

Response Surface Modelling Of Densification, Porosity And Rupture Modulus Of Ceramic Tiles From Nigerian Raw Materials

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ABSTRACT

The modelling of experimental investigations has minimized research complexities in a logical and systematic manner that lead to simplified predictions. In this study, response surface modelling of the densification, porosity and rupture modulus of ceramic tiles was carried out. Formulated slip from local Nigerian mineral raw materials were shaped into ceramic tiles with their particle sizes as well as the Feldspar contents and sintering temperatures varied. 20-runs experiments drawn with DesignExpert® yielded results used to model the density, porosity and rupture modulus of the ceramic tiles. Discussions from 2D Contours and 3D Surface plots revealed detailed effects and interactive influence of the parameters on the response variables. Residual analysis showed Box-Cox plots with Lambda values equal to 1, hence, no data transformation was carried out. The residual analysis demonstrated linear correlations with minimal variation between the experimental data and the predicted results from the second-order polynomial (quadratic) models developed. Remarkably, the three models were statistically fit for predicting the responses of the tiles as the lack of fits were insignificant with respect to the pure errors. Likewise, the Analysis of Variance (ANOVA) results revealed that the parameters, their interactions and squared values were all significant within the 95% confidence limits tested. Also, results from the fit statistics verified the models' adequacy to predict the densification, porosity and rupture modulus of ceramic tiles produced. These findings are highly recommended for research and development of ceramic tiles of

Nigerian origin as a measure advancing their local and global market values.

Keywords: Ceramic tiles; response surface modelling; DOE; density; porosity; rupture modulus

I. INTRODUCTION

As urbanization increases due to rise in global population, the construction industry receives a fair share of rise in demand for construction materials. The ceramic industry has been enlisted as the highest consumers of natural raw materials especially in tile production (Vasic, et al., 2022). Globally, both the production and sales of ceramic tile are steadily increasing according to Ochen et al., (2019). Nigeria as a major consumer of ceramic tiles, still maintains its position as the 13th in ceramic importation rated over 10Billion USD, (Njoku, et al., 2022). With the huge abundance of raw materials that can place Nigeria as the first in global tile production yet overdependence on China, Spain and Brazil has contributed to the stagnant socioeconomic development of Nigerian tile market as well as the high cost of construction despite the huge commercial heritage of tile production raw materials in the country. Hence, development of the market value of Nigerian raw materials for tile production becomes an inevitable necessity. Ceramic tiles play vital roles in everyday life, (El-Maghraby, et al., 2021) and several studies have been carried out on the exploitation of local materials in floor tile production. Wastes and production byproducts to boost the structural performance of ceramic tiles has also been studied. For instance, Vasic, et al., (2022) used aplitic granite waste to augment the

production recipe for floor tile production with favourable outcomes obtained while Ologunwa, (2020), investigated the effect of particle size of raw materials as a major determinant of porcelain product formulation. Zanatta, et al., (2021), used a simplex-centroid experimental design for ternary mixtures to investigate the mapping of porcelain manufacturing byproducts into production of tiles and stoneware. Their laboratory results showed that the developed products satisfied the normative standards. However, the use of statistically designed experiments to evaluate the parametric impact on salient tile properties is presented for the first time.

The use of statistical design of experiments to investigate tile production is scarcely studied despite the method being an established and robust scientific technique with technical advantages over the conventional one-factor-at-a-time approach. Density, porosity and breaking strength (as a function of modulus of rupture) are three principal properties mostly considered in both tile production and commercial value of floor tiles. The lower the porosity and the higher degrees of density and modulus of rupture of ceramic floor tiles, the more their load carrying propensities. In this study, the investigation into factors that shape the density, porosity and rupture modulus of floor tiles is studied for the first time using statistical experimental design. These factors include: particle sizes of the production recipe, flux (Feldspar content) and sintering temperature.

II. EXPERIMENTAL PROCEDURE

2.1 Materials

Raw materials used in this study were all of Nigerian origin: Ball clay was sourced from Nsu in Imo state, Feldspar from Okene in Kogi state while Kaolin and Quartz were from PRODA institute and Iva Pottery respectively at Enugu state. The Ball clay, Quartz and Kaolin were in fixed ratio of 1:2.5:4 respectively while the variation of Feldspar content was a principal variable investigated in this study.

2.2 Methods

These raw materials were initially sun-dried to get rid of physically bound moisture, crushed and subsequently ground prior to sieving into different particle sizes for experimental investigation of the effect of particle size on the properties of the produced ceramic tiles. To remove associated organic matter, the powdered materials were dissolved in distilled water as shown in figure 1a. This organic matter-laden froth was filtered off, 2g of Sodium Silicate (Na_2SiO_3) added to inhibit flocculation of suspended solids and the slurry poured into a plaster of paris mold which absorbed the water to obtain a moldable plastic slip in figure 1b. The slip were kneaded to form the ceramic tiles in figure 1c ready for oven-drying and subsequent firing at different sintering temperatures.



Figure 1: (A) Raw materials soaked in water. (B) Slip for tile making, (C) Produced ceramic tiles

2.3 Design of experiment

To model the Modulus of Rupture, Porosity and Bulk Density of the produced tiles, three major independent variables were studied: effect of sintering temperature ($^{\circ}\text{C}$), effects of Feldspar content (%) and impact of particle sizes (μm) of the raw materials using Design Expert software (version 13, Stat-Ease Inc., Minneapolis, USA). A second order (quadratic) mathematical regression model predicting the individual and

interactive effects of these three independent variables was established as they relate to the response parameters. To reduce the complexities of empirical runs, the experiment was planned using Design of Experiment (DOE) in Central Composite Design (CCD) method of Response surface methodology (RSM). Each of the numeric factors were set to 5 levels: ± 1 axial points, ± 1 factorial points and the center point leading to 20 runs of 6

and 14 center points and non-center points respectively.

