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DEVELOPMENT OF AN AGGREGATE MIX RESPONSE PREDICTIVE MODEL FOR FIRE BRICKS MADE FROM CLAY DEPOSIT IN EDO STATE NIGERIA USING ANALYSIS OF VARIANCE

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ABSTRACT

Experimental procedures used for determining appropriate mix ratios of aggregates for refractory bricks used for making heat furnace is laborious and time consuming. However, use of design of experiment which utilizes statistical data is timely and less cumbersome for such prediction. ANOVA was used in conjunction with experimental procedure to predict suitable mix ratio of clay and sand sourced from Ovia North East River in Edo state, Nigeria. Sample of the clay and sand were collected and prepared in alternating mix ratios of 10% to 90% with binders. The mixes were used to make refractory bricks and their physical properties investigated. Result obtained was used to validate the ANOVA prediction. Split-split plot experimental design and rejection of the null hypothesis indicated that mix ratios of clay/sand of 30/70%, 40/60%, 50/50%, 60/40% and 70/30% respectively were good for refractory bricks with the optimum mix being 60% clay and 40% sand

1. INTRODUCTION

Clay materials are a subset of a group of natural minerals with characteristic properties such as their plasticity, water affinity and catalysis [1]. Edo state in Nigeria has abundance of clay which has been employed in the production of artistic artefacts and domestic wares. Clay composition include metal oxides such as aluminate, magnesium oxide, silicate, calcium oxide [2] and trace amounts of non-metallic oxides. Clay as a soil type evolved from the earth surface arising from weathering due to geological formations. Clay has been employed in making bricks for structural and thermal facilities where it is expected to serve its design purpose of strength and resistance to heat respectively. It therefore means that adequate knowledge of the properties and use of clay in conjunction with other earth materials is significant in enhancing its systematic utilization and or modification to forestall failure or unwarranted degradation in operation in such facilities [3]. Laborious and extensive experimental procedures have been explored to determine the appropriate quantity and or mix ratios of clay and other earth materials for fire bricks making.

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A basic approach for conducting such experiments requires the design and testing various aggregate mix ratios for the main materials composition such as sand (silica) and clay (alumina) of the refractory fire. Important properties of refractory bricks such as apparent porosity, index of plasticity, bulk density [4] and water absorption are affected by clay properties, method of manufacturing and firing temperature. It also depends on composition - i.e. the ratio of clay to non-clay material and grain sizes in the batch, however; few literatures and researchers have dwelled on the normal clay-sand mix for refractories [5]. The use of experiments often involves a trial and error random selection of possible multiple varied mix which may take extended time and effort for many runs, hence there is the need for a nested selection of viable range of mix ratios from which the optimum or other targeted mix ratios can be determined through statistical methods. Efforts have been made towards developing analytical methods rationalizing the initial mixture proportioning into a more logical and systematic process [6]. The mixtures which may be regarded as elements in this case of the refractory brick mix are mainly derived from the sand and clay mixes. The sand and clay elements are often present in their respective materials aggregate as metallic oxides of silicon (silica), aluminium (alumina) and magnesium (magnesia) and are considered as the most important materials used in the manufacturing of refractories [7]. Statistical methods, also often termed as design of experiments methods or statistical experiment design methods or statistical factorial design methods or empirical methods, are used frequently in obtaining the optimum aggregate mixture design [8]. Design of Experiments methods are an improvement over fully experimental methods, in which, instead of selecting one starting mix proportion and then adjusting by trial and error for achieving the optimum solution, a set of trial batches covering a chosen range of proportions for each mixture component is defined according to established statistical procedures or data as applicable in well logs for drilling clay [9][10]. On the use of the specific statistical method, smaller trial batches can then be carried out, test specimens are produced and tested, and experimental results are analyzed using a standard statistical method. These methods include fitting empirical models to the data for each performance criterion. Applying the statistical method in the mix proportioning does not change the overall approach of designing the mix proportion using available standards as it mainly changes the trial batching process [10]. The design of mix ratios and the effects of the respective constituents and their optimums can be ascertained through three major ways which include the followings;

