

Volume: 02 No: 01 | March -2018

ISSN (Online) 2636 – 590 ISSN (Print) 2636 - 591X

DETERMINATION OF OPTIMAL RATIOS OF CHEMICAL COMPOSITION OF CLAY AND ADDITIVES FOR REFRACTORY BRICKS PRODUCTION USING MATHEMATICAL PROGRAMMING TECHNIQUES

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Abstract - The effects of varying ratios of the chemical components of our locally available clays with respect to their applications in furnaces were investigated in this study. Desirability function analysis based on response surface methodology was adopted in analyzing and optimizing the refractoriness (R_e) and linear shrinkage (L_s) of locally made refractory bricks with the oxides of silicon (SiO₂), aluminium (Al₂O₃), iron (Fe₂O₃), magnesium (MgO) and calcium (CaO) as the independent factors. The outcome of this investigation indicated that the oxides of aluminium and iron strongly affected the refractoriness- the former having a direct relationship and the latter, an inverse relationship. These same oxides also showed strong effects on the linear shrinkage; Al₂O₃ indicating an inverse relationship where as Fe₂O₃ showed a direct relationship. An optimum factor setting for the oxides was predicted at 60%, 33%, 2%, 1.5% and 2.1% respectively, with corresponding responses of 1720°C and 3.4% for the refractoriness and linear shrinkage.

Key words: Clay, refractoriness, linear shrinkage, furnace, optimization, model

1.0 INTRODUCTION

The refractories need of Nigeriaan industrially developing nation- is potentially enormous. The Ajaokuta Steel Company and the Delta Steel Company were estimated to, at full capacity; require 43,503 and 25,000 tonnes per annum respectively of fireclay refractories for their various activities; and these products were sourced from abroad (Adondua, 1988). Recently, small and medium scale industries (Onyemaobi, 2002) in Nnewi and elsewhere in the country are now fabricating spare parts using high temperature furnaces (foundry melting furnaces and heat treatment furnaces) that require refractory linings. In his work, he also observed that most of the refractories consumed locally are imported irrespective of the many clay deposits that could be used for

refractories. In 1987, 27 million metric tonnes of refractories were imported (Obadinma, 2003). A lot of clay deposits abound in the country, yet a lot of foreign exchange is expended in importing refractories.

Previous works on Nigerian clay deposits shows many of them to be high in silica and low in alumina content (Hassan, 2001; NMDC, 1991). Besides, a number of deposits have been found suitable for use as raw materials for refractories if properly processed. Hence, the development of our locally available materials for refractory production to meet our industrial and technological requirements is not only justified but imperative. This study is a contribution in that regard.

The estimation of the refractoriness and linear shrinkage of our locally available clay deposits

from their mineralogical content is the focus of this present study.

Refractories are indispensable materials in high temperature applications such as metal extraction/refining, metallurgical heat treatments, glass and ceramic manufacturing, melting practices and foundry power generation (Mark, 2010). The refractoriness of clay samples from certain locations of Nigeria have been established either by individuals or by corporate bodies for the purpose of knowledge gathering and for use in different areas/applications. Clay samples were gathered from different parts of the country and chemical analysis was conducted on them in order to determine their compositions of the oxides of silicon, aluminium, iron, magnesium and calcium. This was followed by the steps described in the flow chart for moulding test pieces which in turn were subjected to refractoriness and linear shrinkage tests and results obtained were used in this research.

Optimization of a single response system is usually simple, but in most practical industrial researches/applications, multiple outputs that are interrelated in a way that improving one will cause deterioration of another (e.g. rate versus consistency; strength versus expense) is always the case. Also, finding settings which will increase yield and decrease the amount of scrap/rework and energy consumption represent opportunities for substantial financial gain in industries. RSM is one of the most useful and outstanding tool for this type of investigation and this is why the method is of great application in industrial researches (Schmidt and Launsby, 1991).

