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1 **Optimisation of Cleaning Detergent use in Brewery Fermenter Cleaning**

2
3 Charlotte Atwell^a, Elaine Martin^b, Gary Montague^{c*}, Jeroen Swuste^d, Mark Picksley^e

4
5
6 (a) Biopharmaceutical and Bioprocessing Technology Centre, School of Chemical
7 Engineering and Advanced Materials, Merz Court, Newcastle University, NE1 7RU,
8 United Kingdom.

9 (b) School of Chemical and Process Engineering, Engineering Building, University of
10 Leeds, Leeds, LS2 9JT, United Kingdom.

11 (c) School of Science and Engineering, Teesside University, Middlesbrough, Tees
12 Valley, TS1 3BA, United Kingdom.

13 (d) Heineken Global Supply Chain, Burgermeester Smeetsweg 1, 2382 PH
14 Zoeterwoude P.O. Box 510, 2380 BB Zoeterwoude, The Netherlands.

15 (e) Heineken UK, 2-4 Broadway Park, South Gyle Broadway, Edinburgh EH12 9JZ,
16 United Kingdom.

17
18
19
20
21 *Corresponding author

22 E.mail : g.montague@tees.ac.uk

23 Phone: +44 7590 371408

24
25
26

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

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

46

47 Highlights

48 Bench scale cleaning analysis of brewery soils

49 Understanding of cleaning chemical effectiveness in brewery cleaning

50 Increasing the time of use of cleaning fluids in a brewery

51

52

53 **Introduction**

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

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

93 When considering the addition of chemicals to enhance cleaning, Eide et al (2003)
94 consider the optimization of cleaning chemical choice demonstrating in their case the

95 enhanced performance provided by sodium hydroxide. Christian and Fryer (2006)
96 studied the impact of changing Sodium Hydroxide concentration and variations in fluid
97 flow to clean whey protein. They observed the need to have long enough exposure of
98 the soil to cleaning agent at sufficient concentration to cause the soil to swell before
99 removal. Constant flow was not necessary and therefore cleaning chemical usage could
100 be reduced. Fryer et al (2011) considered the impact and predictability of cleaning as
101 scale of operation changes and whether predictive performance could be achieved
102 across soils. They observed that for certain soil types predictive performance could be
103 achieved but for others complex relationships existed. Considering brewery cleaning in
104 particular several publications have highlighted the high costs associated with cleaning
105 and options for improvement. For instance, Pettigrew et al (2015) in addition to
106 describing the brewery process and cleaning costs, developed a simulation of the
107 brewery CIP system and formulated an optimisation approach based on the use of an
108 object oriented Petri net to improve water usage in part of the brewery. Goode et al
109 (2010) investigated the optimisation of brewery cleaning with respect to cleaning fluid
110 temperature and cleaning agent concentration and suggested that lower temperature
111 cleaning could be effective.

112 While such scientific studies grow fundamental understanding, practical considerations
113 remain to be addressed. Sodium hydroxide is commonly used as a cleaning detergent
114 in the brewing industry and is known to effectively clean brewery soils but its use is
115 not without problems. These include; i) the level of cleanliness of the equipment
116 surfaces is unknown before or during the clean due to measurement limitations, ii)
117 formation of sodium carbonate in the caustic solution, which reduces the cleaning
118 power and can sometimes result in chemical cleans which are not within specification
119 being performed. A cautious approach however causes excessive and expensive
120 disposal of cleaning chemicals, iii) Uncertainty about the effectiveness of the cleans
121 provided by different types of spray heads in vessels, iv) tanks, filters, and heat
122 exchangers are more complex than ordinary pipe work is to clean.

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

133 Na_2CO_3 is a cleaning agent itself, but its cleaning ability in conjunction with NaOH has
134 not been quantified, neither has there been any investigation as to whether there are any
135 inhibitory effects on the cleaning ability of NaOH . Pre-requisite levels of Na_2CO_3 and
136 NaOH have been put in place at the brewery considered in the study based on industry

137 generic empirical values provided by external cleaning companies. The strength of the
138 NaOH within a CIP cycle is measured continuously online using a measure of
139 conductivity, thus providing feedback information on the chemical cleaning step in
140 place, and the theoretical quantity of active NaOH present during the detergent step.
141 The NaOH strength is increased if the level of conductivity is not sufficient.

