

# Optimizing the Refractory Properties of Alkalari Clay Using Design Expert

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**Abstract:** This study is aimed at optimizing the properties of Alkalari clay mixed with Rice Husk for metal casting using Analysis of Variance. It was discovered that the model adopted is significant for prediction of refractoriness because the R-square value of 0.9268 is close to 1.00 and also the model predicted R-square value of 0.9268 is a reasonable agreement with adjusted R-square of 0.9822. Also the model F-value of 133.41 shows the model is significant value of P-value less than 0.05 indicated model term is significant. However, the model is quadratic model with standard deviation of 0.27 R-square of 0.9958 and predicted and adjusted R-square of 0.9699 respectively. The adjusted R-square is in close agreement with the predicted R-square value while the R-square value of 0.9927 is close to 1.00 this show that the model has 99.27% chance of efficiency prediction of thermal shock resistance and 2.28% chance of not predicting thermal shock resistance efficiently. The model with F-value of 328.81 implies the model is significant; the value of P-value must be less than 0.05 for the model terms to be significant. The cold crushing strength also a quadratic model with standard deviation of 0.017, mean crushing strength of 2.16KN/cm<sup>2</sup>. The predicted R-square of 0.8205 is reasonable agreement with the adjusted R-square of 0.9567 is close to 1.00, this shows 97.4%. The results when compared with ASTM standards of refractory materials, they have proven to be suitable for ferrous metal casting. These standards are apparent porosity 20%-30%, bulk density 1.7g/cm<sup>3</sup>-2.3g/cm<sup>3</sup>, CCS 1.5kN/cm<sup>2</sup>-5.9kN/cm<sup>2</sup>, linear shrinkage 3%-10%, refractoriness 1200°C -1900°C.

**Keywords:** Optimization, Thermal Shock, Cold Crushing, Refractoriness, Rice Husk, Alkalari clay.

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## I. INTRODUCTION

Clay is a natural material obtained from the earth that are powder form the decay fossil, wet, stone-like when baked [1]. Clay is an earthy and soil, although with intricate inorganic blend, whose structure diverges generally depending on the ecological and geographical position. It is a natural material formed as a result earth movement, rock weathering on the earth's surface [2]. Most clay is crystalline, with a definite repeating arrangement of atoms in them. The majority of them are made up of planes of oxygen, aluminum atoms silicon and holding the oxygen together by ionic bonding [3]. The raw materials for the production of various refractory products include kaolinite ( $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ ), Chromite ( $FeOCr_2O_3$ ), Magnesite ( $MgCO_3$ ) and various types of clays [4]. Aluminno-silicate and magnesite refractory products are the major types of refractory used in Nigeria manufacturing industries [4]. Refractory material includes alumino-silicate, magnesite, chrome silica, carbon and dolomite etc. these oxides are classified according to their chemical behavior.

However, in the last few years, there has been tremendous research, geared towards the sustainable and suitability for adoption as refractory material for different metallurgical and process industries. This development is justified by present situation of the nation's economy, the need to meet the technological requirement of the country and to conserve much

needed foreign exchange. The application of clay as a refractory material depends severally on its thermal property of refractoriness, chemical composition, mechanical and physical properties [5]. These properties are responsible for its numerous structural engineering materials in the area of ceramics and refractory materials [6]. The country Nigeria is endowed with vast land, lucrative solid minerals with rich and abundant clays. It is available in commercial quantity but the remained untapped and under-utilized in the metallurgical and ceramic manufacturing industries [7]. It is important to optimize, the desired goal for each factor and its response in order to maximize and minimize target within range, also the most reliable way to evaluate the quality of the model fitting is through the application of analysis of variance (ANOVA) with a central idea to compare the variation due to the change in the combination of variable levels and random errors inherent to the measurements of the generated response.