Noordin *et al.*, (2004) used RSM to fit mathematical models of 97.2% prediction on the effects of feed rate, cutting speed and cutting edge angle on the surface roughness and tangential force when turning AISI 1045 steel. Capobianco *et al.*, (1990) equally developed a response surface model (with 96.77% prediction) that functionally relates sensitivity of an airplane wing crack detector to number of turns, winding distance and wire gauge and also predicted the settings of these

variables that maximized the sensitivity from the model. Also, RSM was also used in NIST Physics Laboratory to determine the best settings of seven factors that maximized luminescent light intensity (NIST/SEMATECH, 2006). Furthermore, Taieb et al., (2010) optimized mechanical clothing tactile comfort (thickness and fabric weight) to 98% success using RSM. Again, Funda, (2009) applied RSM to develop a 96.82% prediction equation for analysis of the relationship between the cutting speed, feed rate, depth of cut and surface roughness of turning process of AISI 4140 steel, whereas Isaac and Nwankwojike, (2016) applied desirability function approach to determine the optimal parameters of a centrifugal pump for heavy end recovery. Similarly, Osi et al., (2017) determined the optimal mix for acid reclamation of used engine oil using the desirability function approach while Oti et al., (2017) carried out a desirability function optimization of garrification process machine.

2.0 MATERIALS AND METHODS 2.1 Chemical Analysis

The analysis carried out in this work has to do with the determination of some of the physical necessary chemical and parameters/properties that are necessary for the application of these clay samples in furnace lining. Representative samples of the clays were analyzed to determine their chemical composition (% of SiO₂, Al₂O₃, etc.). The chemical analyses were done/ conducted at the Soil Science Laboratory of the National Root Crops Research Institute (NRCRI), Umudike, Abia State.

The chemical analysis was done using the Flame Photometer, Atomic Absorption Spectrophotometer (AAS), Murfle-furnance, Gallenkamp electric oven, Digestion Block, and Fume cupboard.

2.2 Processing Of the Clay

The steps involved in processing the clay after sourcing them involve the following procedures:



2.3 Data Analysis Procedure

Evaluation, modelling and optimization of the refractoriness of bricks involves the development of appropriate RSM experimental plan, fitting and selection of the best response surface models, optimization of the selected models and verification/confirmation of the modeling 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 major chemical constituents of clay which are the oxides of silicon (SiO_2) , aluminium (Al₂O₃), iron (Fe₂O₃), magnesium (MgO) and calcium (CaO) - on the performance of clay for refractory applications and linear shrinkage were studied.

The actual high and low levels of the operational factors under investigation were determined from experimental tests and the factor limits are given in Table 1. Thereafter, version 17 of the MINITAB was used to generate and randomize a two coded levels (+1 and -1) half central composite 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.

S/N	Factor Description	Factor Symbols		Factor Values	
		Coded	Actual	High (+1)	Low (-1)
1	Silicon oxide (%)	<i>x</i> ₁	SiO_2	63	48
2	Aluminium oxide (%)	<i>x</i> ₂	Al_2O_3	36	15
3	Iron oxide (%)	<i>x</i> ₃	Fe_2O_3	17	0.5
4	Magnesium oxide (%)	x_4	MgO	1.5	0.1
5	Calcium oxide (%)	<i>x</i> ₅	CaO	2.1	0.1

Table 1: Limits of the operational parameters

The actual high and low levels for each factor was selected based on non-variation of the responses or indicated asymptote behaviour of the responses before or after some combination of the variables.

