Models and Optimization Process of Acid Effects on the Silica and Alumina Contents of Geopolymer Concrete

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Abstract

Geopolymer concrete concept has attracted attentions during the last two decades due to its superior properties, that has made it good and a potential alternative to ordinary Portland cement. The current study therefore, aimed to study the effect of acid on silica and aluminum content of geopolymer concrete and its optimum study. This was achieved by using experimental design tool by adopting central composite design (CCD) technique resulting in a total number of 13 experimental runs for optimum acid concentration effects on the geopolymer concrete mix. Water and acid concentrations were variable parameters used to study their effects on concrete mix, with boundaries of 2-5 ml and 5-10 ml for acid and water concentrations respectively. These two variables were selected to optimize the chosen key factors such as the silicate (SiO_2) and alumina (Al_2O_3) contents of the mix. These were inputted on the design of experiment interface, studied the effects as responses and simulated for ANOVA analysis and optimization. The mass to volume variation trends recorded for SiO_2 and Al_2O_3 were 23.1 to 20.3 kg/m^3 and 6.1 to 5.1 kg/m^3 respectively. The silica to alumina ratio and silica to magnesium ratio of the geopolymer concrete contents were established to be 3.7 and 5.5 respectively which were within the specifications range. It is concluded that, the optimum acid treatment was established as evidence from the R^2-values of both silica and alumina which were close to 1 and the error difference between the predicted and validated values of 2.9 and 3.0 % respectively. The models therefore, are significant as their probability greater than F-value are less than 0.05 in each case.

Keywords
Silica, alumina, Anova, optimization

1. Introduction
Geopolymer is considered is considered as an alternative material to Portland cement owing to its benefits in terms of environmental protection and energy conservation when compared to the other types. This is justified since about 43 to 59 % energy reduction can be achieved during production as compared to conventional concrete (Abdul, *et al*., 2022). In addition to the excellent acid resistance. Geopolymerization is the reactions that occur between silicon (Si), aluminum (Al) and natural source product such as metakaolin or as fly ash which is a byproduct materials with alkali liquids combine together to establish geopolymer binders Ma *et al*., (2018). Furthermore, geopolymerisation increases the strength of geopolymer concrete after being exposed to elevated temperatures (Jabulani *et al*., 2022). Many researches have been reported on the study of geopolymer chemistry, structural performance in terms of geopolymer concrete members when subjected to service loading, such as, beams and columns and steel fiber reinforced geopolymer concrete. However, despite that geopolymer has good resistance to acid, the performance effects on the optimum acid concentration on the material has not been investigated. Therefore, this work aimed to study the effect of acid on silicon and aluminum content of geopolymer concrete.

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1.1 Objectives

The current study therefore, aimed to study the effect of acid on silica and aluminium content of geopolymer concrete and its optimum study. This will be achieved by carrying out the experimental design set up by adopting central composite design (CCD) technique which will result to a total number of 13 experimental runs for optimum acid concentration effects on the geopolymer concrete mix.

2. Literature Review

Climate change and global warming has been a major issue because of the emission of greenhouse gases into the atmosphere as a result of technology. CO_2 is a good example of such gases which is formed during the production of cement as an important ingredient of concrete production. The CO_2 contribute approximately about 65 % of the global warming (Mccaffrey, 2002).

For each ton of Portland cement produced, there is approximately about the same ton of the CO_2 gases into the atmosphere (Greer *et al.,* 2004). This fact is a major concern when the number of tons produce annually is checked. However, cement factories has been reported to be emitting about 1.5 billion tons of the CO_2 into the atmosphere yearly. As such the academies of researchers are looking for an alternative to Portland cement, while geopolymer was found to be promising to tackle or reduce such menace.

As time goes on, the use of geopolymers will reduce the demand for Portland cement since its implementation in concrete technology increases for the purpose of sustainability of concrete. Therefore, there is need to effectively check the suitability of fire resistance of the geopolymer concrete.

