies within the food and drink industry. To enable this, IP ownership must be negotiated, and a detailed cost benefit analysis completed to develop a robust business plan and ensure that return on investment is sufficient.

Chapter 7. Recommendations for Future Work

Benchmarking should always be used in industry regardless of company/sector. This will enable industrialists to identify potential process improvements and provide a measurement system in order to quantify the benefits of any process changes. Recommended process changes should be implemented to achieve the already identified benefits around the areas of recovering and recycling CIP rinse water, and optimising the sodium hydroxide detergent steps to reduce waste and improve cleaning capabilities. All learning from this work and previous process knowledge and research should be considered when developing new CIP sets for current expanding and future developing sites.

The sodium hydroxide investigation may be taken further to determine the relationship between sodium hydroxide and sodium carbonate and the effects on their cleaning efficacies when mixed together in varied volumes. It would also be interesting to find their saturation points in solution and monitoring how this affects cleaning capabilities within the cleaning solution when either of their respective saturation points is achieved. An understanding of the impact of interrupting the Toftejorg cleaning heads mid cycle would be beneficial and provide an incentive to avoid these interruptions in future if it is proven that they are as negatively impacting on the process cleaning capabilities as it is believed and stated by the cleaning head suppliers. This would involve setting up a small scale tank cleaning rig and monitoring the impact when interrupting cleaning with a suitably sized cleaning head. The most beneficial area to investigate around sodium hydroxide cleaning would be to find a way to remove the root cause of the problem. This could involve either cleaning with a detergent that does not react with carbon dioxide, or finding an effective method to remove residual carbon dioxide from the tanks.

The cleaning predictive model needs significant further development before it is ready to be implemented on site. This will involve pushing the boundaries of the experimental design by including additional variable parameters, such as temperatures and different levels and types of fouling. Scaled up site trials are required to be completed, and a user friendly interface which is compatible with the current site control software must be developed. The successful completion of this work could result in a marketable product which could gain substantial return on investment with the development of a robust business plan.

Other interesting areas of investigation which are relevant to this EngD project are:

- The impact of cleaning complex geometries and how they will affect the output of the predictive model.
- The efficacy of acid sanitisers for cleaning process equipment.
- Tank cleaning and the effect that this has on the output of the predictive model.
- A study of the optimal type of cleaning spray head based on cost and cleaning effectiveness.
- A detailed study into alternative cleaning chemicals which may be used more cost effectively, particularly those which do not degrade with exposure to carbon dioxide.
- An investigation of purging equipment with nitrogen to remove carbon dioxide before the sodium hydroxide detergent cleaning step is performed.
- An investigation into regenerating sodium hydroxide and carbon dioxide from sodium carbonate to recycle them both into the cleaning detergent and bottling plant respectively.

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68. **F. Zeman** (2007) '*Energy and Material Balance of* CO_2 *Captured From Ambient Air',* **Environmental Science Technology, Vol. 41**, pp 7558 – 7563

Appendices

Appendix 1 – Composition of Cider Lees

The following details have been taken from Heineken NV records of lab analysis on cider lees (Welten, 2010)

Aluminium	28 mg/kg
Calcium	210 mg/kg
Iron	17 mg/kg
Magnesium	66 mg/kg
Potassium	730 mg/kg
Phosphorous	1200 mg/kg
Phosphate (PO4)	3800 mg/kg
Phosphate (P2O5)	2800 mg/kg
Sodium	85 mg/kg
Sulphur	310 mg/kg
Sulphate	920 mg/kg
Nitrate	7.1 mg/kg
Ash Residue	500-550^{o}C 0.54% m/m
Moisture	83.9 m/m
Chloride	<0.020% m/m
Protein	5.40% m/m
Total Fat	0.72% m/m
Carbohydrates	9.4% m/m
Energetic Value	279 kJ/100g
Energetic Value	66.4 kcal/100g
Solids	11.5%

Fibre, alcohol, organic acids, and polyols have not been taken into account.

The energetic value has been calculated from hydrocarbons, fat, and protein.

Appendix 2 – Composition of Beer Bottoms

The following composition details have been taken from three separate analyses provided by Bryant (2016), Swuste (2016), and King (2016)

“The maturation tank bottoms at Tadcaster are a variable mix of the following from highest to lowest content;

Yeast/isinglass complex- mostly ale but some lager. Isinglass is added at 1 pint to barrel (3.5 millilitres per litre).
Auxiliary finings/protein complex - auxiliary is added to green beer ex fermenter at 0.5 pint per barrel (1.75mls per litre)
Silica hydrogel/protein complex - silica hydrogel is added at 44 grams per litre to green beer ex fermenter).

Isinglass is pure collagen (a protein complex), auxiliary finings at Tadcaster is polymerised alginate based and silica hydrogel is formed from polymerised silica monomers.

The yeast is mainly viable unlike that of cider yeast lees.” Bryant (2016)

Solids 25% w/v
Swuste (2016)

Chloride (mg/l)	119
Sulphate (mg/l)	274
Magnesium (mg/l)	25.7
Potassium (mg/l)	47.3
Sodium (mg/l)	108
Zinc (mg/l)	40

King (2016)

Appendix 3 – Heat Transfer CIP Calculations

Firsty, the energy to heat the CIP fluid stream can be calculated by using:

$$q_{CIP} = \dot{m}tc_P dT$$ (Engineering Toolbox, 2012)

Where,

q_{CIP} = energy required to heat the CIP fluid (kJ/kg)

$\dot{m}$ = mass flow rate of liquid (kg/s)

t = time taken for temperature change to occur (s)

c_P = specific heat capacity of water (4.18 kJ/kg/K)

dT= change in temperature (K)

The mass flow rate can be measured, the specific heat capacity can be assumed to be that of water, the change in temperature can be read from the SCADA, and the total time to reach the higher temperature recorded.

The energy required to heat the CIP stream can be assumed to be equal to the energy used from the steam.

$$q_{CIP} = q_{steam}$$

The mass flow rate of the steam can be calculated by using:

$$\dot{m}_{steam} = \frac{q_{steam}}{h_e}$$

Where h_e is the specific evaporation enthalpy for steam which can be found using steam tables with the known steam pressure.

Once the mass flow rate of steam is known, the time spent heating the CIP fluid can be used to calculate the total quantity of steam.

Appendix 4 – Fermentation Area CIP Flow Diagrams

The following figures are the flow diagrams for the main cost group types of the fermentation area:

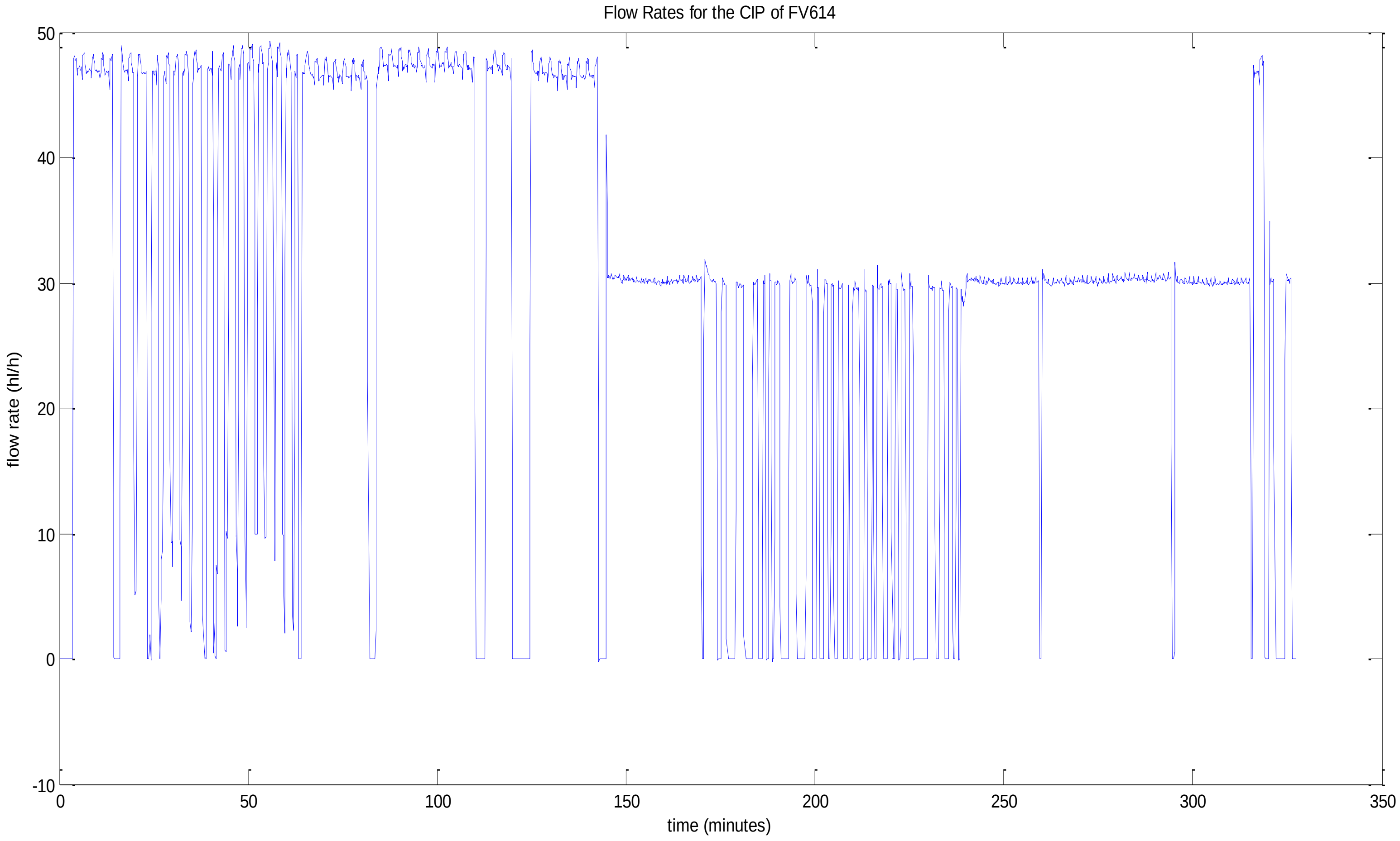


Figure 55: Flow Diagram for Large Fermentation Vessel Clean

Figure 55 represents the typical flows of a large fermentation vessel clean. This graph is discussed in more detail in section 2.6 within the report.

