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Optimization of Calcium Oxide in Water Softening Process

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1.0. Introduction

With the increasing population and the per-capital consumption in the country, there is an increasing difficulty in gaining access to raw water supply of sufficient quantity and quality, to meet the demand of the public. Hence most of the communities have been forced to resort to water supplies that are unfit for consumption in their raw form. Many of these sources are both groundwater and surface water, which have high turbidity and colour, bad taste, or hard and staining. Water hardness requires softening and sanitizing to make it safe for consumption [1] Hardness is a common water quality problem all over the world. Water hardness results from the presence of some positively charged metallic ions in solutions in the water. The most common of these hardness-causing ions are calcium and magnesium. However, hardness can also result from several other dissolved metals; those form divalent or multivalent cations, such as aluminum, barium, strontium, iron, zinc, and manganese. Though they are required in some percentages, their presence in excess has proven to be detrimental to the health of the human [2]. Hardness is of

concern in domestic water consumption as it increases soap consumption, leaving soapy scum in the sink or tub, it also causes water heater electrodes to burn out quickly, causes discoloration of plumbing fixtures and utensils, and makes water less desirable. On an industrial scale, hardness causes boiler scales and damage to industrial equipment [3].

Water quality deals with the physical, chemical, and microbiological characteristics of water. These properties collectively determine the overall water quality and the fitness of the water for a specific use. These properties are either intrinsic to the water or are the result of substances that are dissolved or suspended in the water. Water quality is only meaningful when evaluated with the use of the water. The reason is that water of a certain quality may be fit for a specific use, but also unfit for another use. The World Health Organization's (WHO) drinking water quality guidelines are the international reference points for drinking water quality standards.

The main aim of water treatment is to produce water that is fit for domestic use reliably and consistently from a raw water source at a reasonable cost to the consumers. A water treatment plant employs many individual treatment processes (sometimes called unit processes and unit operations) that are linked in a process train to produce water of the desired quality [4].

Removing hardness from water is called softening. Water softening is a process of ion exchange in which the ions causing hardness in water, such as calcium ions (Ca^{2+}) or magnesium ions (Mg^{2+}) are replaced with other single-charged ions, typically sodium ions ($Na⁺$) or potassium ions ($K⁺$). There are several reasons for softening water. Among these are the aesthetic value of good water, soap consumption, and the elimination of scale from hot water pipes and boilers. The processes of either chemical precipitation or ion exchange process generally achieve water softening. Chemical precipitation is the process of converting calcium hardness to calcium carbonate and magnesium hardness to magnesium hydroxide, and this can only be achieved by determining the best combination that would achieve the result. Hence, optimization in this case deals with the best combination of soda ash and calcium oxide to deal with this problem. Numerical optimization refers to the process of finding the best solution to a problem by systematically evaluating different possibilities using mathematical algorithms. In the context of water softening, numerical optimization can be applied to determine the optimal dosage of quicklime and other process parameters such as reaction time, initial water hardness, temperature, pH, and agitation speed. The study on optimizing quicklime dosage in water softening is crucial for enhancing water quality and efficiency in treatment processes. It aims to determine the precise quicklime dosage and process parameters to remove hardness effectively. This optimization reduces operational costs, minimizes chemical usage, and lessens environmental impact. Additionally, it ensures consistent water quality, aids in regulatory compliance, and advances our understanding of water treatment chemistry, paving the way for future innovations. This is generally practiced using the lime soda ash process or by caustic soda ash process [5].

Therefore, this research determined the level of hardness present in the water samples collected from selected locations in the study area, evaluated the effect of selected input variables, namely, quicklime dose, reaction time, and mixing speed on the overall softening process and also carried out optimization of the softening process and determined the optimum dose of quicklime, exact reaction time, and exact mixing speed required to achieve the recommended level of pH, alkalinity, and calcium carbonate hardness.