142 This paper considers a different aspect to previous cleaning studies. Other studies
143 typified above have concentrated on physical cleaning approaches and their
144 effectiveness at design levels of operation. This paper addresses how these are impacted
145 by degradation in the design conditions. There is no available literature on the
146 recommended limits of the minimum NaOH and maximum Na₂CO₃ levels, before the
147 cleaning ability of the solution is reduced to a point where it ceases to clean effectively.
148 Furthermore, high levels of Na₂CO₃ and low levels of NaOH may provide a sufficiently
149 high conductivity reading to provide 'false' feedback information in terms of it
150 indicating the presence of a theoretically higher quantity of active NaOH. This paper
151 investigates the cleaning abilities of NaOH and Na₂CO₃, measurements required to
152 assess their concentration and limits to optimise the detergent step in a CIP cycle
153 thereby improving confidence in cleaning.

154

155 **Experimental Approach**

156 (a) Methods - A bench scale cleaning rig was developed to represent 'real life' brewery
157 cleaning conditions (Figure 1). The rig consisted of a small tank which contained a
158 4 litre solution of cleaning detergent to be recirculated via peristaltic tubing and a
159 centrifugal pump into a nozzle which sprayed the solution directly onto a suspended
160 5cm square stainless steel 316L coupon. The coupon was prepared by taking 5g of
161 post filtered beer bottoms and spreading it evenly across the surface area leaving a
162 coating of around $1\text{g} \pm 0.01\text{g}$ of dried, evenly spread, post filtered beer bottoms. The
163 soil was allowed to completely dry for two days to complete coupon preparation
164 under ambient conditions. All coupons were prepared at the same time to minimize
165 humidity variation impact. The importance of careful and consistent soil sample
166 preparation was described by Ishiyama et al (2014) and therefore rigorous attention
167 was placed on soil sample preparation as described above. Beer bottoms were used
168 as they represented a worst case soil scenario and provided a repeatability not
169 possible with foam soiling. A bypass valve was used on the peristaltic tubing to
170 enable variation of flow rate through the cleaning nozzle. The hose nozzle is sprayed
171 directly onto the top of the fouled coupon to form a waterfall type effect over the
172 coupon.

173 The design specification of the mini rig was based on scaled down values of the direct
174 forces and shear forces of fluid falling down the walls from the direct impact of a
175 cleaning head spray jet with a shear force of at least 3 Pa (the same as that in a large
176 scale fermenter of 7000hl volume on site (Jensen, 2012)). This involved using flowrates

177 of 50ml/s and 100 ml/s. The jet was directed at an angle of 60° to the coupon at a
178 distance of 30cm from the coupon. This represents a scaled down mimic of an average
179 position in the foam line of the tank. The nozzle diameter was 5mm.

180 A full factorial experimental design was undertaken that covered all combinations of
181 NaOH and Na₂CO₃ at fixed intervals between 0 and 2% w/v and 0 and 12% w/v
182 respectively. Cleaning at two different flow rates was also considered and each
183 combination was performed in triplicate. Table 1 provides details of the design.

184 A total of 90 experimental runs were performed on the rig under ambient temperature
185 conditions consistent with industrial operation. For each run a fresh 4l solution was
186 made and recirculated for 30s to ensure that the solution was well mixed. Fresh
187 solutions for each run ensured that decreased surface tension due to increased quantities
188 of suspended solids within the solutions did not have an impact on the results. A fouled
189 coupon was then suspended in the tank and the transparent Perspex lid closed. The
190 pump was switched on to begin recirculation and the cleaning of the coupon was
191 observed and timed until the coupon was visibly clean. Images of the coupon were
192 taken at this point to document the results. Visibly clean was selected as the measure
193 for cleanliness as the detergent step is used to remove soils and a nitric acid sanitation
194 step is always performed after the detergent step when cleaning brewery equipment and
195 is consistent with the approach adopted in the brewery. Approaches to verify the visibly
196 clean metric were based on the underlying principles found in Nostrand and Forsyth
197 (2005). If the coupon was not visibly clean by 600s it was assumed that this solution
198 would not be sufficient to clean the soil, as no area within a vessel would be exposed
199 to cleaning solution at this force, for this amount of time, in a 'real life' cleaning
200 scenario.