i. Fully experimental
ii. Fully analytical (statistical)
iii. Semi-experimental (half-analytical) methods which are based on combining the experimental database or experimentally developed prediction models and various analytical tools such as genetic algorithm, artificial neural network and mathematical programming [11]. A model was developed by [3] for the evaluation and prediction of mix formulation for production of insulating refractory using Osiele and Ukpor fireclays blended with coconut shell particulates. [12] highlighted the improvement of refractory bricks made from Nigeria clays through aggregate proportioning. [5] Carried out research on effect of clay-sand mix ratios on bricks prepared from two local clay sources viz. Koel River and Naga pond in India. [13] Proposed step-by-step statistical approach to obtain optimum proportioning of aggregate mixtures using the data obtained through a statistically planned experimental program. Scientific study of Ovia clay as a significant natural mineral for refractory bricks making using a concurrent experimental and statistical method has not been extensively explored by researchers. This paper therefore proposes to explore both procedures in predicting responsive aggregate mix ratio of Ovia clay and sand for refractory bricks production.

2.0 MATERIALS AND METHODS

Materials requirement for the initial experimental procedure of the research include refractory castable, clay, sand, distilled water, hoe, shovel, drying oven, mortar and pestle, cubic mold measuring (100 x 100 x 100) mm, rectangular cuboid measuring (100 x 50 x 50) mm, pulverizing machine, grinding machine, mechanized mixer, rammer, sieves, heat furnace, electric digital balance, hydraulic press machine, cold crushing strength machine, Atomic Absorption Spectrometer (AAS) machine, pyrometer.

Preliminary experiment involved sourcing of clay sourced from Ovia river in Ovia North East local of Edo South. The River forms the boundary between Ovia North-west and Ovia South-west Local Government Areas of Edo State, Nigeria. The river flows in a North-Southerly direction, originating from Owan in Ovia North-East Local Government and it empties into the Atlantic Bight of Benin between latitude 05^{o} - 05 40'N and longitude 5 ^{o}00'–60 ^{o}30'E. The river is located within the wet tropical rainforest zone with proportionate dry and rainy seasons.

The method of preparation of the refractory bricks involved the drying, grinding, cooling and washing of the bulk materials of clay and sand before mixing of the aggregates in a random-pre-determined ratio to form refractory bricks using molds of specific sizes. the bulk materials preparation involved the followings:

1. **Atterberg limits** are a basic measure of the nature of a fine-grained soil. The limit can be applied on the followings;
   i. To determine the Liquid limit, plastic limit and plasticity index of fine-grained soils.
   ii. To classify fine-grained soils.
   iii. To correlate with engineering behavior such as compressibility, hydraulic conductivity (permeability), compatibility, shrink-swell, and shear strength.
2. **Drying and grinding**; the clay and sand samples were sun dried in open air to remove moisture. On drying the large clay lumps were crushed to smaller sizes using hammer to enhance its grinding into finer powder with a grinding/pulverizing machine. 50kg each of the material of sand, clay and refractory castable was used in the research.
3. **Bricks making**; Test samples of sand, clay and refractory castable (binder) shown in Figures 1, 2 and 3 respecticely were sieved to fine particles grade of 50µ and mixed with 15% of water to form a homogenous plasticized mortar. Various mix ratio of 90% clay, 10% sand and 90% sand and 10% clay at 10% increment in weight percentage of the clay and sand was formulated into bricks using lubricated cubic steel molds. The produced refractory bricks shown in Figure 4

Figure 1 Ovia clay

Figure 2 River Sand

Figure 3Refratory binder

Figure 4 Produced refractory brick samples

The refractory bricks were dried in a furnace at a temperature of 110^{o}C to ensure further removal of moisture through evaporation. They were fired in a furnace at intervals of 100^{o}C for every 10 minutes till a peak temperature of 1100^{o}C was attained. The test samples were left to remain in the oven at the peak temperature to soak for 8hrs and allowed to cool for another 24hrs inside the furnace before finally removed for analysis.
Initial test operations carried out on the bricks include the followings:

Apparent Porosity (P) given as

$$\frac{W_{w-}W_D}{W_w-W_s} \times 100 \qquad (1)$$

The % Water absorbed (WA) was given as

$$\frac{W_{w-}W_D}{W_D} \times 100 \qquad (2)$$

where;
W_D = Weight of fired specimen, W_S = Weight of fired specimen in water
W_W = Weight of soaked specimen suspended in air

The Cold crushing strength (CCS) which is the amount of load that the clay/refractory material can resist after it has been fired to a temperature of 1200^{o}C. It was expressed as:

$$\frac{Load}{Area} \qquad (3)$$

Bulk density; this was given as

$$\frac{DP_W}{W-S} \qquad (4)$$

where; D = Dried weigh, Pw = Density of water, W = Soaked weight, S = Suspended weigh

Refractoriness Test: This is the measure of the fusibility of a material and indicates the temperature at which the brick softens. It is expressed as its Pyrometric cone equivalent (PCE). Pyrometric cone equivalent is the number which represents the softening temperature of a refractory specimen of standard dimension measuring 38 mm vertical height and 19mm triangular base.

Shrinkage test; the linear shrinkage of the materials was determined as:

Linear Shrinkage in % = $\frac{L_2-L_1}{L_1}$ x 100% (5)

where; L2 and L1 are final and original sizes of bricks respectively.

Slag Attack Resistance Test: This was expressed as a measure of the degree of attack and penetration of metal slag into the refractory brick. A hole was made into the brick, packed full of metal (iron) slag and heated and maintained in a furnace at a temperature of 1300^0C for one hour after which it was cooled sectioned and examined for any attack or penetration.

Loss on ignition (LOI); this was expressed as the weight reduction in percentage in the overall weight of the prepared clay brick. The loss on ignition at given temperature was expressed as

$$\frac{W_1 - W_2}{W_1} \text{ x } 100\% \qquad (6)$$

where: w_1 and w_2 are initial and final weights of bricks respectively.

Thermal Shock Resistance Test: Test piece of the refractory bricks were placed in a cold furnace and heated at the rate of 5^oC/min until the furnace temperature was 1200^oC. the bricks were held at this temperature for 30 minutes after which the test pieces were removed and placed on a cold environment free of draught and allowed to cool down for ten minutes after which they were returned to the furnace for a another 10 minutes. The cycle was repeated N times before thermal crack occurred.

Adsorption of material (total water adsorption) is the increase in the weight of brick due to moisture in air. It was mathematically expressed as

$$W\% = \frac{W_b - W_a}{W_a - W_c} \text{ X } 100\% \qquad (7)$$

where: W = moisture content of specimen (%), Wa = weight of specimen after oven dried for 24 hours (g), Wb = weight of specimen after 24 hours in desiccators (g), Wc=weight of crucible (g)

The design of experiment and the experimental procedure were jointly used to ascertain a suitable mix for a desired refractory brick to be used in a target heat facility. In this case, a fire clay brick for heat treatment furnace was desired with the following characteristics according to ASTM (2015) standard in Table 1

Table 1 International Recommended Refractory characteristics (ASTM, 2015)

Standard refractory bricks characteristics							
Loss on ignition (%)	**Linear shrinkage (%)**	**Apparent porosity (%)**	**Bulk density (g/cm3)**	**Compressive strength (Mpa)**	**Thermal resistance (cycle)**	**Moisture content (%)**	**Refractoriness**
6-15	7-10	20-30	1.71-2.8	15	2330	1-15	1500-1750

Operational temperature of the heat furnace is (0-10500C). The fire clay grades are differentiated by their alumina/silica content ranging from low duty (23%AL, 43%Si) to super duty (49%AL, 80%Si), while intermediary range comprises of medium duty (26-36%AL, 60-70%Si) and high duty (35-40%Al, 50-80%Si). The analytical design abstract for the refractory bricks formulation using the mix ratio in percentages compositions is shown in Figure 5