The following equation 1 was used to effect the coding, where x is the independent variable in

$$X = \frac{x - \left(\frac{x - ax + x_{min}}{2}\right)}{\left(\frac{x - ax - x_{min}}{2}\right)} \quad (1)$$

$$x_1 = \frac{SiO_2 - 55.5}{7.5} \quad (2)$$

$$x_2 = \frac{Al_2O_3 - 25.5}{10.5} \quad (3)$$

natural units; X is the coded variable while
$$x_{max}$$
 and x_{min} are the maximum and minimum values of the independent variables respectively. The independent variables were coded using the transformation equations 2 - 6 relating the coded and actual values of the factors.

$$x_{3} = \frac{Fe_{2}O_{3} - 8.75}{8.25}$$
(4)

$$x_{4} = \frac{MgO - 0.8}{0.7}$$
(5)

$$x_{5} = \frac{CaO - 1.1}{1}$$
(6)

Critical evaluation of the linear order design gave rise to the creation of quadratic order design to augment the possible lapses associated with the linear order design. A central composite design was employed in this study to predict response surface models with quadratic effects and two factor interactions (NIST/SEMATECH, 2012 and Montgomery, 2009) for the linear shrinkage (L_s) and refractoriness (R_e) with respect to the chemical composition of the clay. The response design comprises of two level factorial points (-1, 1), center points (0) and axial points (- α , α) for each independent variable to estimate the second order polynomial as shown in Table 2.

Experime	Coded Values of Factors					
StdOrder	RunOrder	x_1	x_2	<i>X</i> 3	χ_4	x_5
14	1	1	-1	1	1	-1
13	2	-1	-1	1	1	1
26	3	0	0	0	0	2
6	4	1	-1	1	-1	1
12	5	1	1	-1	1	-1
2	6	1	-1	-1	-1	-1
31	7	0	0	0	0	0
11	8	-1	1	-1	1	1
4	9	1	1	-1	-1	1
7	10	-1	1	1	-1	1
19	11	0	-2	0	0	0
16	12	1	1	1	1	1
17	13	-2	0	0	0	0
25	14	0	0	0	0	-2
32	15	0	0	0	0	0
3	16	-1	1	-1	-1	-1
22	17	0	0	2	0	0
28	18	0	0	0	0	0
9	19	-1	-1	-1	1	-1
8	20	1	1	1	-1	-1
30	21	0	0	0	0	0
5	22	-1	-1	1	-1	-1
27	23	0	0	0	0	0
10	24	1	-1	-1	1	1
15	25	-1	1	1	1	-1
21	26	0	0	-2	0	0
20	27	0	2	0	0	0
29	28	0	0	0	0	0
24	29	0	0	0	2	0
23	30	0	0	0	-2	0
1	31	-1	-1	-1	-1	1
18	32	2	0	0	0	0

Table 2: RSM design table

The responses computed from the results of the experimental runs were used with the coded factor levels to fit coded linear functions (main effects) of the performance indicators and the operational variables of the clay using MINITAB. The search for models that will describe the responses adequately started with fitting of linear function because of the desire to quantify the parameters with simplest possible functions. However, 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 approximate the true responses adequately.

The model 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 whilst residual diagnostic plots contains normal probability plots of residuals, histogram of residuals, dot plots of the residuals versus observation order and that of residuals versus fitted response. 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 \mathbf{R}^2 values should be close to 1(100%) and SS of Error should be as small as possible. The analysis of variance was employed in testing the adequacy of the fitted models to be true approximations of the measured data. If the calculated value of $F - statistic(F_{cal})$ for each of the fitted models exceed the tabulated value of $F - statistic(F_{tab})$ i.e $F_{cal} > F_{tab}$ and $P - val < \alpha$, the fitted models are said to be adequate approximation of the data for the performance parameters of the clay. However, if reverse of the above statement is the case; $F_{cal} < F_{tab}$ and $P - val > \alpha$, then the models are inadequate to fit the data. The models lackof-fit test was also conducted to check the goodness of fit of the predicted models for the measured data. If the calculated value of $F - statistic (F_{calLOF})$ for each of the fitted models exceed the tabulated value of F –

statistic (F_{tabLOF}) i.e $F_{calLOF} > F_{tabLOF}$ and $P - val < \alpha$, the fitted models are said to exhibit insignificant lack of fit for the fitted data of the performance parameters of the clay, however if reverse of the above statement is the case; $F_{calLOF} < F_{tabLOF}$ and $P - val > \alpha$, then the models are said to exhibit significant lack of fit.