2. Materials and Method

2.1 Description of Study Area

Ondo South area is an area in Ondo state Nigeria located in the Western part of the country. It is situated on Latitude $5^{\circ}56'78'' N$ to $6^{\circ}46'53'' N$ and on Longitude $4^{\circ}27'42.88'' E$ to $5^{\circ}04'54.41''$ E and comprises of six local government areas namely, Ilaje, Odigbo, Okitipupa, Irele and Ese-odo. The projected population of the area as of 2021 according to Ondo State Bureau of Statistics (2011) [6] is one million eight hundred and seventy-two thousand eight hundred and twenty-one (1,872,821). The Coastal Alluvium underlies the sedimentary basin of Ondo State at the extreme south and along major river floodplains, the Coastal Plain Sands, the Imo Shale, Upper Coal Measures, and Nkporo Shale. These formations have different hydrogeological characteristics. The shallow aquifers within the southern sedimentary portion of Ondo State have been investigated and found to be vulnerable to near-surface contaminants [7] and [8]. [9] observed that potable water supply to inhabitants in some of the communities in the sedimentary rock underlain southern (coastal belt) part of Ondo State had been a major problem due to saltwater intrusion. The Ondo State Water Corporation currently cannot meet the daily water needs of the growing population within the state from its surface water schemes. Only 70.2 million litres of potable water out of the state water requirement of 598 million litres is supplied. This has made groundwater development through borehole drilling inevitable. However, many of the moderately deep (<100 m) boreholes drilled in the coastal area had yielded saline water. Siting deep boreholes in this area requires adequate knowledge and characterization of the aquifer units of the Coastal Plain Sands and the alluvial deposits. The Imo Shale is predominantly an aquiclude and aquitard of low permeability with low groundwater-yielding capacity. Figure 1 shows the areas that make up Ondo South.

2.2 Materials

The constituent materials that were used for this study were water samples and quicklime.

2.3 Sample Collection and Characterization

Water samples were collected from the Agbabu, Aye, Ore, Igbotako, Ilutitun, and Irele areas of Ondo South LGA. Six (6) samples were collected from wells and boreholes and then stored. To maintain hygiene, one-litre plastic containers that were previously cleaned were rinsed three times with the water samples and labelled appropriately as presented in Table 2. Quicklime was added to the water samples, and the reaction was allowed to proceed for the specified time duration while being agitated at the designated mixing speed. The experiments were conducted in duplicate and the mean value of each experimental run was measured and recorded to ensure the reliability of the results. At the end of each experiment, the softened water was analyzed for pH, alkalinity, and calcium carbonate hardness using standard laboratory methods. The data obtained from the experiments were recorded. In each experiment, the quicklime dose, reaction time, and mixing speed were varied according to the experimental design presented in Table 2. The total hardness which includes the calcium and magnesium concentration in the water, the carbon dioxide concentration, alkalinity which is defined in terms of the bicarbonate concentration, and the noncarbonate concentration of the water including the pH of the raw water was determined following the standard procedures recommended by [10].

Figure 1: Map of Ondo South (from Google map)

2.4 Experimental Design and Variable Selection

For the optimization of water softening using quicklime, some of the input variables that can be considered include quicklime dosage, reaction time, initial water hardness, temperature, pH, and agitation speed [11], [12]. For this study, quick-lime dosage, reaction time, and mixing speed were selected as the input variables. After that, Response Surface Methodology (RSM) was employed to design the experiments systematically. Each variable was assigned a range of values to cover the full spectrum of possible conditions. The range and level of the experimental variables used for the statistical design of the experiment are presented in Table 2:

Independent Variables	Range and Levels of Input Variables				
	Lower Range (-1)	Upper Range $(+1)$			
Quicklime Dosage (mg/L) X_1	50	500			
Reaction Time (min) X_2	15	60			
Reaction Speed (rpm) X_4	10	50			

Table 2: Range and Levels of independent variables

To reduce potential risks and account for environmental variability, conservative ranges and levels of experimental variables were selected. These ranges and levels cover a wide range of plausible values, offering resilience to uncertainties and variations. The selections were also informed by past data and following the works of Benjamin and Lawler, (2013) for a hardness level of 400- 750mg/L.

Using the range and levels of the independent variables presented in Table 2, the statistical design of the experiment (DOE) using the central composite design (CCD) method was done with the aid of design expert version 7.01.