The design window was set at upper and lower limits using $\alpha = \pm 1.68179$ while the intermediaries were obtained using:

$$X_i = 2 [2X - (X_{max} + X_{min})] / (X_{max} + X_{min}) \dots\dots\dots(1)$$

Where X_i is the particular coded value needed for variable X while X is a variable between X_{max} and X_{min} . This was used to establish the design space shown in tables 1 for the experiment.

Table 1: Parametric Design Window

Process Parameters	Unit	Symbol	Levels ($\alpha = 1.68179$)				
			$-\alpha$	-1	0	1	α
Sintering Temperature	(°C)	T_s	1100	1200	1250	1300	1330
Feldspar Content	(%)	F_c	13	20	30	40	46
Particle Size	(μm)	P_s	50	75	150	300	375

2.4 Development of mathematical model

The response variables are related to the independent variables following the equation:

$$Y = f (X_1, X_2, \dots, X_n) \pm \epsilon \dots\dots\dots 2$$

where Y is the response variable (Modulus of Rupture, Porosity and Bulk Density) as functions (f) of the independent variables: x_1, x_2 up to x_n while n is the number of the independent variables. In this study, n=3 and x_1, x_2 and x_3 are the Sintering Temperature (T_s), Feldspar Content (F_c), and Particle Size (P_s) respectively. The function (f) is comparable to the response surface since it equitably linked to the set of independent variables. The Response Surface Methodology (RSM) in this study takes these independent variables as surfaces on which a mathematical regression model can be fitted to predict the response variable within the experimental design space. These fittings are within (ϵ) being the residual error representing the limits of experimental errors as expressed in equation 3:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii}^2 x_{ii}^2 + \sum_{i<j}^k \beta_{ij} x_i x_j + \epsilon \dots\dots\dots 3$$

Where Y is the response variable, x is nth independent variable for k number of variables, β_0 is the intercept which is equivalent to the mean value of the response, ϵ is the residual error while β_i, β_{ii} and β_{ij} are respectively the linear, quadratic and interaction coefficients.

III. RESULTS

3.1: Model formulation

Using the Central Composite Design (CCD) settings, the Experimental design matrix shown in Table 2 was developed. Twenty (20) experiments were carried out in the run order of this design matrix in uncoded independent variables generating the corresponding response variable results also displayed in table 2.

Table 2: Experimental design matrix and the response variable

Run No.	Independent Variables			Experimental Result		
	T_s (°C)	F_c (%)	P_s (μm)	Bulk Density (kg/cm^3)	Porosity (%)	MoR (MPa)
1	1250	13	150	2.59	15.1	30.92
2	1200	20	300	2.19	19.04	23.61
3	1250	30	375	2.24	12.29	20.82
4	1200	40	75	2.58	12.98	34.01
5	1300	40	75	3.02	14.81	38.54
6	1250	30	150	2.58	12.22	39.83
7	1300	20	300	2.32	16.41	25.62
8	1250	30	150	2.59	12.64	39.33
9	1330	30	150	2.8	13.01	41.14
10	1100	30	150	1.98	12.43	40.01
11	1250	30	50	2.79	12.91	38.62
12	1200	40	300	2.32	8.21	33.27
13	1200	20	75	2.55	11.32	28.8
14	1250	30	150	2.59	12.62	39.41

15	1250	46	150	2.81	10.24	38.59
16	1250	30	150	2.59	12.76	45.01
17	1250	30	150	2.58	12.63	32.01
18	1250	30	150	2.57	12.66	41.39
19	1300	20	75	2.82	12.1	30.8
20	1300	40	300	2.61	9.67	36.73

3.2 Model development and Verification of developed model adequacy

Using the experimental results in the design matrix, second-order polynomial (quadratic) models of the ceramic tiles' MoR, Porosity and Bulk density were developed in line with equation 3 generating equations 4, 5 and 6 respectively which can be used to make predictions accordingly.

$$\text{MoR} = 196.2844 - (0.2949 * \text{Ts}) + (0.1660 * \text{Fc}) + (0.0951 * \text{Ps}) + (0.0010 * \text{Ts*Fc}) + 5.5264 \times 10^{-6} * \text{Ts*Ps} + (0.0011 * \text{Fc*Ps}) + (0.0001 * \text{Ts}^2) - (0.0210 * \text{Fc}^2) - (0.0004 * \text{Ps}^2) \dots \dots \dots (4)$$

$$\text{Porosity} = -6.6465 + (0.0087 * \text{Ts}) - (0.3809 * \text{Fc}) + (0.2695 * \text{Ps}) + (0.0005 * \text{Ts*Fc}) - (0.0002 * \text{Ts*Ps}) - (0.0027 * \text{Fc*Ps}) \dots \dots \dots (5)$$

$$\text{Bulk Density} = -8.7989 + (0.0175 * \text{Ts}) - (0.1237 * \text{Fc}) + (0.0054 * \text{Ps}) + (8.2500 \times 10^{-5} * \text{Ts*Fc}) - (6.5050 \times 10^{-6} * \text{Ts*Ps}) + (2.1424 \times 10^{-5} * \text{Fc*Ps}) - (6.3595 \times 10^{-6} * \text{Ts}^2) + (0.0004 * \text{Fc}^2) + (1.0536 \times 10^{-6} * \text{Ps}^2)$$

.....(6)

The statistical significance of the goodness of fit of the prediction models (equations 4 to 6) were verified using ANOVA (Analysis of Variance) at a confidence interval of 95%. Tables 3, 5 and 7 show the ANOVA table for MoR, Porosity and Bulk density of the produced ceramic tiles. In addition, tables 4, 6 and 8 show the experimental Fit statistics of the MoR, Porosity and Bulk density results.

