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Physio-chemical properties of Tamiya clay deposit for production of insulating refractory bricks using mixture of rice husk and waste paper as additive

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Abstract

The study aimed at investigating impact of the processing parameters on the physico-chemical and mechanical properties of insulating refractory bricks made from Tamiya clay with admixture of rice-husk/paper-waste. The molding was done with a compression hydraulic press. The molding of the bricks was done by incorporating the varied proportions of the additive of the mixture of rice-husk/paper-waste (10%, 15%, 20wt %) into the clay matrix. The firing temperature varied from 850°C, 950°C, 1000°C. The results were optimized using Taguchi design of experiment method and the effect of the processing parameters (clay, additive, firing temperature) was established using the response Table and means effects plots. The properties of the produced bricks investigated after firing at 850°C, 950°C and 1000°C indicated that samples of mixture of rice husk and waste paper had percentage total apparent porosity increment ranging from 15.90%, - 26.1%. Similarly, samples of the bricks had cold crushing strength ranging from 2.04 MN/m^2 – 3.45 MN/m^2. The estimated refractoriness using Shuen's formula revealed 1554.612°C as a range of refractoriness while the result of the refractoriness using Pyrometric Cone Equivalent (PCE) indicated that samples had refractoriness of 1200°C. On the other hand, thermal insulation improved from 1.759531Wmk to 0.51WmK. Taguchi-based optimization experiment identified Tamiya clay content and temperature as the most considerable factors affecting the cold crushing strength. However, mixture of rice husk and waste paper and temperature are significant parameters influencing thermal conductivity.

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1. Introduction

The demand for cost-effective, energy-efficient, and environmentally friendly insulating materials in high-temperature applications has led to increased interest in the development of lightweight refractory bricks from natural clay deposits and agricultural waste additives. Refractory bricks are essential in furnaces, kilns, incinerators, and reactors due to their ability to withstand high temperatures without deforming or reacting chemically with surrounding media [1]. The physico-chemical properties of the clay

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used, such as plasticity, mineralogy, thermal behavior, and chemical composition, play a critical role in determining the suitability of the raw material for refractory applications [2].

Tamiya clay, a locally sourced kaolinitic clay deposit in Nigeria, is underexplored despite its promising alumino-silicate composition, which is essential for refractory applications. The systematic evaluation of its physico-chemical characteristics provides the foundational data required for its valorization in insulating brick production. Additionally, the integration of sustainable additives such as rice husk (a silica-rich agricultural byproduct) and waste paper pulp (a cellulose-based organic material), has shown potential in improving the thermal insulation and porosity of refractory bricks by acting as pore forming agents during firing [3].

These additives combust during firing, leaving behind voids that reduce thermal conductivity, thereby enhancing the insulating capacity of the brick. This approach not only promotes waste recycling but also aligns with global efforts toward sustainable materials and circular economy practices [4]. Several studies have explored the use of agricultural and industrial by-products to modify clay-based refractories, demonstrating improvements in both thermal resistance and structural performance [3, 5].

Similarly, Ref. [6] showed that the addition of rice husk ash into the mixture of raw clay leads to a significant improvement in thermal insulation and a reduction in thermal conductivity. Again, researchers have explored various approaches to incorporate waste paper into the manufacturing process of refractory bricks to improve their insulating properties and thermal performance [7]. Therefore, the study aims to investigate the physico-chemical properties of Tamiya clay and assess its suitability for insulating refractory brick production through the incorporation of rice husk and waste paper as eco-friendly additives. The outcome will contribute to the development of locally sourced, affordable, and sustainable refractory materials with improved insulating properties for industrial thermal applications.

2. Materials and methods

2.1. Raw material sourcing and preparation

The clay material used in this research work was obtained from tamiya deposit, Ussa LGA in Taraba State, rice husk was obtained from Samaru rice mill Zaria Kaduna State and waste paper was sourced from photocopying centers. The bricks production experimental design matrix was developed based on the L9 Taguchi orthogonal array (OA). The design is to determine the influence of clay content, additive content, and firing temperature (Table 1).