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

207

208 (b) Results - The table of results is too large to be included in this paper, but the trends
209 and general interactions between the variables are discussed. A general linear model
210 was developed using Minitab® 16.2.4 which included the input variables (flow rate,
211 NaOH concentration and Na₂CO₃ concentration) and the output variable, cleaning time.
212 The linear modelling approach is adopted following the good modelling practice
213 approach that the model should be as simple as possible as long as it is effective. All
214 input variables were shown to have first and second order interactions and have been
215 included in the model.

216 Figure 2 shows the interaction plot for the individual variables of NaOH concentration,
217 Na₂CO₃ concentration and cleaning time. This shows that if no NaOH is present, then
218 the detergent generally will not clean, but it will clean slowly with 2-4% Na₂CO₃
219 present, hence water alone will not clean. NaOH >1% will clean well unless the Na₂CO₃
220 level is 12% or more, so Na₂CO₃ does not inhibit cleaning sufficiently until this point.
221 However, sodium carbonate levels present at 12% will still clean with a sufficiently
222 high flow rate. In this figure and subsequent figures the titrations to determine NaOH
223 and Na₂CO₃ concentrations result in errors of concentration determination of ±0.15w/v.
224 The error associated with flowrate measurement is ±2ml/s. The results also show that
225 there is a strong dependency of cleaning ability on the flow rate, showing that higher
226 flow rates improve cleaning abilities.

227 Figure 3 shows the contour plot for NaOH concentration and Na₂CO₃ concentration
228 based on cleaning time. The blue areas are those that cleaned in the shortest time and
229 thus are considered to denote the conditions that give the best cleaning. It can be seen
230 that 1% NaOH and 9% Na₂CO₃ denote the limits of the fastest cleaning times. These
231 are denoted by the red dashed lines. The section between 2 and 4% Na₂CO₃ with less
232 than 1% NaOH also shows a slight cleaning power of Na₂CO₃ alone where cleaning is
233 taking place with no (or little) NaOH present. This section is in a lighter shade of blue
234 which shows that although it does clean at this strength without the presence of
235 NaOH. This will not be sufficient to clean the fermentation vessel effectively as the
236 cleaning time required for cleaning the vessel with this solution will be approximately
237 three times longer than it is currently. This deems it less cost effective and fails to
238 satisfy the objective of cleaning detergent cost based optimization where at least
239 cleaning within the current time frame is the objective.

240

241

242

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

246 Figure 4 shows the interaction plot for NaOH concentration, Na₂CO₃ concentration
247 and conductivity. The error associated with conductivity measurement is ±1.0mS/cm².
248 It can be seen that 1% NaOH gives a conductivity reading which is the same as
249 approximately 5% Na₂CO₃. Due to this, it is possible that readings from a
250 conductivity probe will give a false security of detergent specifications. Readings of
251 NaOH < 1% and Na₂CO₃ > 5% will appear to be within specification.

252 Figure 5 shows the interaction plots for NaOH concentration, Na₂CO₃ concentration,
253 and pH values. The error associated with pH measurement is ±0.008. It can be seen that
254 samples of only water will have a pH of less than 10. Some water samples have pH

255 values as high as 10 due to residual traces of alkaline remaining in the experimental rig
256 pipework from previous runs. Solutions of Na_2CO_3 alone will have a pH of
257 approximately 12 and NaOH solutions will have a pH of approximately 13. When
258 combined solutions of NaOH and Na_2CO_3 are present which contain more than 1%
259 NaOH, the pH of NaOH appears to dominate the overall pH, resulting in a pH of 13-
260 13.5. This is due to the reduction of dissociation of H^+ ions within the solution based
261 on the hydroxide and carbonate ions together, resulting in a higher pH when NaOH is
262 present. This shows that the use of a pH probe will enable the determination of the
263 presence of NaOH or Na_2CO_3 .

264 Discussion

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

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

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

287 The recommended chemical limits within the detergent cleaning step at the minimum
288 required flow conditions are $\text{NaOH} > 1\%$ w/v and $\text{Na}_2\text{CO}_3 < 9\%$ w/v. Implementation
289 of these limits on one of Heineken's sites will yield an estimated 56% chemical cost
290 saving. This value was determined by performing industrial cost benchmark analysis,
291 adopting the techniques developed by Ahmad and Benson (2000) and through analysis
292 of cleaning data that is commercially sensitive although but the underlying principles
293 of Ahmad and Benson cover generic application and transferability of the methods
294 discussed.