Table 3: ANOVA for Quadratic model of the Modulus of Rupture

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	677.02	9	75.22	5.61	0.0063*
A-Firing Temperature	13.23	1	13.23	0.9862	0.3441**
B-Feldspar Content	161.38	1	161.38	12.03	0.0060*
C-Particle Size	60.58	1	60.58	4.52	0.0595**
AB	1.98	1	1.98	0.1476	0.7089**
AC	0.0081	1	0.0081	0.0006	0.9809**
BC	12.23	1	12.23	0.9113	0.3623**
A²	3.98	1	3.98	0.2968	0.5978**
B²	58.98	1	58.98	4.40	0.0624**
C²	204.55	1	204.55	15.25	0.0029*
Residual	134.17	10	13.42		
Lack of Fit	43.99	5	8.80	0.4878	0.7752**
Pure Error	90.18	5	18.04		
Cor Total	811.18	19			

*Significant **Not Significant at 95% Confidence limit

Table 4: experimental Fit statistics

Std. Dev.	3.66	R ²	0.8346
Mean	34.92	Adjusted R²	0.6858
C.V. %	10.49	Predicted R²	0.3245
		Adeq Precision	8.1178

From table 3, the quadratic model's F-value of 5.61 and p-value of 0.0063 which was less than 0.05 demonstrates that the model is significant. Statistically, this shows that there is only a 0.63% chance that an F-value this extent could occur due to noise. Interestingly, an F-value of 0.4878 for the model's Lack of Fit showed that

the lack of fit of the model is not significant relative to the pure error. This shows that the model is adequate for predicting the ceramic tile's MoR. Furthermore, other significant model terms with p-values lower than 0.05 were Feldspar content and the squared value of the Particle size. These demonstrated that Feldspar content and Particle

size play significant roles optimizing the MoR of the produced tiles.

However, table 4 shows that the Predicted regression coefficient (R^2) of 0.3245 was not as close as the Adjusted R^2 of 0.6858 since their difference was above the statistical limit of 0.2. Meanwhile, a low coefficient-of-variation (CV) value of 10.49% was recorded indicating a very high degree of accuracy for determining the MoR of the ceramic tiles using a combination of these parameters. In addition, as the Adequate (Adeq) Precision calculates the S-N ratio (signal to noise ratio), the model's S-N ratio of 8.1178 is a satisfactory signal greater than the standard threshold of 4.00. These results are strong indications that the developed quadratic model is

adequate to navigate the experimental design space for estimating the MoR of the ceramic tiles.

Similarly, table 5 shows an F-value of 262.54 and p-value of 0.0001 showing the model's statistical significance for predicting the porosity of the ceramic tiles. A p-value of 0.3075 shows that the model's lack of fit is not significant compared to the pure error, hence, the model is significantly fit for predicting the ceramic tile's porosity. In addition, the firing temperature, Feldspar content, squared value of the Particle size distribution and their interactive effects were all statistically significant showing that these parameters and their interactions contribute to the porosity of the ceramic tiles.

Table 5: ANOVA for Quadratic model of the Porosity

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	128.61	6	21.44	262.54	< 0.0001*
A-Firing Temperature	0.5943	1	0.5943	7.28	0.0183*
B-Feldspar Content	64.00	1	64.00	783.88	< 0.0001*
C-Particle Size	0.2312	1	0.2312	2.83	0.1163**
AB	0.5725	1	0.5725	7.01	0.0201*
AC	6.18	1	6.18	75.73	< 0.0001*
BC	78.01	1	78.01	955.51	< 0.0001*
Residual	1.06	13	0.0816		
Lack of Fit	0.8857	8	0.1107	3.15	0.1109**
Pure Error	0.1757	5	0.0351		
Cor Total	129.67	19			

*Significant **Not Significant at 95% Confidence limit

Table 6: experimental Fit statistics

Std. Dev.	0.2857	R²	0.9918
Mean	12.65	Adjusted R²	0.9880
C.V. %	2.26	Predicted R²	0.9732
		Adeq Precision	74.5601

The fit statistics for porosity of the tiles in table 6 shows the Predicted R^2 of 0.9918 with closeness less than the statistical limit of 0.2 from the Adjusted R^2 of 0.9880 which demonstrates a strong correlation between the experimental data and predicted values. Table 6 also reports CV value and S-N ratio of 2.26% and 74.5601 respectively where all the results verify the model's adequacy to predict the porosity of the ceramic tiles.

