# Optimisation of Cleaning Detergent use in Brewery Fermenter Cleaning

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This paper investigates improvement possibilities in the cleaning operations undertaken at an industrial brewery. Experiments were performed on a bench scale cleaning rig which was designed to simulate ‘real life’ cleaning conditions of a clean-in-place (CIP) set in the brewery. The rig was used to clean consistently fouled coupons using difficult soils from the brewery. The objective of the experiments was to determine the reduction in effective cleaning performance with varied levels of $Na_2CO_3$ in the detergent from NaOH degradation and the maximum level that may be present before cleaning quality is impacted. The shear force of the cleaning fluid across the surface of the coupon was also varied to determine the impact on cleaning performance. Data collected from these offline measurements has been used to predict the end point of the detergent usage based on cost optimisation within the empirically determined limits. The results show that the NaOH detergent usage can be extended while achieving the same time to clean without impacting the cleaning quality and preventing premature disposal. This will provide an increased confidence level when cleaning fermenters with NaOH. It will also reduce cleaning costs and benefit the environment by reducing chemical effluent and minimising water consumption.

Sodium hydroxide, cleaning-in-place, sodium carbonate, optimisation, fermentation, brewing

Highlights

Bench scale cleaning analysis of brewery soils

Understanding of cleaning chemical effectiveness in brewery cleaning

Increasing the time of use of cleaning fluids in a brewery

## Introduction

Effective process cleaning in a brewery is an essential business requirement to achieve consistently high standards of product quality and hygiene but it can be a costly undertaking. Current Clean-in-Place (CIP) systems can exhibit lengthy cleaning times causing production down time and lost production capacity, increased effluent treatment and higher utility costs. Ineffective cleaning of equipment in the brewing industry is detrimental to the end product quality with respect to taste, appearance and conformance to health and safety legislation. Hence the length of clean is increased and specified to accommodate uncertainty and variation in cleaning behaviour so that such issues do not arise. Variations in cleaning time occur as a consequence of product changeover, where one product requires a more vigorous clean than the other and for the same product general batch to batch variation, where the cleaning parameters may meet the requirements of one batch but it may not be sufficient to clean another batch.

The literature associated with cleaning and CIP improvement considers scales from cleaning fluid - soil –surface interaction to how this impacts on the behaviour of large process plant. A review of process cleaning highlighting the challenges facing industry can be found in Wilson (2005), with a more recent review by Goode et al (2013). At the surface scale Kaye et al (1995) investigated the effect of jet cleaning on a soiled surface. They highlighted the nature of surface removal by jet cleaning and the importance of optimizing operating parameters such as turbulence and jet velocity over the surface to ensure effective cleaning. Palabiyik *et al* (2015) recently similarly considered the mechanisms of soil removal and how shear rate could be varied during the clean to minimise the use of cleaning fluid to deliver the most effective clean. Cleaning fluid temperature and velocity were varied to accommodate the changes in the mechanisms of removal. Lewis et al (2012) considered the cleaning of biofilms from membranes and in studies on yeast observed the relationship between cleaning fluid velocity and thus shear stress and biofilm removal. A more quantitative approach to assessing the effectiveness of process cleaning was taken by Köhler et al (2015) who sought to optimise the cleaning parameters when using a moving jet to clean a Xanthan gum soiled surface. They considered time to clean, fluid used, energy used and overall cost as metrics. It was observed that a global optimum of all four metrics cannot be achieved and a balance is required as specific circumstances dictate. In their studies they considered the design properties of the nozzle (nozzle diameter, gauge pressure and jet moving speed) and the velocity of the fluid. This work was expanded on further by Wilson et al (2015) who developed a mathematical model to provide predictive performance of the system consider by Köhler and achieved good agreement with experimental results. The need to improve cleaning systems and the requirement for more informative measurements in attempts to optimize cleaning when natural variation occurs was considered by Van Asselt et al (2002) who highlighted the benefits of conductivity measurements in dairy process cleaning.