3. Methods

Design of experiment version 12 was used to carry out the experimental design set up by adopting central composite design (CCD) technique resulting in a total number of 13 experimental runs. The variable parameters used to study their effects on geopolymer concrete mix formulated are water concentration and acid concentration which were varied between 2-5 ml for acid and 5-10 ml for water concentrations respectively as the chosen boundary values. These two variables were selected to optimize the chosen key factors such as the silicate and alumina contents of the mix. These were inputted respectively on the design of experiment interface tool. The geopolymer constituents were also analyzed using XRF using standard method.

4. Results and Discussion

4.1 X-Ray Fluorescence Analysis of the Geopolymer Concrete Formulation

Table 1 shows the chemical composition of the geoplolymer concrete formulation in terms of mass to volume ratio. The chemical compositions of the formulated concrete before subjecting it to acid treatment alongside the control sample are presented in Table 1. The silica to alumina (Si/Al) ratio of the sample and that of control are 3.7 and 3.5 respectively as shown in the table, which are within the recommended values of between 2.5 to 4.1 for quality concrete mixed as reported in the work of Hongguang *et al*., (2021). The value of magnesium oxide MgO obtained to be 4.2 and 4.0 Kg/m^3 respectively were within the recommended value of ≥ 1.65 according to BS 12, which further justified the quality of the formulated geopolymer concrete mix. The loss on ignition in both cases were recorded to be 2.8 and 2.6 % respectively as shown in Table 1, which also fell within the recommended specification of < 4. The sodium hydroxide content of the mix was recorded as 28.33 Kg/m^3 for the prepared treatment sample while that of the control was 26.98 Kg/m^3. The sodium hydroxide constituents of the samples were in close agreement with the works of Inocente *et al*., (2021) and Jabulani *et al*., (2022). More so, one of the most important constituents in concrete mixed formulation are the silicates to alumina ratio constituents of metakaolin, which is expected to be in the range of 1.5 to 5.08 upon mixing to form geopolymer solution. Thus, they were obtained to be 3.6 to 3.5 respectively. These two geopolymer important parameters were also investigated further in terms of optimum acid treatment conditions.

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Table 1: The chemical composition of the formulated concrete mix

Compound compositions	Treated Sample (Kg/m^3)	Control Sample (Kg/m^3)
SiO_2	23.1	21.6
Al_2O_3	6.3	6.1
Na_2SiO_3	145.66	141.01
NaOH	28.23	26.98
MgO	4.2	4.0
H_2O	33.00	31.98
LOI	2.8	2.6
Fine aggregate	712.22	702.50
Coarse aggregate	1267.45	1256.00
$SiO_2Al_2O_3$	3.7	3.5
SiO_2/MgO	5.5	5.4

4.2 Effect of Acid Solution in the Formulated Concrete Mix

Figure 1 shows the trends variation in the mass to volume loss in terms of silica and alumina contents of the geopolymer concrete mix due to the effect of acid on the formulated concrete mix. As shown in the figure, the mass to volume of sample treated with different acid concentration in terms of silica content, decrease gradually from 23.1 kg/m^3 to 22.4 kg/m^3 on treatment period 1 through day 3. It continued to decrease in weight from 22.4 kg/m^3 to 22.1, 21.9, 21.3 kg/m^3 between the treatment days 3 to day 8, until it stabilizes at 20.3 kg/m^3 from the 9th day to the 13th day of the investigation. As observed from day 9 to 13, the trend in the weight loss stabilizes; indicating that, the acid effect on the concrete mix has reached it breaking limit, where no further deformation can occur on the structure. However, the alumina content showed that, acid has effect on the geopolymer concrete mix structure as evidence in terms of alumina from its weight to volume loss variation trend as also represented in Figure 1. As shown in the figure for the alumina content, as the acid concentration increases, the weight to the volume ratio of the alumina content increases. This is evidence from the concentration of 2 ml acid content on day 2 as against weight to volume ratio of 6.3 kg/m^3 as compared to 6.0 and 5.5 kg/m^3 recorded on day 4 and 7 with acid concentration of 10 ml and 6 ml respectively, until the acid concentration was reduced to 6 ml from day 9-13, which also influence the weight to volume loss stability of the mix, indicating that hydration process increases the strength of structure.