**II. MATERIALS AND METHODS**

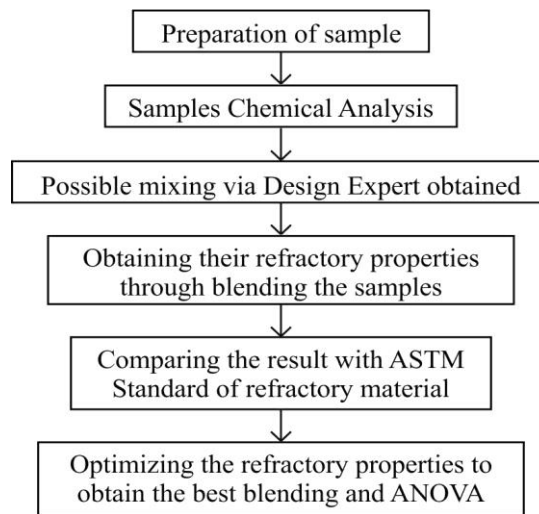
**A. Materials**

The materials used in this research include:

- i. Alkaleri fireclay
- ii. Rice hush ash
- iii. Distilled water

**B. Methods**

Below is the designed procedure that was used for the experiment:



**Fig. 1: Block Diagram of Experiment Process**

**III. RESULTS AND DISCUSSIONS**

**A. Statistical Analysis of Refractoriness**

**Table 1: ANOVA Response Surface Quadratic Model of Refractoriness**

Source	Quadratic
Standard deviation	6.23
R -Squared	0.9896
Adjusted R- Squared	0.9822
Predicted R-Squared	0.9268
PRESS	1916.33
Mean	1627.15
Adequate Precision	35.964

From Table 1 the model is quadratic model with standard deviation of 6.23 and mean refractoriness at 1627.15°C. The model predicted the refractoriness because of its R-square value of 0.9896 was close to 1.00 also the model had predicted the R-square value of 0.9268 which is reasonable in agreement with the Adjusted R-square value of 0.9822. However, the adequate precision measurement of the signal to noise ratio, was a ratio greater than 4 is desirable the model has a ratio of 35.964 indicate an adequate signal. There is 98.96 chance of the model predicting the refractoriness efficiently and 0.74% chance of the model not predicting the refractoriness efficiently.

**Table 2: ANOVA Response Surface Quadratic Model of Refractoriness**

Source	Sum of Squares	dF	Mean Square	F Value	P-Value Prob>F	
Model	25917.71	5	5183.54	133.41	<0.0001	significant
A-RHA	22452.75	1	22452.75	577.87	<0.0001	
B-Clay	327.45	1	327.45	8.43	0.0229	
AB	6.25	1	6.25	0.16	0.7003	
A <sup>2</sup>	3126.54	1	3126.54	80.47	<0.0001	
B <sup>2</sup>	26.45	1	26.45	0.68	0.4365	
Residual	271.78	7	38.85			
Lack of fit	268.78	3	89.59	111.99	0.0003	significant
Pure Error	3.20	4	0.80			
Cor Total	26189.69	12				

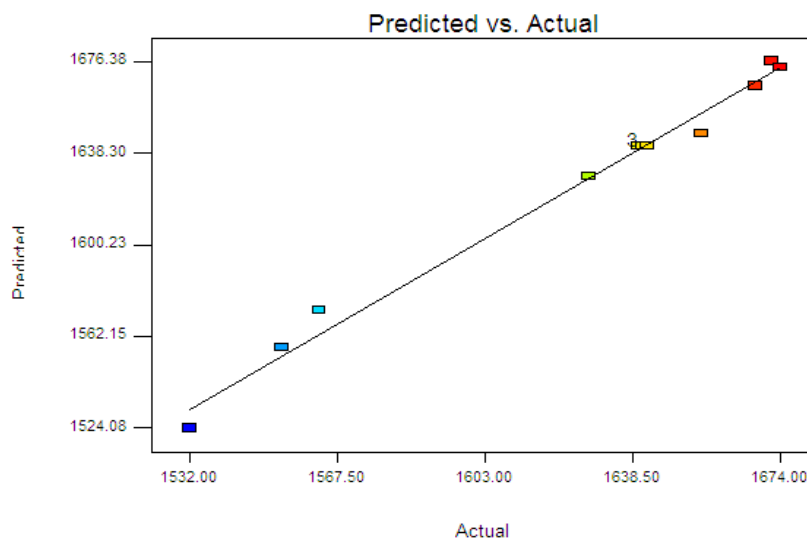
The model F-value of 133.41 this shows the relevance of the model, while the P-values is less than 0.05 make the model term to be of great value. In this case A, B and A<sup>2</sup> are important model terms while AB and B<sup>2</sup> are not significant model terms.

**Equation in terms of code:**

$$\text{Refractoriness} = +1641.40 + 52.98 \times A^2 - 6.40 \times B + 1.25 \times A \times B - 21.20 \times A^2 - 1.95 \times B^2$$

... (1)

From equation 9, A, B, A<sup>2</sup> and B<sup>2</sup> are significant model terms.