295 (b) Online Measurements - The use of conductivity alone as an industrial method of
296 online measurement of the active NaOH concentration present within the detergent is
297 not effective when continuous dosing of NaOH is applied. There is typically more than
298 700 hl of residual CO₂ from the 10% headspace of a 7000 hl fermentation vessel. This
299 is more than sufficient to achieve high levels of Na₂CO₃ when continuous NaOH
300 dosing. If more than 5% Na₂CO₃ is present it will show that the conductivity is
301 sufficiently high when insufficient NaOH is present due to the conductivity associated
302 with Na₂CO₃. This is not a suitable industrial method as incorrect indications of NaOH
303 levels will result in ineffective cleaning which may have an impact on microbial growth
304 within the equipment, resulting in spoilage of product and additional costs to the
305 company. Conductivity does give an indication of the quantity of ions present and can
306 be used in conjunction with further information to provide a better indication of the
307 detergent chemical concentrations.

308 An online pH probe will provide information on the minimum strengths of NaOH and
309 Na₂CO₃ present. Combining this information with the online conductivity information
310 by data fusion will enable confidence that at least 1% NaOH is present and an indication
311 of when Na₂CO₃ strength is increasing. It is sufficient to consider both signals together
312 but conductivity or pH alone is not informative. Additional flow monitoring of any
313 NaOH added to the detergent will be required to ensure that concentrations of Na₂CO₃
314 in excess of 9% may not be achieved. Using this method will provide operational
315 confidence and ensure that cleaning is being performed to an acceptable standard
316 throughout the full duration of the detergent cleaning step.

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

323 **Conclusions**

324 This paper has considered the degradation of NaOH during the cleaning of brewery
325 process equipment. It was known previously that Na₂CO₃ formation degraded cleaning
326 ability and this paper has quantified the extent of this loss of performance. This
327 quantification has enabled a more informed and optimised CIP strategy to be
328 implemented in brewery operations. To do so requires additional on-line measurements
329 to be made to distinguish between NaOH and Na₂CO₃ compositions. It has been shown
330 that with measures of pH and conductivity of the cleaning fluid it is possible to gain
331 this information and consequently be able to determine the current cleaning capability.

332 Considering future work the prime activity is to assess long term returns to ensure that
333 short term gains are maintained before technology 'roll out' to other Heineken sites.
334 Further technical studies also follow in from this work such as the impact of Toftejorg

335 spray head interruptions throughout cleaning procedures to quantify the inhibition to
336 the ability of cleaning the complete surface area with the standard that has been set out
337 by the cleaning head manufacturers. Methods to deal with problem root cause are also
338 worthy of exploration such as the removal of carbon dioxide through nitrogen purging
339 or alternative cleaning detergents which will not react with carbon dioxide for a long
340 term cost effective solution. On the installation of a brand new CIP set, these would be
341 the more cost effective options by removing the root cause of the carbonation formation
342 but for existing equipment costs are prohibitive.

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349

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401 **Tables**

Flow Rate (ml.s ⁻¹)	NaOH (% w/v)	Na ₂ CO ₃ (% w/v)	Flow Rate (ml.s ⁻¹)	NaOH (% w/v)	Na ₂ CO 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