2.5 Water Softening Optimization using RSM

Response Surface Methodology (RSM) is a statistical and mathematical technique used for optimizing processes and improving the performance of systems where the response of interest is influenced by multiple input variables. It's particularly valuable in fields such as engineering, chemistry, and manufacturing, where the goal is to find the optimal combination of input variables to achieve a desired response. It is a powerful tool for optimizing processes and improving performance by systematically exploring the relationship between input variables and response variables.

Response Surface Methodology (RSM) is useful in understanding the relationship between multiple variables with one or more response variables. The technique is popular in industries where process and statistical optimization play a key role.

The objective of the optimization model was to optimize three variables: pH, alkalinity, and calcium carbonate. The pH was to be optimized within a range of 6.5-8.5, alkalinity within a range of 200-600mg/L, and calcium carbonate within a range of 20-200mg/L. The final solution of the optimization process was to determine the optimal values for the input variables of quicklime dose (mg/L), reaction time (min), and mixing speed (rpm) that would optimize the three variables to their respective ranges. The experimental data for the optimization process was generated using the following steps:

- i. A statistical design of the experiment (DOE) was conducted using the central composite design method (CCD). The central composite design (CCD) is a widely used method in experimentation to handle optimization problems that require the use of response surface methodology (RSM).
- ii. Based on the DOE, a design matrix was created with 20 experimental runs, comprising six (6) center points, six (6) axial points, and eight (8) factorial points.

The experiment design and optimization process were carried out with the help of statistical software (Design Expert 7.01). The randomized design included three input variables, namely; quicklime dose (mg/L), reaction time (min), and mixing speed (rpm), and three response variables, namely; pH, alkalinity (mg/L), and calcium carbonate (mg/L).

3.0 Results and Discussion

3.1 Analysis of Water Samples

Results of the analysis of water samples which include calcium and magnesium hardness (Total hardness), bicarbonate alkalinity ($HCO₃²$), sulphate level, chloride ions, pH, and electrical conductivity from the different sources of water (deep well and shallow well) is presented in Table 3.

Table 3: Parameters relevant to the study

From the result of the analysis, it was observed that the pH of the water samples from boreholes was within the range of 6.5 to 7.1, which was within the range of the limit as prescribed by the WHO, but the pH from water samples collected from well water was within the range of 4.3 to 5.3. This shows that they are toxic for human consumption and this is consistent with the findings of [13] . Similarly, water samples from boreholes exhibit Total Hardness values (as CaCO3) ranging from 1.6 to 7.55, Carbonate Hardness values ranging from 64.5 to 89.44, and Non-Carbonate Hardness value is 0 because the Total Hardness is less than the Carbonate Hardness. For well water, Total Hardness varies from 332.4 to 431.08, Carbonate Hardness from 527.28 to 750.4, and Non-Carbonate Hardness is 0. This shows that samples from WW4, WW5, and well WW6 are beyond the acceptable limit of hardness with WW6 being the hardest, hence, the water sample was selected for treatment and the treatment employed is the addition of quicklime, a process known as softening which is aimed at reducing the level of hardness associated with the water. However, according to the Nigeria Standard of Drinking Water Quality (NSDWQ), the acceptable limit of hardness required for water to be consumed is 150 mg/L.

The optimum dosage of quicklime required to soften water with pH 4.3, alkalinity of 750mg/L, and calcium hardness of 320mg/L was determined through optimization using response surface methodology (RSM).

3.2 Water Softening Optimization using RSM

The model design summary of the coded values and real values of the experiment which shows the factors and their lowest and highest values including the mean and standard deviation is presented in Table 4.

The results of Table 4 revealed that the model is of the quadratic type with a minimum value of pH of 6.0 a maximum value of 7.8 and a standard deviation of 0.4826. Also, the minimum value of alkalinity is 350.4mg/L with a maximum value of 550.4mg/L and standard deviation of 46.623. At the same time, the minimum value of calcium carbonate is 50.8mg/L with a maximum value of 170.5mg/L and a standard deviation of 35.994.

To validate the suitability of the quadratic model in analyzing the experimental data, the sequential model sum of squares was calculated for each of the responses as presented in Tables 4a, 4b, and 4c respectively.