To simplify the experimental trials, the experimental results are transformed to Single-To-Noise Ratio, which is recommended to measure the quality characteristic deviating from the desired results. There are three categories of quality characteristics in the analysis of the S/N ratio, i.e. the-smaller-the-better, the-higher-the-better, and the-nominal-the-better.

Table 1: Design for insulating bricks.

Control factors	Unit	Level 1	Level 2	Level 3
A. Clay content	%	90	85	80
B. Additive content	%	10	15	20
C. Firing temperature	°C	800	950	1000

The insulating brick was produced using washed tamiya clay, with a mixture of two ground combustible materials (Rice husk and waste paper). The clay, combustible materials, and temperature were optimized using a Taguchi design of experiment to develop nine different insulating bricks by varying the combustible materials as follows: 10%, 15%, and 20%. The mixture of combustible materials used were rice husk and waste paper in the ratio of 1:1.

Refractory insulating bricks were made from the clay and a mixture of rice husk and waste paper. The rice husk and waste paper were sun-dried to remove moisture present and crushed into particulates, and mixed at the same ratio to obtain the mixture. Clay was milled and immersed in water for three days to form a slurry. Dirt and foreign substances were removed from the filtrate and decanted after three days. The remaining clay material was dried and ground into finer particles. The raw materials were ball-milled to powder and sieved using mesh sieves (212 to 300 μm). The prepared materials were measured and mixed with 50 ml of water (until a uniform consistency was achieved) in a bowl. The clay samples were blended with a mixture of rice husk and waste paper at various proportions of 10%, 15%, and 20% by weight. The test samples were shaped by compressing in a steel cylindrical molding device.

The composition of paper, rice husk, and clay where weighted and mixed to form a semi dry mixture then it is filled in a cylindrical mold, and insulating bricks were made by press forming method using the hydraulic press machine at the Laboratory of the Metallurgical and Material Engineering Department, Ahmadu Bello University, Zaria. The bricks were produced at a pressure of 1750 Psi. The bricks were dried for twenty-four (24) hours, and the values of the dimensions were taken before they were placed in an electric oven for drying at 110 °C for 24 hours. The dimensions were taken and recorded. After oven drying, the samples of the insulating brick were placed in a furnace, then fired at different temperatures (850 °C, 950 °C and 1000 °C). Held for 2 hours. The fired length and weight of each respective sample was measured and recorded for various tests to be carried out.

2.2. Chemical analysis

The major oxide composition of the Tamiya clay fraction was quantified using X-Ray Fluorescence (XRF) Spectrometry at the National Steel Raw Materials Exploration Agency, Kaduna.

2.3. Mineralogical analysis

The bulk mineralogy of powdered clay specimens was determined using XRD at the Metallurgical and Materials Engineering laboratory, A.B.U Zaria.

2.4. Bulk density

It is the weight per unit volume of pore spaces present in the material. The bulk density was calculated using equation (1).

$$\text{Bulk Density} = \frac{W1 \times \rho w}{W2 - S}\ (\text{g/cm}^3), \tag{1}$$

where W1 = Weight of dried sample, S = Weight of sample suspended in water, W2 = Weight of soaked sample suspended in air, ρw = density of water.

2.5. Apparent porosity

The samples were dried in oven for twenty-four (24) hours at 110°C and heated to the various sintering temperatures. The digital weighing balance was used to obtain weights of the dried samples (W1) of the sintered sample bricks. Then the sample bricks were immersed in boiling water for two (2) hours, and afterwards allowed to cool to room temperature while still immersed in water. The test samples were suspended in water and their weights (S) were taken with the aid of a spring balance. The samples were removed from water, cleaned lightly and weighed in air to obtain the saturated weight (W2). The apparent porosity can be calculated using equation (2).

$$\text{Apparent porosity} = \frac{W2 - W1}{W2 - S} \times 100, \tag{2}$$

where W1 = Sample dried weight, S = Weight of the sample suspended in water, W2 = Saturated weight (weight of samples Soaked in water and suspended in air).