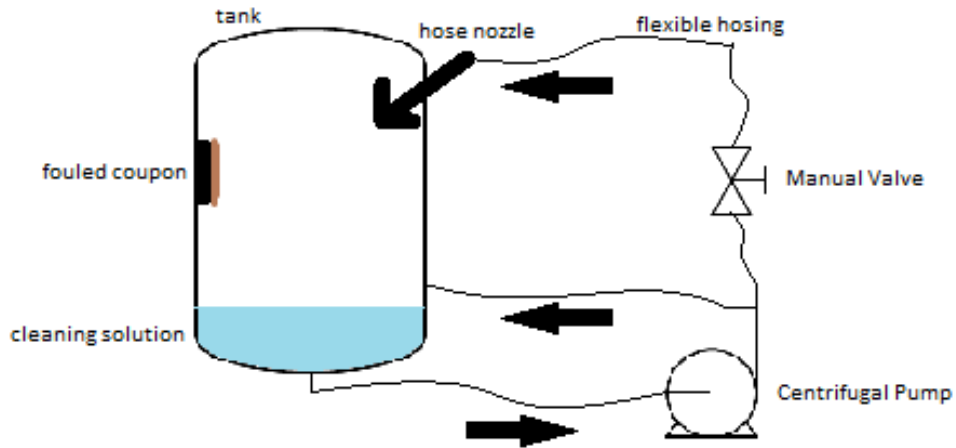
402

403

404 Table 1. Details of full factorial experimental design

405

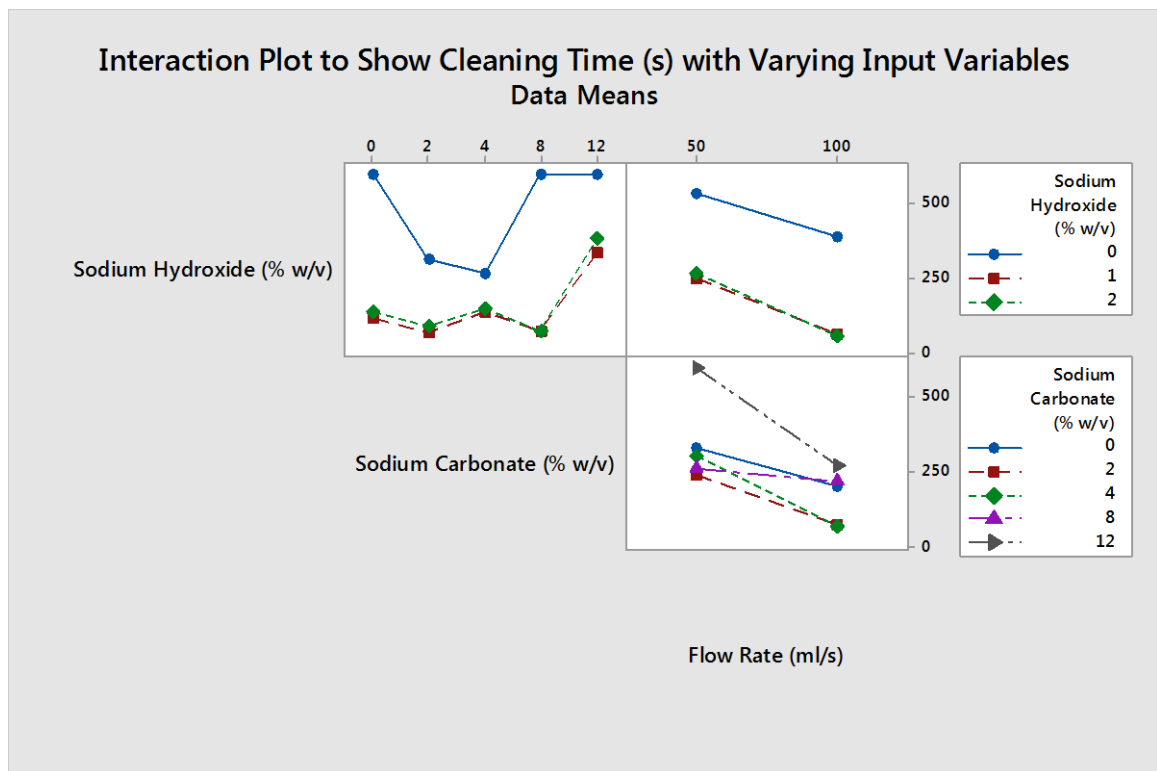
406 **Figures**



407