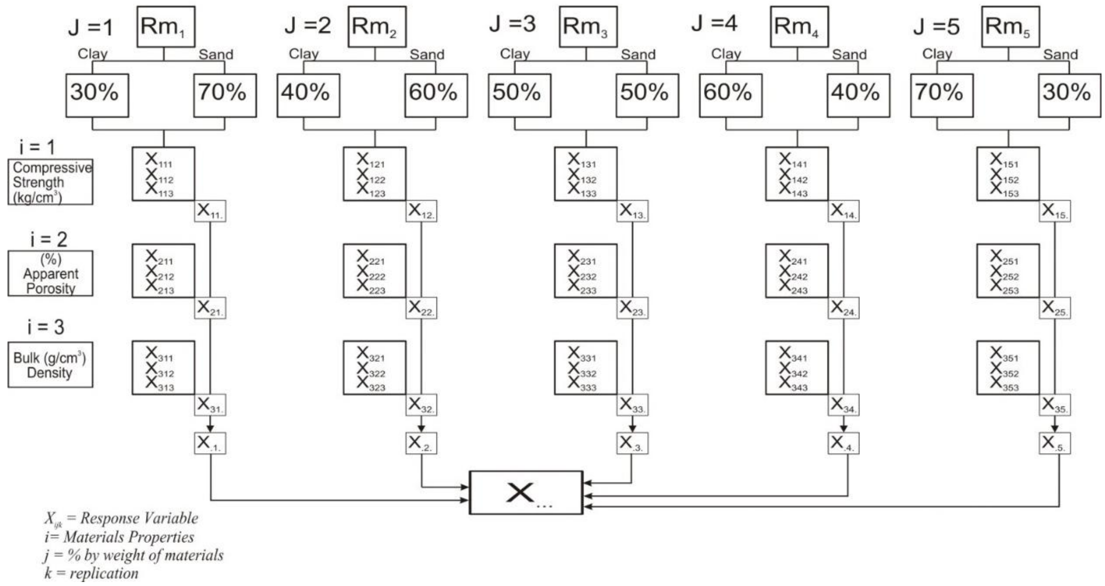

Figure 5 Analytical design abstract of refractory bricks formulation

The Numerical design abstract for refractory bricks formulation is shown in Figure 6.

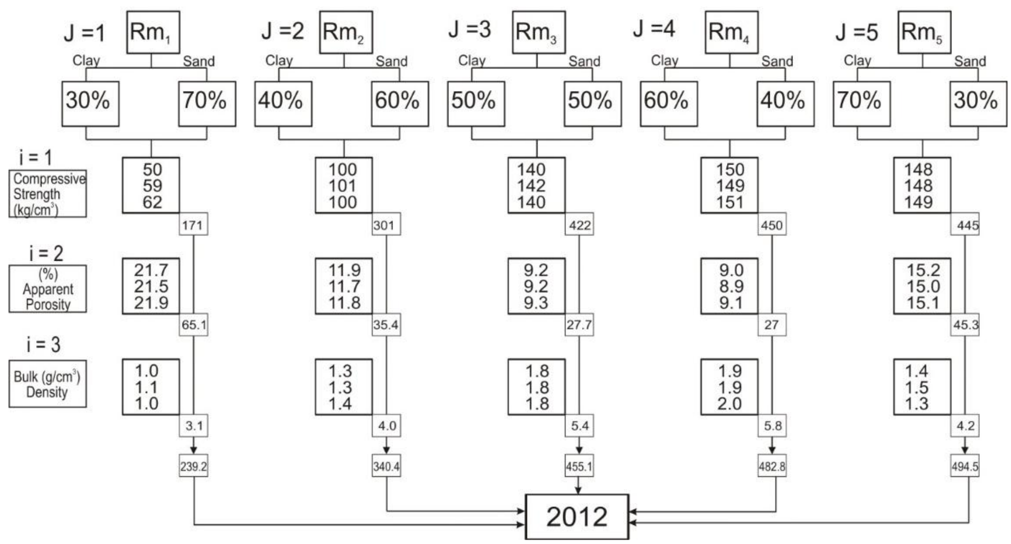

Figure 6. Numerical Design Abstract for Refractory Bricks Formulation

The statistical model developed for the study was expressed as:

$$X_{ijk} = \mu + \alpha_i + \beta_j + (\alpha\beta)_{ij} + \varepsilon_{ij(k)} \begin{Bmatrix} I = 3 \\ J = 5 \\ K = 3 \end{Bmatrix} \tag{8}$$

where, X_{ijk} – Response Variable

$i = Materials$ type and properties

$j = Percentage$ per weight of materials type

$k = replications$

1. Sum of squares for total (SS_T)

$$SS_T = \sum_{i=1}^{I=3}\sum_{j=1}^{J=5} X_{ijk}^2 - \frac{X_{...}^2}{IJK}$$

2. Sum of squares for material types and properties (SS_A)

$$SS_A = \sum_{i=1}^{I=3} \frac{X_{i..}^2}{JK} - \frac{X_{...}^2}{IJK}$$

3. Sum of squares for percentage per weight of materials type (SS_B)

$$SS_B = \sum_{j=1}^{J=5} \frac{X_{.j.}^2}{IK} - \frac{X_{...}^2}{IJK}$$

4. Sum of squares for material types and properties $\times$ percentage per weight of materials type interaction (SS_{AB})

$$SS_{AB} = \sum_{i=1}^{I=3}\sum_{j=1}^{J=5} \frac{X_{ij.}^2}{K} - \sum_{i-1}^{I=3} \frac{X_{i..}^2}{JK} - \sum_{j=1}^{J=5} \frac{X_{.j.}^2}{IK} + \frac{X_{...}^2}{IJK}$$

5. Error sum of squares (SS_E)

$$SS_E = SS_T - SS_A - SS_B - SS_{AB}$$

3.3.4 Statistical Computation

1. Sum of squares for total (SS_T)

$$SS_T = \sum_{i=1}^{I=3}\sum_{j=1}^{J=5} X_{ijk}^2 - \frac{X_{...}^2}{IJK}$$

$$= \begin{bmatrix} 50^2 & + & 59^2 & + & 62^2 & + & . & . & . & + & 1.9^2 & + & 1.9^2 & + & 2.0^2 & + \\ 21.7^2 & + & 21.5^2 & + & 21.9^2 & + & . & . & . & + & 148^2 & + & 148^2 & + & 1.79^2 & + \\ 1.0^2 & + & 1.1^2 & + & 1.0^2 & + & . & . & . & + & 15.2^2 & + & 15.0^2 & + & 15.1^2 & + \\ 100^2 & + & 101^2 & + & 100^2 & + & . & . & .. & + & 1.4^2 & + & 1.5^2 & + & 1.3^2 & \end{bmatrix} - \frac{2012^2}{3\times5\times3}$$

$$= 235{,}949.72 - 89{,}958.75$$

$$= 145{,}990.97$$

2. Sum of squares for material types and properties (SS_A)

$$SS_A = \sum_{i=1}^{I=3} \frac{X_{i..}^2}{JK} - \frac{X_{...}^2}{IJK}$$

$$= \left[\frac{1789^2 + 200.5^2 + 22.50^2}{5 \times 3}\right] - \frac{2012^2}{3 \times 5 \times 3}$$
$$= 216{,}081.83 - 89{,}958.75$$
$$= 126{,}123.08$$

3. Sum of squares for percentage per weight of materials type (SS_B)

$$SS_B = \sum_{j=1}^{J=5} \frac{X_{.j.}^2}{IK} - \frac{X_{...}^2}{IJK}$$
$$= \left[\frac{239.20^2 + 340.40^2 + 455.10^2 + 482.8^2 + 494.50^2}{3 \times 3}\right] - \frac{2012^2}{3 \times 5 \times 3}$$
$$= 95{,}314.54 - 89{,}958.75$$
$$= 5{,}355.79$$