2.6. Shrinkage on firing

The test piece of the refractory clay materials was made into rectangular shape of dimension 13.5 × 2.5 × 2.5 cm in a mold and compacted under a hydraulic pressure of 1750 Psi. A slanted line of length 10 cm was inserted diagonally on each piece and recorded as (L1). The test pieces were then place inside the furnace and fired up to 100 °C and the line drawn across the diagonal axis of the pieces was measured to determine its final length (L2) after firing. The linear shrinkage of the materials was determined using the equation:

$$\text{Linear shrinkage (\%)} = \frac{L1 - L2}{L1} \times 100. \tag{3}$$

2.7. Cold crushing strength of refractory clay

A uniform load was applied on the cylindrical-shaped samples. The load or force at which a crack appears on the brick sample (maximum load) was noted. The CCS was obtained from the formula in equation (4) the test result was then used to Calculated using the formula:

$$\text{CCS} = \frac{\text{Load applied (kg)}}{\text{area of sample (cm}^2)}. \tag{4}$$

2.8. Thermal shock resistance

Test samples of refractory bricks were thoroughly dried and placed in the cold furnace heated at the rate of 5 °C/minute until the furnace temperature reads 1000°C this temperature was kept for 30 minutes after which the test samples were removed with a pair of tong and placed in the environment free from draught and allowed to cool down for about five minutes. The cycle was repeated several times before the thermal crack occur.

2.9. Thermal conductivity

Thermal conductivity test was conducted in Physics Department Ahmadu Bello University Zaria. This test was based on IEEE standard 98-2002 with some slight modifications. The quantity of heat which passes through a homogenous wall was determined using the formula:

$$Q = qF = \lambda F \frac{tw1 - tw2}{\delta} w, \tag{5}$$

$$\lambda = \frac{Q\delta}{F(tw1 - tw2)} \text{ W/m.k}, \tag{6}$$

where λ= Thermal Conductivity (W/m.°C), F= Surface area (m^2), δ= Thickness (m), $tw1$ and $tw2$ = boundaries surfaces of the wall at temperatures tw1 and tw2, Q= Coefficient of thermal conductivity (W/m°C).

3. Result and discussion

3.1. Chemical analysis

Table 2: XRF analysis (concentration in % wt oxides) for Tamiya clays.

Parameters	Clay oxides composition
SiO_2	58.224
Al_2O_3	18.226
Fe_2O_3	14.433
TiO_2	2.203
CaO	1.066
ZrO_2	0.145
MnO	0.071
K_2O	4.276
LOI	0.125
Other oxides	1.231

The chemical analysis of Tamiya clay samples was conducted using X-ray fluorescence (XRF) spectroscopy. The Tamiya clays were found to be predominantly silica (SiO_2 and alumina (Al_2O_3), with concentrations of 58.224% and 18.226%, respectively (see Table 2). Similarly, Ref. [8] revealed Ikere-Ekiti clay to have concentrations of 46.28 and 27.71 % for silica (SiO_2) and alumina (Al_2O_3), respectively. Additionally, notable levels of iron oxide (Fe_2O_3), averaging 14.433%, were detected on the Tamiya clay sample; concentrations of other major oxides, including TiO_2, CaO, ZrO_2 and K_2O, were relatively minor, altogether constituting less than 10% of the average composition. The loss on ignition (LOI) values were consistently below 10.25%.

3.2. XRD phase diagram Tamiya clay

The XRD result in Figure 1 shows the phases present in the clay are mostly high Cristobalite which is high silica phase, pyroxene-ideal phases which have a general formula $XY(Si,Al)_2O_6$ where X represents Ca, Na, Mg, Fe(II),and more rarely Zn, Mn or Li, and Y represents ions of smaller size, such as Cr, Al, Mg, Co, Mn, Sc, Ti, V ore even Fe(II) or Fe(III), the pyroxene-ideal;cristobalite high;magnesiowustite; magnesiowustite phase which is a compound phase where magnesiowustite is a significant host phase for transition metals. Although Nickel is compactable in Magnesiowustite. Finally Pyroxene-ideal; stishovite;stishovite phase where Stishovite is an oxide of silicon bounded with six oxygen which adopts an octahedral coordination geometry Stishovite has resemblance with Rutile (TiO_2) the phases informs the various oxide that are found in Tamiya clay deposit [9].