408 Figure 1. Bench scale cleaning rig

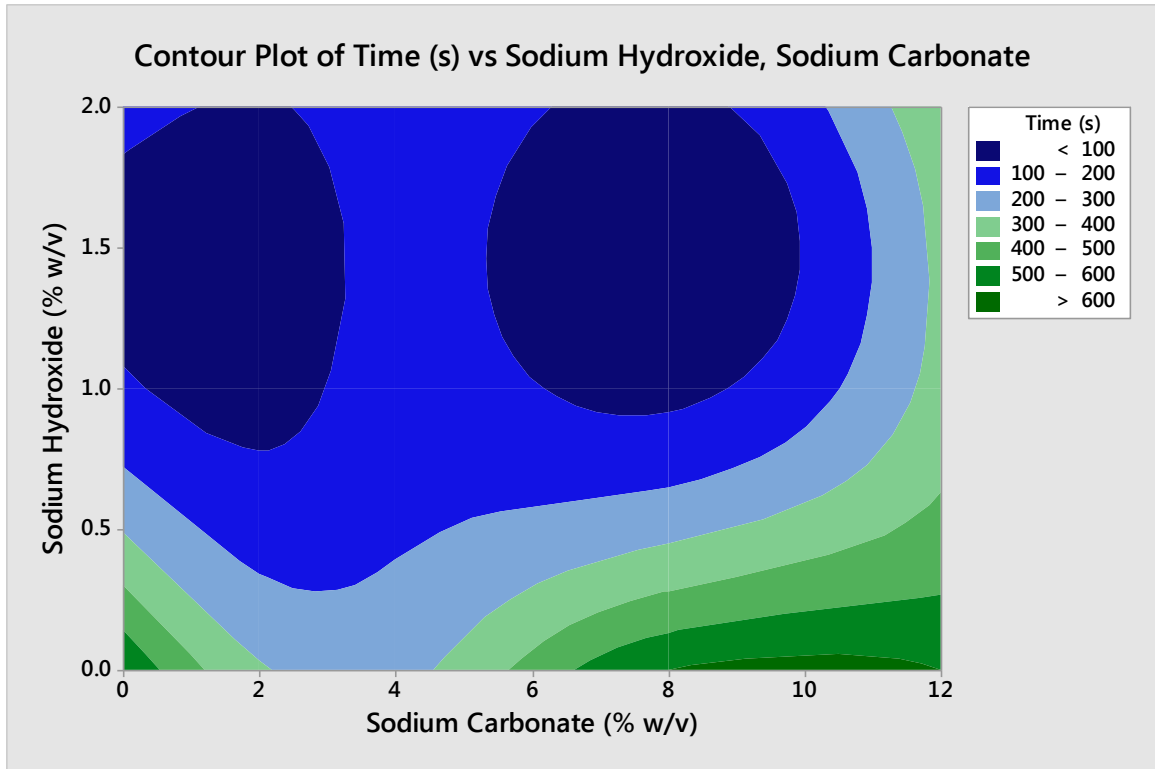
409



410

411 Figure 2. Interaction plot for the variables of NaOH concentration, Na₂CO₃

412 concentration and cleaning time.

