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Soluble Silica Degradation in Mining Effluents Using Magnesium Hydroxide and Acidified Activated Carbon

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ABSTRACT: High soluble silica (SiO_2) concentration in mining effluents from processing plants of most mining companies in Ghana makes most mines noncompliant to the Environmental Protection Agency's (EPA's) soluble silica compliant limit of 20 mg/L. In this research work, magnesium hydroxide and acidified activated carbon have been used to degrade soluble silica in mining effluents to meet the EPA limit. Jar test were performed at three doses of magnesium hydroxide ($Mg(OH)_2$) with concentrations (15-25 g/L) together with three weights of acidified activated carbon (5-10 g) were used at different initial pHs between 10-11. Characterization of the mine water effluent revealed that the silica, pH and conductivity were 124 mg/L, 10.01 and 1132 µS/cm respectively. This implied that soluble silica degradation greater than about 84% was required in other to meet the EPA limit. The outcome of the Jar test revealed that soluble silica degradation rate decreased with increasing $Mg(OH)_2$ addition and pH from 15 g to 19 g and 10 – 10.5 respectively. Beyond the aforementioned $Mg(OH)_2$ and pH range values, the degradation rate increased significantly. Soluble silica degradation rate increased with increasing the activated carbon under conditions of study. Whereas in the case of reaction time, soluble silica degradation remained approximately unchanged between 15 min and 27 min and subsequently increased afterwards, the degradation rate of soluble silica decreased with increasing stirring rate. Response surface methodology (RSM) technique was successfully used to develop models to optimize preparation conditions needed to degrade soluble silica in the mine effluent while maintaining the mine effluent conductivity below the EPA limit of 1500 µS/cm. The optimum conditions for high soluble silica degradation and low solution conductivity was identified to be $Mg(OH)_2$ and activated carbon additions of 20.9 g and 5.0 g respectively, pH of 10.6, reaction time of approximately 44 min and stirring rate of 200 rpm. Under optimized conditions, silica degradation and solution conductivity of 96.0% and 1199.4 µS/cm was achieved experimentally. The cause for the high soluble silica degradation was ascribed to the precipitation of silicon phosphate emanating from reaction between magnesium phosphate and silica, adsorption of silica onto precipitated $Mg(OH)_2$ and the formation of calcium silicate (CaH_2SiO_4). The current study shows that mine effluent containing soluble silica could be degraded while maintaining low solution conductivity by the use of magnesium hydroxide and acidified activated carbon to meet the EPA limit.

KEYWORDS: soluble silica degradation, conductivity, magnesium hydroxide, acidified activated carbon, mining effluent, response surface methodology, precipitation

1. INTRODUCTION

Research has shown that the solid crust of the earth contains 80% to 90% silicates or other compounds of silicon (Anon, 2022). This makes the mining environments prone to high levels of crystalline silica, which is estimated to be more than 95% of the volume of the earth crust (Lehr et al, 2005) as a result of the composition of quartz and other rock-forming silicates. Regarding its geochemistry, silicon forms the largest number of compounds with other elements second only to carbon (Lehr et al, 2005). Over the years, health risk assessments conducted in many mining environments have proved that high concentrations of crystalline silica dust in the air that is inhaled by workers and those living in the catchment area of most mines are at high risk of contracting diseases associated with silica dust; especially long after the exposure has stopped (Anon, 2018). These associated diseases include but not limited to silicosis, lung cancer, chronic bronchitis, and several autoimmune diseases (Anon, 2018). In view of this, many mining sites have adopted scientific ways of dampening the silica dust in air with water to control its effects on the people, who work and live in these environments. However, the challenge for most mining companies is that, as water passes through or over the earth, it dissolves silica from sands, rocks and minerals as one of the impurities it collects (Anon, 2022 and Lottermoser, 2010), which find its way into the ore that is sent to the processing plant to be treated apart from the fact that various ore bodies are quartzites in nature.

Generally, silica in natural waters can be classified in three categories: reactive soluble, nonreactive soluble (colloidal) and nonreactive insoluble (particulate) (Lehr et al, 2005).

Usually, effluents from mining processing plants contain all these forms of silica. Except to state that the reactive soluble and colloidal silica, which go into solution become a challenge to be handled due to the concentration levels in natural waters. It is estimated that silica concentrations in natural waters are 1 – 3 ppm (lakes), 3 – 15 ppm (major rivers), 1 – 10 ppm (seawater), 2 – 60 ppm (wells) and 50 – 300 ppm (wells in volcanic and oil fields) (Lehr et al, 2005). Adopting the admonition given by Hippocrates, the father of medicine, that water contributed much to health (Faust and Aly, 1998) then there's the need for all waters leaving the mine to the environment to be compliant with the regulated standards so as to ensure that human lives as well as the aquatic ecosystem is not overly healthily compromised. When this is achieved, then it would fulfill World Health Organization's water quality guidelines that has an objective to eliminate or reduce to a minimum those constituents in drinking water that are known to be hazardous to the health and well-being of a community and/or country (Faust and Aly, 1998).

Unfortunately, most effluents from mining processing plants treated by wastewater treatment plants (WWTPs) do not meet the World Health Organization (WHO) objective stated above before it is either discharged to the environment or recycled to the plant. At the moment, one of the parameters that most mining processing plants are finding it difficult to handle is the silica in solution, which on the average is between 60 mg/l - ≥ 100 mg/l. This implies that most mining companies have become noncompliant to the Environmental Protection Agency's (EPA's) standard soluble silica discharge limit of 20 mg/L and hence causing pollution to groundwater, nearby wells and other river bodies within their catchment areas. The compliant limit sits well since the recommended daily amount of silica for adults (19-50 years old) is 9-14 mg/day (Anon, 2021). This presupposes that beyond the recommended silica consumption rate threshold for adult it could result in debilitating health challenges; feeding into the belief of some researchers that certain concentrations of soluble silica in water can result in some form of cognitive impairments. Moreover, since the role of silica in drinking water on cognitive function has been little studied and clear results have not yet emerged (Guyannet et al, 2007) and also for the fact that further studies are needed to settle the debate over the link between aluminium or silica in drinking water and neurologic disorders and cognitive impairment (Rondeau et al, 2008), there's the need for all mining companies in Ghana to work within the EPA's compliant limit to reduce the risk of high concentrations of silica finding its way into the groundwater, river bodies and wells that are within the catchment areas of these mines. To ameliorate this health impact, this research was aimed at looking at the possibility of using magnesium hydroxide and acidified activated carbon to remove some amount of the silica in mining effluents to make their water discharged to the environment compliant to the limit of EPA and fulfill the WHO drinking water quality standard objective.

2. EXPERIMENTAL PROCEDURES

2.1 General Reagents

The effluent used for this research was obtained from Zone A (Fig. 2.1) of the storage facility that comes under the Tailings Storage Facility (TSF) of the mining company's process plant. This wastewater is abstracted by means of a high-pressure pump to the Wastewater Treatment Plant (WWTP). Samples were taken and kept under good condition throughout the period that the research was conducted to ensure that the water chemistry does not change within the period of the research. The pure magnesium hydroxide used was procured from a licensed chemical shop whilst the acidified activated carbon was gotten from a colleague, who did his research in this area.

Analytical reagent grade lime was employed as pH modifiers for all experiments.