When considering the addition of chemicals to enhance cleaning, Eide et al (2003) consider the optimization of cleaning chemical choice demonstrating in their case the enhanced performance provided by sodium hydroxide. Christian and Fryer (2006) studied the impact of changing Sodium Hydroxide concentration and variations in fluid flow to clean whey protein. They observed the need to have long enough exposure of the soil to cleaning agent at sufficient concentration to cause the soil to swell before removal. Constant flow was not necessary and therefore cleaning chemical usage could be reduced. Fryer et al (2011) considered the impact and predictability of cleaning as scale of operation changes and whether predictive performance could be achieved across soils. They observed that for certain soil types predictive performance could be achieved but for others complex relationships existed. Considering brewery cleaning in particular several publications have highlighted the high costs associated with cleaning and options for improvement. For instance, Pettigrew et al (2015) in addition to describing the brewery process and cleaning costs, developed a simulation of the brewery CIP system and formulated an optimisation approach based on the use of an object oriented Petri net to improve water usage in part of the brewery. Goode et al (2010) investigated the optimisation of brewery cleaning with respect to cleaning fluid temperature and cleaning agent concentration and suggested that lower temperature cleaning could be effective.

While such scientific studies grow fundamental understanding, practical considerations remain to be addressed. Sodium hydroxide is commonly used as a cleaning detergent in the brewing industry and is known to effectively clean brewery soils but its use is not without problems. These include; i) the level of cleanliness of the equipment surfaces is unknown before or during the clean due to measurement limitations, ii) formation of sodium carbonate in the caustic solution, which reduces the cleaning power and can sometimes result in chemical cleans which are not within specification being performed. A cautious approach however causes excessive and expensive disposal of cleaning chemicals, iii) 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.

This paper addresses one of these issues in particular, the formation of sodium carbonate ($Na_2CO_3$) in sodium hydroxide (NaOH) cleaning detergents commonly used in FMCG process cleaning. This is a common challenge encountered by the brewing and bio-processing industry in the fermentation process, the fundamental engineering and chemistry of which is considered by Hikita et al (1976). It is for this reason we concentrate on fermenter cleaning. Sodium carbonate formation occurs due to the presence of residual carbon dioxide ($CO_2$) in vessels as a by-product of fermentation. Cleaning pre-requisites set maximum $Na_2CO_3$ limits permissible in the detergent which will potentially result in premature disposal of the detergent with increased costs, effluent, environmental impact, and water and utility consumption.

$Na_2CO_3$ is a cleaning agent itself, but its cleaning ability in conjunction with NaOH has not been quantified, neither has there been any investigation as to whether there are any inhibitory effects on the cleaning ability of NaOH. Pre-requisite levels of $Na_2CO_3$ and NaOH have been put in place at the brewery considered in the study based on industry generic empirical values provided by external cleaning companies. The strength of the NaOH within a CIP cycle is measured continuously online using a measure of conductivity, thus providing feedback information on the chemical cleaning step in place, and the theoretical quantity of active NaOH present during the detergent step. The NaOH strength is increased if the level of conductivity is not sufficient.

This paper considers a different aspect to previous cleaning studies. Other studies typified above have concentrated on physical cleaning approaches and their effectiveness at design levels of operation. This paper addresses how these are impacted by degradation in the design conditions. There is no available literature on the recommended limits of the minimum NaOH and maximum $Na_2CO_3$ levels, before the cleaning ability of the solution is reduced to a point where it ceases to clean effectively. Furthermore, high levels of $Na_2CO_3$ and low levels of NaOH may provide a sufficiently high conductivity reading to provide 'false' feedback information in terms of it indicating the presence of a theoretically higher quantity of active NaOH. This paper investigates the cleaning abilities of NaOH and $Na_2CO_3$, measurements required to assess their concentration and limits to optimise the detergent step in a CIP cycle thereby improving confidence in cleaning.