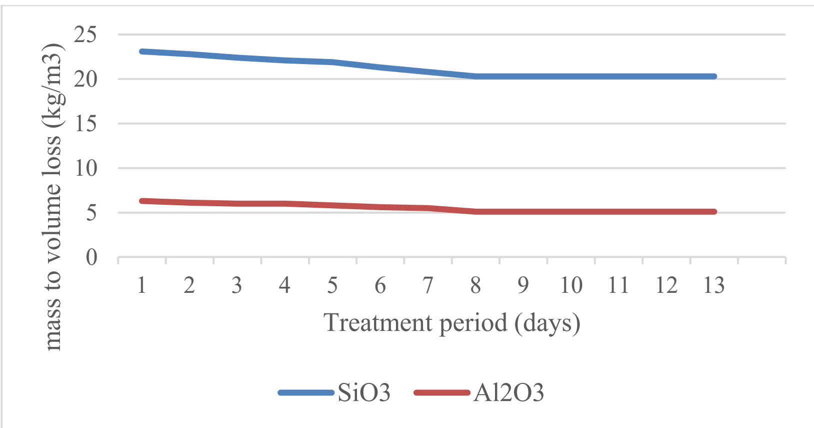

Figure 1: Relationship between the acid treated sample investigation of silica and alumina geopolymer in terms of weight to volume loss

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4.3 Silicate and Alumina Content Analysis of the Simulated Output Results

Table 2 shows the composition of SiO_3 and Al_2O_3 in the formulated geopolymer concrete mix when subjected to different varying acid concentrations. As shown in Table 2, the SiO_3 and Al_2O_3 concentrations varied between 23.1-20.3 and 6.3-5.1 kg/m^3 in each case due to different concentration of acid treatment. As observed from the results displayed in the table, as concentration of acid were 2 ml, the SiO_3 and Al_2O_3 concentration were 23.1 kg/m^3 and 6.3 kg/m^3 respectively, but when the concentration was 10 ml, the SiO_3 and Al_2O_3 were 22.8 and 6.1 kg/m^3 respectively, indicating that the concentration of the solution has variable effects on the SiO_3 and Al_2O_3 contents in the geopolymer concrete formulation.

Table 2: Results of the silicate analysis from the experimental simulation

S/N	Water (ml)	Acid conc. (ml)	SiO_3 (kg/m^3)	Al_2O_3 (kg/m^3)
1	2.00	2.00	23.1	6.3
2	10.00	2.00	22.8	6.1
3	2.00	5.00	22.4	6.0
4	10.00	5.00	22.1	6.0
5	0.34	3.50	21.9	5.8
6	11.66	3.50	21.3	5.6
7	6.00	1.38	20.8	5.5
8	6.00	5.62	20.3	5.1
9	6.00	3.50	20.3	5.1
10	6.00	3.50	20.3	5.1
11	6.00	3.50	20.3	5.1
12	6.00	3.50	20.3	5.1
13	6.00	3.50	20.3	5.1

4.4 Analysis of Variance (ANOVA) Results of the Silicate

The analysis of variance results for silicate content of the geopolymer formulated mix is presented in Table 3. As depicted in the table, the generated quadratic model with Prob > F value of 0.0002 is significant because it is less than the reference recommended value of 0.05. The lack of fit as also shown in Table 3 is not-significant, which is desirable in statistical fit models.