Figure 2.1 Zone A section of the mine where the abstraction of the effluent used for the research is done.

2.2 Experimental Design

The objective of the research was to investigate the degradation of silica in mining effluents to above 85% using magnesium hydroxide and acidified activated carbon to meet the set standard of the Environmental Protection Agency (EPA) while reducing the hike in electrical conductivity that comes with the chemicals used. In this study, the process variables selected were pure magnesium hydroxide ($Mg(OH)_2$), pH modifier (calcium hydroxide ($Ca(OH)_2$), acidified activated carbon, stirring rate and reaction time. The response variables considered were silica concentration and electrical conductivity. A 3-factor 3-level factorial CCD was employed for the study. Tables 2.1 shows the experimental design matrix and their coded levels for the central composite design. The experimental data were analyzed by using statistical software, Design Expert version 13.0 (STATEASE Inc., Minneapolis, Minnesota, USA). In all, forty-four (44) separate experiments were conducted under an ambient temperature of ~25ºC and the results analyzed. The objective for the dependent variables was to maximize the percentage silica degradation and minimize the electrical conductivity.

Table 2.1. Experimental design matrix and their coded levels for the central composite design.

	Levels of coded variables		
	Low -1	Medium 0	High +1
Independent variables			
A-$Mg(OH)_2$, (g)	15	20	25
B-pH	10	10.5	11
C- Activated carbon, (g)	5	7.5	10
D- Reaction time, (min)	15	30	45
E- Stirring rate, (rpm)	200	250	300
Dependent variable objective			
R_1-SiO_2 degradation (%)		***Maximize***	
R_2-Conductivity, (µS/cm)		***Minimize***	

2.3 Statistical analysis

In this study, the Design-Expert software was used for statistical assessment of the results of experimental design. The software was selected for the study since it provides the user with various experimental design options of test work and interpretation of multi factor experiments design statistically (Moghaddam et al., 2015; Khuri and Cornell, 2018; Saeed et al., 2021). The experimental results obtained were validated statistically based on model parameters such as F-value, correlation coefficient (R^2) and adjusted R-squared (R^2Adj). Analysis of variance (ANOVA) provision available in the Design-Expert software was used to check the appropriateness of models. Model F-value with $p<0.05$ and lack of fit F-value with $p>0.05$ for response variables indicate that the model was significant and lack of fit was non-significant relative to the pure error, respectively. Mathematical equations for estimating F-value, R^2, and R^2 (Adj) and AP are presented in Equations 2.12.5 (Singh et al., 2015).

$$Mean\ square\ (MS) = \frac{SS}{Df} \quad (2.1)$$

$$F = \frac{MS_{regression}}{MS_{residual}} \quad (2.2)$$

$$R^2 = \frac{SS_{residual}}{SS_{model} + SS_{residual}} \quad (2.3)$$

$$R^2_{Adj.} = \frac{SS_{residual}/D_f}{(SS_{model}+SS_{residual})/(Df_{model}+Df_{residual})} \quad (2.4)$$

$$Residual \rightarrow e = (y - y_0) \quad (2.5)$$

where SS is sum of square; Df is degree of freedom; y is the predicted value and y_o is actual value.

2. 4 Chemical Characterization of the Acidified Activated Carbon

The activated carbon used for this research is corncob activated carbon ($CCAC_{CI}$) with a phosphoric acid (H_3PO_4) chemical impregnation (Trofymenko et al, (2015). The $CCAC_{CI}$ was produced when the corncob was pyrolized at 500ºC for an hour in an oven and milled to a size range of 2 mm – 0.6 mm after it was allowed to cool. The product after the milling was then soaked in a 40% concentration phosphoric acid (H_3PO_4) for 24 hours (Maulina and Iriansyah, 2018) with an impregnation ratio (IR) of 3½. Research has shown that activated carbon having acidic chemical property is promising for basic gas adsorption such as ammonia (Tadda et al, (2016). The objective for using this acidified activated carbon (AAC) was however not to adsorb gases but to help in the silica degradation and adsorb the metal ions that may find themselves in solution to increase the electrical conductivity (EC).

2.5 Sample Weighing and Jar Test

1000 mL of the effluent was measured into various 1L beakers and 3 dosages of the magnesium hydroxide (from 15 g/L to 25 g/L) were tested at different pHs: 10.0, 10.5 and 11.0. These pHs were chosen because silica degradation with sparingly soluble magnesium hydroxide is effective at high pH (Halka and Nordstrom, 2010). For every test work, the pH of the effluent was first modified by adding $Ca(OH)_2$ to the relevant pH needed and stirred for a minute with the corresponding stirring rate for each test work (i.e. within the range of 200–300 rpm). The magnesium hydroxide dosage and corresponding acidified activated carbon was weighed, added and stirred for a time duration range of 15, 30 and 45 min respectively using the Jar test equipment. When the stirring was done, it was left to settle for 20 min after which portions of the clarified water and dissolved fractions were filtered by Hach Pressure Vacuum Pump Filter and analyzed with the Hach DR6000 UV-VIS Spectrophotometer for the silica concentration whilst the unfiltered but settled portion was analyzed for the output pH and electrical conductivity (EC) with the Hach HQ40d pH and Conductivity Meter respectively. There was a sludge formed as a result of the precipitation. All the 44-test works were done at a room temperature of ~25^{o}C with an error of ± 2^{o}C.

2.6 Analytical Determination of Silica, Electrical Conductivity and pH

2.6.1 Analytical Determination of Silica

All the 44-test works were analyzed for the silica concentration after the Jar Test by using the silicomolybdate method. In achieving this, 10 mL of the filtered sample was measured using a sample cell after which the reagents (i.e., molybdate, acid reagent and citric acid) were added according to laboratory protocols and analyzed with the Hach DR6000 UV-VIS Spectrophotometer to get the soluble silica (SiO_2) in mg/L. Since silica determination using this method can be interfered by colour, iron concentration (i.e., Fe^{2+} and Fe^{3+}), phosphate, sulfides (S^{2-}) and turbidity (Anon., 2007), the machine was zeroed with the corresponding blank sample of each sample before it was read. The principle behind this reaction is that silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic and phosphomolybdic acid complexes. However, the addition of the citric acid destroys the phosphate complexes so that the soluble silica in solution can be measured (Anon., 2007).

2.6.2 Analytical Determination of Electrical Conductivity and pH

Each of the 44-test works was analyzed for the electrical conductivity (EC) as a result of the dissolved metals in solution through the addition of both the magnesium hydroxide and phosphoric acid. This was done by pouring some of the unfiltered clarified water into a beaker after which the cleaned probes of the Hach HQ40d pH and Conductivity Meter were dipped into it and read. To ensure that samples were not contaminated and diluted, deionized water was used to rinse the probes and cleaned thoroughly with a tissue paper after every analysis. Again, each sample was analyzed three times to ensure that the values were accurate.