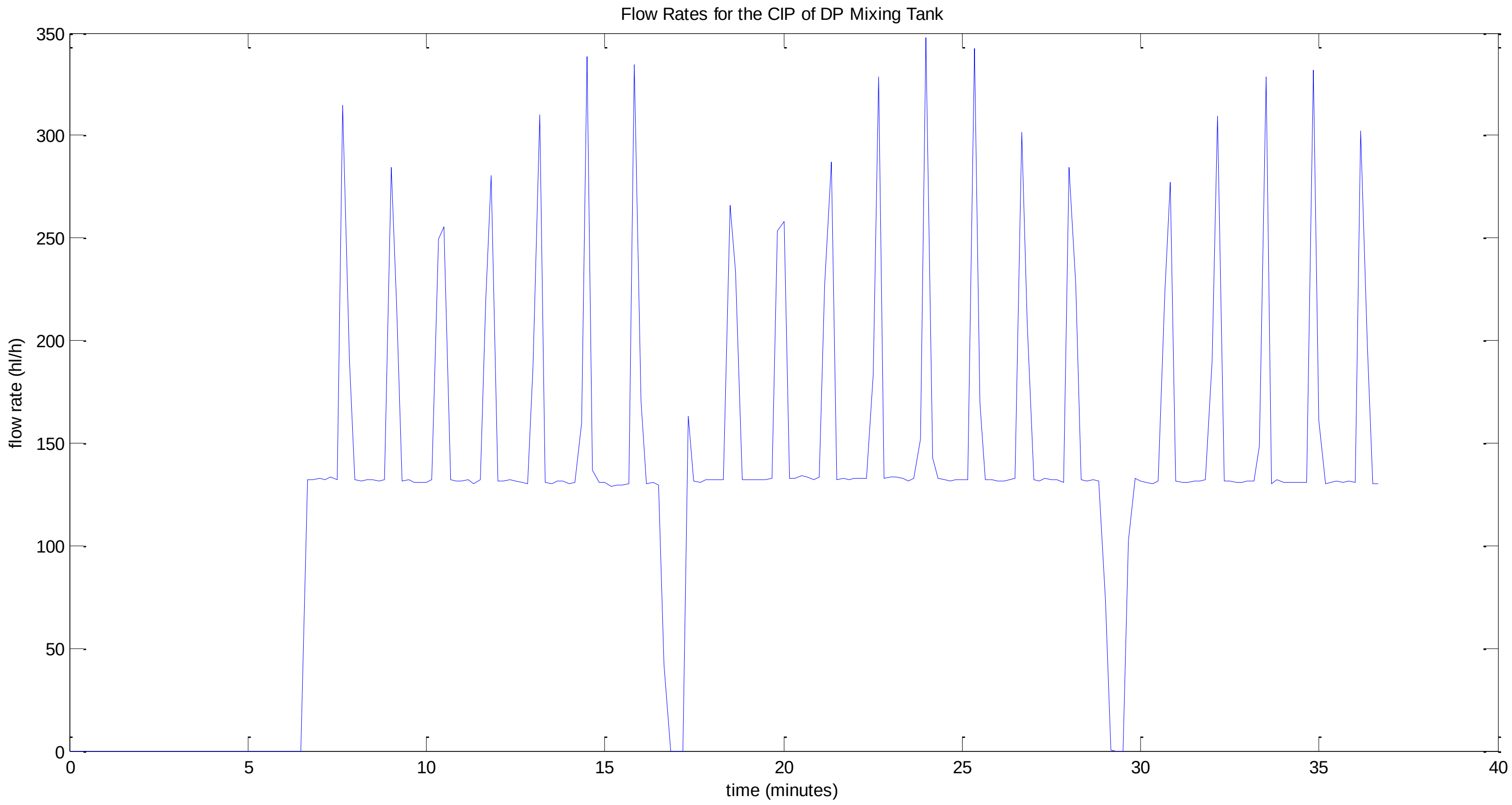


Figure 56: Flow Diagram for small tank CIP

Figure 56 represents the typical flows of the DP mixing tank clean. This clean did not include a caustic step, the gaps with no flow, represent the intermediate down time between swapping between cleaning steps, and the tall spikes represent increased flows though mains cleaning without the back pressure of the DP tank.

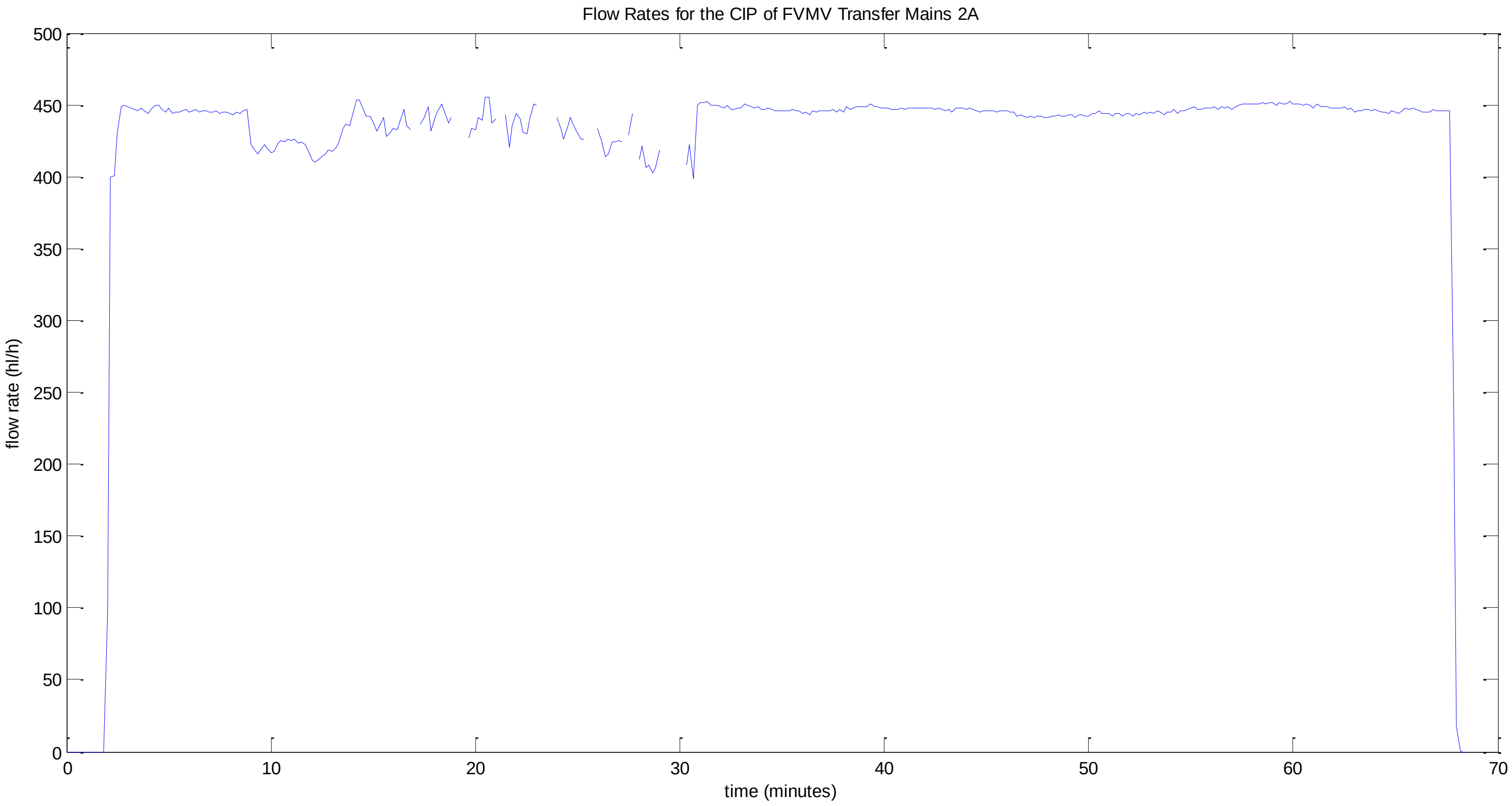


Figure 57: Flow Diagram of High Flow Mains Clean

Figure 57 represents a mains clean on the high flow rate pump. The flow rates are reasonably consistent and the changeover between cleaning steps is small as there is no requirement to wait for vessel draining of pools which accumulate in the bottom of the cone.

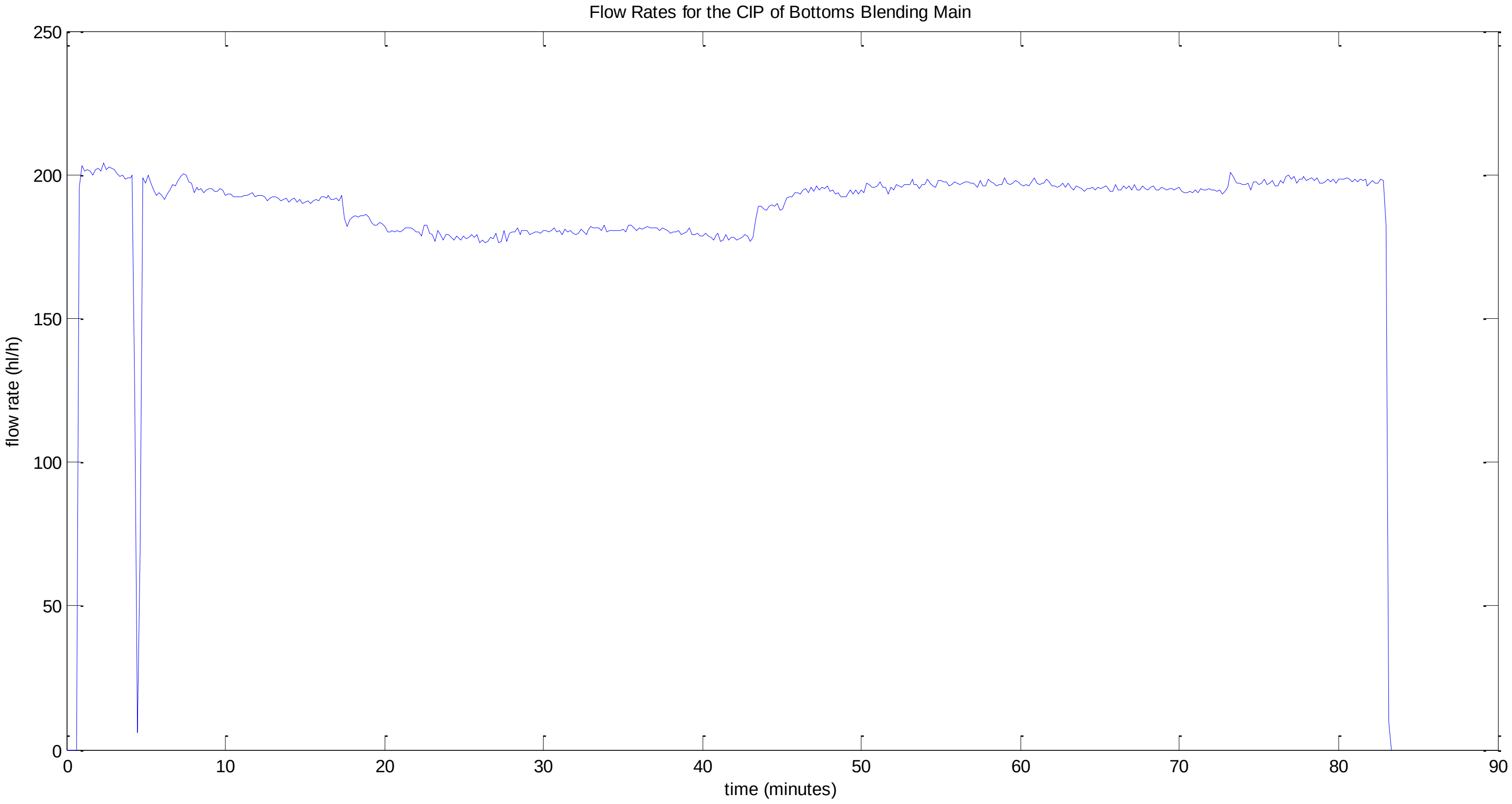


Figure 58: Flow Diagram of Low Flow Mains Clean

Figure 58 represents a mains clean on the low flow rate pump. Again, the flow rates are reasonably consistent and the changeover between cleaning steps is small as there is no requirement to wait for vessel draining of pools which accumulate in the bottom of the cone. There is one short hold period during the first rinse – it is likely that this is due to the mains being outside and not lagged.