Likewise, table 7 shows an F-value and p-value of 1108.47 and 0.0001 respectively showing the model's statistical significance for predicting the Bulk density of the ceramic tiles. Also, an F-value of 2.46 shows the non-significance of the model's lack of fit compared to the pure error, hence, the model is fit to predict the ceramic tile's

bulk density. Remarkably, the sintering Temperature, Feldspar Content, the Particle Size, their interactions and squared values were all significant showing that these parameters, their interactions and squared values contribute to the bulk density of the ceramic tiles. Similarly, the fit statistics in table 8 shows the Predicted R^2 of 0.9944 with closeness less than the statistical limit of 0.2 from the Adjusted R^2 of 0.9981 which demonstrates a strong correlation between the experimental data and predicted values. With Table 8 reporting CV value and S-N ratio of 0.4199% and 138.1385 respectively, these results jointly verify the model's adequacy to predict the ceramic tiles' bulk density.

Table 7: ANOVA for Quadratic model of the Bulk Density

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1.15	9	0.1277	1108.47	< 0.0001*
A-Ts	0.2629	1	0.2629	2282.72	< 0.0001*
B-Fc	0.0838	1	0.0838	727.54	< 0.0001*
C-Ps	0.4050	1	0.4050	3516.34	< 0.0001*
AB	0.0136	1	0.0136	118.18	< 0.0001*
AC	0.0112	1	0.0112	97.13	< 0.0001*
BC	0.0049	1	0.0049	42.15	< 0.0001*
A²	0.0125	1	0.0125	108.21	< 0.0001*
B²	0.0224	1	0.0224	194.38	< 0.0001*
C²	0.0013	1	0.0013	11.50	0.0069**
Residual	0.0012	10	0.0001		
Lack of Fit	0.0008	5	0.0002	2.46	0.1733**
Pure Error	0.0003	5	0.0001		
Cor Total	1.15	19			

*Significant **Not Significant at 95% Confidence limit

Table 8: experimental Fit statistics

Std. Dev.	0.0107	R²	0.9990
Mean	2.56	Adjusted R²	0.9981
C.V. %	0.4199	Predicted R²	0.9944
		Adeq Precision	138.1385

3.3. Residual analysis

To validate the models' adequacy using ANOVA, diagnostic analysis of the designed models' fitness for predicting the MoR, Porosity and Bulk Density of the ceramic tiles were carried out.

Figure 2a_i, a_{ii} and a_{iii}, show the Normal plots of the residuals of the MoR, Porosity and Bulk Density respectively showing the residuals and the distribution of their fitted values. These results indicate that the data are closely fitted around the regression line with a normal distribution similar to the results in Rahiman, et al., (2022). Furthermore, since the values of Lambda = 1 in the Box-Cox plots in figures 2b_i, b_{ii} and b_{iii}, for the MoR, Porosity and Bulk Density respectively, these agree with standard recommendation that no

power law transformation for the experimental data obtained was needful. As the optimum lambda line (blue) lies between the lower and upper C.I. lines (red), this means that the model is properly fitted to the experimental data presented as obtained in Tong, et al., (2022); Uchegbulam and Jack, (2023). More importantly, since the lower and upper C.I. limits accommodates the optimum lambda value of 1, this demonstrates a distribution normality hence no need for power transformation of the obtained data to obtain variance stability. Also, figure 2c_i, c_{ii} and c_{iii}, demonstrate linear correlations with minimal variation between the predicted and experimental data for the MoR, Porosity and Bulk Density respectively.