3. RESULTS AND DISCUSSION

3.1 Silica Degradation and Solution Conductivity

Figure 3.1 shows the percent degradation and conductivity after the treatment of the mine effluent at varied pH (10-11), activated carbon addition (5 -10 g/L), stirring rate (250 – 300 rpm) and reaction time (15 – 45 min). Based on the initial silica concentration of the mine effluent and after treatment, percent degradation for each for all the 44 test runs were estimated (Fig 3.1a). The red dotted line represents EPA threshold for the percent silica degradation and conductivity, which was estimated based on the initial SiO_2 content of the mine effluent, and conductivity. The data in Figure 4.1 shows that it is possible to degrade silica in the mine effluent to meet the EPA limit of 20 mg/L at conductivity levels < 1500 µS/cm. For the 44 test runs performed in this study, the percent degradation (Fig.3.1a) and conductivity (Fig. 3.1b) varied between 30% - 99% and 1132 µS/cm - 1587 µS/cm respectively. Four (4) of the test runs gave percent degradation levels of soluble silica greater than 90%. The conductivity levels for most of the test runs fell below the EPA limit. It is however important to mention that the mine effluent conductivity after treatment for all the test runs increased by about 14%-29% relative to the initial value. This suggests that optimization of the operating variables is necessary to achieve high silica degradation and low conductivity levels for the mine effluent considered in this study.

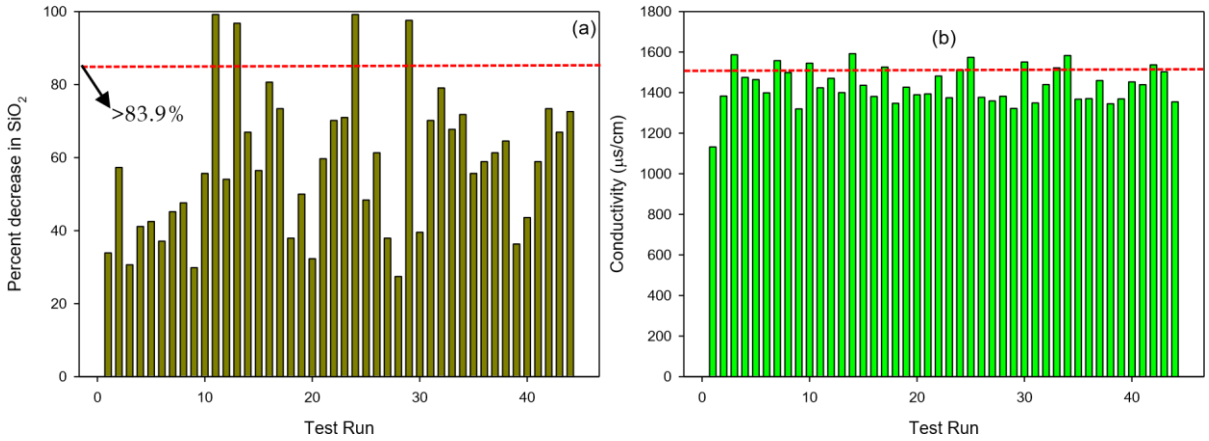

Figure 3.1. Percent silica removal (a) and (b) conductivity for various test runs [(pH (10-11), activated carbon addition (5 -10 g/L), stirring rate (250 – 300 rpm) and reaction time (15 – 45 min)]

3.2 Regression Model development

Regression analysis of experimental data (Figure 3.1) were performed using the Design Expert software (version 13.0). The regression equations for response variables obtained from the response surface methodology in terms of coded values of the experimental factors are presented in Eqns. (3.1) and (3.2). The negative and positive sign indicates the antagonistic and synergistic effects respectively.

$$\%Si_{degradation} = +36.69 + 10.89A + 0.0393B + 3.60C + 14.52D - 10.08E + 3.47AB + 3.02AC - 4.60AD - 2.01AE + 4.33BC + 2.23BE - 1.82CD + 6.21CE - 2.71DE + 22.58A^2 + 39.11B^2 + 15.73D^2 + 14.52E^2 - 6.21ABD - 1.92ACD + 3.72BCD - 15.78A^2D + 8.48A^2E - 9.53AB^2 - 10.29ABCD + 5.91ACDE + 5.91BCDE - 68.10A^2B^2 \quad (3.1)$$

$$Conductivity\ (\mu s/cm) = +1478.75 + 31.50A + 6.50B + 87.50C - 36.00D + 52.50E + 12.62AB + 31.75AC - 5.19AD + 9.69AE + 6.31BC + 6.25BE - 8.88CD + 37.37CE + 15.56DE - 70.25A^2 + 40.75B^2 - 87.75D^2 - 67.25E^2 + 3.94ABC - 12.37ACD - 14.87ACE - 5.81ADE + 14.44BCD - 8.12CDE - 29.13A^2B - 49.75A^2C + 40.44A^2D - 20.69A^2E - 34.31AB^2 - 12.69ABCD + 6.44ABCE + 5.62ACDE + 10.31BCDE + 149.81A^2B^2 \quad \ldots\ldots(3.2)$$

Where; A, B, C, D and E represent $Mg(OH)_2$, pH, activated carbon, reaction time and stirring rate respectively.

3.3 Analysis of variance (ANOVA)

ANOVA analysis was employed to study the significance of the regression models, individual model coefficients and lack of fit. As indicated in Table 3.1, the Model F-values and lack of fit F-values for the soluble silica degradation and conductivity, respectively, were 16.01 ($p<0.0001$) and 0.28 ($p>0.05$), and 65.24 ($p<0.0001$) and 0.37 ($p>0.05$). This indicates that the models for silica degradation and conductivity were significant and lack of fit was not significant relative to pure error, depicting the correctness of the data. The appropriateness of the regression models was examined by the correlation coefficient (R^2) and adjusted coefficient of determination (adj. R^2) values. Generally, R^2 value for a good statistical model should be close to 1 (Worapun et al., 2012). The R^2 and adjusted R^2 values respectively for silica degradation (0.97 and 0.90) and conductivity (0.99 and 0.98) were high, an indicative of high accuracy for the regression models and strong correlation between the experimentally observed and predicted values. The significance of the model terms could be inferred from their p-values. Thus, P-value<0.05 indicates that the model terms is significant for the confidence limit of 95%. From Table 4.1, it is clear that silica degradation rate and conductivity were influenced significantly by the quantity of $Mg(OH)_2$ and acidified activated carbon added, pH, reaction time and stirring rate. The interplay between operating variables such as AB, AC, AD, BC, BE, CE, DE, A^2, B^2, D^2, E^2, ABD, BCD, A^2D, AB^2, ABCD, ACDE, BCDE, A^2B^2 are significant model terms (P-values<0.05) when it comes to soluble silica degradation from mine effluent. In the case of conductivity, the interaction terms AB, AC, AD, AE, BC, BE, CD, CE, DE, A^2, B^2, D^2, E^2, ACD, ACE, ADE, BCD, CDE, A^2B, A^2C, A^2D, A^2E, AB^2, ABCD, ABCE, ACDE, BCDE, A^2B^2 were identified as being very significant.

3.4 Statistical and Diagnostic Analysis

Diagnostic plots were plotted to examine the goodness of fit of proposed model. Figures 3.2 and 3.3 show the predicted vs. actual values and studentized residuals vs. predicted for silica degradation and conductivity. The predicted vs. actual plots (Figs. 3.2a and 3.3a) show that the graphs were highly linear, which implied that the experimentally observed values for silica degradation and conductivity were in close agreement with the model predicted values (Montgomery, 2017, Dean et al., 2015). This indicates the developed models would predict the response variables with high levels of accuracy. The externally studentized residuals vs. predicted

values also suggest that colour points describing silica degradation and conductivity (Fig. 3.2b and 3.3b) fell within the limits close to zero-axis, which is indicative of the absence of constant error (Montgomery, 2017). This indicates that the assumptions regarding the residuals (linearity, independence, homoscedasticity and uncorrelated) have been satisfied. Additionally, the residuals are fairly distributed around zero such that they show no obvious indication of nonlinear, increasing or decreasing patterns.