The clean is a heated clean and if the temperature drops below the specification it is put in hold until the temperature is reached again. In winter this can cause problems with clean times on these mains.

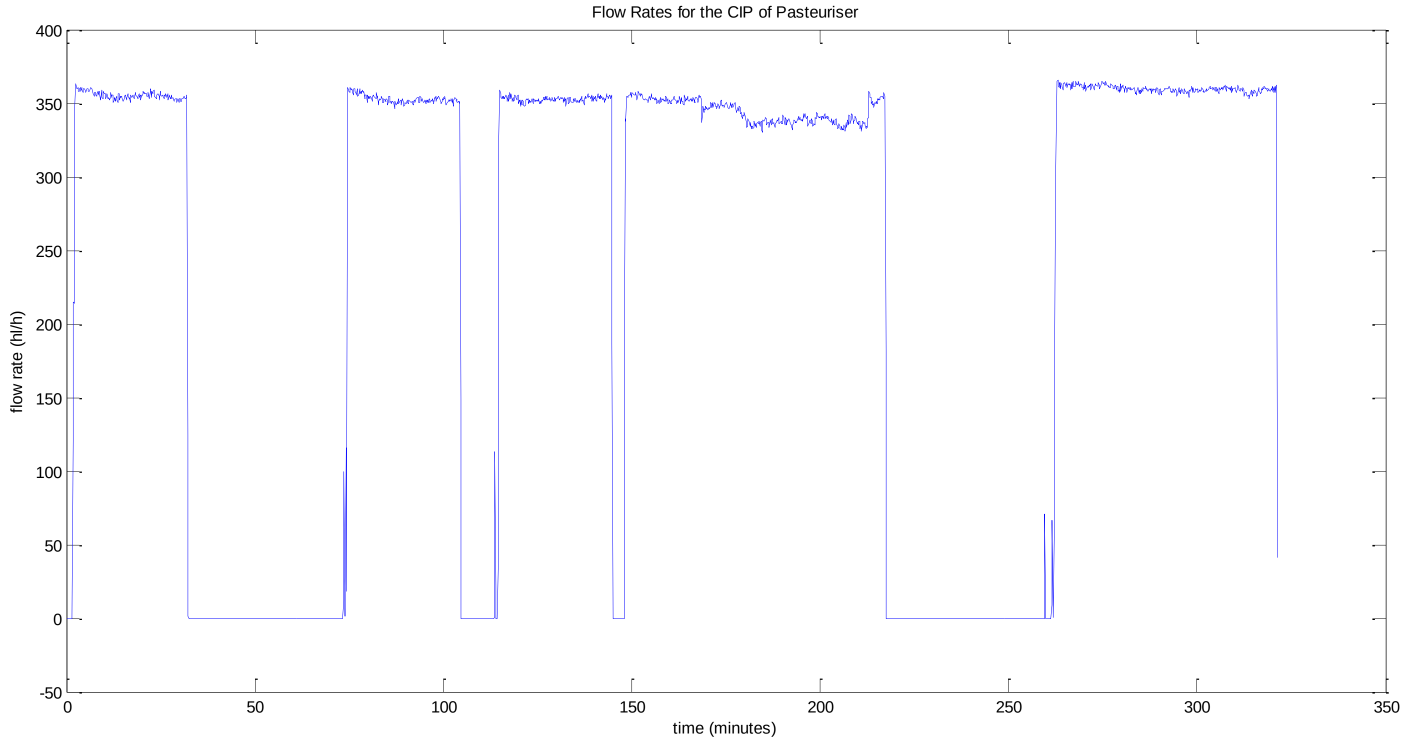


Figure 59: Flow Diagram of Pasteuriser Clean

Figure 59 shows a typical pasteuriser clean. The flow rates are consistent much like with the mains clean, but the cleaning temperature on this equipment is 70°C. This makes it more difficult to reach and maintain the cleaning temperature and so hold times can be more of an issue here as can be seen with all of the extended periods of time with no flow

Appendix 5 – Fermentation CIP Thermal Energy Requirements

Table 9 shows the calculated thermal energy requirements of the fermentation area heated cleans based on the theory provided in appendix 3.

Table 9: Thermal Energy Requirements for Fermentation Area Heated Cleans

Equipment	Water rinse time (s)	Caustic cycle time (s)	Energy required (kJ)	Thermal Energy Usage kWh
Transfer Mains	600	660	2350	5.33
Maturation Vessel Fill Main	600	720	784	1.62
Bottoms Blender Mains	1200	1152	870	0.57
Pasteuriser	1200	828	2682	2.35
Fresh Fruit Racking Lees Main	600	1152	609	0.71
Micro Filter Lees Main	1200	600	1218	1.35
Yeast Propagation Main	1200	1200	976	0.61
Lactic Acid Main	1200	600	976	1.08
Glucose Unloading	1200	639	2195	2.34
Culinary Concentrate Unloading	1200	900	1218	0.99
Bittersweet concentrate mains	2400	1152	2437	0.70
Bittersweet unloading	2400	1152	2437	0.70
North Planet Fill Mains	2400	756	3722	1.35

The calculations are based on the assumption that all water is required to be heated to the specified temperatures, and the caustic tank is required to be heated until the whole tank has been heated. Then it is assumed that the caustic tank will not lose its heat for the remainder

Appendix 6 – Capital Cost Calculation for CIP Water Sacrificial Tank

			cost	
using 4300 hl tank as example	4300	hl	£ 335,000	
	100		£ 35,072	(scaled to actual size)
	Civ/Inst/Eng	38%	£ 13,327	
	Total		**£ 48,400**	

Estimations based on knowledge from experienced capital project industrialist within Heineken (Swuste, 2013).

Appendix 7 – NPV H20

Opco revenue			13,000	£/Annum									
			years										
Process costs			**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Total investment costs		£ * 1000	48										
Total operational cost		£ * 1000	0	1	1	1	1	1	1	1	1	1	1
Total cost		£ * 1000	**48**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**
Costs benefits													
Opco Revenue		£ * 1000		13	13	13	13	13	13	13	13	13	13
Total benefits		£ * 1000	**-48**	**13**	**13**	**13**	**13**	**13**	**13**	**13**	**13**	**13**	**13**
Net result		£ * 1000	**-48**	**12**	**12**	**12**	**12**	**12**	**12**	**12**	**12**	**12**	**12**
WACC			7.7										
discounted cash flows			-48.0	11.1	10.3	9.6	8.9	8.3	7.7	7.1	6.6	6.2	5.7
NPV is sum DCF			33.6										
Operational NPV			**82**										
NPV		£ * 1000	**34**										

Appendix 8 – Diversey Sodium Hydroxide and Sodium Carbonate Titrations

The Diversey titration method (Diversey, 2011) is as follows:

1. Take a 2.5ml sample of solution.
2. Add three drops of phenolphthalein.
3. Titrate using 0.1M hydrochloric acid until colour change from purple to colourless.
4. Add three drops of bromothymol blue.
5. Titrate using 0.1M hydrochloric acid until colour change from blue to yellow.
6. The strength of sodium carbonate (%w/v) = (titre2 – titre 1) x 0.464
7. The strength of sodium hydroxide (%w/v) = (2 x titre 1 – titre 2) x 0.160

Target:

1.5% NaOH ± 0.2

<4% Na_2CO_3

Appendix 9 – Titration Method Verification

To verify the titration method in place, it was necessary to derive the method from basic principles. The product of the strength of acid present and the volume of acid present is equal to the product of the strength of base present and the volume of base present.

$$M_{acid}V_{acid} = M_{base}V_{base} \quad (1)$$

Substituting in from the values known in appendix 8,

$$0.1 \times V_{acid} = M_{base} \times 2.5 \quad (2)$$

There are potentially two bases present (sodium hydroxide and sodium carbonate. To distiunguish between the two, two indicators are used; phenolphthalein and bromothymol blue.

Phenolphthalein ($C_{20}H_{14}O_4$) colour changes between the pH of 8.2 and 10. Titrating to this value will give an approximation of the quantity of hydrochloric acid required to neutralise the sodium hydroxide present and form sodium chloride (equation 3). Bromothymol blue ($C_{27}H_{28}Br_2O_5S$) colour changes between the pH of 6 and 7.6. This will then provide an indication of the remaining acid required to completely neutralise all of the remaining bases by first converting sodium carbonate to sodium bicarbonate and sodium chloride (equation 4), and then the remaining sodium bicarbonate to sodium chloride (equation 5). The first titration value of the hydrochloric acid volume will be known as T1, and the second as T2 and will include the total volume of acid required to neutralise the whole solution (the difference between T1 and T2 is the volume required for the second half of the titration).

$$HCl + NaOH \rightarrow NaCl + H_2O \quad (3)$$

$$HCl + Na_2CO_3 \rightarrow NaCl + NaHCO_3 \quad (4)$$

$HCl + NaHCO_3 \rightarrow NaCl + H_2CO_3$

(5)

The difference between the two titrations shows the salt formed (at a ratio of 1:1 in each titration with HCl). This then provides the ratio of sodium hydroxide and sodium carbonate.

The acid and base balance with both sodium hydroxide and sodium carbonate becomes:

$M_{acid}V_{acid} = M_{NaOH}V_{NaOH} + M_{Na2CO3}V_{Na2CO3}$

(6)

$M_{Na2CO3} = (M_{HCl} \text{ x } V_{HCl})/V_{Na2CO3}$

(7)

Substituting in the known values

$M_{Na2CO3} = 0.1\ (T2 - T1)/2.5$

(8)

Then convert the strength of sodium carbonate into a percentage by multiplying through by the molar mass of sodium carbonate (106).