3.3. Physical properties

Physical testing reveals the physical properties possess by the raw Tamiya clay (Table 3). It was seen from Table 3 that the bulk density value of the clay was 2.0293 g/cm^3. Bulk density of refractory brick is influenced by some oxides like CaO and MgO. They lead to the formation of chemical components of low fusion temperature which would eventually flow into the pores and result in an increase in the density of the brick. it is also seen that the clay had apparent porosity value of 15.90% w. This showed that Tamiya clay is more moderately porous. Apparent porosity means proportion of voids or pores per unit volume of the material. It depends on clay mineralogy and internal structure. The apparent porosity of the samples as a function of additives and the firing temperature is indicated in Table 4. The samples with the legend M, M1, M2 and M3 represent additives in weight percentage (wt. %) 0, 10, 15 and 20 wt. % of mixture of rice husks and waste paper respectively. The results showed that the porosity increased from 15.90% to 26.1% with an increase in the composition of mixture of rice husk and waste paper at 1000°C. This is due to the fact

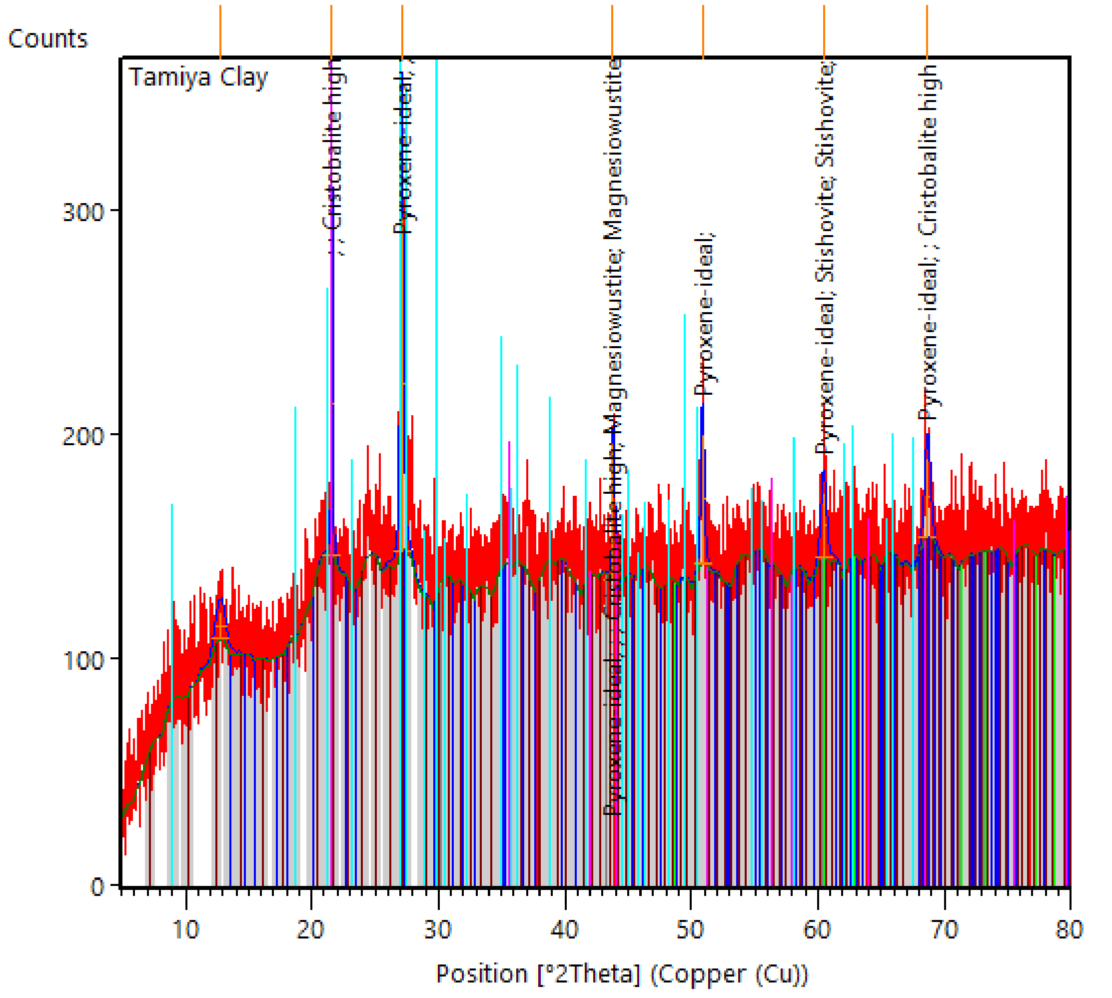

Figure 1: XRD pattern for Tamiya clay.