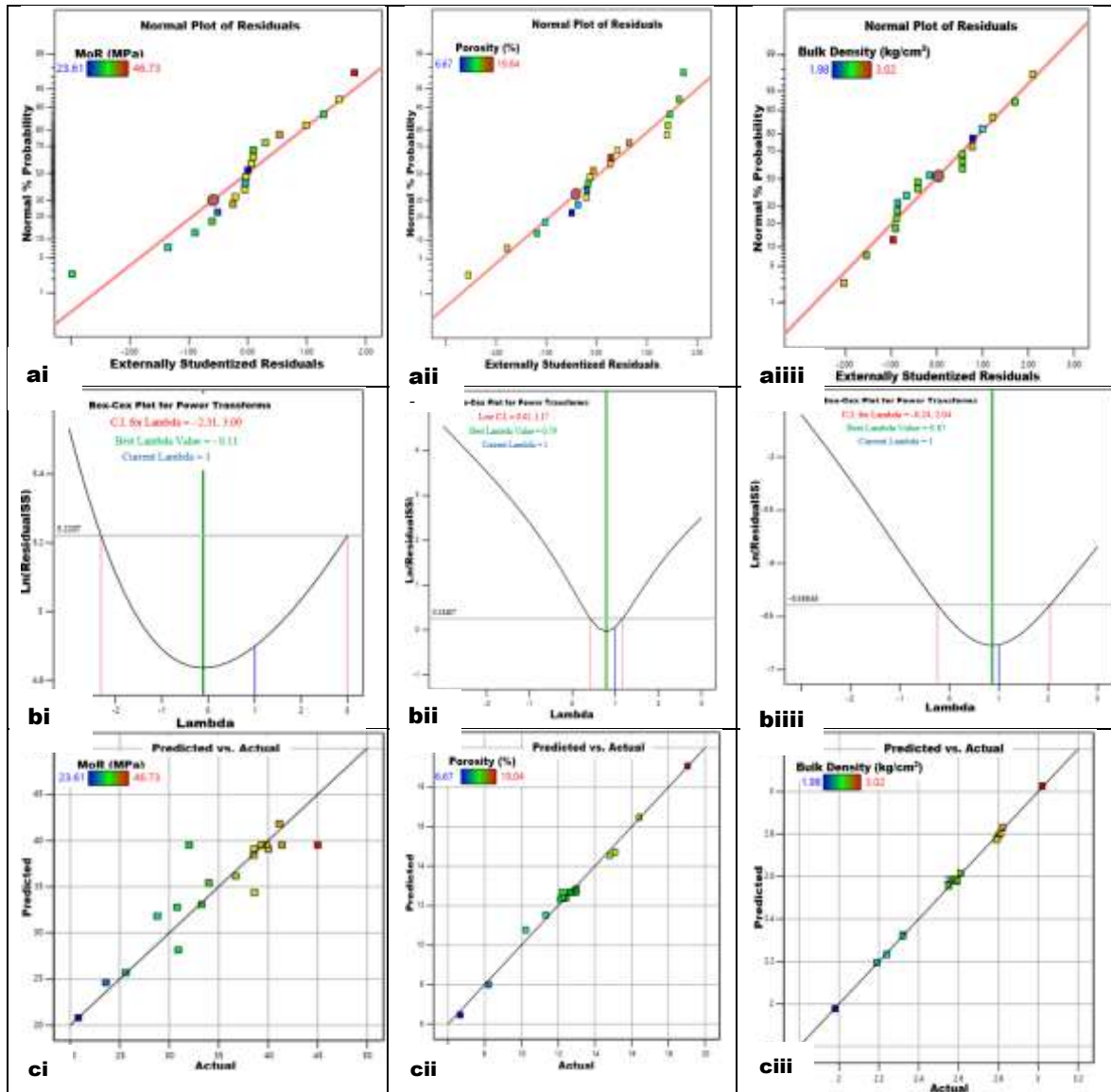


Figure 2: Residual plots (a) Normal probability plot of residuals (b) Box-cox Plot for power transformation (c) Plot of predicted versus actual data.

3.4 Influence of parameters

The parametric influences of Feldspar content, Particle size and Sintering Temperature as independent variables on the MoR, Porosity and Bulk Density as the response variables were

studied. To do these, one of the three factors was fixed at its central point, the other two were drawn on the x and z-axis while the response variables were plotted on the y-axis.

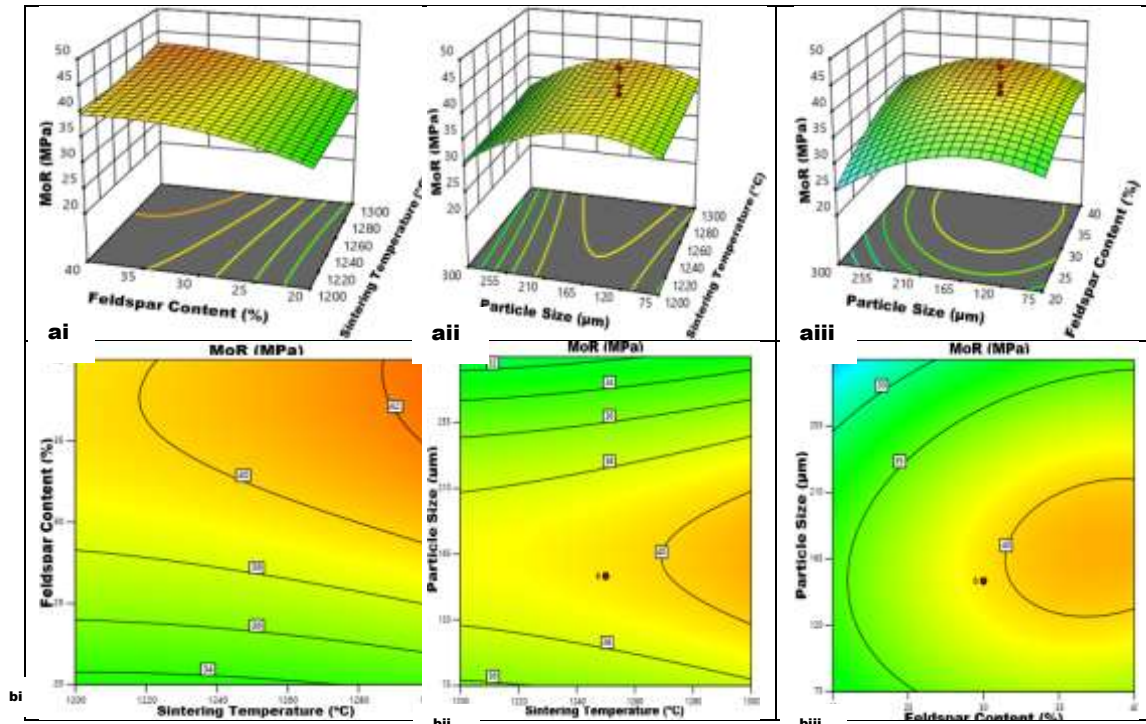


Figure 3: Contour and surface plots of effects of Feldspar content, Particle size and Firing Temperature effects on the MoR of ceramic tiles

To predict the MoR values of the ceramic tiles produced, the 3D surface plots and 2D contour plots in figure 3 were used to show the effects and interaction of two independent variables on the MoR at a time when the other independent variable was fixed at its center point.