$\%\ Na_2CO_3 = (M_{Na2CO3} \text{ x } 106)/10$

(9)

$\%Na_2CO_3 = 0.1\ (T2 - T1)/2.5 \text{ x } 106/10$

(10)

$\underline{\%Na_2CO_3 = (T2 - T1) \text{ x } 0.424}$

Similarly with sodium hydroxide

$M_{NaOH} = (M_{HCl} \text{ x } V_{HCl})/V_{NaOH}$

(11)

As the titration for the sodium hydroxide also includes the titration for the sodium carbonate, this must be accounted for in the equation.

M_{NaOH} = 0.1T1/2.5 – M_{Na2CO3}

(12)

M_{NaOH} = 0.1T1/2.5 – 0.1(T2 – T1)/2.5 = 0.1/2.5 x (2T1 – T2)

(13)

Then this must be converted to a percentage by multiplying through by the molar mass of sodium hydroxide (40).

%NaOH = 0.1/2.5 x (2T1 – T2) x 40/10

(14)

<u>%NaOH = (2T1 – T2) x 0.160</u>

Appendix 10 – Mini Cleaning Rig Design Calculation

The cleaning requirements of the mini cleaning rig were that the shear force over the foul coupon was equivalent to 3 mPa. The flow rate and height of the liquid stream were calculated as follows:

$$\tau(y) = \mu \frac{du}{dy}$$ (Engineering Toolbox, 2012)

Where

τ = Shear force of the liquid (Pa)

μ = kinematic viscosity of water (1.002 x 10^{-3} N.s/m^2)

du = change in velocity of the liquid being fired at the tank wall (m/s)

dy= distance between the point where the liquid hits the wall and the bottom of the fouled coupon (m)

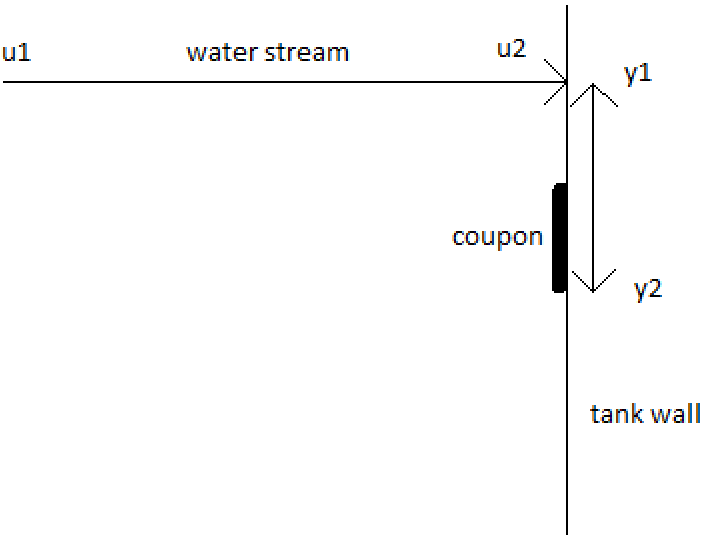


Figure 60: Diagram of Liquid Direction for Shear Force Calculation

This equation is integrated to give:

$$\int_{y_2}^{y_1} \tau \, dy = \int_{u_2}^{u_1} \mu \, du$$

It can be assumed that the velocity of the fluid when it hits the cell wall is 0, and the bottom of the fouled coupon is 0 to give the height required above the coupon.

$$\tau[y_1 - 0] = \mu[u_1 - 0]$$

The maximum height above the coupon is 17 cm, so substituting that into the equation allows the initial fluid velocity to be calculated:

$$3 \times 10^{-3}[(17 \times 10^{-2}) - 0] = 1.002 \times 10^{-3}u_1$$

$$u_1 = 0.509 \text{ m/s}$$

The cross sectional area of the nozzle can be calculated knowing that the diameter is 5 mm. This can then be used to calculate the minimum volumetric flow rate required:

$$A = \frac{\pi(5\times10^{-3})^2}{4} = 1.963 \times 10^{-5} \text{ m}^2$$

$$v = Au_1 = 1.963 \times 10^{-5} \times 0.509 = 4.712 \times 10^{-6} \text{ m}^3\text{/s}$$

$v = 10$ ml/s

Appendix 11 – Mini Cleaning Rig Standard Operating Procedure

1. While performing experiments, ensure that gloves and safety goggles are worn at all times when dealing with caustic.
2. Set the tank up ensuring that the bottom of the tank is higher than the height of the pump, and that all pipes are connected correctly in the configuration which can be seen in figure 61.

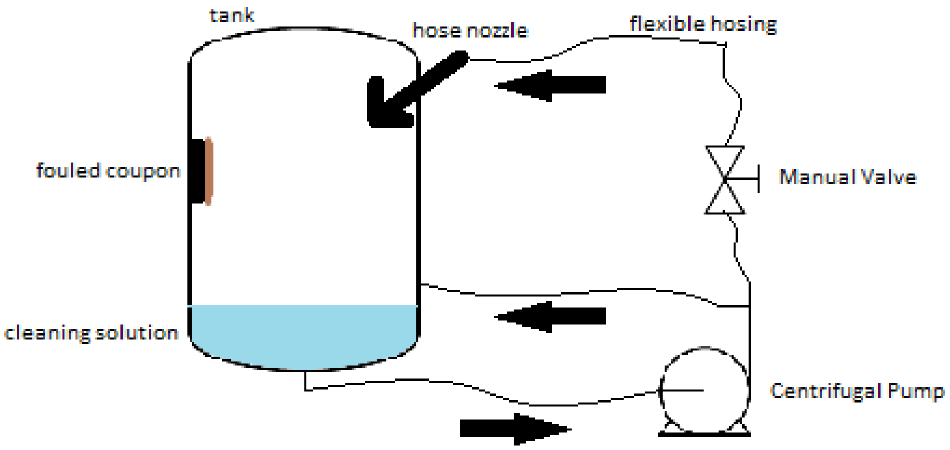


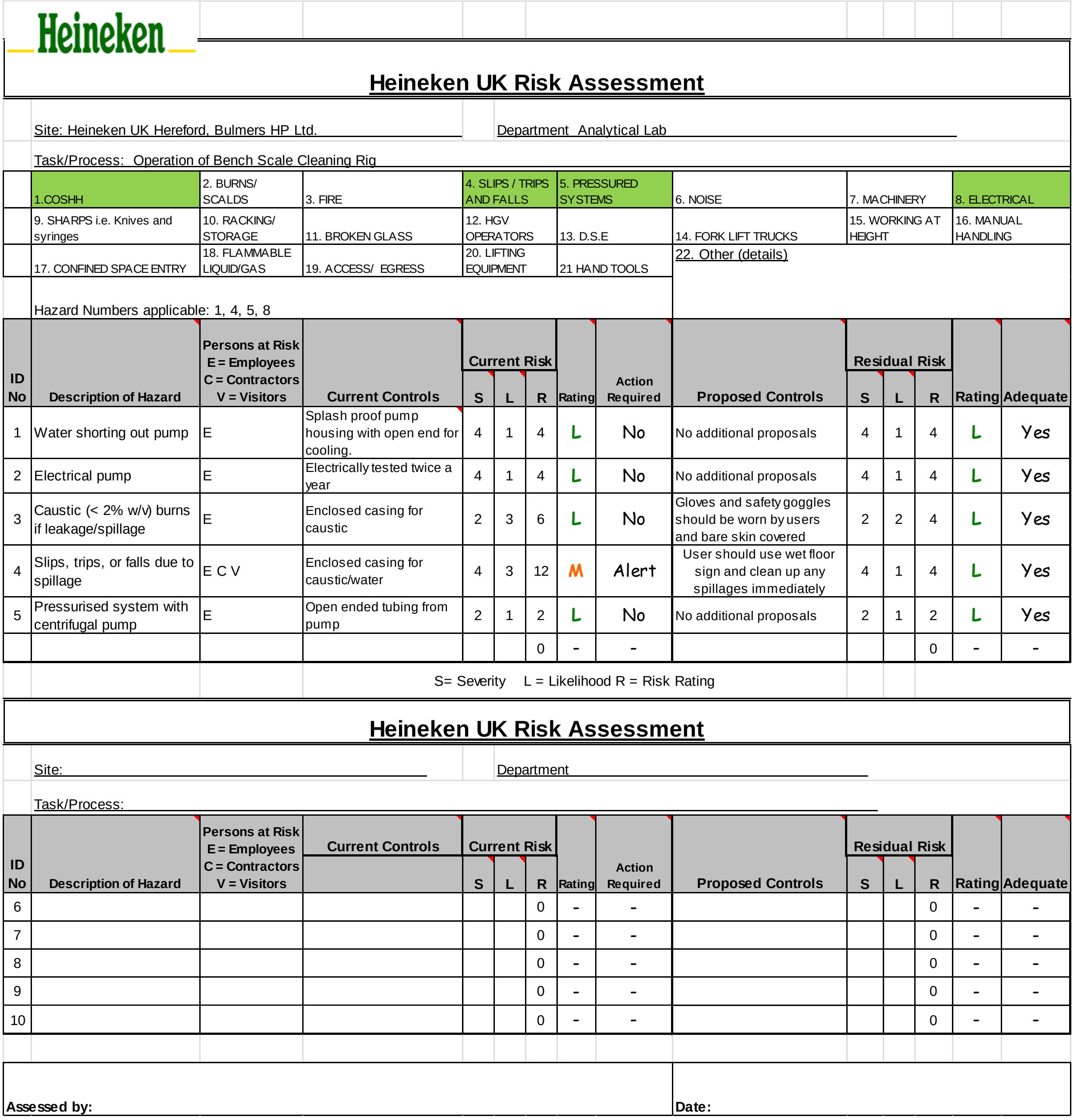
Figure 61: Diagram of Mini Cleaning Rig

3. Ensure the pump casing is on to protect the pump from any water splashes which may occur, and plug pump into 240V mains supply, but do not switch on.
4. Fill the tank with 4 litres of towns water.
5. Switch on the pump by the mains supply to recirculate, but have the hose nozzle pointing directly into a measuring cylinder above the open tank. Use a stop watch and adjust the hose nozzle and bypass valve until a flow rate of 50 or 100 ml/s is reached. This will require two people.
6. Put the nozzle in position in the fitting on the tank wall and run the pump to ensure that liquid hits tank wall just above the place where the fouled coupon will be suspended.
7. Drain the tank by removing nozzle and holding your thumb over the end of it. Point the nozzle into a large beaker (>4l) at a level where the top of the beaker is lower than the bottom of the tank. Remove your

thumb from the hose nozzle and allow the tank contents to drain into the beaker.

8. Make up 4l of solution of the required strength, but put in enough caustic/carbonate for a 4.5 l solution, as 0.5 l will be remaining in the pipes and pump.
9. Pour the solution carefully into the tank and run the pump for a few seconds to mix it with the water in the system.
10. Place a fouled coupon on the hook on the wall, and suspend another within the solution.
11. Place the clear lid on the top of the tank, then start the stopwatch and pump.
12. Monitor the time taken to clean both coupons visually.
13. Drain the system.
14. Flush the system with water and drain.
15. Repeat from points 8 to 14 if more experiments are to be performed.
16. When experiments are completed and system has been flushed with water and drained, unplug the pump and put equipment away.