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 the 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 data.

Residual is the difference between the respective observed responses and their model predicted values. If a model is adequate, the points on the normal probability plots of the residuals should form a straight line. Small departure from the line in the normal probability plot is common, but a clearly "S" shaped curve indicates bimodal distribution of the residuals. Breaks near the middle of this graph are also indications of abnormalities in the residual distribution. The plots of the residuals versus run order and that of residuals versus fitted response should exhibit scatter feature without any obvious pattern (i.e. structureless) while histogram of the residuals is expected to portray dumb-bell shape.

Analytically, a multiple response optimization model was formulated using mathematical programming method with linear shrinkage (L_s) response as the objective functions to be minimized while maximizing refractoriness (R_e) of the clay. The formulated optimization model was solved using Desirability function approach.

3.0 RESULTS AND DISCUSSION

Graphical representation of the significant test using the main effects plot is shown in figures 1 and 2.

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Main effects plot are used for comparing the magnitude of data means of responses. The points in the plots represent the data means of the response variables at various levels of each factor with a reference line drawn as the grand means of the response data. Main effects plot comprises of plot line and the magnitude of the effect of each factor to the response is measured by the angle of inclination of the plot line to the centre line where positive inclination means positive effect and negative inclination means negative effect while lines horizontally parallel to the x- axis means the factor has no or insignificant effect on the response. Hence, it can be seen from the main effects plot in figures 1 and 2 for refractoriness and linear shrinkage respectively that x_2 and x_4 (figure 1) have positive inclination, i.e. an increase in

the oxides of aluminium and magnesium will result to an increase in the refractoriness of the clay while x_1 and x_3 (figure 1) have negative inclination and consequently, an increase in the oxides of silicon and iron will reduce the refractoriness of clay. From figure 2 which is the main effects plot for linear shrinkage, it was observed that x_3 has a positive effect while x_2 has a negative effect on the linear shrinkage. That means an increase in aluminium oxide will lead to a decrease in the linear shrinkage of the refractory material whereas an increase in iron oxide will increase the linear shrinkage. The two term interaction between the factor models of the refractoriness and linear shrinkage are shown in figures 3 - 4.



Figure 3: Interaction Plot for refractoriness





The characteristics of these plots are the possession of factor pairs representing the levels of the central composite design- high (+2) and low (-2) – of each factor and if the

lines are parallel to each other, the interaction between the factors is said to be statistically insignificant on the response. Also, if the lines intersect, the interaction between the factors are said to be statistically significant to the response. Positive and negative interaction effects occur when the intersection is on the high level of the factors and the low levels of the factors respectively. Inspection of the figures 3 and 4 shows some level of interaction between factors and corresponding responses and hence, the linear model was augmented to a second order (or quadratic) model to analyse the effects of the factor interactions on the corresponding responses.

In order to determine the optimum chemical composition of clay desirable for refractory application, experimental design methods were used. Table 3 shows the result of the experimental trial on the experimental plan for factor combinations.