## Experimental Approach

(a) Methods - A bench scale cleaning rig was developed to represent 'real life' brewery cleaning conditions (Figure 1). The rig consisted of a small tank which contained a 4 litre solution of cleaning detergent to be recirculated via peristaltic tubing and a centrifugal pump into a nozzle which sprayed the solution directly onto a suspended 5cm square stainless steel 316L coupon. The coupon was prepared by taking 5g of post filtered beer bottoms and spreading it evenly across the surface area leaving a coating of around 1g ± 0.01g of dried, evenly spread, post filtered beer bottoms. The soil was allowed to completely dry for two days to complete coupon preparation under ambient conditions. All coupons were prepared at the same time to minimize humidity variation impact. The importance of careful and consistent soil sample preparation was described by Ishiyama et a1 (2014) and therefore rigorous attention was placed on soil sample preparation as described above. Beer bottoms were used as they represented a worst case soil scenario and provided a repeatability not possible with foam soiling. A bypass valve was used on the peristaltic tubing to enable variation of flow rate through the cleaning nozzle. The hose nozzle is sprayed directly onto the top of the fouled coupon to form a waterfall type effect over the coupon.

The design specification of the mini rig was based on scaled down values of the direct forces and shear forces of fluid falling down the walls from the direct impact of a cleaning head spray jet with a shear force of at least 3 Pa (the same as that in a large scale fermenter of 7000hl volume on site (Jensen, 2012)). This involved using flowrates of 50ml/s and 100 ml/s. The jet was directed at an angle of $60^{o}$ to the coupon at a distance of 30cm from the coupon. This represents a scaled down mimic of an average position in the foam line of the tank. The nozzle diameter was 5mm.

A full factorial experimental design was undertaken that covered all combinations of NaOH and $Na_2CO_3$ at fixed intervals between 0 and 2% w/v and 0 and 12% w/v respectively. Cleaning at two different flow rates was also considered and each combination was performed in triplicate. Table 1 provides details of the design.

A total of 90 experimental runs were performed on the rig under ambient temperature conditions consistent with industrial operation. For each run a fresh 4l solution was made and recirculated for 30s to ensure that the solution was well mixed. Fresh solutions for each run ensured that decreased surface tension due to increased quantities of suspended solids within the solutions did not have an impact on the results. A fouled coupon was then suspended in the tank and the transparent Perspex lid closed. The pump was switched on to begin recirculation and the cleaning of the coupon was observed and timed until the coupon was visibly clean. Images of the coupon were taken at this point to document the results. Visibly clean was selected as the measure for cleanliness as the detergent step is used to remove soils and a nitric acid sanitation step is always performed after the detergent step when cleaning brewery equipment and is consistent with the approach adopted in the brewery. Approaches to verify the visibly clean metric were based on the underlying principles found in Nostrand and Forsyth (2005). If the coupon was not visibly clean by 600s it was assumed that this solution would not be sufficient to clean the soil, as no area within a vessel would be exposed to cleaning solution at this force, for this amount of time, in a 'real life' cleaning scenario.

Samples of each solution were taken at the end of each run from the discharge point and titrated to verify the correct combination of chemicals within the solutions had been used. pH and conductivity readings were also taken to investigate the relationships between these measurements and the strength of the individual solution components. The conductivity probe used was an Omega CDH-280 and the pH meter was a Mettler Toledo Five Easy FE20. Both were desktop offline probes.

(b) Results - The table of results is too large to be included in this paper, but the trends and general interactions between the variables are discussed. A general linear model was developed using Minitab® 16.2.4 which included the input variables (flow rate, NaOH concentration and $Na_2CO_3$ concentration) and the output variable, cleaning time. The linear modelling approach is adopted following the good modelling practice approach that the model should be as simple as possible as long as it is effective. All input variables were shown to have first and second order interactions and have been included in the model.