Appendix 12 – Mini Cleaning Rig Risk Assessment



Heineken

Heineken UK Risk Assessment

Site: Heineken UK Hereford, Bulmers HP Ltd.

Department Analytical Lab

Task/Process: Operation of Bench Scale Cleaning Rig

1.COSHH	2. BURNS/ SCALDS	3. FIRE	4. SLIPS / TRIPS AND FALLS	5. PRESSURED SYSTEMS	6. NOISE	7. MACHINERY	8. ELECTRICAL
9. SHARPS i.e. Knives and syringes	10. RACKING/ STORAGE	11. BROKEN GLASS	12. HGV OPERATORS	13. D.S.E	14. FORK LIFT TRUCKS	15. WORKING AT HEIGHT	16. MANUAL HANDLING
17. CONFINED SPACE ENTRY	18. FLAMMABLE LIQUID/GAS	19. ACCESS/ EGRESS	20. LIFTING EQUIPMENT	21 HAND TOOLS	22. Other (details)		

Hazard Numbers applicable: 1, 4, 5, 8

ID No	Description of Hazard	Persons at Risk E = Employees C = Contractors V = Visitors	Current Controls	Current Risk			Rating	Action Required	Proposed Controls	Residual Risk			Rating	Adequate
				S	L	R				S	L	R		
1	Water shorting out pump	E	Splash proof pump housing with open end for cooling.	4	1	4	L	No	No additional proposals	4	1	4	L	Yes
2	Electrical pump	E	Electrically tested twice a year	4	1	4	L	No	No additional proposals	4	1	4	L	Yes
3	Caustic (< 2% w/v) burns if leakage/spillage	E	Enclosed casing for caustic	2	3	6	L	No	Gloves and safety goggles should be worn by users and bare skin covered	2	2	4	L	Yes
4	Slips, trips, or falls due to spillage	E C V	Enclosed casing for caustic/water	4	3	12	M	Alert	User should use wet floor sign and clean up any spillages immediately	4	1	4	L	Yes
5	Pressurised system with centrifugal pump	E	Open ended tubing from pump	2	1	2	L	No	No additional proposals	2	1	2	L	Yes
						0	-	-				0	-	-

S= Severity L = Likelihood R = Risk Rating

Heineken UK Risk Assessment

Site:

Department

Task/Process:

ID No	Description of Hazard	Persons at Risk E = Employees C = Contractors V = Visitors	Current Controls	Current Risk			Rating	Action Required	Proposed Controls	Residual Risk			Rating	Adequate
				S	L	R				S	L	R		
6						0	-	-				0	-	-
7						0	-	-				0	-	-
8						0	-	-				0	-	-
9						0	-	-				0	-	-
10						0	-	-				0	-	-

Assessed by:

Date:

Figure 62: Mini Cleaning Rig Risk Assessment

Appendix 13 – Diversey NaOH Cleaning Effectiveness Graph

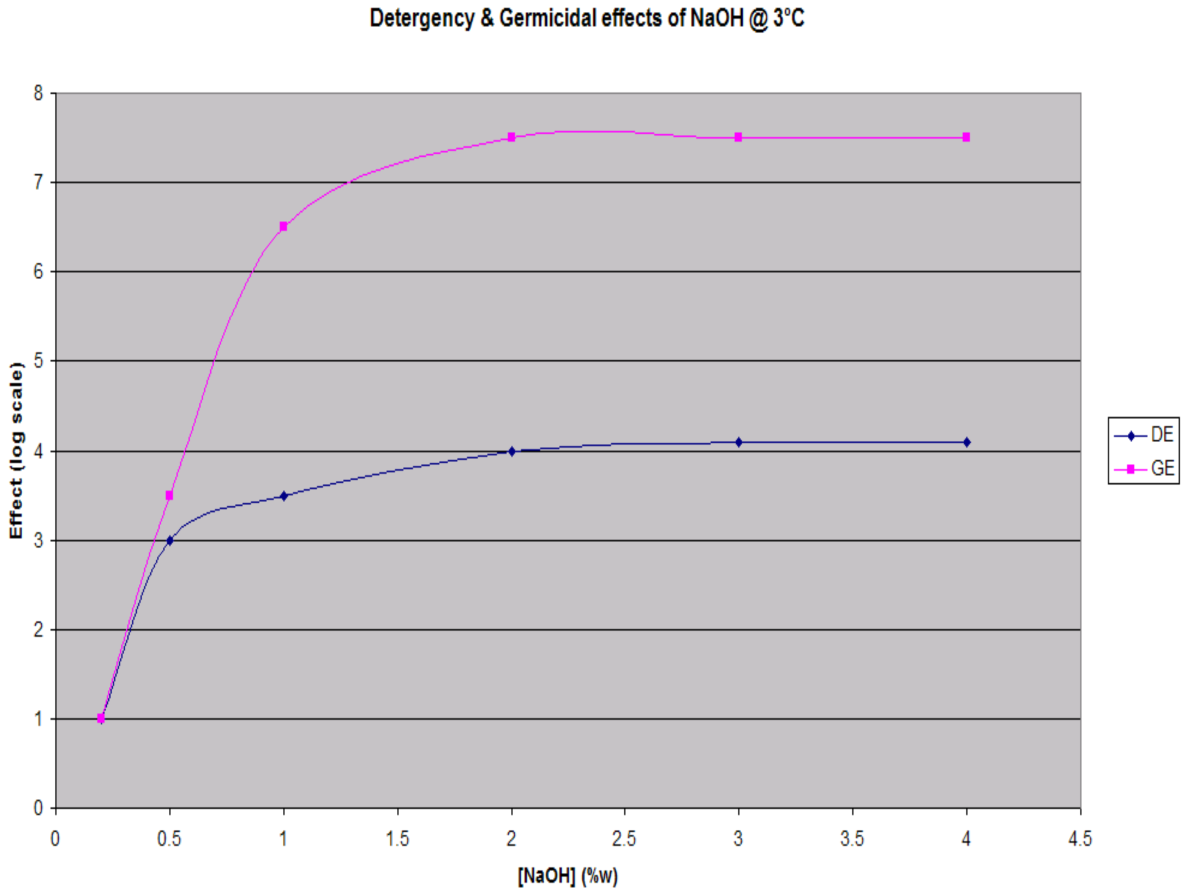


Figure 63: Graph of Cleaning Effects of NaOH (Edwards, 2011)

Figure 63 (Edwards, 2011), is the graph from the work Diversey has completed on the cleaning detergent and germicidal effects of sodium hydroxide with varying concentration. It can be seen that the detergent effects in this work closely match that done in this report in section 4.6.1 where the cleaning detergent effect does not improve by much with concentrations greater than 1%.

Appendix 14 – FV611 pH and Conductivity Verification Results

Table 10 shows the results from monitoring fermentation vessel 611 to verify the conductivity and pH values at set intervals against the model developed in section 4.6. These results are discussed in more detail in section 4.6.2.

Table 10: Results of pH and Conductivity Values for FV611 Clean

Sample Time (mins)	NaOH conc (% w/v)	Na2CO3 conc (% w/v)	pH (actual)	Conductivity (mS) (actual)
0	1.50	1.87	13.01	31.9
5	1.35	2.37	13.10	32.4
10	1.23	2.54	13.09	32.3
15	1.42	2.59	13.12	32.5
20	1.33	3.05	13.11	32.5
25	1.46	2.93	13.11	32.9
30	1.41	3.35	13.12	33.2
30	1.28	3.43	13.11	32.7
35	1.26	4.07	13.03	32.9
40	0.99	4.79	13.02	33.0
45	1.15	5.05	12.92	32.6
50	0.86	5.98	12.92	32.9
55	1.20	5.72	12.81	32.6
60	0.78	6.49	12.66	32.3
65	0.90	6.44	12.80	32.8

Appendix 15 – Capital Cost Calculation for Tank with same Volume as Realised Capacity

Time saved		250	min		
Time in working week	Quantity	10080	min]	Volumes	
Large Tank	11	8000		88000	hl
Small Tank	4	2700		10800	hl
				98800	hl
			Total	2450.397	hl
			cost		
using 4300 hl tank as example	4300	hl	£ 335,000		
	2450.396825		£ 239,059	(scaled to actual size)	
	Civ/Inst/Eng	38%	£ 90,843		
	Total		**£ 329,902**		

Estimations based on knowledge from experienced capital project industrialist within Heineken, Swuste (2013).

Appendix 16 – Estimated Installation Costs for Carbonate Benefits Realisation

Automation	£5,000
Piping	£12,000
pH probes	£13,000

Estimations based on knowledge from experienced capital project industrialist within Heineken, Swuste (2013).

Appendix 17 – Net Present Value Calculations for Carbonate in Caustic Benefits with Downtime Saving Opportunities

Opco revenue			959000	£/Annum									
			years										
Process costs			**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Total investment costs		£ * 1000	30										
Total operational cost		£ * 1000		1	1	1	1	1	1	1	1	1	1
Total cost		£ * 1000	**30**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**
Costs benefits													
Opco Revenue		£ * 1000		959	959	959	959	959	959	959	959	959	959
Total benefits		£ * 1000	**-30**	**959**	**959**	**959**	**959**	**959**	**959**	**959**	**959**	**959**	**959**
Net result		£ * 1000	**-30**	**958**	**958**	**958**	**958**	**958**	**958**	**958**	**958**	**958**	**958**
WACC			7.7										
discounted cash flows			-30.0	889.5	825.9	766.9	712.0	661.1	613.9	570.0	529.2	491.4	456.3
NPV is sum DCF			6486.2										
Operational NPV			**6516**										
NPV		£ * 1000	**6,486**										

Appendix 18 – Net Present Value Calculations for Carbonate in Caustic Benefits with Consumable Cost Savings Only

Opco revenue			59,000	£/Annum									
			years										
Process costs			**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Total investment costs		£ * 1000	30										
Total operational cost		£ * 1000		1	1	1	1	1	1	1	1	1	1
Total cost		£ * 1000	**30**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**	**1**
Costs benefits													
Opco Revenue		£ * 1000		59	59	59	59	59	59	59	59	59	59
Total benefits		£ * 1000	**-30**	**59**	**59**	**59**	**59**	**59**	**59**	**59**	**59**	**59**	**59**
Net result		£ * 1000	**-30**	**58**	**58**	**58**	**58**	**58**	**58**	**58**	**58**	**58**	**58**
WACC			7.7										
discounted cash flows			-30.0	53.9	50.0	46.4	43.1	40.0	37.2	34.5	32.0	29.7	27.6
NPV is sum DCF			364.5										
Operational NPV			**395**										
NPV		£ * 1000	**365**										

Appendix 19 – Sodium Carbonate Rate Formation Calculation

A clean was monitored on a large fermentation vessel at Bulmers, and based on the current CIP setup, the carbonate formation rate was monitored (including with dosing).