Table 3.1. ANOVA for soluble silica degradation and conductivity after quartic fitting (modified).

Term	Soluble Silica Degradation (%)		Conductivity, µs/cm^3	
	F-value	p-value	F-value	p-value
Model	16.01	< 0.0001*	65.24	< 0.0001*
A-$Mg(OH)_2$ (g)	6.84	0.0195*	15.77	0.0032*
B-pH	0.0015	0.9694	0.6715	0.4337
C-Activated Carbon (g)	12.7	0.0028*	121.69	< 0.0001*
D-Reaction time (min)	12.16	0.0033*	20.6	0.0014*
E-Stirring rate (rpm)	5.87	0.0286*	43.81	< 0.0001*
AB	11.12	0.0045*	40.53	0.0001*
AC	8.4	0.011*	256.35	< 0.0001*
AD	19.5	0.0005*	6.84	0.028*
AE	3.72	0.0728	23.87	0.0009*
BC	17.28	0.0008*	10.13	0.0111*
BE	4.58	0.0492*	9.93	0.0117*
CD	3.07	0.1002*	20.03	0.0015*
CE	35.6	< 0.0001*	355.24	< 0.0001*
DE	6.8	0.0198*	61.59	< 0.0001*
A^2	19.62	0.0005*	52.29	< 0.0001*
B^2	58.87	< 0.0001*	17.6	0.0023*
D^2	9.52	0.0075*	81.59	< 0.0001*
E^2	8.11	0.0122*	47.92	< 0.0001*
ABC	-	-	3.94	0.0783
ABD	35.6	< 0.0001*	-	-
ACD	3.42	0.0843	38.94	0.0002*
ACE	-	-	56.27	< 0.0001*
ADE	-	-	8.59	0.0167*
BCD	12.79	0.0028*	53.01	< 0.0001*
CDE	-	-	16.79	0.0027*
A^2B	-	-	12.69	0.0061*
A^2C	-	-	37.02	0.0002*
A^2D	13.54	0.0022*	24.46	0.0008*
A^2E	3.9	0.0669	6.4	0.0322*
AB^2	4.94	0.0421*	17.61	0.0023*
ABCD	97.81	< 0.0001*	40.94	0.0001*
ABCE	-	-	10.54	0.0101*
ACDE	32.21	< 0.0001*	8.05	0.0195*
BCDE	32.21	< 0.0001*	27.04	0.0006*
A^2B^2	31.27	< 0.0001*	41.66	0.000*1
Lack of Fit	0.2809	0.9199	0.3665	0.8628

Significant value p<0.05

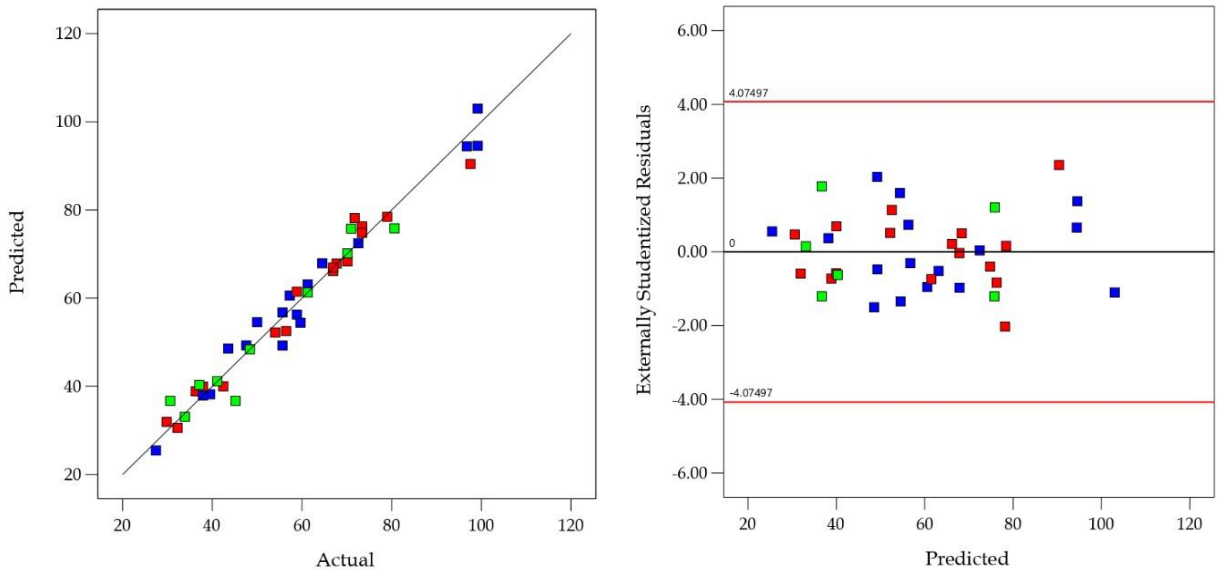

Figure 3.2. A plot of (a) predicted vs. actual values and (b) externally studentized residuals vs. predicted value for yield.

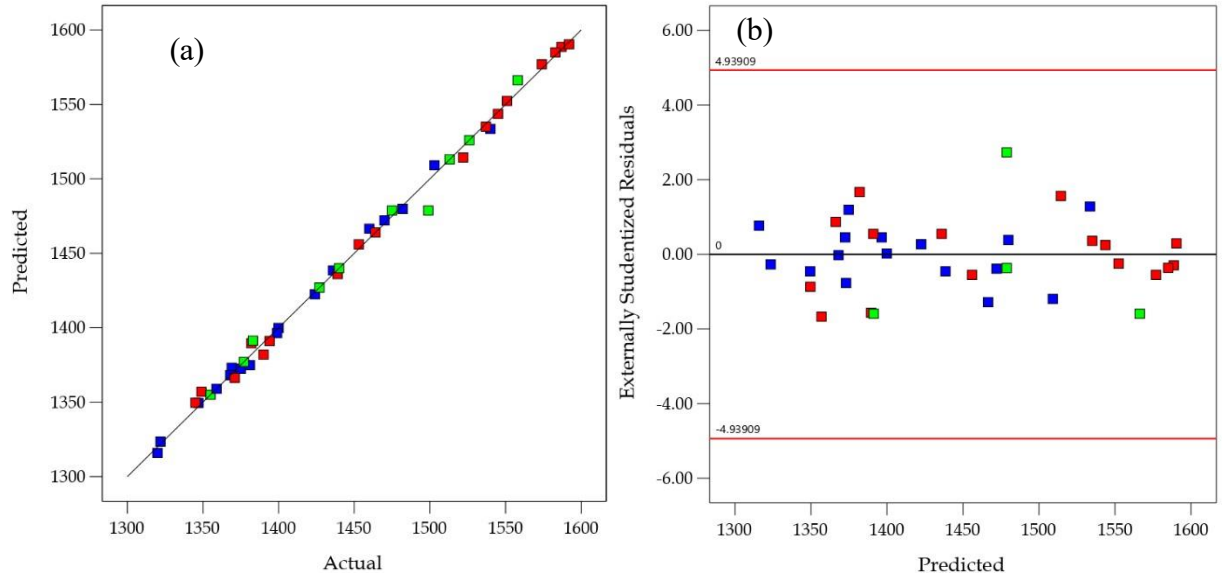

Figure 3.3. A plot of (a) predicted vs. actual values and (b) externally studentized residuals vs. predicted value for yield.

