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Development of standard formulations for porcelain production from Ohiya clay

Ibeh M. Imagwuike[a], Nwankwojike B. Nduka[a], Obi I. Anthony[a], Nwadinobi P. Chibundo*[b], and Lewachi A. Augustine[a]

[a]Department of Mechanical Engineering, Michael Okpara University of Agriculture, Umudike, Nigeria.
[b]Department of Mechanical Engineering, Abia State University, Uturu, Nigeria.

Abstract

Optimal designs for producing standard porcelains from Ohiya clay were developed in this study to scale up industrial utilization of this alumina-silicate dominated natural clay reserve. This involved experimental evaluation, modeling and optimization of the effects of firing temperature and varying Ohiya clay-feldspar-silica mix on the properties of porcelains produced from it using desirability optimization methodology. Apparent porosity, linear shrinkage, apparent density, water absorption rate, cold compressive strength and modulus of rupture constitutes the porcelains' properties evaluated. Results revealed 42:32:20, 42:33:17, 41:32:27 as standard formulation of the clay, feldspar and silica into low/high voltage insulators, tiles and tableware respectively while 1250/1300°C, 1000°C and 1300°C constitute their respective optimal firing temperatures. Six hundred and twenty-five naira (₦625), three hundred and twenty-two naira (₦322), three hundred and four seven (₦347) were used for producing an 11 kV electric insulator, 400×400 mm floor tile and serving dish based on these standard design respectively against their respective market prices of one thousand naira (₦1000), three hundred and fifty naira (₦350), four hundred and twenty naira (₦420) This implies 60%, 9% and 21% profit boost from the developed porcelain designs. Hence, adoption of the models/optimal formulations developed in study for porcelains' production from ohiya clay is recommended for proficient exploitation of this natural resource.

Keywords: Porcelain products; Performance parameters; Investors' interest; Ohiya clay; Standard formulation

1. Introduction

Clay is defined as earth or soil that is plastic and tenacious when moist and that becomes permanently hard when baked or fired [1]. It is also described as a fine-grained natural rock or soil material that combines one or more minerals with traces of metal oxides and organic matter [2-6]. According to a report by [7], clay is formed due to the decomposition of an igneous rock by some geologic hypogenic actions that result from the mixture of gases and vapor in the interior of the earth's crust over a very long period which must be about some million years ago. Thus, Clay consists of hydrous alumino-silicate minerals formed by the weathering of feldspathic rocks, such as granite [8].

Clays vary in plasticity, all being more or less malleable and capable of being molded into any form when moistened with water. They are used for making various kinds of pottery, tobacco pipes, bricks and tiles etc. The commoner varieties of clay and clay rocks are china clay, kaolin, pipe clay, potter's clay, sculptor's clay, brick clay and fireclay [9]. The application of clay-based ceramics to mankind is important [10]. Ceramic applications of clay materials include but not limited to bricks, fireclay refractories, earthenware, stoneware, porcelain, sanitary ware and tableware [11-13]. Apart from serving the immediate needs of man such as household utensils and decorative tableware [14], it is also applied in various other diverse ways to satisfy the demands of mankind.

In this research, ceramic applications of clay materials in industrial production of ceramic porcelain products were undertaken. These products include electric insulators, tableware and tiles.

For technical purposes, porcelain products are designated as electrical, chemical, mechanical, structural and thermal wares [15].

*Corresponding author. Email: chibundop@gmail.com

Porcelain is an inorganic compound made by heating a blend of Kaolin, Quartz and Feldspar, each of which reacts when subjected to appropriate heat temperature [16,10]. Kaolin serves as the plastic material while quartz (silica) and feldspar are the non-plastic materials. When fired, porcelain (made of a clayey body) becomes very hard, strong and translucent (when glazed) though unreactive and containing little or no iron impurities [10].

Porcelain insulators are used in electrical power transmission system due to their high electrical, mechanical and thermal properties [17]. These are the reasons for their continued use over the years despite the emergence of new materials like plastics and composites [18]. Clay-based porcelain insulators are the commonly used ceramic insulators for both low and high tension insulation and they are considered as one of the most complex ceramic materials [18]. For electrical insulation applications, porcelains are expected to meet minimum specifications values of 16 kV/mm and 69 MPa for dielectric strength and modulus of rupture, respectively [15].

Furthermore, tiles and tableware are also categorized as porcelain products. Tiles are used for interior and exterior decoration and they belong to a class of ceramics known as white wares. The raw materials used to form tile consist of clay minerals, natural minerals and chemical additives. These minerals and additives are often refined or beneficiated and they are required for the shaping process and used to lower the firing temperature. Ceramic tiles are consumed in bulk by their single largest end-user i.e. the construction industry and the constituent raw materials are classified by their particle size. For many ceramic products, including tile, the body composition is determined by the amount and type of raw materials. Therefore, it is important to mix the right amounts together to achieve the desired properties. Continuing, tableware is the dishes or dishware used for setting a table, serving food and dining. It includes cutlery, glassware, serving dishes and other useful items for practical as well as decorative purposes [19].

Despite the extensive research in the use of clay deposits from other parts of the country, it is clear that the study of Ohiya clay which is rich in kaolinite with a kaolin deposit bulk reserve of about 74.38 million metric tonnes [25] is undermined partly due to its location in the country or ignorance of its potentials. Efforts have been made in the use of this abundant clay in refractory applications and palm kernel bleaching [25-27] but other ceramic applications of these clay deposits have not been well explored. Even with its study for refractory applications, its optimal mix for various ceramic applications is yet to be established, thereby making it difficult for potential investors to access valid information about this abundant natural resource at Ohiya.