The volume of the dilute caustic tank is 10,000l, filled to a working capacity of 80% (800l). Based on monitoring the flow rate and time of a small fermentation vessel clean, the volume of dilute sodium hydroxide which passes through the vessel is 27,000l. Therefore it can be assumed that the whole dilute tank passes through the vessel 3.4 times.

If sodium hydroxide starts at 1.7%, then

1.7 x 8000 = 13600g NaOH

13600/40 = 340 moles NaOH

Based on ratio from

$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$

170 moles of Na_2CO_3 formed

170 moles of CO_2 used, so around 38hl CO_2 used.

Assuming head space of fermentation vessel was completely full of CO_2 and all of that remains in the vessel after it has been emptied and the head space was 15%, then

15% of 7000 = 1050hl CO_2

If 38hl CO_2 mopped up per dilute tank, then 28 full cleans required to mop up all CO_2

Appendix 20 – MSDS for Diversey Spektak G

Date: 08/08/08

Revision No: 1

Page 1 of 2

SAFETY DATA SHEET

1.

IDENTIFICATION OF THE SUBSTANCE / PREPARATION AND O

F THE COMPANY / UNDERTAKING

Diversey E

astern and Central

A

frica

Limited

Kaptagat Road, Loresho PO Box 41939, 00100 GPO Nair

obi Tel +254 20 4224000 Fax +254 20 4224888

SPECKTAK G VC1

Clearance Code

Prof Cleaning/maintenance product for Food & Beverage Industries

Product Code

DL10398

MSDS 1850

2.

COMPOSITION / INFORMATION ON INGREDIENTS

CAS No

EINECS No

1310-73-2

215-185-5

Sodium Hydroxide

C: R35

15-30%

3.

HAZARDS IDENTIFICATION

C

- Corrosive

4.

FIRST AID MEASURES

Eyes :

Wash immediately with copious amounts of water and obtain medical attention

Inhalation :

Remove from source of exposure. Obtain medical attention if symptoms develop. In case of unconsciousness bring patient into stable side

position for transport

Skin :

Wash skin immediately and thoroughly with water. Remove contaminated clothing. Obtain medical attention if symptoms develop

Ingestion :

Remove material from mouth. Drink one or two glasses of water or milk and obtain medical attention without delay

5.

FIRE FIGHTING MEASURES

Suitable Extinguishing Media

– CO2,xtinguishing powder or water jet>Fight larger fires with water jet or alcohol resistant foam

Protective Equipment

- No special measures required

6.

ACCIDENTAL RELEASE MEASURES

Protection of Personnel

-Wear suitable protective clothing, gloves and eye/face protection

Environmental Precautions

-Dilute with plenty of water

Method of Disposal

-Ensure adequate ventilation. Collect Mechanically. Dispose off the collected material according to regulations. Use neutralizing Agent

7.

HANDLING & STORAGE

Safe Handling-

Use common rules for working with chemicals

Explosions and fires

-No special measures required.

Storage

-According to local Legislation

8.

EXPOSURE CONTROL / PERSONAL PROTECTION

Hand :

Chemical –resistant protective gloves made of butyl or nitrile rubber

Eyes :

Wear tight fitting Safety Glasses

Skin :

Avoid contact with skin

:

: 08/08/2008

Revision Number: 1

Respiration :

Personal Protection is not normally required.

9.

PHYSICAL & CHEMICAL PROPERTIES

Appearance :

Clear pale yellow Liquid

Odour :

Characteristic

pH :

>12.5

Solubility :

Fully Miscible in water

Density :

1.47g/cm3

10.

STABILITY & REACTIVITY

Thermal Decomposition

-No decomposition if used according to specifications

Dangerous Reactions

-Reacts with Acids

Hazardous Decomposition Products

-None Known

11.

TOXICOLOGICAL INFORMATION

Eyes :

Causes Severe or permanent Damage

Skin :

Causes Severe Burns

Inhalation :

Severe Irritant, may cause pulmonary oedema

Ingestion :

Causes Severe Burns .Ingestion will lead to a strong caustic effect on mouth and throat and to the danger of perforation of esophagus and stomach

12.

ECOLOGICAL INFORMATION

When used for its intended purpose this product should not cause adverse effects in the environment.

13.

DISPOSAL CONSIDERATIONS

Product Recommendation

-Dispose off observing official regulations

Uncleaned Packaging

Recommendation

-Dispose of observing official regulations.

Recommended Cleaning Agent

-Water, if necessary with cleaning agent

14.

TRANSPORT INFORMATION

EEC Regulation :

C, CORROSIVE, Contains Sodium Hydroxide

IMDG/UN :

Sodium Hydroxide UN 1824 PG 11

RID/ADR :

8 corrosive substances

ICAO/IATA :

Class 8

15.

REGULATORY INFORMATION

Hazard symbol :

C, CORROSIVE, Contains Sodium Hydroxide

Risk phrases :

R35

Causes Severe Burns

Safety phrases :

S26

In case of contact with eyes, rinse immediately with plenty of water and seek medical advice

S28

After contact with skin, wash immediately with plenty of water.

S36/37/39

Wear suitable protective clothing, gloves and eye/face protection.

S45

In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

Appendix 21 – Rotational Fouling Device Standard Operating Procedure

1. Ensure device is plugged in to 240V mains supply.
2. Place o ring and blank end cap on the end of the pipe to be fouled and secure with a tri-clover clamp.
3. Stand the pipe up so that the blanked end is on the floor.
4. Measure 400 ml of well mixed beer bottoms and pour into pipe.
5. Cover open end of pipe and move gently from side to side and rotate slightly to distribute the fouling within the pipe.
6. Place o ring and end cap with a hole on the open end of the pipe and secure with a tri-clover clamp.
7. Open the safety case of the rotational device and carefully lower the pipe onto the rollers of the device with the end with a hole facing away from the motor (keeping horizontal to avoid spillage of bottoms through the hole).
8. Unscrew the tri-clover clamp on the end with the blank end cap, remove the clamp, but hold the end cap in place with your finger.
9. Remove the end cap, but not the o ring, and slide the pipe quickly into position with the o ring between the pipe and the fitting for connection on the device. Hold this securely in place with one hand (to stop the fouling from pouring out) whilst placing the tri-clover clamp in place with the other, and then securing.
10. Close the safety case of the device, and ensure that the motor settings are set to “off” and low rpm.
11. Enable the device by pressing the green button. The light will show green if the device is ready to be used. If the green light does not show, and the safety case is not shut, then there is a fault with the device.
12. Move the switch to the “on” position, and then slowly increase the rpm to roughly 10 rpm.
13. Place the fan at the end of the device so that it is facing down the pipe towards the motor.
14. Switch on the fan to its highest setting and leave the pipe to foul for 2 hours (time to be confirmed).
15. If at any point the safety case is opened during operation, the device will automatically cut out. If this happens, then the device may be restarted by closing the safety case, and repeating from point 10.

16. When fouling is complete, switch off the fan, and also the device by pressing the red button.
17. Open the safety case and remove the tri-clover clamp which is closest to the motor.
18. The dry, fouled pipe may now be removed and all o rings and end caps removed.
19. If the device is not going to be used again, switch off the device at the mains supply, ensure it is cleaned to avoid any microbial growth from the fouling, close the safety case and put away.

Appendix 22 – Pilot Plant Standard Operating Procedure at Ambient Temperature with No Caustic

1. Fill tank 23 from tank 21 using route 4a – ensure level is above filling inlet pipe (for good mixing and to minimise foaming) and is approximately 55 cm, measure with tape measure (so particulate concentration and NaOH strength can be calculated).
2. Half fill tank 22 from tank 21 using route 8a so that this water may be used for flushing the system later on.
3. Ensure plant is configured correctly with clean 1 m pipe in place in test section. Run tank 23 to drain via test section using route 4c to fill pipes with fluid (only specified liquid will come into contact with test section then – not previous fluid).
4. Drain test section using bucket at point near pump so that there is no liquid remaining in test section piece.
5. Remove clean 1 m length of pipe from test section and replace with readily fouled 1 m length pipe. Ensure that if there is any point where fouling is thicker (e.g. where settled at bottom of pipe), that it is placed in the test section so it remains at the bottom of the pipe. Also check that the end of the pipe is not fouled so that it prevents proper connection with the o ring to the rest of the plant to avoid leakage.
6. Record turbidity reading from Kemtrak turbidity probe and take a sample of the tank water.
7. Return content of bucket to tank 23.
8. Run the pilot plant on a recirculation via tank 23 using route 9 for 10 minutes. This will enable the pipe in the test section to be cleaned and the increase of particles in a known volume to be calculated. One tank should be used to clean one set of experiments (e.g. constant NaOH concentration and temperature, but varied flows in triplicate), provided that turbidity readings on the Kemtrak probe are well below saturation which is known to be 550 FTU. If this is exceeded, a fresh tank should be made by draining tank 23 without test section using route 8c until empty and cleaning with hosepipe whilst draining, then flushing system using tank 22 on route 7, and then continuing from point 1 without re-filling tank 22 unless it is empty).

9. After the run is complete, drain the test section using the bucket near the pump. Take a sample from this water for particle count analysis offline.
10. Remove the pipe and assess cleanliness.
11. Replace pipe with new pre-fouled pipe for next run.
12. Repeat points 5 to 9 until all experimental runs in the given set have been completed.
13. Replace clean pipe in test section. Run tank 23 to drain without test section using route 8c until empty, cleaning with hosepipe whilst flushing. Then flush test section using tank 22 on route 7.
14. Fill tank 23 from tank 21 using route 4a so that this will not need to be done at the start of the next set of experiments.
15. Half fill tank 22 from tank 21 using route 8a so that this will not need to be done at the start of the next set of experiments.
16. Switch off pilot plant if end of the day.