Experime	(Coded Values of Factors					Responses	
Std Order	Run Order	x_1	x_2	<i>x</i> ₃	χ_4	<i>x</i> ₅	R	Ls
14	1	1	-1	1	1	-1	1225	9.31
13	2	-1	-1	1	1	1	1350	9.63
26	3	0	0	0	0	2	1450	6.5
6	4	1	-1	1	-1	1	1170	9.21
12	5	1	1	-1	1	-1	1600	2.86
2	6	1	-1	-1	-1	-1	1400	5.75
31	7	0	0	0	0	0	1370	5.95
11	8	-1	1	-1	1	1	1720	3.85
4	9	1	1	-1	-1	1	1600	2.2
7	10	-1	1	1	-1	1	1450	9.1
19	11	0	-2	0	0	0	1270	8.72
16	12	1	1	1	1	1	1440	6.8
17	13	-2	0	0	0	0	1520	6.3
25	14	0	0	0	0	-2	1470	6.1
32	15	0	0	0	0	0	1370	5.9
3	16	-1	1	-1	-1	-1	1670	4.78
22	17	0	0	2	0	0	1350	9.8
28	18	0	0	0	0	0	1370	5.76
9	19	-1	-1	-1	1	-1	1500	4.62
8	20	1	1	1	-1	-1	1436	6.7
30	21	0	0	0	0	0	1370	5.94
5	22	-1	-1	1	-1	-1	1508	7.9
27	23	0	0	0	0	0	1370	6.2
10	24	1	-1	-1	1	1	1700	6
15	25	-1	1	1	1	-1	1510	5.75
21	26	0	0	-2	0	0	1700	2.65
20	27	0	2	0	0	0	1560	4.26
29	28	0	0	0	0	0	1360	6.01
24	29	0	0	0	2	0	1500	5.98
23	30	0	0	0	-2	0	1390	6.5
1	31	-1	-1	-1	-1	1	1380	4.76
18	32	0	0	-2	0	0	1400	6.05
14	1	1	-1	1	1	-1	1225	9.31

Table 3: Design Table for the RSM study.

Test of significance of individual terms in the models is carried out on 95% significance level with the value of T_{tab} obtained using statistical Tables as 2.201. Individual terms in the model are said to be statistically insignificant to the responses if P - val > 0.05 and therefore, those statistically insignificant terms are

eliminated from the model as shown in equations 7 and 8.

The reduced empirical relationships between the factors and responses developed and analysed using the MINITAB 17 are shown in equations 7 to 8 for the refractoriness and linear shrinkage respectively.

$$R_{e} = 1369.33 - 31.54x_{1} + 73.87x_{2} - 90.88x_{3} + 27.13x_{4} - 3.29x_{5} + 21.92x_{1}^{2} + 10.67x_{2}^{2} + 38.17x_{3}^{2} + 18.17x_{4}^{2} + 21.92x_{5}^{2} - 36.06x_{1}x_{3} + 17.94x_{1}x_{4} + 33.56x_{1}x_{5} - 12.69x_{2}x_{4} - 31.81x_{3}x_{4} - 31.19x_{3}x_{5} + 49.31x_{4}x_{5}(7)$$

$$L_{s} = 6.1135 - 0.0858x_{1} - 1.0025x_{2} + 1.8283x_{3} - 0.1092x_{4} + 0.1950x_{5} + 0.0920x_{2}^{2} - 0.5175x_{1}x_{2} + 0.2375x_{1}x_{4} - 0.2937x_{1}x_{5} - 0.3412x_{2}x_{4} - 0.0788x_{3}x_{4} + 0.3925x_{3}x_{5} + 0.2250x_{4}x_{5}(8)$$

Table 4: Coefficients of determination and error standard deviation for the quadratic models

Responses	S	R-sq	R-sq(adj)
R_e	6.4945	99.89	99.76
L_s	0.2003	99.39	98.95

The reduced empirical relationship between the factors and responses developed in natural values is given in equations 4.7 to 4.8 below.