Hence, this study applied RSM –based desirability function analysis to establish mathematical models and optimal framework for composing Ohiya clay and additives into various ceramic porcelain products. Thus making its overall production cost to be low and thus attract investors to this sector.

2. Materials and methods

The clay sample was sourced from Ohiya clay deposit in Umuahia South LGA of Abia State, Nigeria. Preparation/production of test specimens for the analysis of Ohiya clay and ceramic porcelain products from it was done at Project Development Institute (PRODA) Enugu, Enugu State, Nigeria. The silica and feldspar used in this study were sourced from Ishiagu in Ebonyi State, Nigeria.

The clay sample obtained from Ohiya was analysed to determine its mineralogical and chemical composition using the X-ray diffraction (XRD) and X-ray fluorescence (XRF) machines. The XRD analysis was done at Nigeria Geological Laboratory Zaria, Kaduna State, Nigeria while the XRF analysis was done at Central laboratory Ahmadu Bello University, Zaria, Kaduna State, Nigeria. A sample of 200 g of the Ohiya clay was crushed and milled to a fine particle. The mineral phases within the sample were identified by powdered X-ray diffractometry method. The sample was first subjected to X-ray scanning using the Philips PW 1830 X-ray diffractometer with a Cu-anode. After the X-ray scanning of the sample, mineral peaks were identified using XPert High Score plus Software. The background and peak-positions were identified and based on the peak positions and intensities, a search-match routine was performed and mineralogical composition of the clay was determined.

Quantitative analysis of chemical composition of the sample was also done by X-ray Fluorescence Spectroscopy using a Magi X Pro XRF Spectrometer. For this purpose, 200 g of the powdered clay sample was mixed with Herzog organic binder. The organic binder contained cellulose and wax. The mixture was homogenized by milling. The homogenized sample was placed in an aluminium cup and hydraulically pressed into pellets. This was done to ensure sample integrity under the vacuum and a consistent surface to receive the X-rays.

2.1. Production of porcelain electric insulators and tableware

The lumps of Ohiya clay were crushed using a clay crushing mill, sun-dried, and granulated using a pan mill, and also sieved through 0.425 mm sieve. The porcelain body was formulated using the optimal mixing ratio for clay, feldspar and silica in wt % of 42:32:20 respectively, at a firing temperature of 1250 °C as shown in the desirability plot (Fig. 1). Porcelain insulator bodies (for the low and high voltage each respectively) were formulated in accordance with weight percentage obtained in desirability plot, and placed in the porcelain molds using a process known as slip casting. Square

Table 1: Response Surface Design Layout.

Experimental Runs		Coded values of factors			
Std Order	Run Order	X_1	X_2	X_2	X_4
27	1	0	0	0	0
5	2	-1	-1	1	-1
30	3	0	0	0	0
25	4	0	0	0	0
17	5	-2	0	0	0
19	6	0	-2	0	0
14	7	1	-1	1	1
1	8	-1	-1	-1	-1
3	9	-1	1	-1	-1
18	10	2	0	0	0
24	11	0	0	0	2
23	12	0	0	0	-2
29	13	0	0	0	0
13	14	-1	-1	1	1
2	15	1	-1	-1	-1
6	16	1	-1	1	-1
22	17	0	0	2	0
9	18	-1	-1	-1	1
20	19	0	2	0	0
21	20	0	0	-2	0
7	21	-1	1	1	-1
10	22	1	-1	-1	1
26	23	0	0	0	0
3	24	1	1	1	-1
12	25	1	1	-1	1
16	26	1	1	1	1
11	27	-1	1	-1	1
4	28	1	1	-1	-1
15	29	-1	1	1	1
28	30	0	0	0	0

mold of 80 mm × 80 mm × 80 mm. was produced with plaster of Paris (POP) and the setup was left for two days to air dry and this was followed by Bisque firing in a gas-fired kiln at the rate of 150 °C/hr before glaze and vitrification was applied to each sample. This process was followed by firing and thereafter experimental validation of the optimal settings of insulation resistance, polarization index and dielectric testing of the samples were carried out, [15]. Similar process was adopted for the production of the tableware. This was done at the optimal mixing ratio for clay, feldspar and silica in wt % is 41:32:27 at a firing temperature of 1000 °C.

2.2. Production of tile

The mixture of clay, feldspar and silica was made using the optimal weight percent obtained from desirability plot. The clay body of 8 kg was milled and mixed with 1litre of water to form a slurry. Excess water was removed using the hydraulic filter press followed by spray drying. The dry powder obtained after spray drying was placed into tile molds. The formulated body of the tile was pressed to the size and shape of the mold before drying. Heat was applied to the tiles by convection using hot gases from the furnace. After drying, the tiles entered a temporary storage facility known as the racking and deracking chamber. This chamber aids in removing excess moisture, it presents and stabilizes the quality of the product before glazing. The firing of the tiles was carried out in a furnace at the vitrification temperature of 1300°C as obtained in the desirability plot for experimental evaluation.