Table 4: RSM design summary

Sequential Model Sum of Squares [Type I]"0+: Select the highest order polynomial where the additional terms are significant and the model is not aliased

Table 4b: Sequential model sum of square for alkalinity

"Sequential Model Sum of Squares [Type I]"0+: Select the highest order polynomial where the additional terms are significant and the model is not aliased

Table 4c: Sequential model sum of squares for calcium carbonate

Source	Squares	df	Square	Value	Prob > F	
Mean vs Total	297021.6		297021.6			
Linear vs Mean	6537.414	3	2179.138	1.799701	0.1878	
2FI vs Linear	2163.744	3	721.2479	0.544826	0.6602	
Quadratic vs					$\,<\,$	
2FI	17173.52	3	5724.507	1587.141	0.0001	Suggested
Cubic vs						
Quadratic	22.99092	$\overline{4}$	5.74773	2.637155	0.1388	Aliased
Residual	13.07711	6	2.179519			
Total	322932.3	20	16146.62			
"Sequential Model Sum of Squares [Type I]"0+: Select the highest order polynomial where the additional terms are significant and the model is not aliased						

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The sequential model sum of squares Table shows the accumulating improvement in the model fit as terms are added. Based on the calculated sequential model sum of square, the highest order polynomial where the additional terms are significant and the model is not aliased was selected as the best fit. From the results of Tables 4a, 4b, and 4c, it was observed that the cubic polynomial was aliased and hence cannot be employed to fit the final model. In addition, the quadratic and 2FI models were suggested as the best fit thus justifying the use of quadratic polynomials in this analysis.

To obtain the optimal solution, we first consider the coefficient statistics and the corresponding standard errors. The computed standard error measures the difference between the experimental terms and the corresponding predicted terms. Coefficient statistics for each response are presented in Tables 5a-c.

	Coefficient		Standard	95% CI	95% CI	
Factor	Estimate	Df	Error	Low	High	VIF
Intercept	6.598402		0.038406	6.512828	6.683977	
A-Quicklime Dose	0.352801		0.025482	0.296024	0.409578	
B-Reaction Time	-0.08088		0.025482	-0.13765	-0.0241	
C-Mixing Speed	0.1764		0.025482	0.119624	0.233177	
AB	-0.425		0.033293	-0.49918	-0.35082	
AC	-0.125		0.033293	-0.19918	-0.05082	
BC	0.05		0.033293	-0.02418	0.124182	
A^2	0.222573		0.024806	0.167303	0.277844	1.018265
$B^{\wedge}2$	0.028119		0.024806	-0.02715	0.08339	1.018265
$C^{\wedge}2$	0.081152		0.024806	0.025882	0.136423	1.018265

Table 5a: Coefficient estimates statistics for optimizing pH to a range of 6.5-8.5

	Coefficient		Standard	95% CI	95% CI	
Factor	Estimate	Df	Error	Low	High	VIF
Intercept	458.7424		4.419489	448.8952	468.5896	
A-Quicklime Dose	-5.39918		2.93223	-11.9326	1.134233	
B-Reaction Time	-8.28119		2.93223	-14.8146	-1.74777	
C-Mixing Speed	-9.58121		2.93223	-16.1146	-3.0478	
AB	27.4625		3.831143	18.92618	35.99882	
AC	-12.4625		3.831143	-20.9988	-3.92618	
BC	57.4125		3.831143	48.87618	65.94882	
A^2	3.891818		2.854447	-2.46829	10.25192	1.018265
$B^{\wedge}2$	11.12198		2.854447	4.761881	17.48209	1.018265
$C^{\wedge}2$	-15.5006		2.854447	-21.8607	-9.14048	1.018265

Table 5b: Coefficient estimates statistics for optimizing alkalinity to a range of 200- 600mg/L

Table 5c: Coefficient estimates statistics for optimizing calcium carbonate to a range of 20-200mg/L