3.5 Response Surface Analysis

3.5.1 Influence of $Mg(OH)_2$ and pH on Silica degradation

The prediction profile for soluble silica degradation of treated mine effluent is presented in Figures 3.4. The solid black lines show all the possible what-if scenarios if that input were to change, and all other inputs held constant. The blue and pink lines represent the 95% confidence band and prediction band respectively. The data in Figure 4.4a shows that soluble silica degradation rate decreased with increasing $Mg(OH)_2$ addition from 15 g to 19 g after which the degradation rate increased with increasing $Mg(OH)_2$ dosage. For pH, the silica degradation rate decreased for pH range of 10 to 10.5 and increased for pH 10.5 – 11 (Fig. 3.4b). The high silica degradation rate observed at pH 10.5-11 is consistent with previous finding reported by Batchelor et al. (1991) where the optimal pH for soluble silica degradation was between 10.5-11.0. Other studies (Kotz et al. 2006; Chao and Westerhoff, 2002) also revealed that pH of around 11 was optimal for soluble silica degradation with $Mg(OH)_2$.

The improvement in soluble silica degradation at pH>10.5 with increasing $Mg(OH)_2$ addition could be related to the adsorption of silica onto precipitated $Mg(OH)_2$ (Sheikholeslami et al., 2001). The solubility of magnesium hydroxide drops steeply (Eqn. 1) as the pH rises above 9.5 and approaches zero a little below 10.5 (James, 2008). This implies that at pH > 10.5, $Mg(OH)_2$ added will be precipitating out of solution with the silica since $Mg(OH)_2$ is an effective silica adsorbent due to it amorphous structure, high surface area, and porous surface (Lawler and Kweon 2004; Russell et al. 2009). Also, at pH >9.5 silica in the form of monosilicic acid dissolution increases with silicates species such as $H_3SiO_4^-$ and $H_2SiO_4^{2-}$ dominating the solution (Sheikholeslami et al., 2001). $Mg(OH)_2$ is positively charged hence, it could attract the negatively charged silicate

ions present in solution at pH > 10.5 thereby facilitating the degradation rate of silica from solution.

$Mg(OH)2\ (s) \Leftrightarrow Mg2+(aq) + 2OH-(aq)$ ` (3.3)

Furthermore, silica degradation in the form of calcium silicate (CaH_2SiO_4) precipitate might have contributed to the high silica removal rate at pH >10.5 since the pH modifier (lime) used in the study could yield high Ca^{2+} in solution (Batchelor et al., 1991). Low silica degradation rate observed at pH 10-10.5 is not very clear at this stage. However, it could mean that a certain pH is required for effective silica degradation. Based on the results, it is fair to say that for mine effluents in which pH is above 10, higher dosages of $Mg(OH)_2$ would lead to better degradation of the soluble silica in solution.

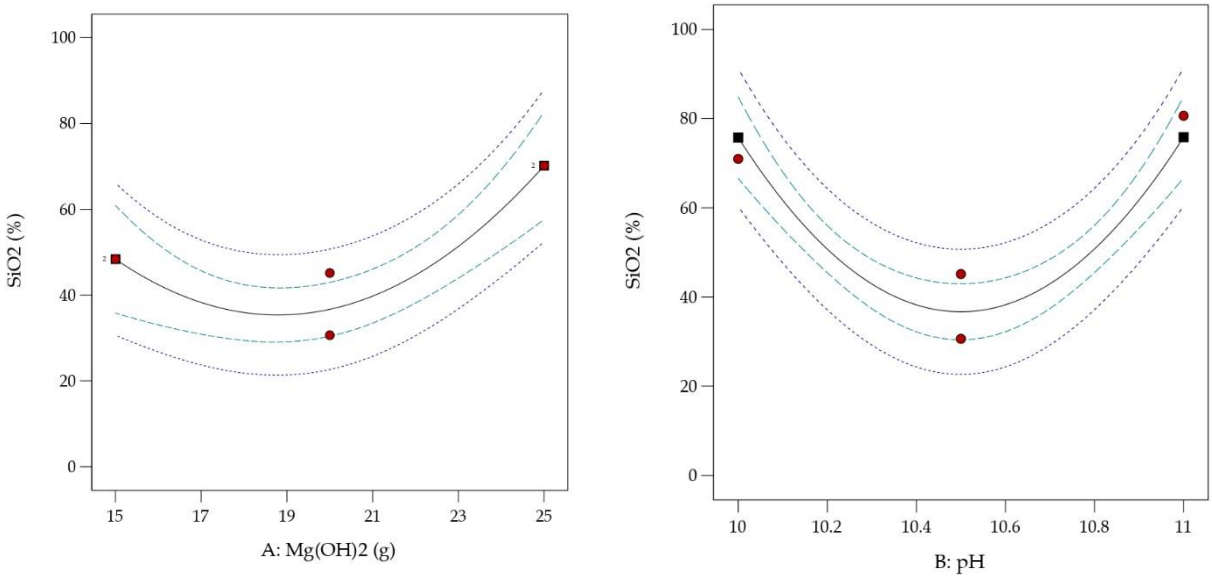

Figure 3.4 Soluble silica degradation as a function of $Mg(OH)_2$ and pH.

3.5.2 Influence of activated carbon on Silica degradation

Figure 3.5 shows the influence of activated carbon on soluble silica degradation rate. The data shows that soluble silica degradation rate increased with increasing the activated carbon addition from 5 g to 10 g. As mentioned earlier (Section 2), activated carbon used in this study was acidified with phosphoric acid. Hence, the phosphoric acid can react with the magnesium hydroxide to form magnesium phosphate and water (Eqn. 3.4) which then reacted with the soluble silica to produce a silicon phosphate precipitate (Eqn. 3.5), contributing to the improvement in silica degradation rate from solution. Increasing the acidified activated carbon addition translates into more phosphoric acid solution, which favour reaction 3.4 and subsequently 3.5 to yield more silicon phosphate precipitate. This acidified activated carbon option was chosen as it is a cheap option to increase dissolved magnesium hydroxide and, could lower the extent of final water electrical conductivity than soluble magnesium compounds (Latour et al, 2018). Interestingly, the 10 g of acidified activated carbon used produced the four best silica degradation of 99.2%, 96.8%, 99.2% and 97.6% (Figure 3.1).