2.3. Modelling, optimization and performance test procedure

Evaluation, modelling and optimization of ceramic production process involves: Development of appropriate RSM experimental design, fitting and selection of the best response models, optimization of the selected models and verification/ confirmation of the modelling and optimization results. The choice of the experimental design was based on the study objectives, number of variables, resource availability and source of data collection. Effects of the operational parameters of the clay products on the performance parameters of Modulus of rapture (H), apparent density (D_A), cold compression strength (CCS) or the minimum values of the apparent porosity (P_a), water absorption rate (W) and linear shrinkage (L_s)in the plots were studied. Version 17 of the MINITAB was used to generate and randomize a two coded level (+1 and -1) factorial design layout in which "+1"and "-1" indicate the high and low level of the factors respectively with "0" as the midpoint of the factors. Thereafter, the actual high and low levels of the operational factors being investigated were determined from experimental tests.

A two-level full factorial composite design (2^k) was employed in the study and the response surface layout is given in Table 1. The choice of this factorial design layout is because of its economic viability, desirable properties, orthogonality and it permits marginally small experimental runs to be analyzed for high factorial points.

Minitab® Release 17 software with experimental study variable number (K = 4), for independent variables of quantity of clay (Q_c), quantity of feldspar (Q_f), temperature (T) and quantity of silica (Q_s) was used for the design and analysis of the results. The transformation equation 1 was used to effect the coding, where x is the independent variable in natural units; *x* is the coded variable while $x_{\max}$ and $x_{\min}$ are the maximum and minimum values of the independent variables respectively.

$$x = \frac{X - \left(\frac{X_{\max} + X_{\min}}{2}\right)}{\left(\frac{X_{\max} - X_{\min}}{2}\right)} \tag{1}$$

The response surface design layout generated (Table 1) was used to develop a linear model for the operational parameters. The main effects plots, model adequacy measures and residual diagnostic plots displayed by the software along with the fitted linear models was used to evaluate if the functions approximated the true responses adequately, after which non-linear models were found appropriate. The generated data allowed analysis of non-linear interactions of the initial linear designs and was used to develop non-linear models (quadratic) for the ceramics performance responses (Equation 2).

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j x_j + \sum \beta_{jj} x_j^2 + \sum_{j<} \sum_{j=2}^{k} \beta_{ij} x_i x_j \tag{2}$$

The quadratic models adequacy measures used for the statistical verification of the fitted functions include regression analysis of model coefficients, analysis of variance (ANOVA) and lack-of-fit tests. A good model must be significant (i.e. P-value < 0.05), lack-of-fit must be insignificant, various coefficients of determination, R^2 and adjusted R^2 values should be close to 1(100%) and Sum of Square Error (SSE) should be as small as possible.

The coefficient of determination, R^2 and adjusted coefficient of determination, $adj - R^2$ for each of the response models were determined to know how properly the models fit the measured data. The values of R^2 lies between zero and one $(i.e\ 0\% \leq R^2 \leq 100\%)$ and the more the value of R^2 approaches one (1), the better the estimated model fits the measured data.

Table 2: Chemical Composition of Ohiya clay.

S.N.	Element	Concentration (wt %)
1	Na_2O	0.280
2	Mg_2O	1.340
3	Al_2O_3	32.160
4	SiO_2	49.02
5	P_2O_5	0.319
6	SO_3	0.303
7	Cl	0.004
8	K_2O	1.970
9	CaO	0.220
10	TiO_2	0.798
11	Cr_2O_3	0.029
12	Mn_2O_3	0.007
13	Fe_2O_3	0.970
14	ZnO	0.005
15	SrO	0.025
16	H_2O	12.550

Furthermore, insignificant terms were eliminated from the models using stepwise model building approach. The confirmation trials were conducted using the same procedure as in the determination of the factor levels. The Modulus of rapture (H), apparent density (D_A), cold compression strength (CCS) or the minimum values of the apparent porosity (P_a), water absorption rate (W) and linear shrinkage (L_s)were predicted based on various factor combinations of confirmation experimental plan using point prediction capability of MINITAB. When the models have been fully validated, optimal settings of all responses and factors were determined.

In addition, the dielectric strength test for the insulator was done at Enugu Electricity Distribution Company (EEDC), Umuahia, Abia State, Nigeria according to [15]. An insulation tester with model number "Fluke 1550c/1555" and rated capacity of 5000 volts was used in determining the insulation resistance of the porcelain insulator samples. The live and earth props were connected to the respective ports in the tester and the tester was switched on. The other end of the props were connected to the insulator material. For the insulation resistance test, the timer was set to sixty seconds (60 seconds) and the injected voltage was varied from 250 volts to 5000 volts. Values of the insulation resistance at the end of the timer countdown for each injected voltage was digitally displayed by the instrument and recorded. The polarization index of the insulator was determined by setting the timer to 10 minutes and injected voltage kept constant at 5000 volts. Values of the resistance obtained at the end of every one minute were recorded and the polarization index was calculated using equation 3 [15].

$$Polarization\ index = \frac{Resistance\ at\ the\ 10th\ minute}{Resistance\ at\ the\ 1st\ minute} \tag{3}$$

3. Results and discussion

Table 2 shows that the alumina-silicate oxides content of the clay constitute about 81 % of the total chemical composition of the clay. There are also traces of other chemical oxides like manganese, chromium, zinc, potassium, phosphorus, sodium, magnesium, calcium, titanium and iron. Hence, Ohiya clay is rich in alumina-silicate and hence suitable for ceramic porcelain products applications.