	Coefficient		Standard	95% CI	95% CI	
Factor	Estimate	Df	Error	Low	High	VIF
Intercept	151.9712		0.774569	150.2454	153.6971	
A-Quicklime Dose	3.252095		0.513909	2.107036	4.397155	
B-Reaction Time	-0.52054		0.513909	-1.6656	0.624518	
C-Mixing Speed	21.6297		0.513909	20.48464	22.77476	
AB	-1.4625		0.671454	-2.95859	0.033593	
AC	-6.1375		0.671454	-7.63359	-4.64141	
BC	-15.1875	1	0.671454	-16.6836	-13.6914	
A^2	5.014088		0.500276	3.899403	6.128773	1.018265
$B^{\wedge}2$	-25.9042		0.500276	-27.0188	-24.7895	1.018265
$C^{\wedge}2$	-23.1995		0.500276	-24.3142	-22.0848	1.018265

Variance inflation factor (VIF) value of 1.00 for the individual and combined terms, and 1.018265 for the quadratic terms as observed in Tables 5a-c indicate a significant model in which the variables are adequately correlated with the responses (pH, alkalinity (mg/L) and calcium carbonate (mg/L)).

The optimal equations show the individual effects, and the combined interactions of the selected input variables, namely; quicklime dose (mg/L), reaction time (min), and mixing speed (rpm) against the measured responses, namely; pH, alkalinity (mg/L) and calcium carbonate (mg/L) is presented based on actual factors as follows;

 \boldsymbol{p} H = 5.66068 + 0.003131 x_1 + 0.011993 x_2 + 0.00011944 x_3 – 8.39505 $e^{-5}x_1x_2$ –

 $2.77778e^{-5}x_1x_3 + 0.000111x_2x_3 + 4.39651e^{-6}x_1^2 + 5.55438e^{-5}x_2^2 + 0.00020288x_3^2$ (1)

 $\boldsymbol{Alkality} = 671.9724 - 0.186620x_1 - 7.335044x_2 - 2.176751x_3 + 0.005425x_1x_2 -$

 $0.0027694x_1x_3 + 0.1275833x_2x_3 + 7.68754e^{-6}x_1^2 + 0.02196935x_2^2 - 0.038751463x_3^2$

(2)

Calcium carbonate = $-52.445165 + 0.0117297x_1 + 4.90646198x_2 + 6.2021004x_3$ – $0.00028880x_1x_2 - 0.00136389x_1x_3 - 0.03375x_2x_3 + 9.90437e^{-6}{x_1}^2 - 0.0511687{x_2}^2 0.05799868x_3^2$ (3)

Where; x_1 is pH level x_2 is alkalinity level (mg/L) x_3 is calcium carbonate level (mg/L)

Finally, numerical optimization was performed to ascertain the desirability of the overall model. Numerical optimization plays a crucial role in optimizing the water-softening process to ensure the efficient removal of hardness ions like calcium and magnesium from water. The first step was to define the problem mathematically. In the case of water softening, the objective was to minimize the concentration of hardness ions in the treated water while considering constraints such as the availability of softening agents, quicklime, pH levels, and other parameters. Constraints are conditions that must be satisfied during the optimization process. These constraints can include limitations on the amounts of softening agents that can be used, pH constraints, equipment capacity constraints, or regulatory limits on the concentration of certain ions in the treated water.

However, numerical optimization being a powerful tool for solving problems, it has limitations that researchers and practitioners must recognize. Firstly, relying on mathematical models may oversimplify problems and lead to discrepancies when actual conditions deviate from assumptions. Secondly, choosing an objective function is crucial, but a single function may oversimplify problems with competing objectives and neglect critical factors. Thirdly, constraints are essential, but managing them, particularly with complex constraints, can be challenging and may lead to infeasible or suboptimal solutions. Fourthly, the accuracy of numerical optimization depends on reliable data inputs, which can be affected by noise, uncertainty, or errors. Finally, while numerical optimization offers systematic approaches, human judgment, and expertise are still necessary for interpreting results and validating solutions. Recognizing these limitations enables practitioners to make informed decisions and ensure the practical relevance and feasibility of optimization outcomes.

In this research, the objective of numerical optimization was to determine the optimum quicklime dose (mg/L), reaction time (min), and mixing speed (rpm) that will optimize the pH to a range of 6.5-8.5, alkalinity to a range of 200-600mg/L and calcium carbonate to a range of 20-200mg/L. The interphase of the numerical optimization showing the objective function is presented in Figures 2 a-c.