Appendix 23 – Pilot Plant Standard Operating Procedure at Ambient Temperature with Caustic

1. Fill tank 23 from tank 21 using route 4a – ensure level is above filling inlet pipe (for good mixing and to minimise NaOH foaming) and is approximately 55 cm, measure with tape measure (so particulate concentration and NaOH strength can be calculated.
2. Half fill tank 22 from tank 21 using route 8a so that this water may be used for flushing the system later on.
3. From the measured height of the liquid, calculate the total volume of water in tank 23.
4. Use this value to calculate the required dosing volume of NaOH. Add this volume to the tank using the plastic container with a lid whilst getting into position, wearing gloves and safety goggles. Mix the tank well.
5. Ensure plant is configured correctly with clean 1 m pipe in place in test section. Run tank 23 to drain via test section using route 4c to fill pipes with fluid (only specified liquid will come into contact with test section then – not previous fluid).
6. Drain test section using bucket at point near pump so that there is no liquid remaining in test section piece.
7. Remove clean 1 m length of pipe from test section and replace with readily fouled 1 m length pipe. Ensure that if there is any point where fouling is thicker (e.g. where settled at bottom of pipe), that it is placed in the test section so it remains at the bottom of the pipe. Also check that the end of the pipe is not fouled so that it prevents proper connection to the rest of the plant to avoid leakage.
8. Record turbidity reading from Kemtrak turbidity probe and take a sample of the tank water.
9. Return content of bucket to tank 23.
10. Run the pilot plant on a recirculation via tank 23 using route 9 for 5 minutes or until readings level off at a maximum value. This will enable the pipe in the test section to be cleaned and the increase of particles in a known volume to be calculated. One tank should be used to clean one set of experiments (e.g. constant NaOH concentration and temperature, but varied flows in triplicate (provided that turbidity readings on the Kemtrak probe are well below

saturation which is known to be 550 FTU. If this is exceeded, a fresh tank should be made by draining tank 23 without test section using route 8c until empty and cleaning with hosepipe whilst draining, then flushing system using tank 22 on route 7, and then continuing from point 1 without re-filling tank 22 unless it is empty).

11. After the run is complete, drain the test section using the bucket near the pump. Take a sample from this water for particle count analysis offline and titrations to verify NaOH concentration.
12. Remove the pipe and assess cleanliness.
13. Replace pipe with new pre-fouled pipe for next run.
14. Repeat points 5 to 9 until all experimental runs in the given set have been completed.
15. Replace clean pipe in test section. Run tank 23 to drain without test section using route 8c until empty, cleaning with hosepipe whilst flushing. Then flush test section using tank 22 on route 7.
16. Fill tank 23 from tank 21 using route 4a so that this will not need to be done at the start of the next set of experiments.
17. Half fill tank 22 from tank 21 using route 8a so that this will not need to be done at the start of the next set of experiments.
18. Switch off pilot plant if end of the day.

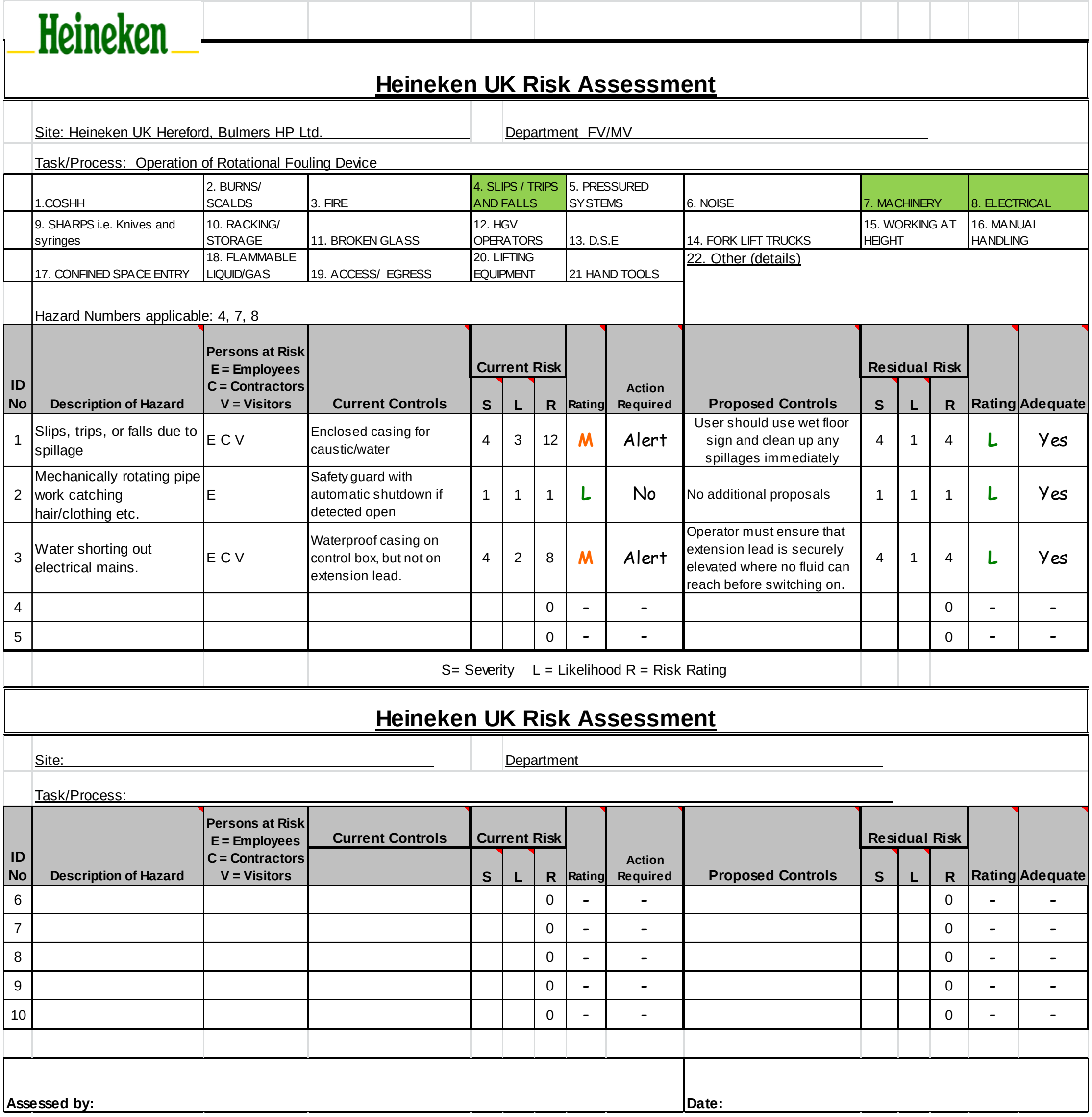
Appendix 24 – Pilot Plant Standard Operating Procedure for Heated Cleaning

1. Fill tank 23 from tank 21 using route 4a – ensure level is above filling inlet pipe (for good mixing, and to minimise foaming) and is approximately 55 cm, measure with tape measure (so particulate concentration and NaOH strength can be calculated.
2. Half fill tank 22 from tank 21 using route 8a so that this water may be used for flushing the system later on.
3. Heat tank 23 without the test section to the desired temperature.
4. Ensure that the manual steam valve is opened for heating, and closed again when not heating.
5. Ensure plant is configured correctly with clean 1 m pipe in place in test section. Run tank 23 to drain via test section using route 4c to fill pipes with fluid (only specified liquid will come into contact with test section then – not previous fluid).
6. Drain test section using bucket at point near pump so that there is no liquid remaining in test section piece.
7. Remove clean 1 m length of pipe from test section and replace with readily fouled 1 m length pipe. Ensure that if there is any point where fouling is thicker (e.g. where settled at bottom of pipe), that it is placed in the test section so it remains at the bottom of the pipe. Also check that the end of the pipe is not fouled so that it prevents proper connection to the rest of the plant to avoid leakage.
8. Record turbidity reading from Kemtrak turbidity probe and take a sample of the tank water.
9. Return content of bucket to tank 23.
10. Run the pilot plant on a recirculation via tank 23 using route 9 for 5 minutes or until readings level off at a maximum value. This will enable the pipe in the test section to be cleaned and the increase of particles in a known volume to be calculated. One tank should be used to clean one set of experiments (e.g. constant NaOH concentration and temperature, but varied flows in triplicate (provided that turbidity readings on the Kemtrak probe are well below saturation which is known to be 550 FTU. If this is exceeded, a fresh tank should be made by draining tank 23 without test section using route 8c until empty and cleaning with hosepipe whilst draining, then flushing system using

tank 22 on route 7, and then continuing from point 1 without re-filling tank 22 unless it is empty).

11. After the run is complete, drain the test section using the bucket near the pump again. Take a sample from this water for particle count analysis offline.
12. Remove the pipe and assess cleanliness.
13. Replace pipe with new pre-fouled pipe for next run.
14. Repeat points 5 to 9 until all experimental runs in the given set have been completed.
15. Replace clean pipe in test section. Run tank 23 to drain without test section using route 8c until empty, cleaning with hosepipe whilst flushing. Then flush test section using tank 22 on route 7.
16. Fill tank 23 from tank 21 using route 4a so that this will not need to be done at the start of the next set of experiments.
17. Half fill tank 22 from tank 21 using route 8a so that this will not need to be done at the start of the next set of experiments.
18. Switch off pilot plant if end of the day and double check that manual steam valve is closed.

Appendix 25 – Risk Assessment for Rotational Fouling Device



Heineken

Heineken UK Risk Assessment

Site: Heineken UK Hereford, Bulmers HP Ltd. Department FV/MV

Task/Process: Operation of Rotational Fouling Device

1.COSHH	2. BURNS/ SCALDS	3. FIRE	4. SLIPS / TRIPS AND FALLS	5. PRESSURED SYSTEMS	6. NOISE	7. MACHINERY	8. ELECTRICAL
9. SHARPS i.e. Knives and syringes	10. RACKING/ STORAGE	11. BROKEN GLASS	12. HGV OPERATORS	13. D.S.E	14. FORK LIFT TRUCKS	15. WORKING AT HEIGHT	16. MANUAL HANDLING
17. CONFINED SPACE ENTRY	18. FLAMMABLE LIQUID/GAS	19. ACCESS/ EGRESS	20. LIFTING EQUIPMENT	21 HAND TOOLS	22. Other (details)		

Hazard Numbers applicable: 4, 7, 8

ID No	Description of Hazard	Persons at Risk E = Employees C = Contractors V = Visitors	Current Controls	Current Risk				Action Required	Proposed Controls	Residual Risk				
				S	L	R	Rating			S	L	R	Rating	Adequate
1	Slips, trips, or falls due to spillage	E C V	Enclosed casing for caustic/water	4	3	12	M	Alert	User should use wet floor sign and clean up any spillages immediately	4	1	4	L	Yes
2	Mechanically rotating pipe work catching hair/clothing etc.	E	Safety guard with automatic shutdown if detected open	1	1	1	L	No	No additional proposals	1	1	1	L	Yes
3	Water shorting out electrical mains.	E C V	Waterproof casing on control box, but not on extension lead.	4	2	8	M	Alert	Operator must ensure that extension lead is securely elevated where no fluid can reach before switching on.	4	1	4	L	Yes
4						0	-	-				0	-	-
5						0	-	-				0	-	-