Table 3 showed the actual low and high levels of the independent factors required for the empirical analysis of the ceramic properties/performance parameters of product mix. These are the limits used in variation of quantity of Ohiya clay, feldspar, tempera-

Table 3: Limits of the operational parameters.

S.N.	Factor Description	Coded Symbols	Factor Values	
			Low (-1)	High (+1)
1	Quantity of clay (wt %)	x_1	20	50
2	Quantity of feldspar (wt %)	x_2	10	25
3	Temperature (°C)	x_3	1000	1250
4	Quantity of silica (wt %)	x_4	10	40

ture and silica to ascertain how significantly they affect apparent porosity (%), linear shrinkage (%), apparent density (g/cm^3), water absorption rate (%), cold compression strength (MPa) and rupture modulus (kg/cm^2) of the ceramic products.

The actual low and high levels of the factors were determined from experiments required for the empirical analysis of the ceramic properties of Ohiya clay using the equations 4 to 7 to effect the coding and are tabulated in Table 3.

$$x_1 = \frac{Q_c - 35}{15} \tag{4}$$

$$x_2 = \frac{Q_f - 17.5}{7.5} \tag{5}$$

$$x_3 = \frac{T - 1125}{125} \tag{6}$$

$$x_4 = \frac{Q_s - 25}{15} \tag{7}$$

Where Q_c is quantity of clay (wt %); Q_f is quantity of feldspar (wt %); T is temperature (°C) and Q_s is quantity of silica (wt %).

A two level single replicate, single block completely randomized full central composite design with six center points and four factors shown in Table 4 is the design table comprising the natural factors and their responses.

The reduced empirical relationships between the factors and responses developed and analyzed using the MINITAB 17 are shown in equations 8 to 13 in natural units. The dependent variables are defined as modulus of rapture (H), apparent density (D_A), cold compression strength (CCS), the minimum values of the apparent porosity (P_a), water absorption rate (W) and linear shrinkage(L_s).

$$\begin{aligned} Pa \;=\;& 12.38 + 0.11\,Q_c - 5.51 \times 10^{-2} Q_f - 2.91 \times 10^{-3}\,T \\ & + 0.11\,Q_s + 1.28 \times 10^{-4} Q_c^2 - 3 \times 10^{-6}\,Q_c T \\ & - 4.05 \times 10^{-3}\,Q_c Q_s + 7.6 \times 10^{-5}\,T * Q_s \end{aligned} \tag{8}$$

$$\begin{aligned} DA \;=\;& 3.65 - 3.03 \times 10^{-2}\,Q_c - 8.89 \times 10^{-3} Q_f \\ & - 1.27 \times 10^{-3} T - 1.97\,Q_s \times 10^{-2} \\ & + 9.8 \times 10^{-5} Q_c^2 + 1.23 \times 10^{-4} Q_f^2 \\ & + 3.2 \times 10^{-5} Q_s^2 + 2.44 \times 10^{-4}\,Q_c Q_f \\ & + 4 \times 10^{-6}\,Q_c T + 2.71 \times 10^{-4}\,Q_c Q_s \\ & + 1.1 \times 10^{-5}\,Q_f T - 2.79 \times 10^{-4}\,Q_f Q_s \\ & + 1.8 \times 10^{-5}\,T Q_s \end{aligned} \tag{9}$$

$$\begin{aligned} W \;=\;& 11.06 - 0.13\,Q_c + 1.77 \times 10^{-2}\,Q_f \\ & - 3.97 \times 10^{-3}\,T + 5.93 \times 10^{-2}\,Q_s \\ & + 1.543 \times 10^{-3} Q_c^2 - 1.28 \times 10^{-3} Q_f^2 \\ & + 2 \times 10^{-6} T^2 - 3.7 \times 10^{-5} Q_s^2 \\ & + 1.228 \times 10^{-3}\,Q_c Q_f + 9 \times 10^{-6} Q_c T \\ & - 1.890 \times 10^{-3}\,Q_c Q_s - 1.9 \times 10^{-5}\,Q_f T \\ & + 6.94 \times 10^{-4}\,Q_f Q_s - 1.3 \times 10^{-5} T Q_s \end{aligned} \tag{10}$$

$$\begin{aligned} Ls \;=\;& -5.1 + 0.21\,Q_c - 1.64 \times 10^{-2}\,Q_f + 6.27 \times 10^{-3}\,T \\ & + 2.6 \times 10^{-4}\,Q_s + 6.67 \times 10^{-4}\,Q_c Q_s \\ & - 1.5 \times 10^{-5} Q_f T - 8 \times 10^{-6}\,T Q_s \end{aligned} \tag{11}$$

$$\begin{aligned} H \;=\;& 28.72 - 0.2\,Q_c + 0.22\,Q_f + 1.41 \times 10^{-2}\,T \\ & - 3.26 \times 10^{-2} Q_s + 1 \times 10^{-6} T^2 \\ & - 1.75 \times 10^{-4}\,Q_f T + 5.17 \times 10^{-3}\,Q_f Q_s \\ & - 1.02 \times 10^{-4}\,T Q_s \end{aligned} \tag{12}$$

$$\begin{aligned} CCS \;=\;& -19.53 + 0.71\,Q_c + 0.99\,Q_f + 1.51 \times 10^{-3} T \\ & + 0.14\,Q_s - 1.14 \times 10^{-2} Q_c^2 \\ & - 1.14 \times 10^{-2} Q_f^2 - 3 \times 10^{-6} T^2 \\ & - 6.165 \times 10^{-3} Q_s^2 + 2.24 \times 10^{-4}\,Q_c T \\ & + 8.54 \times 10^{-3} Q_c Q_s - 2.15 \times 10^{-4}\,Q_f T \\ & + 8.1 \times 10^{-5}\,T Q_s \end{aligned} \tag{13}$$

3.1. Model validation/confirmatory test result

The coefficient of determination and error standard deviation of the quadratic models are shown in Table 5. The results thereby implied that the developed models are adequate to statistically fit the data for the ceramic responses.