Table 3: Analysis of variance for the trisilicate content

Source	Sum of square	DF	Mean value	F-value	Prob>F	Remark
Model	2.44	5	0.49	25.95	0.0002	Significant
A	0.35	1	0.35	18.67	0.0035	
B	0.13	1	0.13	6.99	0.0333	
A^2	1.56	1	1.56	82.68	< 0.0001	
B^2	0.40	1	0.40	21.39	0.0024	
AB	0.18	1	0.18	9.37	0.0183	
Residual	0.13	7	0.019			
Lack of Fit	0.13	3	0.044	224	1.0012	Not significant
Pure Error	0.000	4	0.000			

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As also shown in Table 3, the model terms A, B, D, A^2, B^2 and AB with model prob > F values of 0.0035, 0.0333, <0.0001 and 0.0024 respectively are all significant as they are less than 0.05.

More so, the interactions between the independent variable parameters were further checked for their consistency using the model adequacy parameters as shown in Table 4. As it is observed in the table, the R^2 value of 0.9488 and the adjusted R^2 of 0.9122 shows that, the model is good because their percentage error difference is 3.9 %, this also influence its adequate precision of 13.532 to be greater than 4, which further that the model is good.

Table 4: Adequacy test of the model

Parameter	Values
R-Squared	0.9488
Adj R-Squared	0.9122
Pred R-Squared	0.9359
Adequate Precision	13.532

Moreover, the model generated form the independent variable parameters for silicate content of the formulation is presented in Equation 1. The significance of the model equation is to predict desired response of a proposed experimental design prior to the practical scientific experiment, it also reduced the numbers of trials and minimize error as much as possible.

$$SiO_3 = 53.88 - 0.21A - 0.12B + 0.47A^2 + 0.24B^2 + 0.21AB \quad (1)$$

4.5 Relationship between the Predicted and Experimental Silicate Plots

Figure 2 described the relationships between the predicted and actual responses. The figure presented the design expert parity plots of the responses against its independent variables.

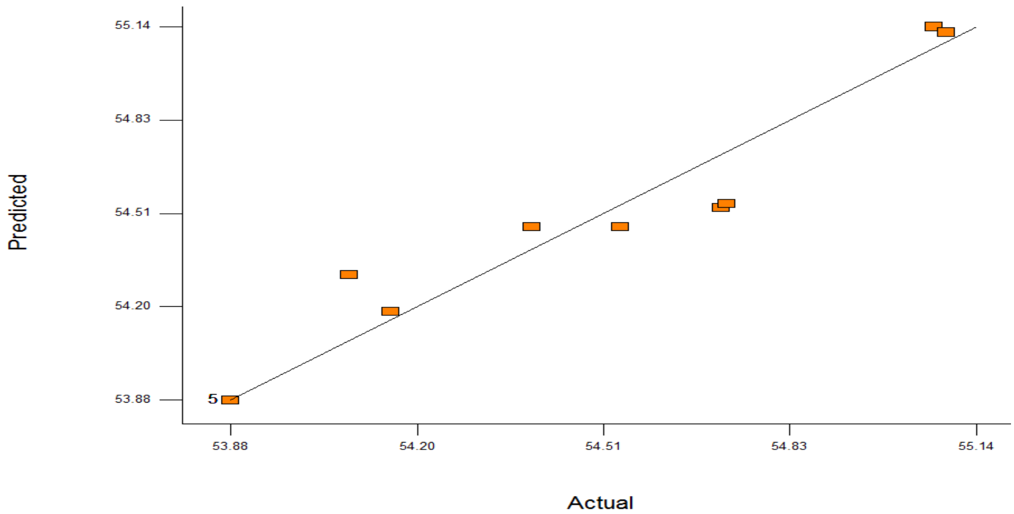

Figure 2: Parity plot relatiosnhip of predicted and actual for the geopolymer silicate content

4.6 3D-Surface Plots of Variable Parameters and Responses Studied

The 3D response surface plots generally illustrate the effects of the independent variables and their interactive effects on the responses as reported by Li *et al*., (2020). Also, the shape of the corresponding contour plots usually indicates whether the mutual interactions between the independent variables with their responses are significant or not, in which an elliptical nature of the contour plots usually indicates that the models are significant. Thus, the 3D plots shown in Figure 3 illustrate the effect of acid solution and water on the formulated geopolymer concrete mix. The elliptical contour shape as shown in the figure further justified that, the model developed is significant and a fit model representing a good interaction between the independent variables and the response.