Table 3: Result of physical properties of Tamiya clay.

Test	Clay oxides composition
Bulk density	2.0293 g/cm^3
Apparent porosity	15.90%
Water absorption	55%
Plasticity index	24.52%
Shrinkage on firing	2%
Thermal shock resistance	20 cycles
Loss on ignition	0.125%
Thermal conductivity	1.759531W/mK
Refractoriness	1554.612°C
Cold crushing strength	2.7 MN/m^2

that the mixture of rice husks and waste paper escaped during firing and create pores in the brick [8]. However, the thermal shock resistance value of the clay is mildly 20 cycles. It has been reported that the presence of oxides like phosphorus oxide improves thermal shock of clay material [10]. Therefore, absent of the phosphorus oxide is responsible for the low resistance. Again, Tamiya clay was fired to the temperature of 1200 °C and shrinkage on firing recorded was 2% which is considered low, indicating that the clay bricks would have minimal shrinkage after firing. This is desirable, leading to high quality bricks, minimal cracking, better dimensional stability and improved strength [11]. Atterberg limit tests conducted on the Tamiya clay sample reveal liquid limits of 48.56%, plasticity limits of 24.04%, and plasticity index value of 24.52%. Based on the Casagrande plasticity chart classification.

Table 4: L9 design of experiment.

Clay	RH&PW (M)	Temp (°C)	CCS (MN/m^2)	P (%)	TC (W/mK)	SNRA1	SNRA2
80	10	850	2.04	19.1	0.54	6.1926	5.35212
80	15	950	2.06	20.6	0.51	6.2773	5.84860
80	20	1000	2.20	26.1	0.52	6.8485	5.67993
85	10	850	2.36	14.2	0.53	7.4582	5.51448
85	15	950	2.62	22.4	0.53	7.4582	5.51448
85	20	1000	2.14	18.3	0.53	8.3660	5.51448
90	10	850	3.45	15.6	0.54	6.6083	5.35212
90	15	950	3.35	18.0	0.53	10.7564	5.51448
90	20	1000	2.30	22.2	0.52	7.2346	5.67993