4. Sum of squares for material types and properties $\times$ percentage per weight of materials type interaction (SS_{AB})

$$SS_{AB} = \sum_{i=1}^{I=3}\sum_{j=1}^{J=5} \frac{X_{ij.}^2}{K} - \sum_{i=1}^{I=3} \frac{X_{i..}^2}{JK} - \sum_{j=1}^{J=5} \frac{X_{.j.}^2}{IK} + \frac{X_{...}^2}{IJK}$$

$$= \left[\frac{171^2 + 65.1^2 + 3.1^2 + ... + 445^2 + 45.3^2 + 4.2^2}{3}\right] - 216{,}081.83 - 95{,}314.54 + 89{,}958.75$$
$$= 235{,}865.53 - 216{,}081.83 - 95{,}314.54 + 89{,}958.75$$
$$= 14{,}427.91$$

5. Error sum of squares (SS_E)

$$SS_E = SS_T - SS_A - SS_B - SS_{AB}$$
$$= 145{,}990.97 - 126{,}123.08 - 5355.79 - 14{,}427.91$$
$$= 84.19$$

RESULTS AND DISCUSSION

The summary of the ANOVA for the computations is shown in Table 2

Table 2: ANOVA result for refractory bricks production

Sources of Variation	**Sum of squares**	**Degree of freedom**	**Mean sum of squares**	**F_{cal}**	**F_{tab}** $\alpha = 0.05$	**Decision**
Material types and properties (A)	126,123.08	(I-1) = 2	63061.54	$\frac{MS_A}{MS_E} = 22{,}441.83$	$F_{2,30,0.05} = 3.32$	$F_{cal} > F_{tab}$ Reject H_0

Percentage per weight of materials type (B)	5,355.79	(J-1) = 4	1338.95	$\frac{MS_B}{MS_E} = 476.49$	$F_{4,30,0.05} = 2.69$	$F_{cal} > F_{tab}$ Reject H_0
Material types and properties $\times$ Percentage per weight of materials type ($A \times B$)	14,427.91	(I-1) (J-1) = 8	1803.49	$\frac{MS_{AB}}{MS_E} = 641.81$	$F_{8,30,0.05} = 2.27$	$F_{cal} > F_{tab}$ Reject H_0
Error	84.19	IJ(K-1) = 30	2.81			
Total	145,990.97	IJK-1 = 44				

Statement of Hypothesis was:

H_0 : There is no differential treatment effect among the material types and properties

H_1 : There is differential treatment effect among the material types and properties

i. ***Examination of block effect of material types and properties***

$$H_0 : \alpha_i = 0, \ \mathrm{H_i} : \alpha_i \neq 0$$

Since F_{cal} = 22,441.83 > F_{tab} = 3.32, we lack sufficient evidence for us to accept the null hypothesis $H_0 \ @ \ \alpha = 0.05$. It was therefore concluded that the materials properties as well as the type of materials employed each have differential treatment effect on the quality of refractory bricks produced.

ii. ***Examination of treatment effect of percentage per weight of materials type***

$$H_0' : \beta_j = 0, \ \mathrm{H_j}' : \beta_i \neq 0$$

F_{cal} = 476.49 > F_{tab} = 2.69. This suggests that our experimental data do not provide enough proof for us to accept the null hypothesis H_0^1. The implication of this is that the differential treatment effect produced by the refractory brick characteristics in different experimental runs is largely due to the percentage per weight of materials type as well as the composition of the materials employed.

iii. ***Examination of material types and properties × percentage per weight of materials type* interaction**

$$H_0^{II} : (\alpha\beta)_{ij} = 0, \ \mathrm{H_j}^{II} : (\alpha\beta)_{ij} \neq 0$$