S= Severity L = Likelihood R = Risk Rating

Heineken UK Risk Assessment

Site: Department

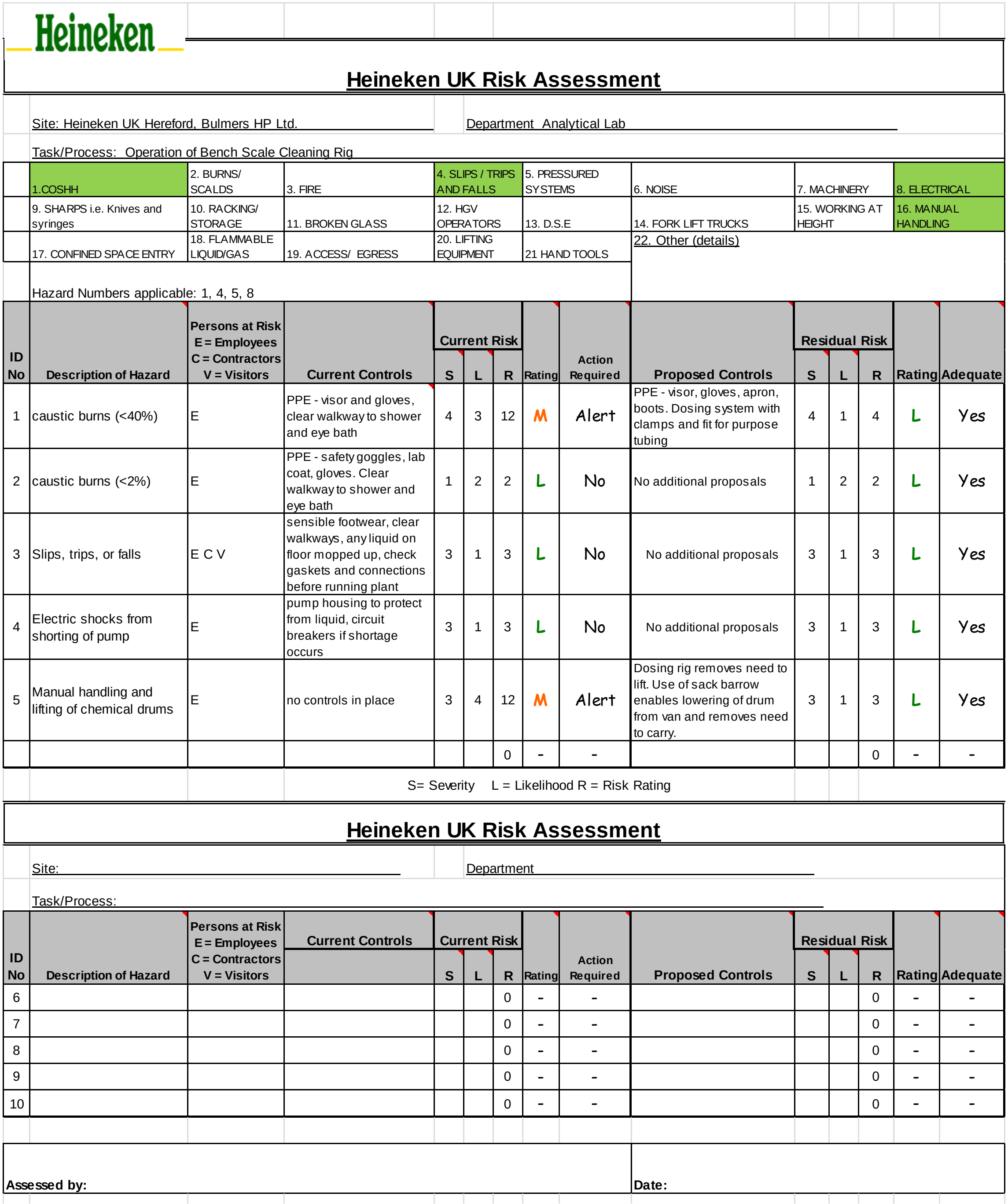
Task/Process:

ID No	Description of Hazard	Persons at Risk E = Employees C = Contractors V = Visitors	Current Controls	Current Risk				Action Required	Proposed Controls	Residual Risk				
				S	L	R	Rating			S	L	R	Rating	Adequate
6						0	-	-				0	-	-
7						0	-	-				0	-	-
8						0	-	-				0	-	-
9						0	-	-				0	-	-
10						0	-	-				0	-	-

Assessed by: **Date:**

Figure 64: Risk Assessment for Rotational Fouling Device

Appendix 26 – Risk Assessment for Pilot Plant Experiments



Heineken UK Risk Assessment

Site: Heineken UK Hereford, Bulmers HP Ltd.

Department Analytical Lab

Task/Process: Operation of Bench Scale Cleaning Rig

1.COSHH	2. BURNS/ SCALDS	3. FIRE	4. SLIPS / TRIPS AND FALLS	5. PRESSURED SYSTEMS	6. NOISE	7. MACHINERY	8. ELECTRICAL
9. SHARPS i.e. Knives and syringes	10. RACKING/ STORAGE	11. BROKEN GLASS	12. HGV OPERATORS	13. D.S.E	14. FORK LIFT TRUCKS	15. WORKING AT HEIGHT	16. MANUAL HANDLING
17. CONFINED SPACE ENTRY	18. FLAMMABLE LIQUID/GAS	19. ACCESS/ EGRESS	20. LIFTING EQUIPMENT	21 HAND TOOLS	22. Other (details)		

Hazard Numbers applicable: 1, 4, 5, 8

ID No	Description of Hazard	Persons at Risk E = Employees C = Contractors V = Visitors	Current Controls	Current Risk			Rating	Action Required	Proposed Controls	Residual Risk			Rating	Adequate
				S	L	R				S	L	R		
1	caustic burns (<40%)	E	PPE - visor and gloves, clear walkway to shower and eye bath	4	3	12	M	Alert	PPE - visor, gloves, apron, boots. Dosing system with clamps and fit for purpose tubing	4	1	4	L	Yes
2	caustic burns (<2%)	E	PPE - safety goggles, lab coat, gloves. Clear walkway to shower and eye bath	1	2	2	L	No	No additional proposals	1	2	2	L	Yes
3	Slips, trips, or falls	E C V	sensible footwear, clear walkways, any liquid on floor mopped up, check gaskets and connections before running plant	3	1	3	L	No	No additional proposals	3	1	3	L	Yes
4	Electric shocks from shorting of pump	E	pump housing to protect from liquid, circuit breakers if shortage occurs	3	1	3	L	No	No additional proposals	3	1	3	L	Yes
5	Manual handling and lifting of chemical drums	E	no controls in place	3	4	12	M	Alert	Dosing rig removes need to lift. Use of sack barrow enables lowering of drum from van and removes need to carry.	3	1	3	L	Yes
						0	-	-				0	-	-

S= Severity L = Likelihood R = Risk Rating

Heineken UK Risk Assessment

Site:

Department

Task/Process:

ID No	Description of Hazard	Persons at Risk E = Employees C = Contractors V = Visitors	Current Controls	Current Risk			Rating	Action Required	Proposed Controls	Residual Risk			Rating	Adequate
				S	L	R				S	L	R		
6						0	-	-				0	-	-
7						0	-	-				0	-	-
8						0	-	-				0	-	-
9						0	-	-				0	-	-
10						0	-	-				0	-	-

Assessed by:

Date:

Figure 65: Risk Assessment for Pilot Plant Trials

Appendix 27 – Safety Brief for Pilot Plant Experiment Assistants

Introduction

The pilot plant trials in Birmingham are part of a project for Heineken for optimising their cleaning-in-place (CIP) procedures. The CIP pilot plant will be used to clean test section pipes which have been fouled with beer/cider fouling which is typically found in the plant after production.

The pilot plant will be monitoring these cleans using online measurements (particle count, turbidity, conductivity, flow, temperature, etc.). The cleaning parameters of detergent temperature, chemical strength and flow rate will be varied so that the effects of varying the parameters can be determined.

Cleans on site are currently based on cleaning the equipment for fixed periods of time which are known to clean the equipment fully. The data collected from these experiments will be used to develop a predictive model which may be implemented within the Heineken CIP systems to optimise their cleaning times and improve the confidence of their cleans. This will result in reduced cleaning times and associated costs of chemicals, water usage, effluent, utilities, etc.

SOPs

Standard operating procedures for the pilot plant in three different phases have been provided to give a general idea of the tasks which will be taking place. You do not need to learn these, it is just for your information and training will be provided on the arrival.

Health and Safety

A risk assessment has also been provided in a separate document, but the key risks you need to be aware of are highlighted below.

Key Risks

Chemicals

We will be handling sodium hydroxide solutions of up to 40%w/v. This will burn you or worse if you get it on your skin so extra care has to be taken. The solutions in the plant will not be greater than 2% maximum which is unlikely to harm you but care must still be taken. Lab coats and safety goggles should be worn at all times whilst the plant is in operation. Gloves must be worn to handle the pipework and equipment.

Prior to making a batch of solution, concentrated sodium hydroxide must be added to the tank. A fit for purpose dosing rig has been put in for this to remove the need for human contact and reduce the risk. Whilst connecting the dosing system to the drum, the operator should be wearing nylon gloves and apron, protective footwear and a visor in addition to the lab coat.

If an accident should happen, the sodium hydroxide must be rinsed off with cold water immediately and medical attention sought from a first aider in the chemical engineering building. There is a shower and eye bath next to the pilot plant. It is important to keep the walkway to this clear at all times. These will be shown to you on your first day.

Manual Handling

The chemical drums are approximately 30kg and outside of the manual handling limits. A sack barrow will be used to lower these drums from the van, with two people working together to lower the drum onto the wheels on the ground. The drums may then be wheeled inside with no need to lift them at all.

Slips, trips and falls

Care must be taken to ensure that all walkways are cleared to avoid trips, and wet floors are mopped up immediately. Bags and personal belongings must be tucked away from the walkways.

PPE

You will need to bring your own lab coat and safety goggles. Everything else will be provided.

- Safety goggles
- Lab coats
- Gloves
- Visor and apron if handling concentrated caustic

What You Need

- Old clothes and comfy shoes
- Lab coat and safety goggles
- Learn as you go so no need to spend lots of time preparing

Appendix 28 – Experimental Design for Predictive Model Cleaning Trials

The experimental design for the later cleaning predictive model trials at the Birmingham Pilot plant is as follows:

Full fouling (300ml beer bottoms evenly coated and dried on with the rotational device and a fan).

Table 11: Experimental Design for Detailed Predictive Model Experiments (Heavy Fouling)

Flow Rate ($m^3.h^{-1}$)	**NaOH Concentration (%w/v)**			
	0	**0.5**	**0.75**	**1.0**
10	1	2	5	2
12	0	2	5	2
14	0	2	5	2
16	2	4	4	2

Intermediate fouling (40ml beer bottoms evenly coated and dried on with the rotational device and a fan).

Table 12: Experimental Design for Detailed Predictive Model Experiments (Intermediate Fouling)

Flow Rate ($m^3.h^{-1}$)	**NaOH Concentration (%w/v)**	
	0.5	**1**
10	2	2
12	2	2
14	2	3
16	3	2

The values were selected based on time constraints of the experimental trials, prioritising runs which were of more interest to the investigation. This includes runs at the boundaries of the experimental design to ensure that the optimal cleans were selected within those boundaries.