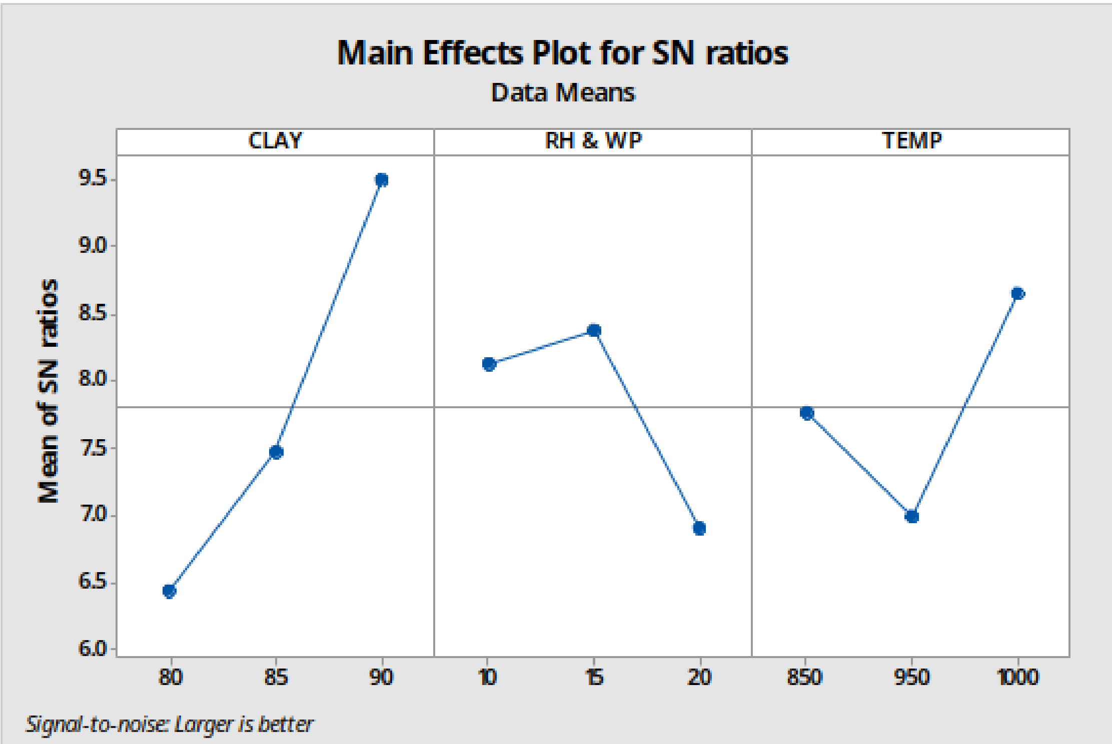

Figure 2: SN plot for cold crushing strength (MN/m^2) versus clay, RH & WP, temp.

Table 5: Response table for signal to noise ratios (larger is better).

Level	Clay (A)	RH&PW (B)	Temperature (C)
1	6.439	8.136	7.767
2	7.478	3.381	6.990
3	9.497	6.897	8.657
Delta	3.058	1.484	1.667
Rank	1	3	2

Thus, the clay (Tamiya clay) has high degree of plasticity meaning it can be easily molded and shaped when moist, and retains its shape when dried. The refractoriness estimates derived from the bulk chemical composition of the Tamiya clays was 1554.612°C. This refractoriness temperature signifies the Tamiya deposits comprise of minerals with elevated melting points, including quartz, kaolinite and hematite [11]. Similar physical properties fot otukpo clay were highlighted by Ref. [12].

On the other hand, the cold crushing strength and thermal conductivity of the clay were found to be 2.7 MN/m^2 and 1.759531W/mK respectively. Accordingly, Table 4 demonstrated the impact of composition and sintering temperature on the cold crushing strength (CCS) of mixture of rice husk and waste paper brick samples. It is observed that the cold crushing strength at 10% show maximum strength of 3.45MN/m^2 at 850°C, this agrees with Ref. [13], which state that compressive strength decrease with increase rice husk