The models confirmatory test shown in Fig. 1 indicates that the fitted functions are good fits for the ceramic responses and can be used for optimization of the clay constituents with the predicted values having a percentage error of plus and minus five percent($\pm$5%).

3.2. Development of Optimal Formulations for Producing Standard Porcelain Products

Production of porcelain insulator using the optimal formulations obtained from the optimality test (Fig. 2) was done. Inspection of the desirability plot (Fig. 2) indicates that the established empirical models [20] were able to predict factor settings required to obtain the responses at composite desirability of 93.6 % with individual desirabilities of each response as 99 %, 97 %, 99 % and 80 % for compressive strength, modulus of rupture, linear shrinkage and apparent porosity respectively. The values of individual desirability and the composite desirability respectively approximate to 1 which signifies that the optimization result is highly desirable. Therefore, it is seen that the insulators produced from Ohiya clay performed optimally at the factor settings of 0.4242, 2.0, 0.4646 and -0.3384 for quantity of clay, feldspar, temperature and silica respectively. The optimal mixing ratio for clay, feldspar and silica in wt % is 42:32:20 at a firing temperature of 1250 °C as shown in the desirability plot.

Table 4: RSM Experimental Result showing Natural Values and Responses.

Experimental Runs		Natural Values of Factors				Responses						
Std Order	Run Order	Q_c	Q_f	T	Q_s	P_a	D_A	W	L_s	H	CSS	R
27	1	40	25	850	30	13.47	2.15	6.7	8.65	35.7	21	1500
5	2	30	15	1300	15	12.44	2.01	7	8.84	42	10.2	1560
30	3	40	25	850	30	13.4	2.15	6.69	8.53	35.5	21	1500
25	4	40	25	850	30	13.4	2.16	6.7	8.5	35.6	20.5	1492
17	5	20	25	850	30	13.5	2.28	7.83	4	40	11.5	1370
19	6	40	5	850	30	14.6	2.04	6.03	9.1	31.7	12.5	1530
14	7	50	15	1300	45	13.7	2.29	5.51	14	36.5	23.5	1470
1	8	30	15	400	15	14.1	2.36	7.82	3.66	31	9.5	1570
3	9	30	35	400	15	13	2.45	7.72	3.2	35.1	17.6	1550
18	10	60	25	850	30	13.5	2.1	6.77	13	32	21.5	1480
24	11	40	25	850	30	13.7	2.23	6.27	9.1	35.6	21	1272
23	12	40	25	850	0	13.2	2.13	7.03	8	36	10	1697
29	13	40	25	850	30	13.46	2.15	6.7	8.5	35.7	21.3	1490
13	14	30	15	1300	45	14.92	2.31	6.79	9.5	40	14	1345
2	15	50	15	400	15	15.35	2.1	7.57	8.14	27.5	10	1623
6	16	50	15	1300	15	13.6	1.83	6.86	13.5	38	14	1630
22	17	40	25	1750	30	12.8	2.34	7.07	13.7	45	20	1523
9	18	30	15	400	45	14.5	2.16	8.02	4.2	31	11	1365
20	19	40	45	850	30	12.3	2.36	6.31	8	40.5	20.5	1427
21	20	40	25		30	14.13	2.27	9.38	3.35	27	16	1487
7	21	30	35	1300	15	11.34	2.31	6.5	8.3	44	13	1580
10	22	50	15	400	45	13.35	2.06	6.61	9	27	18	1457
26	23	40	25	850	30	13.47	2.15	6.65	8.7	35.8	21	1492
8	24	50	35	1300	15	12.58	2.22	6.85	12.7	40	18	1595
12	25	50	35	400	45	12.25	2.09	7.335	8.84	35	23	1326
16	26	50	35	1300	45	12.6	2.51	6	13.4	40.2	26	1375
11	27	30	35	400	45	13.47	2.09	8.31	3.5	39.5	19	1290
4	28	50	35	400	15	14.27	2.29	7.93	7.6	31.2	17	1540
15	29	30	35	1300	45	13.82	2.43	6.7	8.5	44	17.3	1320
28	30	40	25	850	30	13.46	2.15	6.7	8.53	36	20	1490