$3Mg(OH)_2 + 2H_3PO_4 \rightarrow Mg_3(PO_4)_2 + 6H_2O$ (3.4)

$2Mg3(PO4)2 + 3SiO2 \rightarrow 6MgO + Si3(PO4)4$ (3.5)

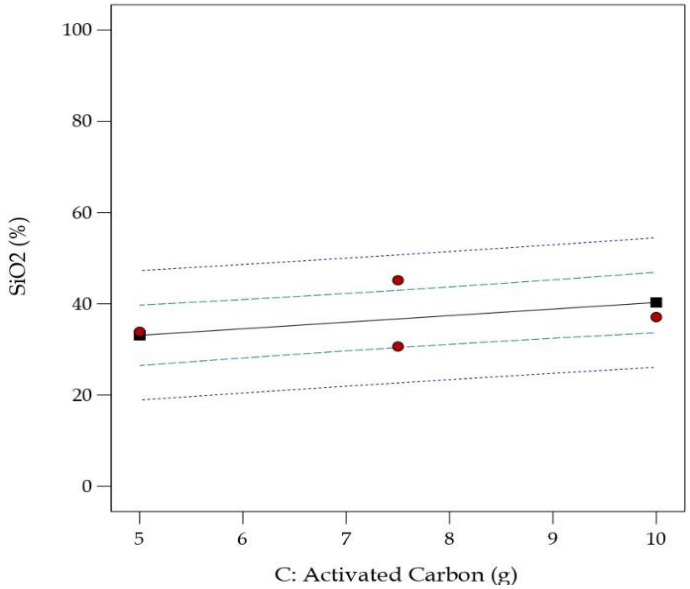

Figure 3.5 Soluble silica degradation rate as a function of acidified activated carbon.

3.5.3 Influence of reaction time and stirring rate on Silica degradation

In the case of reaction time, the data in Figure 3.6a shows that soluble silica degradation remained approximately unchanged between 15 min and 27 min and subsequently increased afterwards. This implies that allowing more time provides enough room for the silica to interact with $Mg(OH)_2$ and Ca^{2+} as well as phosphoric acid dissolution from carbon and its subsequent reaction with $Mg(OH)_2$ and silica to form various silicates precipitates in solution. Soluble silica degradation rate however, decreased with increasing stirring rate from 200 rpm to about 260 rpm after which further increase resulted in a slight increase in soluble silica degradation from solution (Figure 3.6b). The significance of the reaction time and stirring rate when it comes to silica degradation from the mine effluent is not very surprising since for the silica to degrade effectively, there was the need for the silica in solution to interface well with the chemicals for them to react very well.

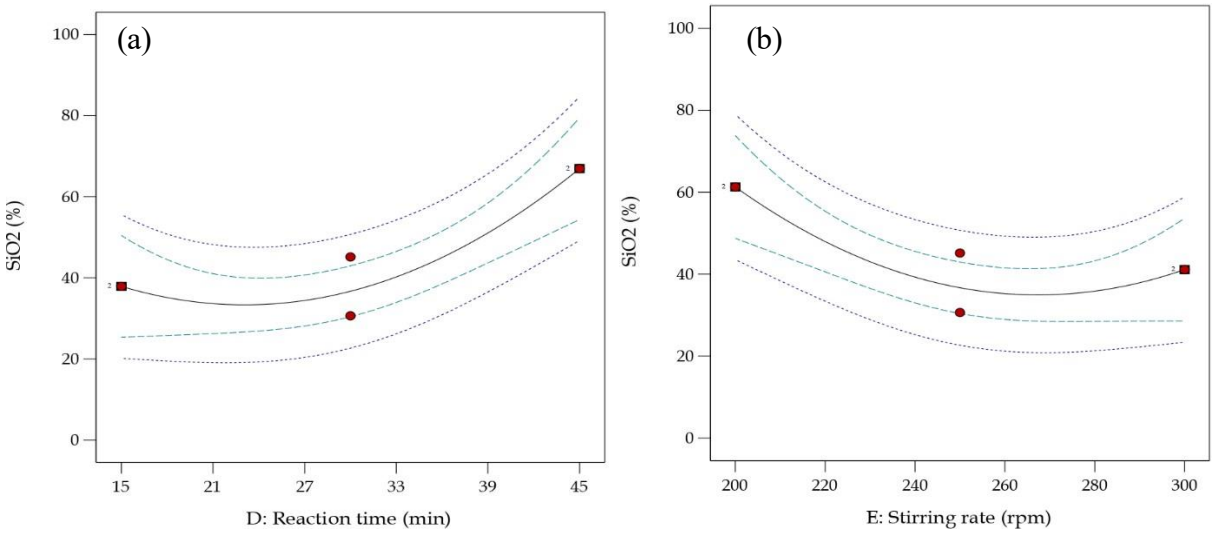

Figure 3.6 Soluble silica degradation rate as a function of reaction time and stirring rate.

3.5.4 Influence of magnesium hydroxide dosage, pH and activated carbon on Electrical Conductivity

Ordinarily, the use of sparingly soluble magnesium hydroxide has the advantage of adding less conductivity to the water while causing no further scaling problems it does not add counter ions to the total dissolved solids (Anon., 1992). Interestingly, Figure 3.7a confirms this assertion except to add that, at a low dosage of the magnesium hydroxide (i.e., 15 g/l), the electrical conductivity was lower than the EPA's standard limit of 1500 µS/cm. However, as the dosage of the magnesium hydroxide increased, the electrical conductivity also increased until it got to about 19 g/L and started decreasing again. This perhaps was due to the fact that the magnesium ions that were in solution at saturation point started a reverse reaction by precipitating into either magnesium oxide or magnesium hydroxide. Hence, at even a high magnesium hydroxide dosage of 25 g/L, the electrical conductivity is not expected to be high while at the same time it would ensure that the silica is degraded sufficiently. But on a whole, the observed increase in conductivity could be related to the Mg^{2+} and Ca^{2+} released from $Mg(OH)_2$ and $Ca(OH)_2$ (Slake slime) into solution.

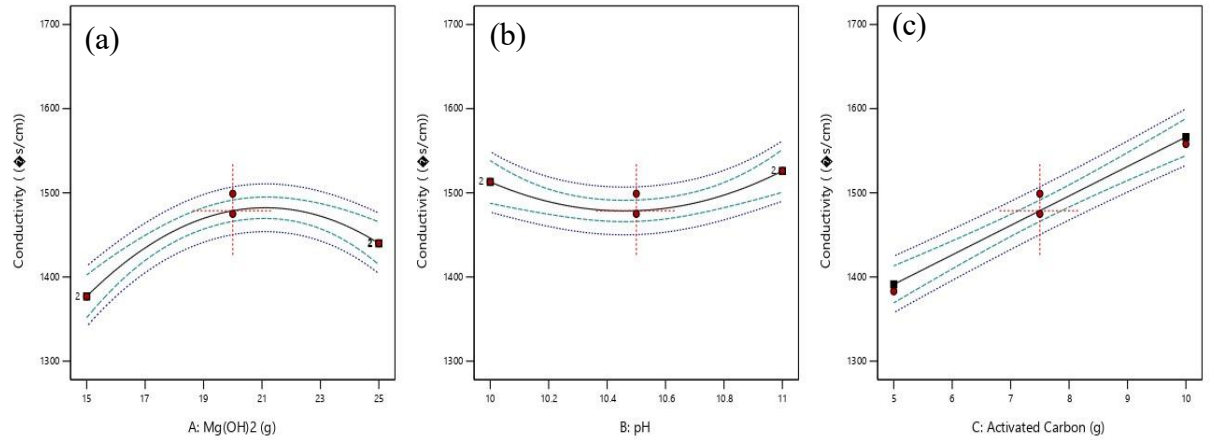

Figure 3.7 Influence of $Mg(OH)_2$ dosage, pH and activated carbon on electrical conductivity.