Figure 2 shows the interaction plot for the individual variables of NaOH concentration, $Na_2CO_3$ concentration and cleaning time. This shows that if no NaOH is present, then the detergent generally will not clean, but it will clean slowly with 2-4% $Na_2CO_3$ present, hence water alone will not clean. NaOH >1% will clean well unless the $Na_2CO_3$ level is 12% or more, so $Na_2CO_3$ does not inhibit cleaning sufficiently until this point. However, sodium carbonate levels present at 12% will still clean with a sufficiently high flow rate. In this figure and subsequent figures the titrations to determine NaOH and $Na_2CO_3$ concentrations result in errors of concentration determination of ±0.15w/v. The error associated with flowrate measurement is ±2ml/s.The results also show that there is a strong dependency of cleaning ability on the flow rate, showing that higher flow rates improve cleaning abilities.

Figure 3 shows the contour plot for NaOH concentration and $Na_2CO_3$ concentration based on cleaning time. The blue areas are those that cleaned in the shortest time and thus are considered to denote the conditions that give the best cleaning. It can be seen that 1% NaOH and 9% $Na_2CO_3$ denote the limits of the fastest cleaning times. These are denoted by the red dashed lines. The section between 2 and 4% $Na_2CO_3$ with less than 1% NaOH also shows a slight cleaning power of $Na_2CO_3$ alone where cleaning is taking place with no (or little) NaOH present. This section is in a lighter shade of blue which shows that although it does clean at this strength without the presence of NaOH. This will not be sufficient to clean the fermentation vessel effectively as the cleaning time required for cleaning the vessel with this solution will be approximately three times longer than it is currently. This deems it less cost effective and fails to satisfy the objective of cleaning detergent cost based optimization where at least cleaning within the current time frame is the objective.

Two further general linear models were developed in Minitab® 16.2.4 based on the offline measurements of conductivity and $H^+$ ions which were recorded from each of the experimental samples.

Figure 4 shows the interaction plot for NaOH concentration, $Na_2CO_3$ concentration and conductivity. The error associated with conductivity measurement is ±1.0mS/cm$^2$. It can be seen that 1% NaOH gives a conductivity reading which is the same as approximately 5% $Na_2CO_3$. Due to this, it is possible that readings from a conductivity probe will give a false security of detergent specifications. Readings of NaOH < 1% and $Na_2CO_3$ > 5% will appear to be within specification.

Figure 5 shows the interaction plots for NaOH concentration, $Na_2CO_3$ concentration, and pH values. The error associated with pH measurement is ±0.008. It can be seen that samples of only water will have a pH of less than 10. Some water samples have pH values as high as 10 due to residual traces of alkaline remaining in the experimental rig pipework from previous runs. Solutions of $Na_2CO_3$ alone will have a pH of approximately 12 and NaOH solutions will have a pH of approximately 13. When combined solutions of NaOH and $Na_2CO_3$ are present which contain more than 1% NaOH, the pH of NaOH appears to dominate the overall pH, resulting in a pH of 13-13.5. This is due to the reduction of dissociation of $H^+$ ions within the solution based on the hydroxide and carbonate ions together, resulting in a higher pH when NaOH is present. This shows that the use of a pH probe will enable the determination of the presence of NaOH or $Na_2CO_3$.

## Discussion

(a) Chemical Limits - The investigation based on the chemical concentrations within the cleaning detergent has shown that NaOH needs to be at least 1% w/v for the clean to be effective. NaOH concentrations greater than 1% make no significant improvements in terms of the cleaning abilities demonstrating that it is not cost effective to clean in industry with NaOH strengths of greater than 1% w/v as there is no additional cleaning benefit.

$Na_2CO_3$ has been shown to have a cleaning ability on brewery soils between 2-4% w/v but is not sufficient for cleaning brewery equipment as a sole detergent. Increasing concentrations of $Na_2CO_3$ appear to inhibit the cleaning abilities of NaOH slightly, but not enough to prevent sufficient cleaning until concentrations of greater than 9%. Although concentrations of $Na_2CO_3$ up to 9% will have some impact on cleaning abilities, it will be most cost effective to allow the strength to reach 9% before replacing the detergent as cleaning will still be effective enough to visibly clean a worst case scenario brewery soil up until this point.