It can be seen from figure 3ai that, at fixed center point of 187.5μm particle size, the MoR values of the tiles increased by 18.14% when Feldspar content increase from 20 to 40% at low temperature of 1200°C while the MoR rose by 3.02% when firing temperature increased from 1200 to 1300°C at low Feldspar content of 20%. This showed that Feldspar content is a principal factor in MoR of ceramic tiles with its statistical significance been validated in table3. Furthermore, in figure 3bi, the interactive effect of Feldspar content and Firing Temperature demonstrated a 26.98% rise in MoR. Similarly, by fixing the Feldspar content at the center point of 30% and increasing temperature from 1200 to 1300°C increased the MoR by 5.06%. Remarkably, decreasing the particle size increased the MoR values. This showed that finer particle size increases packing density which in turn increases

MoR as supported by Osonwa, et al., (2017). However, this particle size reduction from 300 to 75μm did not make a linear increase in MoR values of the ceramic tiles, instead, the MoR increased gradually as particle size was reduced from 300μm down to 150μm and declined as the particle sizes further reduced to 75μm. Consequently, the interactive effect of firing temperature and particle size made peak MoR value of 40.85MPa at 1300°C and 150μm.

Similarly, at a fixed point of firing temperature at 1250°C as seen in figure 3aiii, the MoR increased gradually by 37.24% as particle size was reduced from 300μm down to 146.05μm. After this, the MoR values declined by 28.2% as the particle sizes were further reduced to 75μm. Similarly, the MoR increased gradually by 15.73% as Feldspar content increases from 20% up to 35.68%. Then the MoR declined by 14.41% as the Feldspar content increases further increases to 40%. However, a convex interaction with elliptical contours between the particle size and Feldspar content in figure 3biii made peak MoR value of 40.87MPa at 172.80μm and 38.14% respectively.

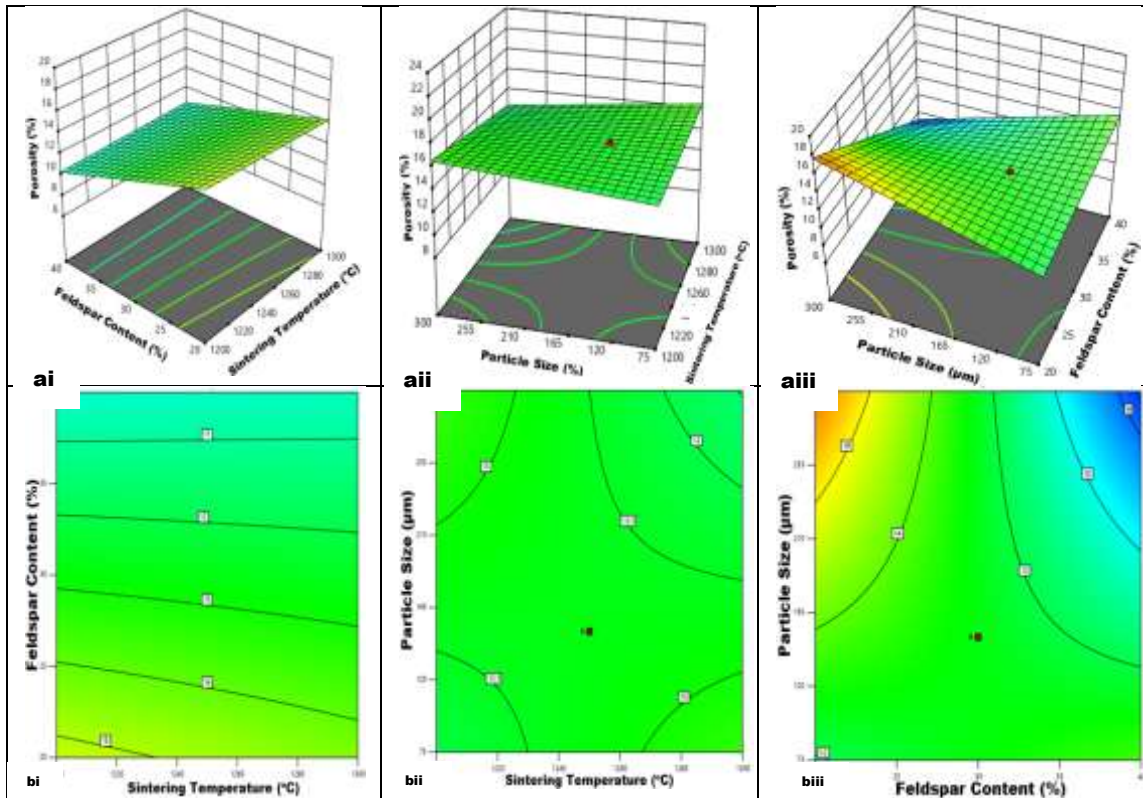


Figure 4: Contour and surface plots of the effects of Feldspar content, Particle size and Firing Temperature effects on the Porosity of ceramic tiles

With Particle size at fixed center point (187.5 μm), figure 4ai show that both Feldspar content and firing temperature as well as their interaction were linearly proportional to Tile Porosity. From figure 4ai, increasing temperature at low Feldspar content of 20% decreased the porosity by 5.89%. This can be attributed to the glass-forming mechanism at elevated temperatures reported in (El-Gamal, et al., 2021), which infiltrates open pores to further reduce the apparent porosity of tiles.

Similarly, at low firing temperature of 1200°C, increasing the Feldspar content reduced the porosity by 32.37% as this can be linked to the melting behaviour of Feldspar at low temperatures as a fluxing agent in line with (Kimambo, et al., 2014; Desouky, et al., 2020). This melts fill the existing open pores thus reduces the inter particle spacing leading to lower porosity. However, the

interactive effect of firing temperature and Feldspar content reduced the porosity by 31.26%.