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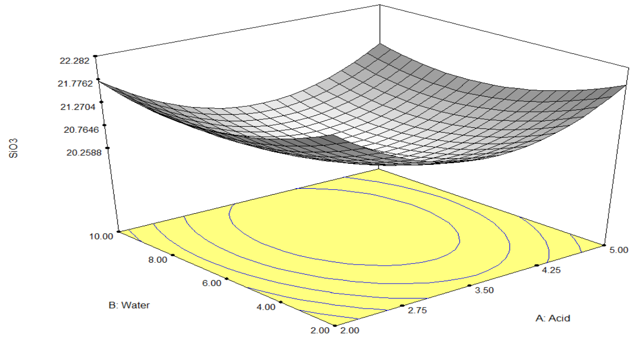

Figure 3: 3D plot of SiO_3 against acid solution and water concentration effects

4.7 Pertubation Plot Relationship of the Variable Parameters

Figure 4 depicts the interaction relationship of water (A) and acid (B) effects on the formulated geopolymer concrete mix. As shown in the figure, the two parameters are highly dependent as observed in the plot that, they not parallel to each other in their interaction but met at a reference point of 0.000.

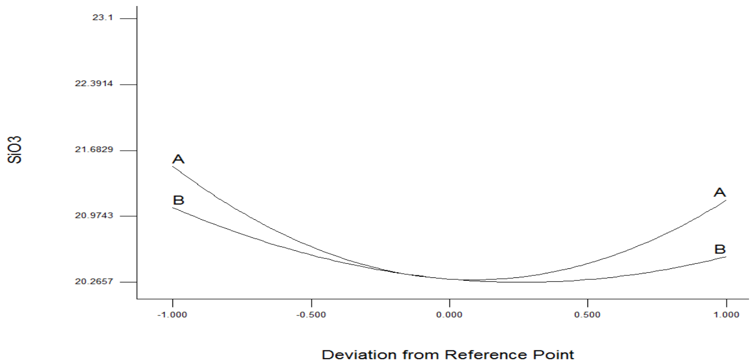

Figure 4: Pertubation plot relationship between the two variable parameters

4.8 Analysis of Variance (ANOVA) Results of the Alumina Content of the Geopolymer Concrete (Al_2O_3)

The silicate **(**Al_2O_3) contents analysis of variance results of the formulated geopolymer concrete mix are presented in Table 5. As it can be seen from Table 5, the model with Prob > F value of <0.0001 is significant as it is less than 0.05. Also, the lack of fit value of 0.9767 is not-significant but desirable in for a fit model. The model terms A, B, D, A^2, B^2 and AB with model prob > F values of <0.0001 in each case as also shown in Table 5 are all significant as they are less than 0.05.

Table 5: Results of analysis of variance of alumina content of the geopolymer concrete mix

Source	Sum of square	DF	Mean value	F-value	Prob>F	Remark
Model	1.46	5	0.29	2842.50	< 0.0001	Significant
A	0.13	1	0.13	1263.52	< 0.0001	
B	0.40	1	0.40	3951.61	< 0.0001	
A2	0.59	1	0.59	5787.25	< 0.0001	
B2	0.33	1	0.33	3269.29	< 0.0001	
AB	0.096	1	0.096	938.45	< 0.0001	
Residual	7.168E-004	7	1.024E-004			
Lack of Fit	7.168E-004	3	2.389E-004	652	0.9767	Not significant
Pure Error	0.000	4	0.000			

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Also, Table 6 shows the consistency of the model adequacy parameters. As it is observed in the table, the R^2 value of 0.9995 and the adjusted R^2 of 0.9992 shows that, the model is consistent with high level of good interaction as their percentage error difference is 0.03 %. More so, the adequate precision of 148.125 is greater than 4, which further justified that the model is good.