Further, as explained earlier, the activated carbon used for this research was activated by using phosphoric acid H_3PO_4. Hence, it is expected that this acid would dissolve into solution and play a part in the chemical reaction. As shown in equations (3.4) and (3.5), the phosphoric acid actually became the catalyst to speed up the reaction when it came to the dissolution of the magnesium hydroxide for the silica to degrade. In view of this, more hydrogen ions found themselves into solution making the output pHs of the test works that was conducted with the activated carbon to be low. This suggests that the behaviour of the electrical conductivity with respect to the activated carbon is actually related to the pH. On this premise, it can be seen from Figures 3.7b and 3.7c that the electrical conductivity show a symmetrical parabolic function with the pH whilst in the other, it has a proportional relationship. In Figure 3.7c for instance, it is clear that the electrical conductivity increased with increase in the weight of the acidified activated carbon (AAC), which would correspond to the amount of phosphoric acid that would be dissolved into solution to lower the pH. Consequently, when you consider the best four soluble silica degradation results in Figure 3.1 with acidified activated carbon (AAC) weights of 10 g each, all had output pHs of 9.38, 7.97, 8.41 and 9.58 deviating from their original input pHs of 11.0, 11.0, 10.0 and 11.0 respectively. As a result of the lowering of their pHs, their corresponding electrical conductivities increased from the initial electrical conductivity of the effluent, which was 1132 µS/cm to that of 1470 µS/cm, 1592 µS/cm,1574 µS/cm and 1551 µS/cm respectively accounting for increments of 29.9%, 40.6%, 39.0% and 37.0% respectively. So in a nutshell, electrical conductivity increases at 15<Mg(OH)2<19 g and decreases beyond 19 g. with regards to the pH, it decreases and increases slightly for pH 10-10.5 and 10.5-11 and then correlates positively to activated carbon addition rate.

3.5.5 Influence of stirring rate and reaction time on Electrical Conductivity

Stirring rates in liquid-liquid or liquid-solid solution mixing ensures that the dissolution of the constituents have taken place. Hence, as the stirring rate increases, it is expected that the dissolution of the chemicals would also increase. This implies that there would be more ions in solution to facilitate the rate at which the solution can conduct electrical current. As shown in Figure 3.8a, it can be seen that increments in the stirring rate resulted in the increase in electrical conductivity. However, the electrical conductivity started declining when the stirring rate was about 270 rpm. This notwithstanding, from Figure 4.1 under Appendix, the highest electrical conductivity (i.e., 1592 µS/cm) recorded occurred at a stirring rate of 300 rpm. This affirms theoretically that stirring rate is proportional to electrical conductivity. On the other hand, the reaction time rather shows a parabolic relationship with the electrical conductivity that is anticlimax (Fig. 3.8b). From the reaction time of 15 min, the electrical conductivity increased steadily until it started decreasing around a reaction time of 27 min. This suggests that an increase in the reaction time doesn't translate into an increase in the formation of ions to increase the electrical conductivity. Hence, for all the four best silica degradation results (Fig. 3.1), which also produced some of the highest ECs, only one of them had a reaction time of 45 min with the rest taking place within 15 min.

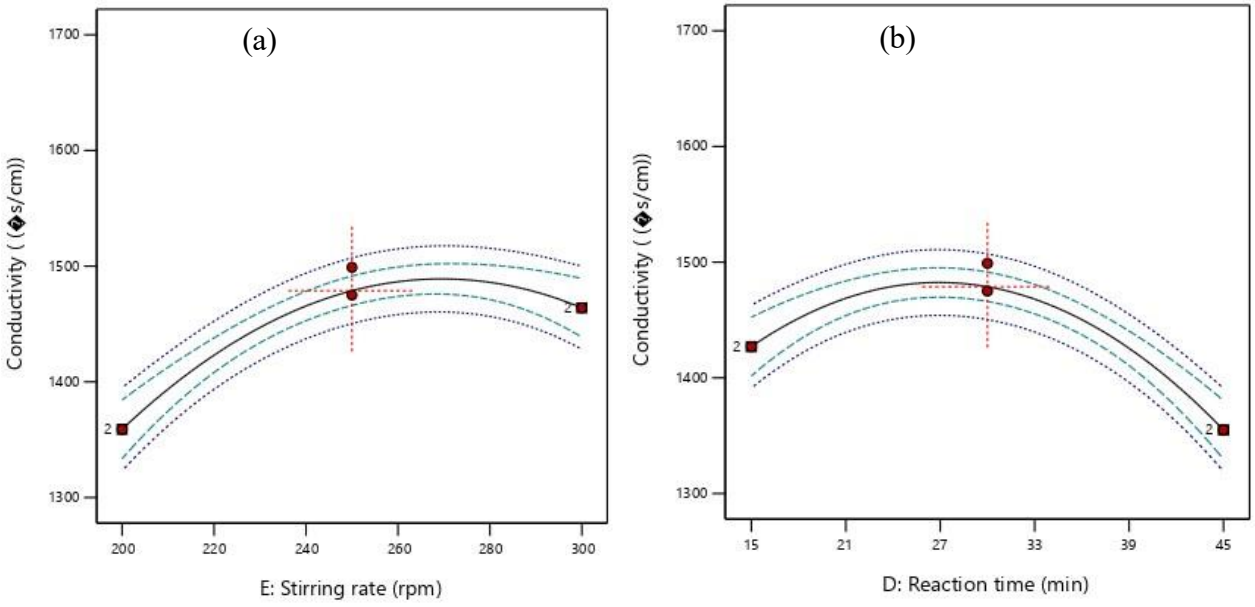

Figure 3.8 Electrical conductivity performance in respect of reaction time and stirring rate.

3.5.6 Optimization of process parameters

From the response surface analysis (section 3.5), it was clear that high soluble silica degradation rate could be achieved at $Mg(OH)_2$ dosage, pH and reaction time greater 19 g, 10.5, and 27 min respectively and high activated carbon addition at low stirring rate. In the case of conductivity, $Mg(OH)_2$

dosage, pH and reaction time greater 19 g, 10.5, and 27 min respectively, and less activated carbon addition and stirring rate was required to achieve low values. Therefore, to establish the optimized conditions for the operating parameters, numerical optimization was executed by using the desirability function of Design Expert Software. Figure 3.9 shows the multi-objective optimization ramps for input and output variables. The optimization report gave 90 solutions which contain different levels of independent variables. Solutions that gave maximum desirability value of 1 was selected as the optimized condition for necessary to achieve high silica degradation and low solution conductivity. The response values for one of the selected solutions at optimized preparation conditions for silica degradation and low solution conductivity is presented Table 3.2.

Table 3.2 Optimized Conditions Proposed by RSM.