$$\begin{split} R_e &= 2942.5 - 50.02SiO_2 + 3.481Al_2O_3 + 20.08Fe_2O_3 - 195.5MgO - 323.2CaO \\ &\quad + 0.3897SiO_2{}^2 + 0.0968Al_2O_3{}^2 + 0.5608Fe_2O_3{}^2 + 37.08MgO^2 + 21.92CaO^2 \\ &\quad - 0.5828SiO_2Fe_2O_3 + 3.417SiO_2MgO + 4.475SiO_2CaO - 1.726Al_2O_3MgO \\ &\quad - 5.509Fe_2O_3MgO - 3.780Fe_2O_3CaO \\ &\quad + 70.45MgOCaO \end{split}$$

$$\begin{split} L_{s} &= -2.29 + 0.1630SiO_{2} + 0.2638Al_{2}O_{3} + 0.1802Fe_{2}O_{3} - 1.717MgO + 1.695CaO \\ &+ 0.000834Al_{2}O_{3}^{2} - 0.006571SiO_{2}Al_{2}O_{3} + 0.04524SiO_{2}MgO - 0.03917SiO_{2}CaO \\ &- 0.04643Al_{2}O_{3}MgO - 0.01364Fe_{2}O_{3}MgO + 0.04758Fe_{2}O_{3}CaO \\ &+ 0.3214MgOCaO \end{split}$$

The model adequacy are confirmed graphically using the residual plots which include the normal probability plot, histogram, residual versus fitted values and residual versus observation order in a 4-in-1 format as shown in figures 5 - 6.***



Figure 5: Confirmatory test and Residual plots for second order model of the refractoriness





An analytical view of the figures 5-6 revealed that the introduction of the second order terms improved the adequacy of the models and the plots of the normal probability tends to fall in a straight line and the histogram gives the required dumb bell shape. Hence, the second order models are adequate to statistically fit the data with little outliers and reduced skewness.

Desirability function approach eliminates the rigour associated with most other optimization techniques such as the optimization using contour and surface plots. It is a multi - response multi -factor optimization technique which operates on the principle established by Derringer Harrington. It optimizes a set of responses and defines the best factor settings for a solution of a multivariate objective function. The objective of this study is to determine the optimum clay constituents required to minimize linear shrinkage while maximising refractoriness. *** The response optimizer capability of MINITAB 17 was employed for this purpose and the optimization plot is given in fgure 7.



Figure 7: Optimization plot of the clay parameters.

The value of individual desirability and the composite desirability respectively approximate to 1 which signifies that the optimization result is highly desirable. The desirability plot in Figure 7 above can be visualized and the values of the optimal settings graphically presented. Therefore, it is seen that the clay will perform optimally at the factor settings of 60%, 33%, 2%, 1.5% and 2.1% for oxides of silica, aluminium, iron, magnesium and calcium respectively. The optimal response is obtained as 3.4% linear shrinkage and 1720°C refractoriness.

4.0 CONCLUSION

Refractory bricks of different clay samples from different locations were produced and factors considered in fired. The this investigation were the amount of silicon oxide, aluminium oxide, iron iii oxide, magnesium oxide and calcium oxide. while the refractoriness, R_e and linear shrinkage, L_s of the bricks were analyzed and optimized using response surface methodology. The results obtained from the conducted experiments were employed in developing the quadratic models

which described the performance parameter of the clays adequately above 95% prediction level. The results obtained showed that refractoriness of 1720°C and linear shrinkage of 3.4% could be obtained at 60%, 33%, 2%, 1.5% and 2.1% of SiO₂, Al₂O₃, Fe₂O₃, MgO and CaO, which is desirable enough for refractory bricks. Also, the main effects of these metallic oxides on the response variables and the developed models were shown in this work, the insignificant parameters of the response models as revealed by the ANOVA test being eliminated from the developed models.

It is therefore recommended that the models developed in this study be applied in the determination of the suitability of clays for refractory applications. Also, additives that are rich in alumina content can be used in the production of refractory bricks with a view to increasing its refractoriness.

As shown in the main effects plot, increase in the alumina content not only increases the refractoriness but also decreases the linear

shrinkage of the bricks; and this is highly recommended for refractory bricks in service.

It is also recommended that studies on the effects of these oxides on other physical properties of refractory bricks such as the apparent porosity be carried out by researchers so as to improve on the quality of these bricks.

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