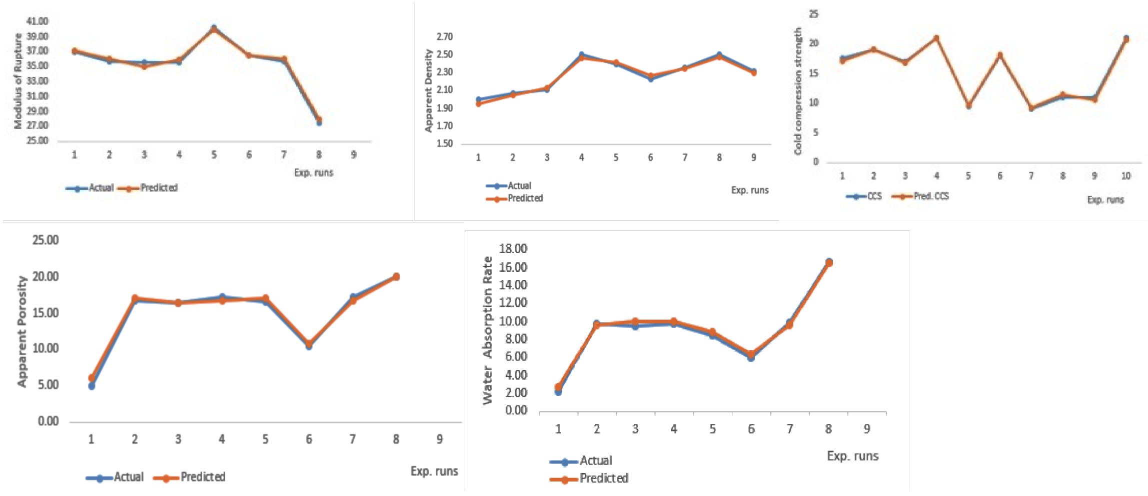

Figure 1: Model confirmatory test of response parameters.

Table 5: Coefficients of determination and error standard deviation for the quadratic models.

Responses	S	R-sq	R-sq(adj)
P_a	0.0293	99.88	99.83
D_A	0.0037	99.97	99.93
H	0.5027	99.20	98.94
W	0.0245	99.95	99.90
CCS	0.6768	98.76	97.89
L_s	0.1224	99.89	99.85

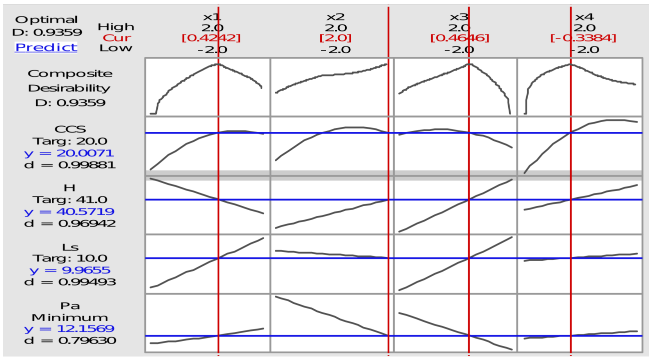

Figure 2: Optimization plot of the insulator parameters.

The optimal mixing ratio for Ohiya clay, feldspar and silica as predicted above was used in the clayey body formulations for low and high voltage insulators shown in Fig. 3.

Experimental validation of the performance of the porcelain insulator shown in Fig. 4, 5 and 6 respectively indicates that the porcelain insulator had a maximum breakdown voltage of 26 kV/mm when fired at 1300 °C (Fig. 4), insulation resistance of 6.96 GΩ when injected 5000 volts (Fig. 5) and a polarization index of 1.46 (Fig. 6). However for the low voltage insulator, temperature was obtained as 1250 °C and the breakdown voltage (dielectric strength) at this temperature is 17 kV/mm.

Corroborating this result, [21] reported that porcelain insulators produced at temperatures exceeding 1300 °C experienced a drastic reduction in their dielectric strength. Hence, Ohiya clay deposits together with its additives offered good prospects for their exploitation in the production of high voltage and low voltage electric porcelain insulators using the optimal mix ratio, since the values of the breakdown voltage satisfies the required standards as recommended by [22,17,23].

Table 6 shows the experimental validation of electrical insulator from Ohiya clay formulated at optimal mix ratio.

Furthermore, inspection of the desirability plot for tiles produced from Ohiya Clay (Fig. 7) indicates that the established empirical models [20] were able to predict factor settings required to obtain the responses at composite desirability of 96% with individual desirability of each response as 99 %, 100 %, 87 % and 100 % for modulus of rupture, linear shrinkage, water absorption rate and apparent porosity respectively.

The values of individual desirability and the composite desirability respectively approximate to 1 which signifies that the optimization result is highly desirable. Therefore, it is seen that the tile produced from Ohiya clay performed optimally at the factor settings of 0.47, 2.0, 1.33 and -0.52 for quantity of clay, feldspar, temperature and silica respectively. The optimal mixing ratio for clay, feldspar and silica in wt % is 42:33:17 at a firing temperature of 1300 °C as shown in the desirability plot. The optimal mixing ratio for Ohiya clay, feldspar and silica as predicted above and the addition of diatomite and talc (7 wt. %) was used in the clayey body formulations for tiles shown in Fig. 8. The experimental validation of tile sample carried out using tile specimens formulated at the optimal mix ratio is shown in Table 7. The percentage error for all the responses studied is within ± 5 %, hence the optimality test is validated.

As shown in Table 8, the tile produced can be used as porcelain tile, wall tile, floor tile and exterior tiles since it meets the required properties in terms of modulus of rupture, water absorption rate, porosity and linear shrinkage.

It is also observed that the optimum temperature had a beneficial effect on the development of certain technological properties such as its contribution to the increased mechanical strength since decreasing porosity also reduced crack formation. This formulation gave high variety of tile application as against the formulation of [24] that obtained responses suitable for floor tile only.