Figure 4aii and 4aiii show that porosity reduced with decreasing particle size. This can be linked to the higher packing factor that occurs at reduced particle sizes and this enhances the tendency of finer particles to fit into micropores thereby reduce the tile Porosity even at low firing temperatures. It can be noticed that temperature rise increased porosity at low particle size due to bloating, a situation where entrapped gases expand as temperature increases and creates pores when they evaporate as reported in (Kimambo, et al., 2014). Also, porosity increased as Feldspar content increased at finer particle sizes in figure 4aiii. Likewise, the interactive effects of firing temperature and particle size as well as Feldspar content with particle size reduced the porosity by % and % respectively.

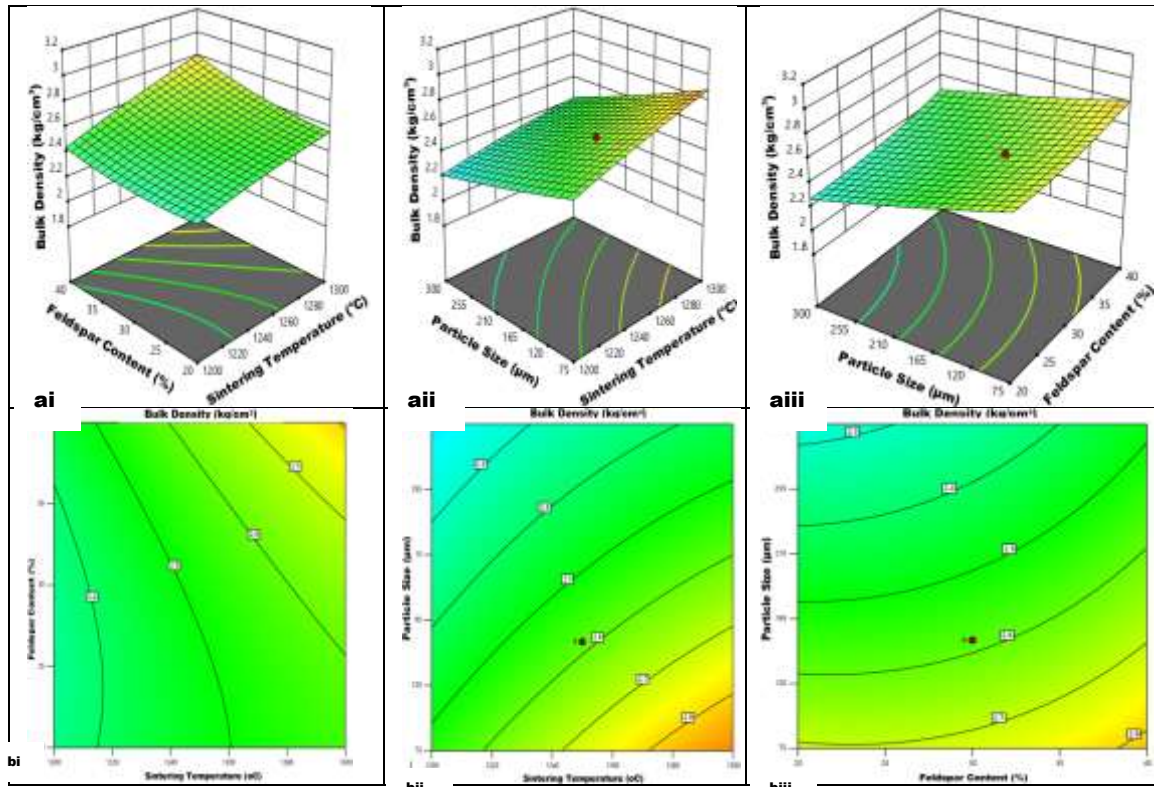


Figure 5: Contour and surface plots showing the effects of Feldspar content, particle size and Firing Temperature effects on the Bulk Density of ceramic tiles

Figure 5 displayed 2D contours and 3D response surface plots of Tiles' Bulk density presenting the interaction of firing temperature and Feldspar content. It is clear that Bulk density rose by 3.39% with increase in Feldspar content due to the viscous flow of melts formed by increasing Alkaline oxides in Feldspar which fills up voids and increases the mass by unit volume vis a viz increased bulk density. These results agree with previous studies, (Kimambo, et al., 2014; Xu, et al., 2015; El-Gamal, et al., 2021). Likewise, Bulk density increased by 8.47% due to rise in firing temperature as reported in (El-Maghraby, et al., 2021). With these rise in densification simultaneously through temperature and Feldspar content increments, an optimal Bulk density of 2.81kg/cm^3 can be noticed at the interaction of these two parameters at fixed particle size of $187.5\mu\text{m}$. Similarly, increasing the fineness of the raw materials by reducing the particle size also increased the bulk density due to increased packing factor. This was responsible for optimal bulk density increased by 14.23% at low particle size and high firing temperatures. Likewise, in figure 5 aiij, the interactive effect of decreasing particle size and increasing Feldspar content led to optimal Bulk densities up to 2.82kg/cm^3 . Hence, the three parameters studied: Increasing firing temperature,

Reducing particle size and Increasing Feldspar content made significant contributions to rise in bulk density in line with their statistical significance in table 3.