Cleaning flow rate is important when cleaning and this has been verified in the work of Goode et al (2010). Industrial cleaning with higher flow rates will enable a higher $Na_2CO_3$ limit to be put in place and the cleaning detergent to be replaced less frequently. It is necessary to ensure that the process can consistently achieve the required flow rate when cleaning all equipment before selecting a higher $Na_2CO_3$ limit. If the minimum flow/pressure requirements specified by the cleaning head manufacturers are not reached then the $Na_2CO_3$ levels will have more of an impact on the NaOH cleaning at lower levels.

The recommended chemical limits within the detergent cleaning step at the minimum required flow conditions are NaOH $>$ 1% w/v and $Na_2CO_3$ $<$ 9% w/v. Implementation of these limits on one of Heineken's sites will yield an estimated 56% chemical cost saving. This value was determined by performing industrial cost benchmark analysis, adopting the techniques developed by Ahmad and Benson (2000) and through analysis of cleaning data that is commercially sensitive although but the underlying principles of Ahmad and Benson cover generic application and transferability of the methods discussed.

(b) Online Measurements - The use of conductivity alone as an industrial method of online measurement of the active NaOH concentration present within the detergent is not effective when continuous dosing of NaOH is applied. There is typically more than 700 hl of residual $CO_2$ from the 10% headspace of a 7000 hl fermentation vessel. This is more than sufficient to achieve high levels of $Na_2CO_3$ when continuous NaOH dosing. If more than 5% $Na_2CO_3$ is present it will show that the conductivity is sufficiently high when insufficient NaOH is present due to the conductivity associated with $Na_2CO_3$. This is not a suitable industrial method as incorrect indications of NaOH levels will result in ineffective cleaning which may have an impact on microbial growth within the equipment, resulting in spoilage of product and additional costs to the company. Conductivity does give an indication of the quantity of ions present and can be used in conjunction with further information to provide a better indication of the detergent chemical concentrations.

An online pH probe will provide information on the minimum strengths of NaOH and $Na_2CO_3$ present. Combining this information with the online conductivity information by data fusion will enable confidence that at least 1% NaOH is present and an indication of when $Na_2CO_3$ strength is increasing. It is sufficient to consider both signals together but conductivity or pH alone is not informative. Additional flow monitoring of any NaOH added to the detergent will be required to ensure that concentrations of $Na_2CO_3$ in excess of 9% may not be achieved. Using this method will provide operational confidence and ensure that cleaning is being performed to an acceptable standard throughout the full duration of the detergent cleaning step.

Implementation of the determined chemical limits within the detergent, and application of a cost optimisation technique incorporating the data fusion of pH, conductivity, and flow monitoring of concentrated NaOH will provide cost savings on one Heineken site of 56% in cleaning chemical costs, which contributes to 10% of total cleaning costs on the fermentation vessels. The resulting operational savings provide a payback time on capital investment by the business for this change of less than eight months.

## Conclusions

This paper has considered the degradation of NaOH during the cleaning of brewery process equipment. It was known previously that $Na_2CO_3$ formation degraded cleaning ability and this paper has quantified the extent of this loss of performance. This quantification has enabled a more informed and optimised CIP strategy to be implemented in brewery operations. To do so requires additional on-line measurements to be made to distinguish between NaOH and $Na_2CO_3$ compositions. It has been shown that with measures of pH and conductivity of the cleaning fluid it is possible to gain this information and consequently be able to determine the current cleaning capability.

Considering future work the prime activity is to assess long term returns to ensure that short term gains are maintained before technology ‘roll out’ to other Heineken sites. Further technical studies also follow in from this work such as 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. Methods to deal with problem root cause are also worthy of exploration such as the removal of carbon dioxide through nitrogen purging or alternative cleaning detergents which will not react with carbon dioxide for a long term cost effective solution. 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 but for existing equipment costs are prohibitive.