In addition, the tableware performance parameters optimized using the desirability plot (Fig. 9) indicates that the established empirical models [20] were able to predict factor settings required to obtain the responses at composite desirability of 93% with individual desirability of each response as 99%, 83%, 91% 90% and 99% for compressive strength, modulus of rupture, linear shrinkage, water absorption rate and apparent density respectively.

The values of individual desirability and the composite desirability respectively approximate to 1 which signifies that the optimization result is highly desirable. Therefore, it is seen that the tableware produced from Ohiya clay performed optimally at the factor settings of 0.46, 2.0, -0.91 and 0.15 for quantity of clay, feldspar, temperature and silica respectively.

In natural units the optimal mixing ratio for clay, feldspar and silica in wt % is 41:32:27 at a firing temperature of 1000 °C as shown in the desirability plot. The optimal mix ratio was used in the clayey body formulations for dishes fired at 1000 °C as shown in Fig. 10.

The experimental validation of tableware sample carried out using tableware specimens formulated at the optimal mix ratio is shown in Table 9.

The costs incurred in the production of the ceramic products from Ohiya clay is shown in Table 10. From the cost analysis presented in Table 10, the sum required to produce 11 kV electric insulator, 400×400 mm tile and serving dish are respectively six hundred and twenty-five naira (₦625), three hundred and twenty-two naira (₦322) and three hundred and four seven (₦347) only. However, the market price of 11 kV electric pot insulator, 400×400 mm tile and serving dish are respectively one thousand naira (₦1000), three hundred and fifty naira (₦350) and four hundred and twenty naira (₦420) only.

Hence, the production of electrical insulator, tiles and tableware at the optimal settings can save about three hundred and seventy five naira (₦375), twenty eight naira (₦28) and seventy three naira (₦73) respectively which translates to 16%, 60%, 9%, 21% and 69% in profit for these products.

4. Conclusion

This study revealed optimal formulations and firing temperatures for producing standard low/high voltage insulators, tiles and tableware from ohiya clay-feldspar-silica mix as 42:32:20 at 1250/1300°C, 42:33:17 at 1000°C and 41:32:27 at 1300°C respectively. Application of these designs in the production of 11 kV electric insulator, 400×400 mm floor tile and serving dish yielded 60%, 9% and 21% profit boost. Thus, production of standard porcelains from ohiya clay based on the models/designs developed in this work is highly recommended to attract investment for scaling up industrial utilization of an alumina-silicate dominated natural clay reserve at Ohiya in Abia State of Nigeria.

Figure 3: High and low Voltage Insulators.

Table 6: Experimental Validation/Comparative analysis of Porcelain electrical insulator from Ohiya Clay.

S.N.	Responses	Level		% Error	NIS/ISO Specifications
		Predicted	Actual		
1	Modulus of Rupture (kg/cm^2)	40.00	41.00	1.05	≥20 kg/cm^2
2	Apparent Porosity (%)	12.20	12.72	4.30	≤15 %
3	Compressive Strength (MPa)	20.00	21.00	4.70	≥10 MPa
4	Linear Shrinkage (%)	9.96	10.10	0.40	7-12 %
5	Insulation Resistance (GΩ)	-	6.96	-	≥5 GΩ
6	Dielectric Strength (kV/mm)(Low Voltage)	-	17.00	-	10-20 kV/mm
7	Dielectric Strength (kV/mm)(High Voltage)	-	26.00	-	21-30 kV/mm
8	Polarization Index	-	1.46	-	-

Table 7: Experimental Validation/Comparative analysis of Tiles from Ohiya Clay.

S.N.	Responses	Level		% Error	NIS/ISO Specifications
		Predicted	Actual		
1	Modulus of Rupture (kg/cm^2)	44.75	45.33	1.20	≥12 kg/cm^2
2	Linear Shrinkage (%)	10.0	9.93	0.70	7-10 %
3	Water Absorption rate (%)	6.0	5.73	4.70	1-10 %
4	Apparent Porosity (%)	11.34	11.70	3.10	1-15 %

Table 8: Ohiya clay tile Applications.

S.N.	Tile Applications	NIS/ISO Specifications	Remarks
1	Porcelain	Low Porosity (≤12 %) High modulus of rupture(≥30 kg/cm^2) Water absorption rate (≤10 %)	Suitable
2	Interior		
	a. Wall tile	MOR ≥12 kg/cm^2 Water absorption rate (≤10 %)	Suitable
	b. Floor tile	MOR ≥30 kg/cm^2 Low porosity	Suitable
3	Exterior tile	MOR ≥40 kg/cm^2 Low absorption rate	Suitable

Table 9: Experimental Validation/Comparative analysis of tableware (serving dish) from Ohiya Clay.

S/N	Responses	Level		% Error	NIS/ISO Specifications
		Predicted	Actual		
1	Modulus of Rupture (kg/cm^2)	41.85	42.01	0.35	≥21 kg/cm^2
2	Apparent density (g/cm^3)	2.00	2.05	2.40	2.0-2.5 g/cm^2
3	Compressive Strength (mPa)	21.97	21.7	1.24	≥15 mPa
4	Linear Shrinkage (%)	7.64	7.50	1.80	≤10 %
5	Water absorption rate (%)	7.23	7.20	0.41	≤10 %

Table 10: Cost Analysis of Optimal Ceramic Products from Ohiya Clay.