$Mg(OH)_2$ (g)	pH	Activated Carbon (g)	Reaction time (min)	Stirring rate (rpm)	SiO_2 (%)	Conductivity *µS/cm*	Desirability
20.9	10.6	5.0	43.8	200	99.0	1189.4	1.0

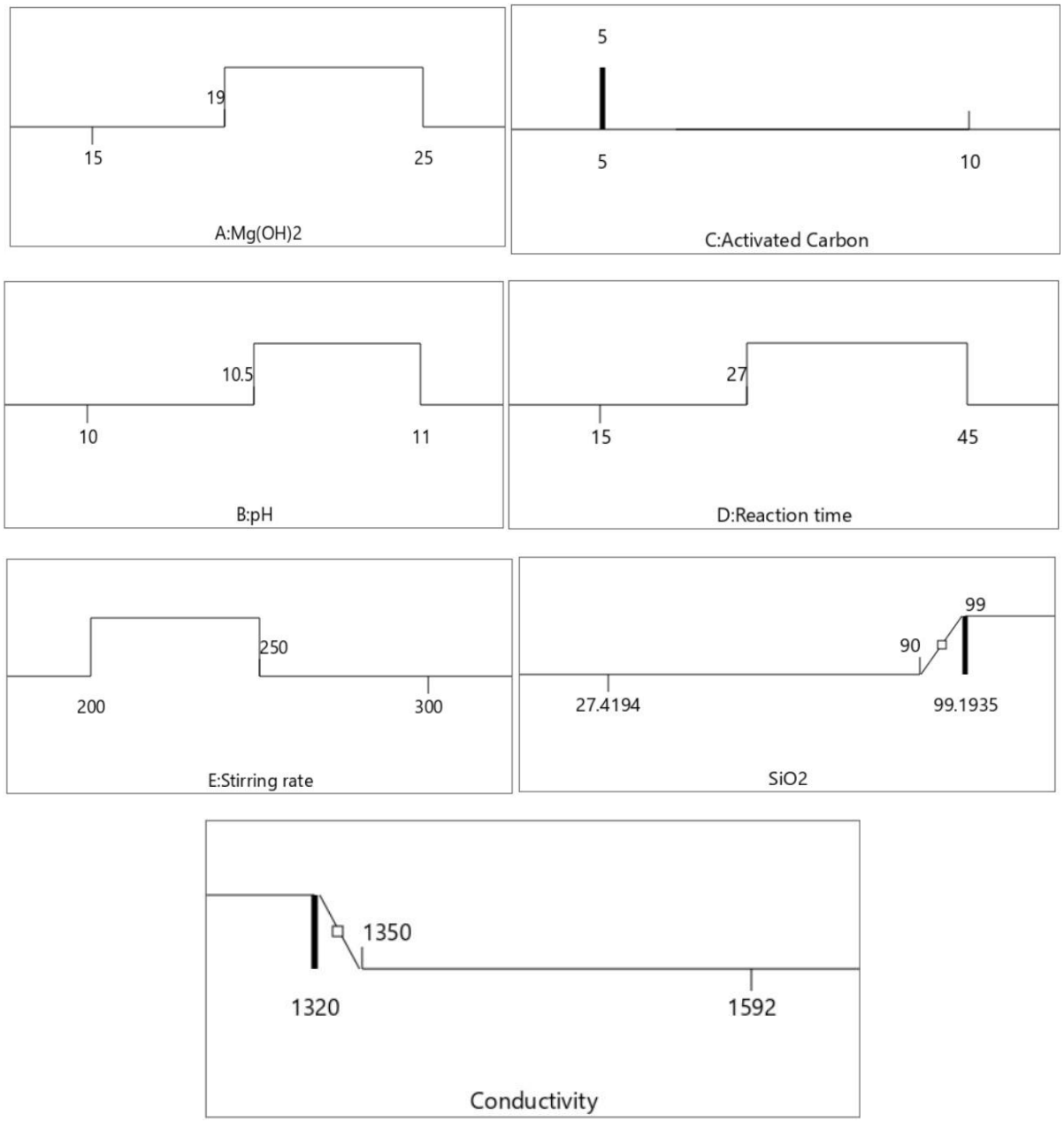

Figure 3.9 multi-objective optimization Ramps for input variable and output variables.

3.5.7 Response Surface Methodology (RSM) Model validation

To check the suitability and accuracy of the model for prediction of response variables, the optimized preparation conditions proposed by the RSM were validated by performing experiments under optimized conditions. The experimental values at optimized preparation conditions gave approximate soluble silica degradation and conductivity of 99.0% and 1189.4 respectively (Table 3.3). The experimental values for soluble silica degradation rate and conductivity align very well with the model predicted values. The results obtained clearly show that high soluble silica degradation rate of mine effluent could be treated at low solution conductivity. The relative error associated with soluble silica degradation and conductivity were error of 3.03% and -0.84% respectively.

Table 3.3 RSM predicted and experimental value for response variables at optimized conditions.

RSM predicted Results		Laboratory test confirmation results		Relative Error (%)	Relative Error (%)
SiO_2 (%)	Conductivity *µs/cm*	SiO_2 (%)	Conductivity *µS/cm*	SiO_2	Conductivity
99.0	1189.4	96.0	1199.4	3.03	-0.84

4. CONCLUSION

The study focused on the use of magnesium hydroxide and acidified activated carbon to degrade soluble silica in mining effluents to meet the EPA limit and fulfill the WHO drinking water quality standard. Characterization of the mine water effluent revealed that the soluble silica, pH and conductivity were 124 mg/L, 10.0 and 1132 µS/cm respectively. This meant that soluble silica degradation greater than about 84% was required in other to meet the EPA limit. The employment of response surface methodology (RSM) technique was successfully used to develop models to optimize preparation conditions needed to degrade soluble silica in the mine effluent while maintaining the mine effluent conductivity below the EPA limit of 1500 µS/cm. Significant model F-values ($p<0.05$) and nonsignificant lack of fit F-value ($p>0.05$) for the response variables (i.e., percent silica degradation and conductivity) indicated the accuracy of the models. The predicted vs. actual plots revealed graphs with high linear function, which signified that the experimental observed values for the response variables were in close agreement with the predicted values. Externally studentized residuals vs. predicted values of response parameters revealed absence of constant error.

The study revealed that silica degradation rate decreased with increasing $Mg(OH)_2$ addition and pH from 15 g to 19 g and 10 – 10.5 respectively. Beyond the aforementioned $Mg(OH)_2$ and pH range values, the degradation rate increased significantly. Soluble silica degradation rate increased with increasing the activated carbon under conditions of study. Whereas in the case of reaction time, soluble silica degradation remained approximately unchanged between 15 min and 27 min and subsequently increased afterwards, the degradation rate of soluble silica decreased with increasing stirring rate. The optimum conditions for high soluble silica degradation and low solution conductivity was identified to be $Mg(OH)_2$ and activated carbon additions of 20.9 g and 5.0 g respectively, pH of 10.6, reaction time of approximately 44 min and stirring rate of 200 rpm. Based on the optimized conditions, soluble silica degradation and solution conductivity of 96.0% and 1199.4 µS/cm was achieved experimentally. Based on the study, it is clear that mine effluent containing soluble silica could be degraded while maintaining low solution conductivity by the use of magnesium hydroxide and acidified activated carbon to meet the EPA limit. The proposed mechanism for soluble silica degradation was ascribed to the precipitation of silicon phosphate emanating from reaction between magnesium phosphate and silica, adsorption of silica onto precipitated $Mg(OH)_2$ and the formation of calcium silicate (CaH_2SiO_4).

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