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**Tables**

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

Table 1. Details of full factorial experimental design

**Figures**

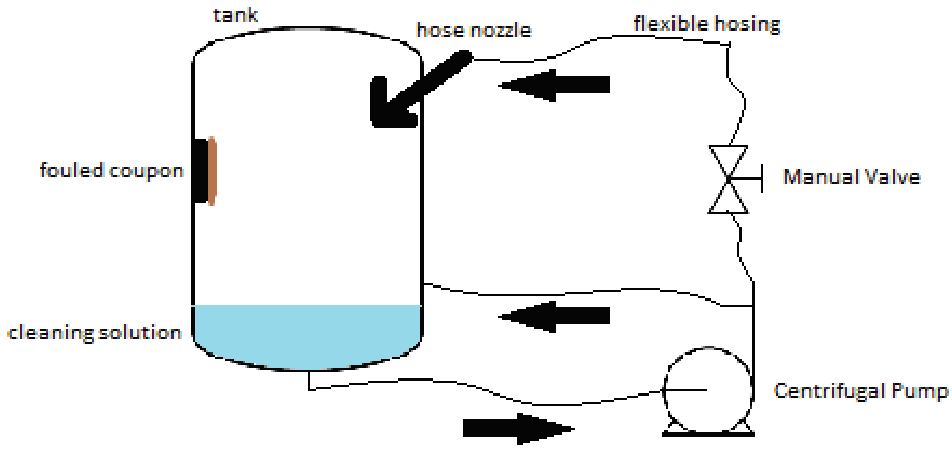


Figure 1. Bench scale cleaning rig

Figure 2. Interaction plot for the variables of NaOH concentration, $Na_2CO_3$ concentration and cleaning time.