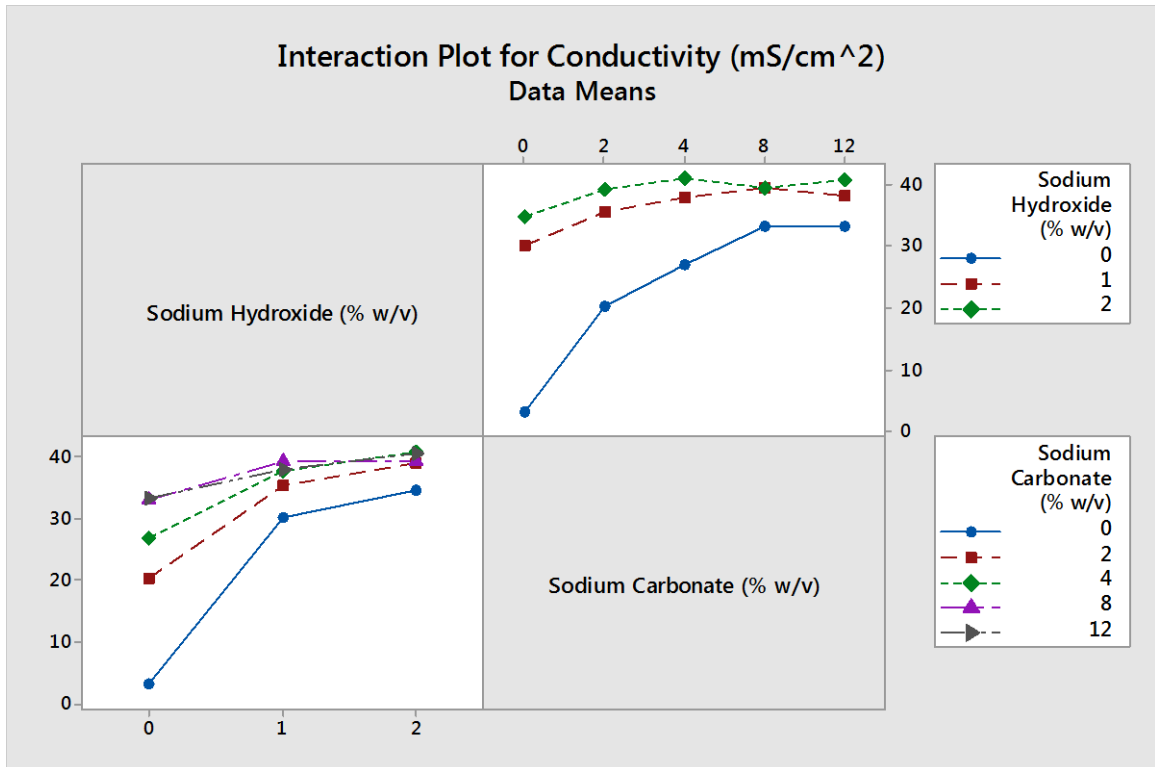


413

414 Figure 3. Contour plot for NaOH concentration and Na₂CO₃ concentration based on
 415 cleaning time

416

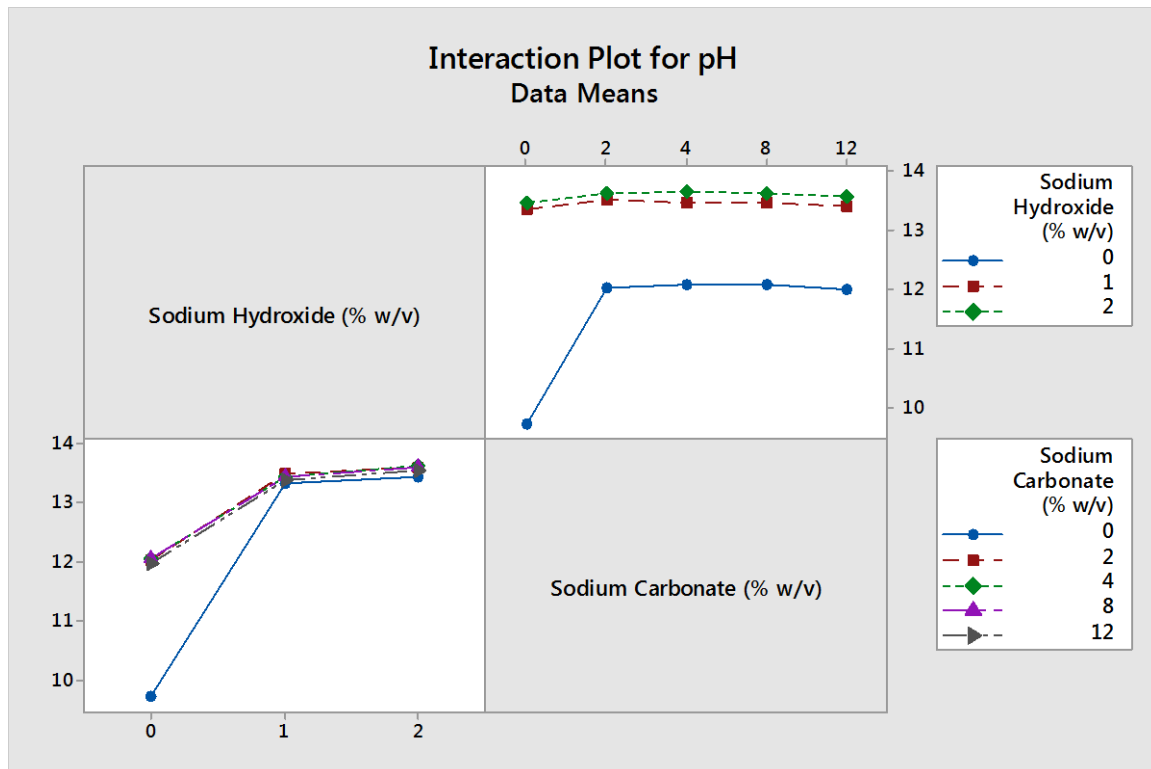
417



418

419 Figure 4. Interaction plot for NaOH concentration, Na₂CO₃ concentration and
 420 conductivity.

421



422

423 Figure 5. Interaction plots for NaOH concentration, Na₂CO₃ concentration, and pH
424 values

425

426