**Fig. 2: Graph of predicted value against actual value of refractoriness**

The above figure shows the graph of predicted value versus actual value of refractoriness. From the graph both the predicted value and actual values are in relative conformity. Refractoriness of the predicted value is 1638.30°C while for actual value is 1638.50°C.

**B. Statistical Analysis of Thermal Shock Resistance**

Table 3 shows the summary statistical analysis of thermal shock of the samples.

**Table 3: Model Summary Statistics for Thermal shock of the Samples**

Source	Quadratic
Standard deviation	0.27
R -Squared	0.9958
Adjusted R- Squared	0.9927
Predicted R-Squared	0.9699
PRESS	3.68
Mean	12.00

From the table 3, the result show that is a quadratic model with standard deviation of 0.27, R-square value of 0.9958 and the predicted and adjusted R-square values was 0.9699 and 0.9927 respectively. Another one is a press of 3.68 and means thermal shock resistance of 12 cycles. The adjusted R-square value is in close concord with the predicted R-square value while the R-square value of 0.9927 is close to 1.00; this implies that the model has 99.27% chance of competent prediction of thermal shock resistance and 2.28% chance of not predicting thermal shock resistance efficiently.

**Table 4: ANOVA Response Surface Quadratic Model of Thermal shock**

Source	Sum of Squares	dF	Mean Square	F Value	P-Value Prob>F	
Model	121.48	5	24.30	328.81	<0.0001	significant
A-RHA	113.57	1	113.57	1536.95	<0.0001	
B-Clay	2.91	1	2.91	39.44	0.0004	
AB	1.00	1	1.00	13.53	0.0079	
A <sup>2</sup>	1.74	1	1.74	23.54	0.0019	
B <sup>2</sup>	1.74	1	1.74	23.54	0.0019	
Residual	0.52	7	0.074			
Lack of fit	0.52	3	0.17	1417.6	<0.0001	significant
Pure Error	0.000	4	0.000			
Cor Total	122.00	12				

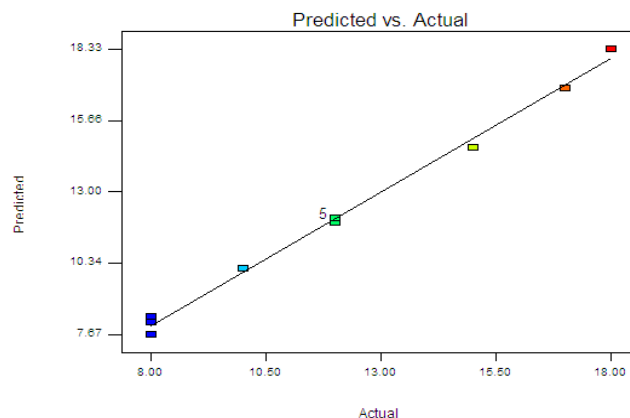
The model with F-value of 328.81 this shows the important of the model, while the P-values must be less than 0.05 for the model terms are to be significant. Hence A, B, AB, A<sup>2</sup> and B<sup>2</sup> are significant model terms.

**Equation in terms of code:**

$$\text{Thermal Shock Resistance} = + 12.00 - 3.77 \times A + 0.6 \times B - 0.5 \times A \times B + 0.5 (A)^2 - 0.5(B)^2 \dots(2)$$

From equation 2, both RHA and Alkaleri clay are significant model terms.

Fig. 3: Graph of predicted value against actual value of thermal shock resistance



From Fig. 3, both predicted and the actual values of thermal shock resistance is 12 cycles.

C. Statistical Analysis of Cold Crushing Strength

Table 5: Model Summary for Cold Crushing Strength of the samples

Source	Quadratic
Standard deviation	0.017
R –Squared	0.9747
Adjusted R- Squared	0.9567
Predicted R-Squared	0.8205
PRESS	0.015
Mean	2.16
Adequate Precision	22.225

Table 5, show that the model is a quadratic model with standard deviation of 0.017, mean CCS of 2.16 kN/cm<sup>2</sup>. The predicted R-square of 0.8205 is realistic with the adjusted R-square of 0.9567 and the R-square value of 0.9747 is close to 1.00, this implies that 97.47% chance of the model predicting CCS capably and 1.7% chance of the model not predicting CCS efficiently. Adequate precision of 22.225 is sufficient for the model forecast of CCS.

Table 6, shows the ANOVA response surface quadratic model.