Table 6: Adequacy test of the alumina model

Parameter	Values
R-Squared	0.9995
Adj R-Squared	0.9992
Pred R-Squared	0.9965
Adequacy Precision	148.125

Equation (2) shows the interaction between the independent variables and alumina content of the geopolymer concrete response. The significance of this equation showed that, the model it represents can be repeatable, validated and predict the desired response of the experimental design details.

$$Al_2O_3 = 19.10 - 0.13A - 0.22B + 0.29A^2 + 0.22B^2 + 0.16AB \quad (2)$$

4.9 Parity Plot Relationship of the Experimental and Predicted Alumina Response

The relationships between experimental and predicted data of alumina response are presented in Figure 5. As shown in the figure, all the data points fell on the regression line and also superimposed with the predicted data, indicating that the model is significant and the experimental data are in good agreement with the predicted data.

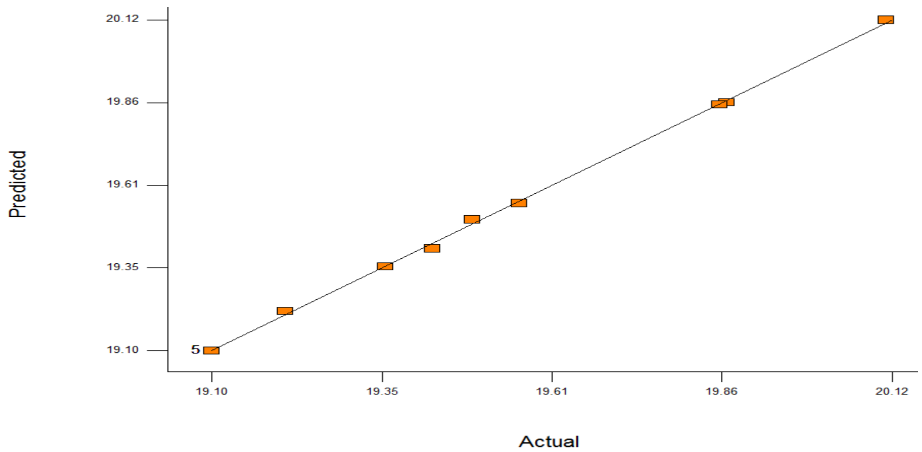

Figure 5: Parity plot of the experimental and predicted data of alumina content

Figure 6 further explained the significance of the model generated in terms of the alumina content of the geopolymer concrete mix

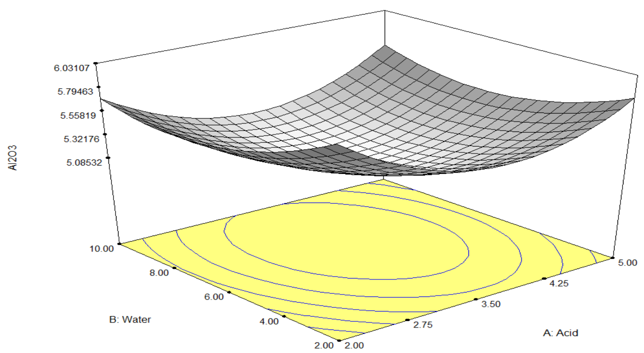

Figure 6: Three-dimensional plot of water and acid solution effects on alumina content

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The 3D plot shown in Figure 6 further proved that, the model generated is significant as evidence from the elliptical nature of the contour surface. This indicates that, there is a good interaction between the independent variables and the alumina response. The significance level of the model and good interaction between the two independent parameters was further justified from the data relationship graph shown in Figure 7.

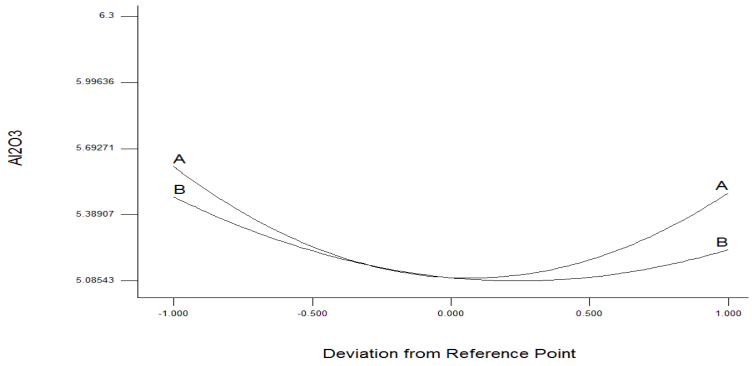

Figure 7: Perturbation plot of the independent variable parameter relationships.