Since F_{cal} = 641.81 > F_{tab} = 2.27. It was concluded that the experimental analysis provide paucity of evidence to accept the null hypothesis H_0^{II}. This suggest that there seems to be interaction between the type of materials and the percentage per weight of the materials to significantly influence the quality of refractory bricks produced in this study. From the design of experiment in and the experimental analysis of the clays, the hypothetical 40/50%, 50/50% and an optimum 60/40% clay to sand ratio were strongly suggested to have better potential of producing fire bricks with the recommended significant characteristics of bulk density, apparent porosity, cold crushing strength and thermal resistance. Actual mix of clay and sand for production of good fire bricks could be predicted on the premise of the optimum 60/40% clay mix for different clays judging by the test characteristics of prepared samples as analyzed in Table 3. For the different clays with varying Alumina (Al) and Silica (Si), contents, the 60/40% optimum mix ratio may be achieved by increasing the % by weight of one constituent even up the mix ratio in the 60/40% by weight ratio. For example, 3 parts by weight of Ovia clay may be added to 1.8 parts by weight of sand to maintain the 60/40% optimum mix ratio. This mix ratio was arrived at after randomization.

Table 3. Clay to sand recommended mix ratio

Clays	Al/Si ration	Total weight (w) in %	Clay/sand ratio (w/97)%	60/40% multiplier factor
Ovia	29/58	87	1/1.11	3/1.8 (clay/sand)
Sand	3/94	97	-	-

Observation made from preliminary tests carried out on the bricks, showed noticeable defects and instability of some of the bricks that were prepared outside the boundary of the recommended mix ratio of clay and sand. Such bricks were eliminated leaving the better bricks for further testing to ascertain the suitability of characteristics for conventional fire bricks. Initial test bricks prepared with the sand/clay mixture are shown in Tables 4 and 5

Table 4 Test samples of prepared refractory bricks

Test samples	1	2	3	4	5	6	7	8	9
Material mix ratio (% wt.)	10/90	20/80	30/70	40/60	50/50	60/40	70/30	80/20	90/10
Initial observation from tests on plasticity, stability on drying	poor	Poor	Good	Good	Good	Good	Good	Poor	Poor
Remarks	No	No	Yes	Yes	Yes	Yes	Yes	No	No

Table 5 Selected refractory test bricks for further resting

Test samples	A	B	C	D	E	F-Imported
Clay/sand mix ratio (% wt.)	30/70	40/60	50/50	60/40	70/30	Anonymous

The refractory castable binder was kept constant at 10% of the entire mix, while water mix ratio was within recommended 8-15% when plasticity of the bulk mix of material was attained. This was within standard brick preparation procedure [14]. The output data of the experiment carried out on the bricks is shown in Table 6.

Table 6 Physical properties of brick test samples

Sample	Bulk density (g/cm^3)	Apparent Porosity (%)	C.C.S (kg/cm^2)	Shrinkage (%)	LOI(%)	Slag resistance	Thermal resistance
A	1.024210526	21.66302	148	4.76	8.124357657	poor	Good
B	1.333998669	11.86253	150	4.76	11.86034913	Good	Very good
C	1.796999117	9.204545	140	4.76	1.453831041	Good	Very good
D	1.865561694	9.010011	100	1.9	2.783810464	Good	Very good
E	1.392832045	15.20737	60	4.76	3.834510595	Good	Good

F	2.4	2.843602	311	1.9	0.672690763	Very good	Very good

Based on the outcome of the split-split plot experimental design carried out and rejection of the null hypothesis it was concluded that mix ratios of clay/sand of 30/70%, 40/60%, 50/50%, 60/40% and 70/30% respectively were good for refractory bricks with the optimum mix given as 60% clay and 40% sand. This was further validated by the experiment outcome of the physical tests of the bricks as shown in Table 6. It was also inferred that alumina from clay and silicate from sand are the major and significant constituents of a refractory brick materials as indicated in Table 3. The thermal resistance of the bricks suggests what aggregate mix ratio will be appropriate for a desired thermal facility such as a heat treatment furnace.

CONCLUSION

There are other constituents contained in sand and clay which are often regarded as impurities. They are present in trace quantities with insignificant effect on the major characteristics of refractory bricks depending on their percentage composition. The presence of high quantities of these impurities may sometimes necessitate beneficiation at an extra cost and time consumption. This research paper was focused on suing statistical analysis (Analysis of Variance) to predict mix ratio of clay sourced from Ovia in Edo state and sand for the production of fire bricks viable for use in heat treatment furnace.

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