Table 6: ANOVA response surface quadratic model

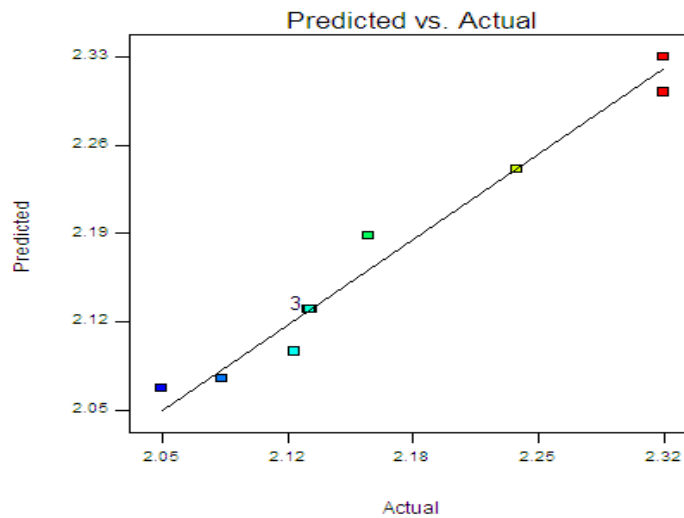
Source	Sum of Squares	dF	Mean Square	F -Value	P-Value Prob>F
Model	0.082	5	0.016	54.01	<0.0001
A-RHA	0.069	1	0.069	227.98	<0.0001
B-Clay	3.414E-003	1	3.414E-003	11.23	0.0122
AB	4.000E-004	1	4.000E-4	1.32	0.2891
A <sup>2</sup>	8.280E-003	1	1.463E-003	4.81	0.0644
Residual	2.126E-003	7	3.041E-004		
Lack of fit	2.126E-003	7	7.0088E-004	1417.6	<0.0001
Pure Error	2.000E-006	4	5.000E-007		
Cor Total	0.084	12			

The Model F-Value of 54.01 and P-value less than 0.0001 shows the model is good. The value of P-value was less than 0.05 which indicated that the model terms are important in this situation both A and B with P-value of less than 0.001 and 0.0122 respectively are important model term while AB and A<sup>2</sup> with P-values of 0.2891 and 0.0644 respectively are not significant term.

Equation in terms of code

$$C.C.S = 2.13 + 0.093 (A) - 1.00E-002 (B) (A) + 0.035 (A)^2 + 0.015 (B)^2 \dots(3)$$

From equation 3, RHA and clay are significant terms.



**Fig. 4: Graph of Predicted values against Actual values of CCS**

Fig. 4 predicted the C.C.S value to be 2.12kN/cm<sup>2</sup> and for actual value was 2.14kN/cm<sup>2</sup>. This implies that the statistical analysis of CCS was correct in terms of constant error.

**D. Optimization Using Design Expert 7.1.6**

**Table 7: Numerical optimization using Design-Expert 7.1.6**

Constraints						
Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
RHA	In range	10	30	1	1	3
Alkalari Clay	In range	70	90	1	1	3
Bulk Density	In range	1.8303	1.94	1	1	3
Apparent Porosity	In range	20.0719	22.7267	1	1	3
Linear Sharinkage	minimize	2.4	6	1	1	3
C.C.S	In range	2.048	2.320	1	1	3
Thermal Shock resist	maximize	8	18	1	1	3
L.O.I	Minimize	8.6	9.4	1	1	3
Refractoriness	maximize	1532	1674	1	1	3

Table 7 shows the constraints, goals and limits of the numerical optimization of the result while table 8 shows the solution of the numerical optimization of the result.

**Table 8: Numerical optimization using Design-Expert 7.1.6 Solution**

Solutions	
RHA	14.22
Clay	88.75
Bulk density, g/cm <sup>3</sup>	1.92084
Apparent Porosity, %	22.3536
Linear shrinkage, %	3.214
CCS, KN/cm <sup>2</sup>	2.084
Thermal shock resistance	14.7424
Refractoriness, °C	1595.99
L.O.I, %	9.309
Desirability	0.617

Table 8 shows the optimization result, the optimal solution was achieved at desirable point of 0.617. The optimal blending was 88.75g of Alkalari clay and 14.22g of RHA and the equivalent optimal refractory properties are as follow bulk density of 1.9208g/cm<sup>3</sup>, apparent porosity of 22.3536%, linear shrinkage of 3.3140%, CCS of 2.084kN/cm<sup>2</sup>, thermal shock resistance of 14.7424, refractoriness of 1595.99°C and L.O.I of 9.309%.