As shown in Figure 7, the two independent variables A and B are in good interaction with each other, meeting at a reference point as shown in the figure where A represents the acid concentration and B is the water concentration.

4.10 Optimization and Validation of Results of the Responses

The optimization solutions of the silicate and alumina constituents of the geopolymer concrete mix are presented in Table 7.

Table 7: optimization solutions of the responses using numerical technique

Parameters	Water conc (ml)	Acid conc. (conc)	SiO_2 (%)	Al_2O_3 (%)	Desirability
1	6.71	3.32	24.66	6.5	0.92
2	6.59	3.50	19.78	5.9	0.88

As shown in Table 7, the optimization processes reduced the number of experimental runs from 13 to 2 after several hundreds of simulations. Thus, the two optimized solutions as shown in Table 6 have different desirability. However, the optimized solution number one (1) was selected based on its highest desirability of 0.92 for validation when compared to solution two (2) with desirability value of 0.88. Also, the other criteria used to select solution (1) is that, it predicted highest values of SiO_2 and Al_2O_3 of 24.66 kg/m^3 and 6.5 kg/m^3 when compared to 19.78 and 5.9 kg/m^3 respectively despite using lower concentration of acid. The validated results are also shown in Table 8. As presented in the table, the validated solutions are in a very good agreement with the optimized values. This is evidence from the error differences of the responses investigated whose silica contents optimized value and its validated error difference is 2.9 % and that of alumina content is 3.0 %. It can therefore, be proved that the predicted optimized results are consistent with the validated analysis as observed from the data generated.

Table 8: Validated optimized results of the responses

Parameters	SiO_2	Al_2O_3
Optimized values	24.66	6.5
Validated values	23.95	6.7
% Error difference	2.9	3.0

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5. Proposed Improvement

The present investigation can be improved by increasing the numbers of independent variable parameters to compare their effects and efficiencies on their respective geopolymer properties or responses numerically or graphically. The graphs illustrated in the Figures 8 to 10 can be used as references for improvement. The deviation of experimental data from the predicted as shown in Figure 8 can be improved with several incorporated independent variable parameters in future studies. Also, the Box-Bohnken Design (BBD) as shown in Figure 9 can be tested and compared with Central Composite Design (CCD), in terms of their improved efficiency.