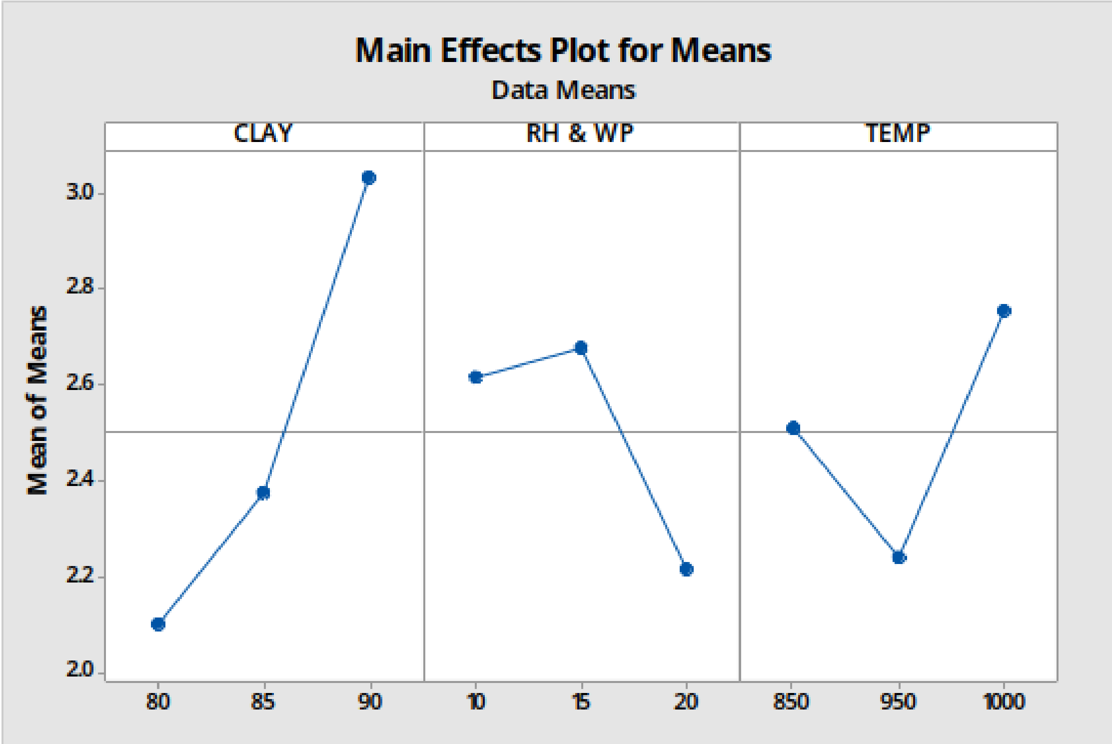

Figure 3: Mean plot for cold crushing strength (MN/m^2) versus clay, RH & WP, temp.

Table 6: Response table for means.

Level	Clay (A)	RH&PW (B)	Temperature (C)
1	2.100	2.617	2.510
2	2.373	2.677	2.240
3	3.033	2.213	2.757
Delta	0.933	0.463	0.517
Rank	1	3	2

Table 7: Response table for signal to noise ratios (smaller is better).

Level	Clay (A)	RH&PW (B)	Temperature (C)
1	5.627	5.406	5.460
2	5.514	5.626	5.681
3	5.516	0.5233	5.516
Delta	0.112	0.220	0.221
Rank	3	2	1

Table 8: Response table for means.

Level	Clay (A)	RH&PW (B)	Temperature (C)
1	0.5233	0.5367	0.5333
2	0.5300	0.5233	0.5200
3	0.5300	0.5233	0.5300
Delta	0.0067	0.0133	0.0133
Rank	3	1	2

addition, because higher porosity and low bulk density, for rice husk ash addition of 2% by weight shows maximum compressive strength of 6.20 MPa . This can be attributed to the fact that an increase in porosity led to a decrease in load-bearing capacity, resulting to a decrease in the CCS [13]. Additionally, the thermal conductivity values (Table 4) of the bricks containing mixture of rice