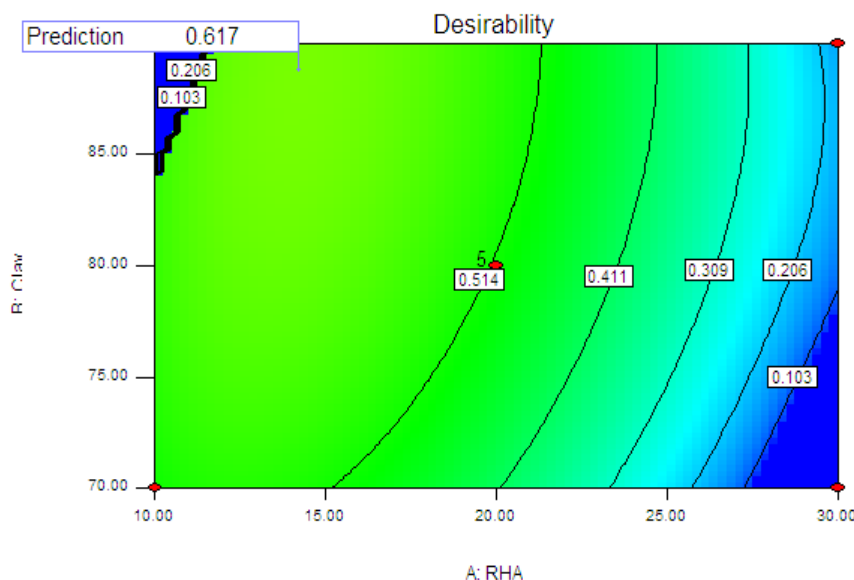


Fig. 5: Graph of Clay against RHA at 0.617 desirability

However, Fig. 5 shows the contour graph representation of the optimal solution at 0.617 desirably point. The optimal blend is 88.75g of Alkalari clay and 14.22g of RHA. This result agreed with the confirmatory test result of 80.00g of Alkalari clay and 20.00g of RHA with the following responses 1.900g/cm<sup>3</sup> bulk density, 21.64% apparent porosity, 5.0% linear shrinkage, 2.13kN/cm<sup>2</sup> CCS, 12 cycles, 1643°C, and 9.2% L.O.I.

#### IV. CONCLUSION

The refractory properties of Alkalari clay at various RHA mixtures using design expert 7.1.6 Central Composite Design (CCD) was determined and the result was compared with ASTM standard of refractory materials for casting ferrous metal. From the result obtained, the following conclusions were drawn:

- a. The characterization of the Alkalari clay and RHA using ED-XRFS shows that the Alkalari clay is alumino-silicate clay, due to its 33.2% of Al<sub>2</sub>O<sub>3</sub> and 46.35% of SiO<sub>2</sub>, also the analysis of the RHA shows that the 74.2% of SiO<sub>2</sub> and 8.52% of K<sub>2</sub>O gives the RHA its binding property effect with clay while 2.0% of MgO and 4.09% of CaO was responsible for the increase in crushing strength.
- b. Design expert 7.1.6 was successfully employed in achieving the possible mixing proportion of Alkalari clay and RHA. The refractory properties such as bulk density, apparent porosity, L.O.I, thermal shock resistances, CCS, refractoriness and liner shrinkage were determined.
- c. Using design expert 7.1.6 CCD, the optimum blend was achieved at desirability of 0.617. At 0.617 desirability, the optimal blend was 88.75g of Alkalari clay and 14.22g of RHA, which has refractory properties of apparent porosity of 22.3536%, CCS of 2.0838kN/cm<sup>2</sup>, firing shrinkage of 3.21%, bulk density of 1.9208 g/cm<sup>3</sup>, L.O.I of 9.3995%, thermal shock resistance of 14.74 cycles, refractoriness of 1595.99°C. Hence, recommended for cast iron works.

The result was compared with ASTM standard of refractory material for ferrous casting and the result conforms to the standard. These standards are apparent porosity 20%-30%, bulk density 1.7g/cm<sup>3</sup>-2.3g/cm<sup>3</sup>, CCS 1.5kN/cm<sup>2</sup>-5.9kN/cm<sup>2</sup>, linear shrinkage 3%-10%, refractoriness 1200°C -1900°C.

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