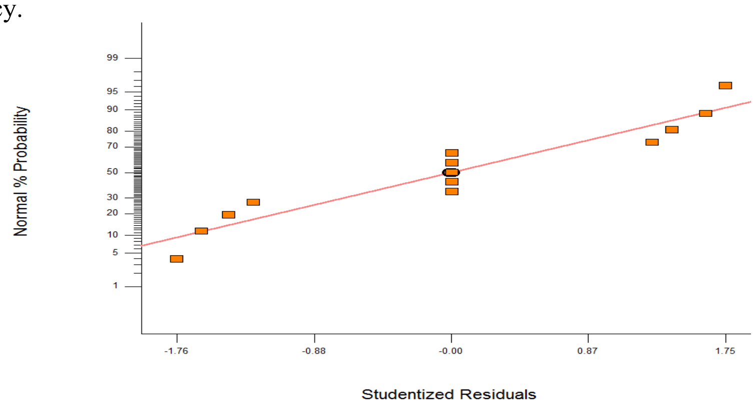

Figure 8: Normal probability and residual of geopolymer properties

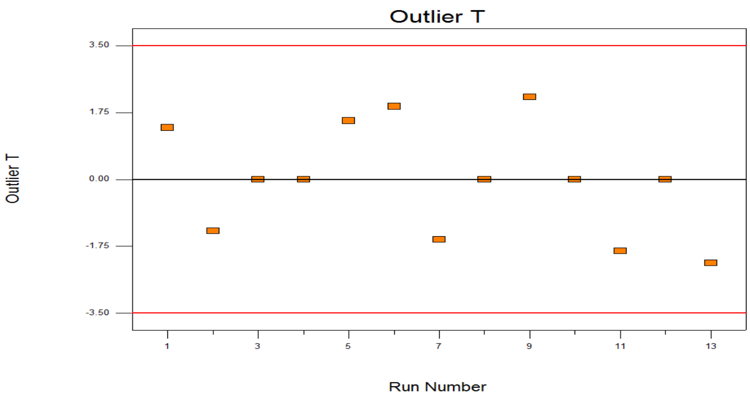

Figure 9: The outlier graph showing deviation from ideal data

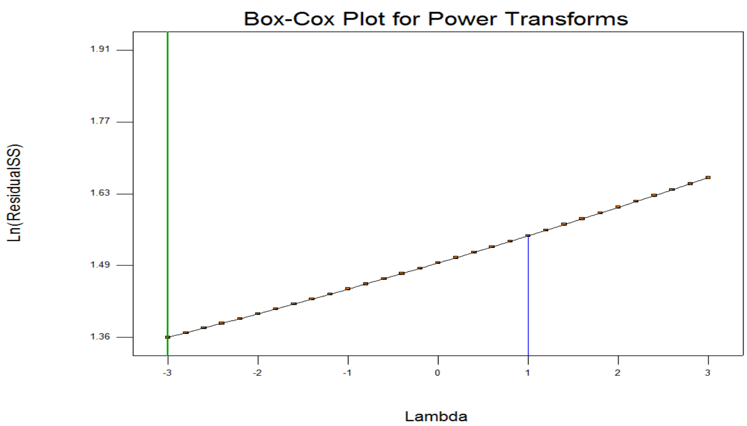

Figure 10: Box-cox plot

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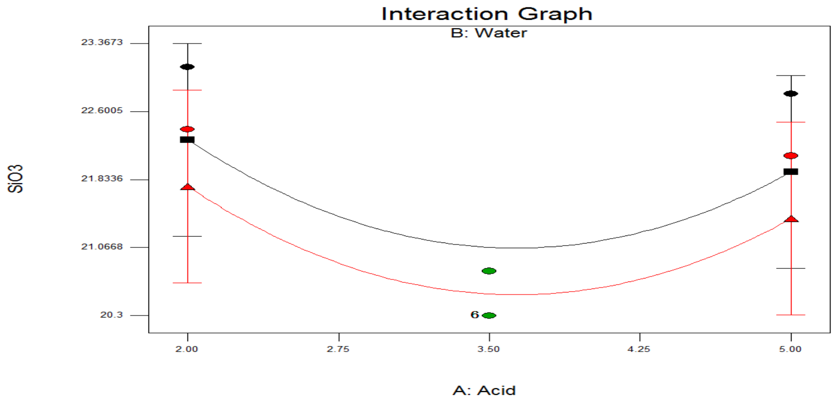

Figure 11: The interaction between acid and water against silica content.

6. Conclusions

In this study, the effect of acid on geopolymer concrete mix was established as the silica and alumina contents of the formulation were reduced from 23.1 to 20.3 kg/m^3 and 6.1 to 5.1 kg/m^3 respectively within the investigation period. The silica to alumina ratio and silica to magnesium ratio of the geopolymer concrete contents were established to be 3.7 and 5.5 respectively which were within the range of specifications. It is also concluded that, the optimum acid treatment was established as evidence from the R^2-values of both silica and alumina which were close to 1 and the error difference between the predicted and validated values of 2.9 and 3.0 % for the silica and alumina respectively. The models therefore, are significant as their probability greater than F-value are less than 0.05 in each case.

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