S.N.	Description	Unit Price (₦/kg)	Electrical Insulator		Tiles		Tableware (Serving Dish)	
			Quantity (kg)	Amount (₦)	Quantity (kg)	Amount (₦)	Quantity (kg)	Amount (₦)
1	Ohiya Clay	15	1.26	15	0.76	11	1.07	16
2	Feldspar	100	0.96	96	0.59	59	0.83	83
3	Silica	100	0.6	60	0.3	30	0.7	70
4	Pyrophylite	300	0.18	54	-	-	0.16	48
5	Sawdust	50	-	-	-	-	-	-
6	Talc	300	-	-	0.14	42	-	-
7	Labour Cost	-	-	300	-	80	-	80
8	Miscellaneous	-	-	100	-	100	-	50
	Total			625		322		347

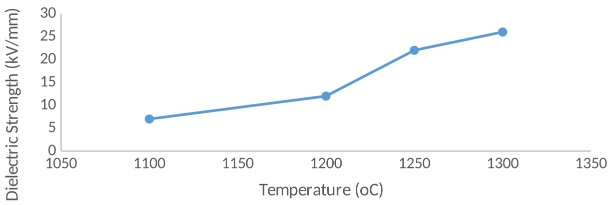

Figure 4: Dielectric Strength Test of the Porcelain Insulators.

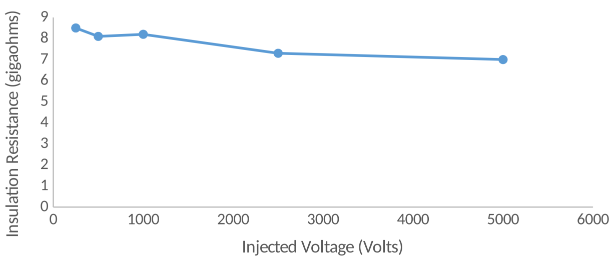

Figure 5: Insulation Resistance Test of the Porcelain Insulators.

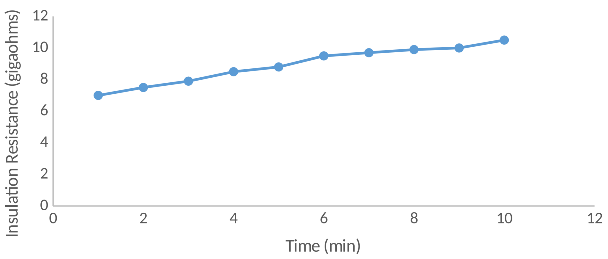

Figure 6: Polarization Index Test.

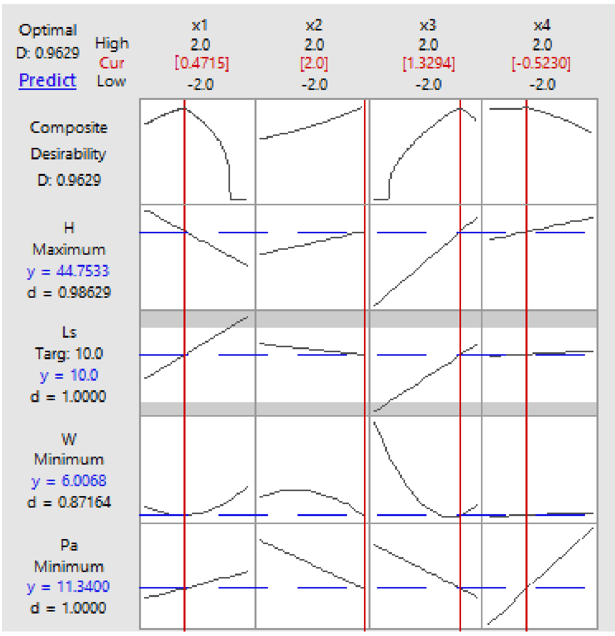

Figure 7: Optimization plot of the tile parameters.

Figure 8: Tiles from Ohiya Clay.

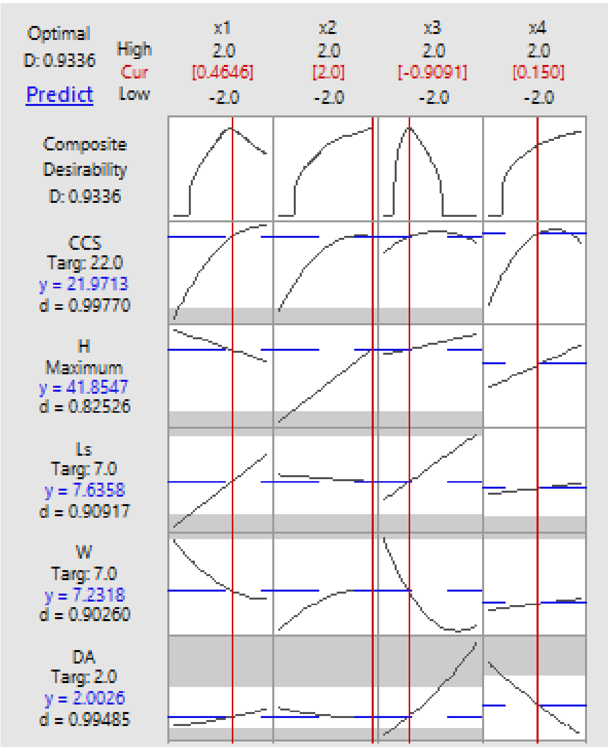

Figure 9: Optimization plot of the tableware parameters.

Figure 10: Serving dishes from Ohiya Clay.

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