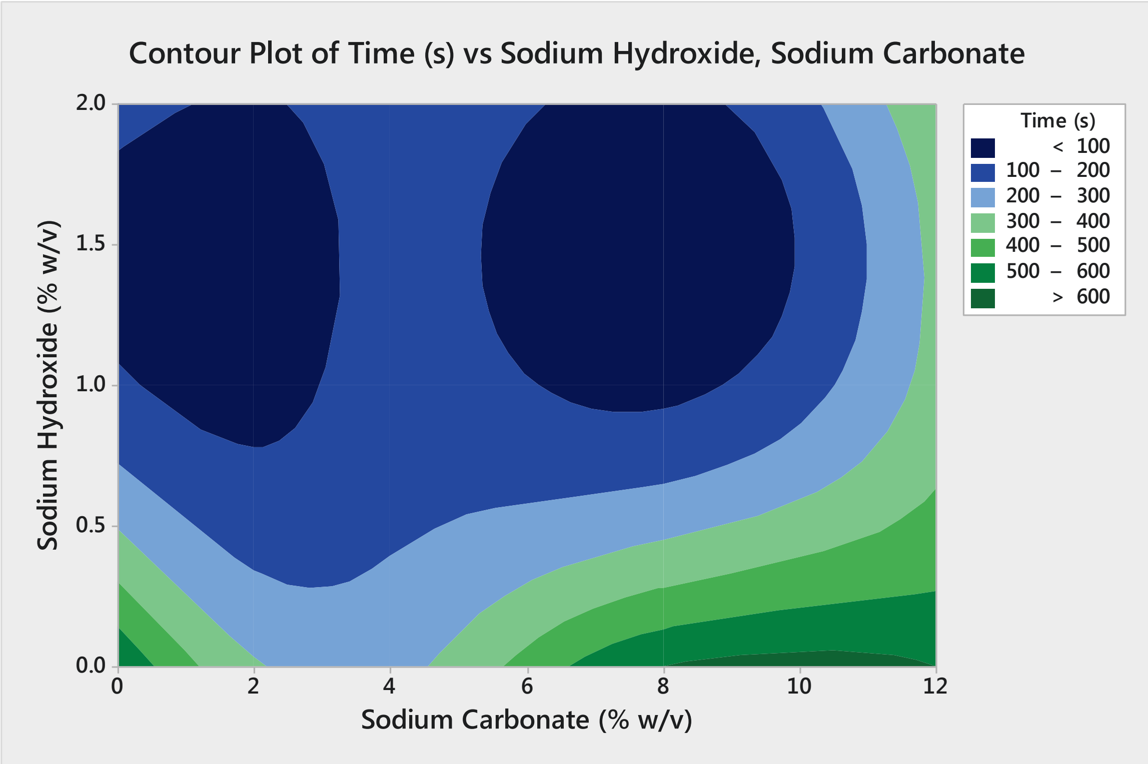


Figure 3. Contour plot for NaOH concentration and $Na_2CO_3$ concentration based on cleaning time

Figure 4. Interaction plot for NaOH concentration, $Na_2CO_3$ concentration and conductivity.

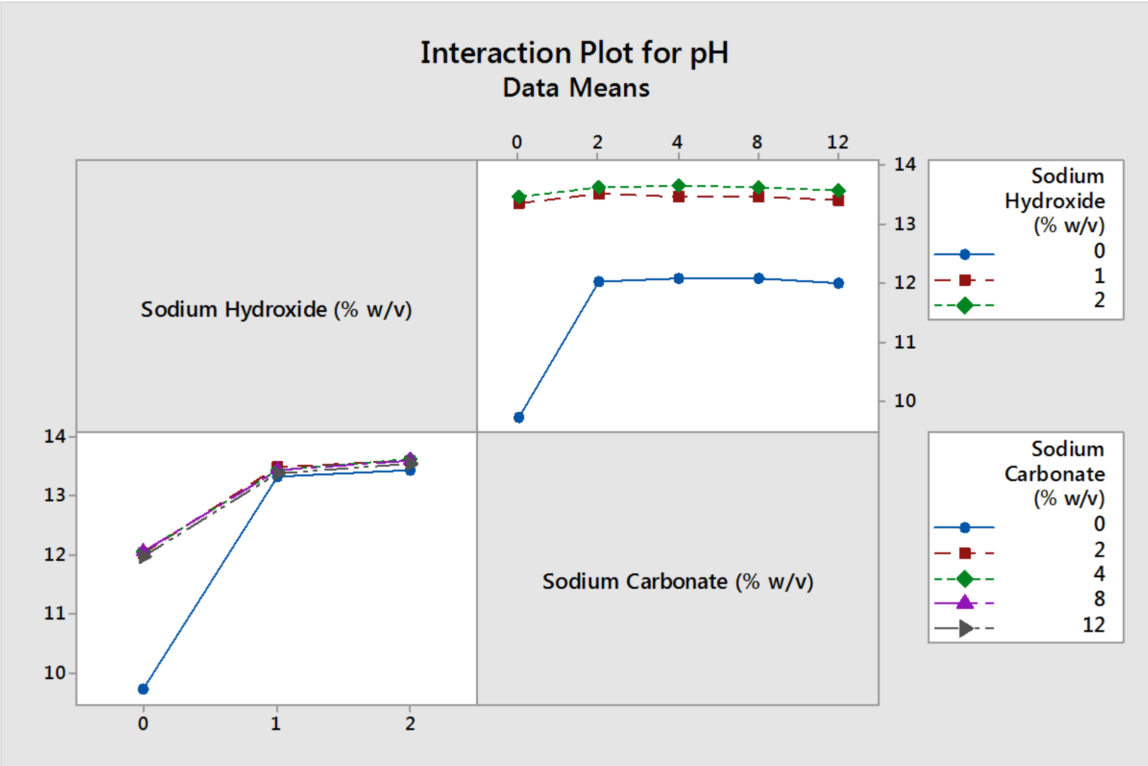


Figure 5. Interaction plots for NaOH concentration, $Na_2CO_3$ concentration, and pH values