Figure 2a: Interphase of a numerical optimization model for optimizing pH to a range of 6.5-8.5

Figure 2b: Interphase of a numerical optimization model for optimizing alkalinity to a range of 200-600mg/L

Figure 2c: Interphase of a numerical optimization model for optimizing calcium carbonate to a range of 20-200mg/L

The optimization objective was to optimize the pH to a range of 6.5-8.5, alkalinity to a range of 200-600mg/L, and calcium carbonate to a range of 20-200mg/L. The relative importance was set at the optimum value of 5.0 and the lower and upper boundary conditions were set at 6.5-8.5 for pH, 200-600mg/L for alkalinity, and 20-200mg/L for calcium carbonate hardness. The final solution of numerical optimization is presented in Table 6.

Table 6 Optimal solutions of numerical optimization

From the results of Table 6, it was observed that a quicklime dose of 221.04mg/L, a reaction time of 36min., and mixing speed of 43rpm will treat water with an initial pH of 4.3, alkalinity of 750mg/L and calcium carbonate hardness of 320mg/L to pH of 6.7, alkalinity of 447.3mg/L and calcium carbonate hardness of 157.2mg/L. A design expert selected this solution as the optimal solution with a desirability value of 100%.

Optimizing the dosage of quicklime, reaction time, and mixing speed as presented in Table 6, is crucial in water treatment processes like water softening or pH adjustment. Quicklime, also known as calcium oxide, is commonly used for these purposes. The effectiveness of the treatment process heavily relies on the proper dosage of quicklime. Inadequate use can result in insufficient water quality improvement while overdosing may lead to issues such as elevated pH levels or excessive precipitation of hardness ions.

Optimizing the quicklime dosage allows for achieving the desired water quality improvement while minimizing chemical usage and associated costs. For instance, an optimal quantity of 221.04mg/L, as presented in Table 6, can prevent overdosing or insufficient treatment.

The duration for which water interacts with quicklime, or the reaction time, is crucial for efficient impurity removal. Insufficient reaction time can lead to incomplete removal of impurities, including hardness ions. By optimizing reaction time, the treatment process can maximize impurity removal while enhancing operational efficiency.

Mixing speed plays a vital role in ensuring uniform distribution of quicklime in water, facilitating chemical reactions. Proper mixing prevents issues like localized overdosing or incomplete mixing, thereby promoting consistent high-quality treated water.

Generally, optimizing quicklime dosage, reaction time, and mixing speed are essential for improving water quality in treatment processes. Achieving the right balance among these factors ensures effective impurity removal, efficient chemical usage, and consistent production of highquality treated water, aligning with research objectives.

The ramp solution which is the graphical presentation of the optimal solution is presented in Figure 3 while the desirability bar graph which shows the accuracy with which the model can predict the values of the selected input variables and the corresponding responses is presented in Figure 4.

It can be deduced from the result of Figure 4.7 that the model developed based on response surface methodology and optimized using numerical optimization method, predicted the

- i. pH with an accuracy level of 100%
- ii. Alkalinity with an accuracy level of 1000% and
- iii. Calcium carbonate hardness with an accuracy level of 100%

Figure 3: Ramp solution of numerical optimization

Figure 4: Desirability Bar Graph

4. Conclusion

The study aimed to enhance the water softening process by investigating the effects of quicklime dosage, reaction time, and mixing speed on water quality parameters. Analysis of pH, alkalinity, and calcium carbonate hardness revealed notable variations between borehole and well water samples. Borehole water exhibited pH levels within WHO standards, whereas well water pH indicated unsuitability for consumption. Similarly, hardness levels varied, with borehole water showing lower values compared to well water. Optimization efforts identified a quicklime dose of 221.04 mg/L, a reaction time of 36 minutes, and a mixing speed of 43 rpm as optimal conditions, resulting in significant improvements in water quality. These findings underscore the importance of balancing input parameters for effective water softening. Recommendations for future research include conducting further assessments on the long-term environmental impact of quicklime and validating optimization results on a larger scale and with diverse water sources. Additionally, a comprehensive cost-benefit analysis is recommended to evaluate the economic feasibility of implementing optimized water softening conditions, considering factors such as raw material costs and potential savings in operational expenses.

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