IV. CONCLUSION

In this study, a three-factor, five-level Central Composite Design based Response Surface Methodology was used to draw a 20-run experimental design space for modelling the density, porosity and rupture modulus of the ceramic tiles of Nigerian origin. By varying the particle sizes, Feldspar contents and sintering temperatures, ceramic tile samples were shaped and mechanical test results generated data for developing quadratic models using Design-Expert-13 statistical software. Based on Box-Cox plots recommendations from the residual analysis, no data transformation was carried out. Also, diagnostic analysis of the designed model demonstrated a normal distribution with the points closely fitted around the regression line. Using ANOVA, statistical analysis revealed a validation of the developed models' consistency with the experimental data. 2D Contour and 3D Surface plots revealed various interactive effects of the parameters on the MoR, Porosity and Bulk Density of the ceramic tiles produced. In addition, fit

statistics within the design space also validated the adequacy of the models in predicting the densification, porosity and rupture modulus of the produced ceramic tiles at 95% Confidence Interval. The findings from this study is recommendable for manufacturing ceramic tiles with predictable performance.

REFERENCES

- [1]. Desouky, O.A., Belal, E., El-Gamal, S.M.A., Abd-Allah, M.A. and Eliyan, T., (2020). Improving the insulating and physical characteristics of HV porcelain dielectric materials using nano silica. *Journal of Materials Science: Materials in Electronics*, **31**: 12649–12660 <https://doi.org/10.1007/s10854-020-03815-8>
- [2]. El-Gamal, S.M.A., Abd-Allah, M.A., Belal, E., Eliyan, T. and Desouky, O.A., (2021). Amelioration of The Dielectric Properties of Ceramic Insulators Using Nano-alumina. *International journal of Engineering Education*, **3**(1): 1-10. <http://dx.doi.org/10.14710/ijee.3.1>
- [3]. El-Maghraby, M.S., Ismail, A.I.M. & Shalaby, B.N.A., (2021). Utilization of some Egyptian Raw Materials in Ceramic Tiles. *Silicon*, **13**: 985–992. <https://doi.org/10.1007/s12633-020-00459-5>
- [4]. Kimambo, V., Philip, J.Y.N. and Lugwisha, E.H., (2014). Suitability of Tanzanian Kaolin, Quartz and Feldspar as Raw Materials for the Production of Porcelain Tiles. *International Journal of Science, Technology and Society*, **2**(6): 201-209. <https://doi.org/10.11648/j.ijsts.20140206.17>
- [5]. Njoku, R.E., Ocheri, C., Chukwuka, C., Nnamchi, P.S. and Ude, S.N., (2020). Physical and Mechanical Properties of Porcelain Tiles Manufactured Using Nrobo- Uzo Uwani Nigerian Clay and local Raw Materials. *Journal of Material Science and Metallurgy*, **1**(1): 1-8.
- [6]. Ochen, W., D’ujanga, F.M. and Oruru, B., (2019). Effect of Quartz Particle Size on Sintering Behavior and Flexural Strength of Porcelain Tiles Made from Raw Materials in Uganda. *Advances in Materials*, **8**(1): 33-40. <https://doi.org/10.11648/j.am.20190801>.
- [7]. Ologunwa, T.P., (2020). Process and Development of Electrical Porcelain Insulator Using Edo State, Nigerian Raw Materials. *International Journal of Engineering and Manufacturing*, **3**: 43-55. <https://doi.org/10.5815/ijem.2020.03.04>
- [8]. Osonwa, N.O., Nwabinele, E.O., Ekwueme, E., Ogbn, C.C. and Alegu, F.N.F., (2017). The Effect of Particle Size And Particle Size Distribution on The Modulus of Rupture of Some South East Nigeria Clays. *Journal of Sciences and Multidisciplinary Research*, **9**(3):51-60.
- [9]. Rahiman, M.K., Santhoshkumar, S., Mythili, S., Barkavi, G.E., Velmurugan, G. and Sundarakannan, R., (2022). Experimental analysis of friction stir welded of dissimilar aluminium 6061 and Titanium TC4 alloys using Response Surface Methodology (RSM). *Materials Today: Proceedings*, **66**: 1016–1022. <https://doi.org/10.1016/j.matpr.2022.04.822>
- [10]. Tong, Q., Yan, S., Wang, S. and Xue, J., (2022). Optimization of process technology and quality analysis of a new yogurt fortified with Morchella esculenta. *Food Science and Technology*, **42**(e45822). <https://doi.org/10.1590/fst.45822>
- [11]. Uchegbulam, I. and Jack, T.A., (2023). Statistical Modelling and Optimization of Fs-Welded 6061-T651 Aluminum Alloy, *Scientia Africana*, **21**(4).
- [12]. Vasic, M.V., Mijatovic, N. and Radojevic, Z., (2022). Aplitic Granite Waste as Raw Material for the Production of Outdoor Ceramic Floor Tiles. *Materials*, **15**(3145):1-17. <https://doi.org/10.3390/ma15093145>
- [13]. Xu, X., Lao, X., Wu, J., Zhang, Y., Xu, X. and Li, K., (2015). Microstructural evolution, phase transformation, and variations in physical properties of coal series kaolin powder compact during firing. *Applied Clay Science*, **115**: 76-86. <http://dx.doi.org/10.1016/j.clay.2015.07.031>
- [14]. Zanatta, T., Santa, R.A.A.B., Padoin, N., Soares, C. and Riella, H.G., (2021). Eco-friendly ceramic tiles: development based on technical and market demands. *Journal of Materials Research and Technology*, **11**: 121-134. <https://doi.org/10.1016/j.jmrt.2020.12.081>