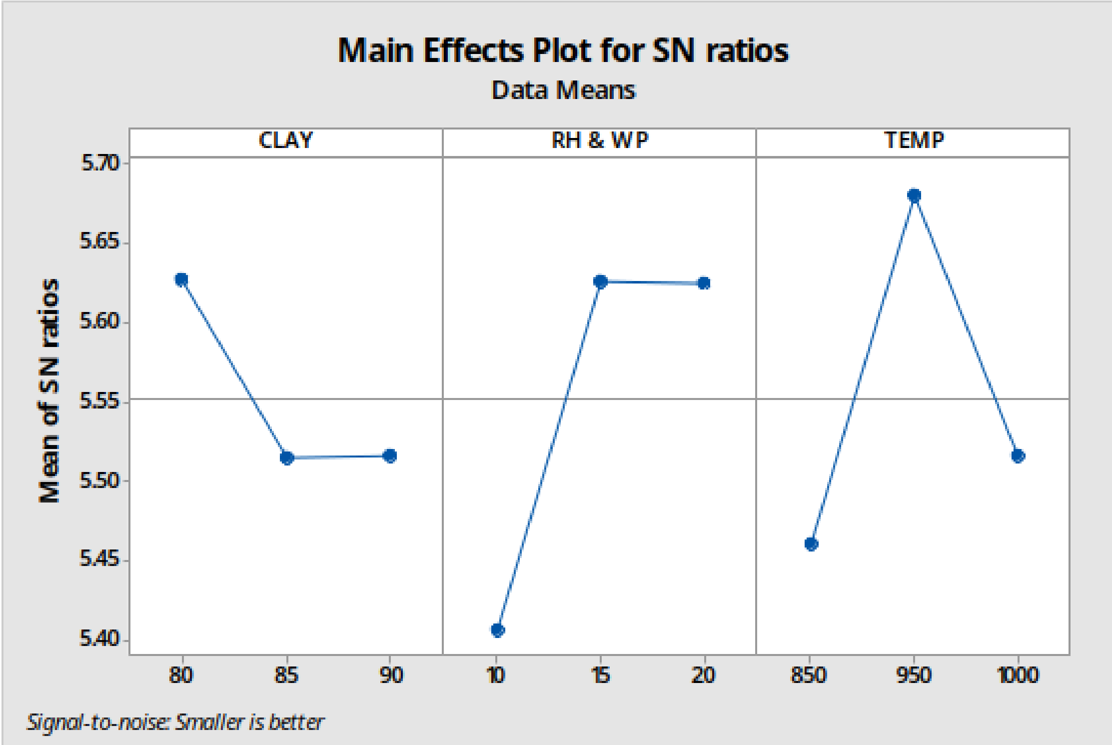

Figure 4: SN plot for thermal conductivity (W/mK) versus clay, RH & WP, temp.

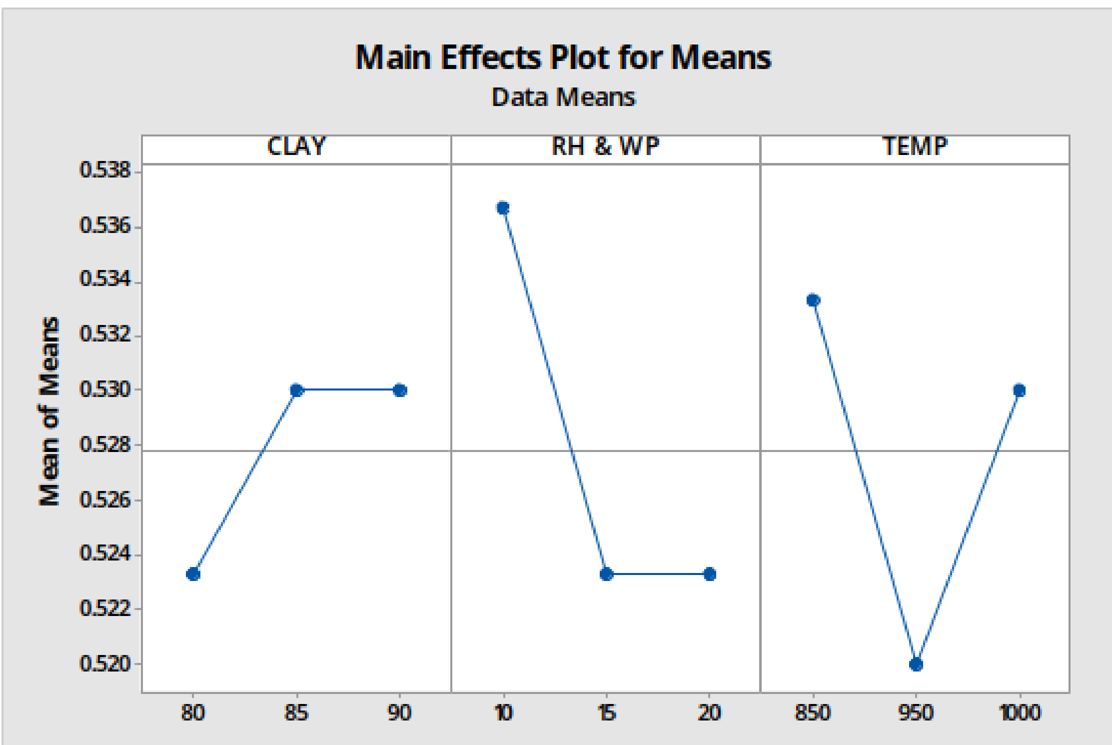

Figure 5: Mean plot for thermal conductivity (W/mK) versus clay, RH & WP, temp.

husk and waste paper shows that the thermal conductivity reduces from 1.759531 W/mK at 0 wt% mixture of rice husk and waste paper (M) to 0.51 W/mK at 15 wt% mixture of rice husk and waste paper and 80wt% clay (M2) at 950°C. This could be a result of the pores created by the mixture of rice husk and waste paper on the bricks during sintering. These pore spaces or voids present in

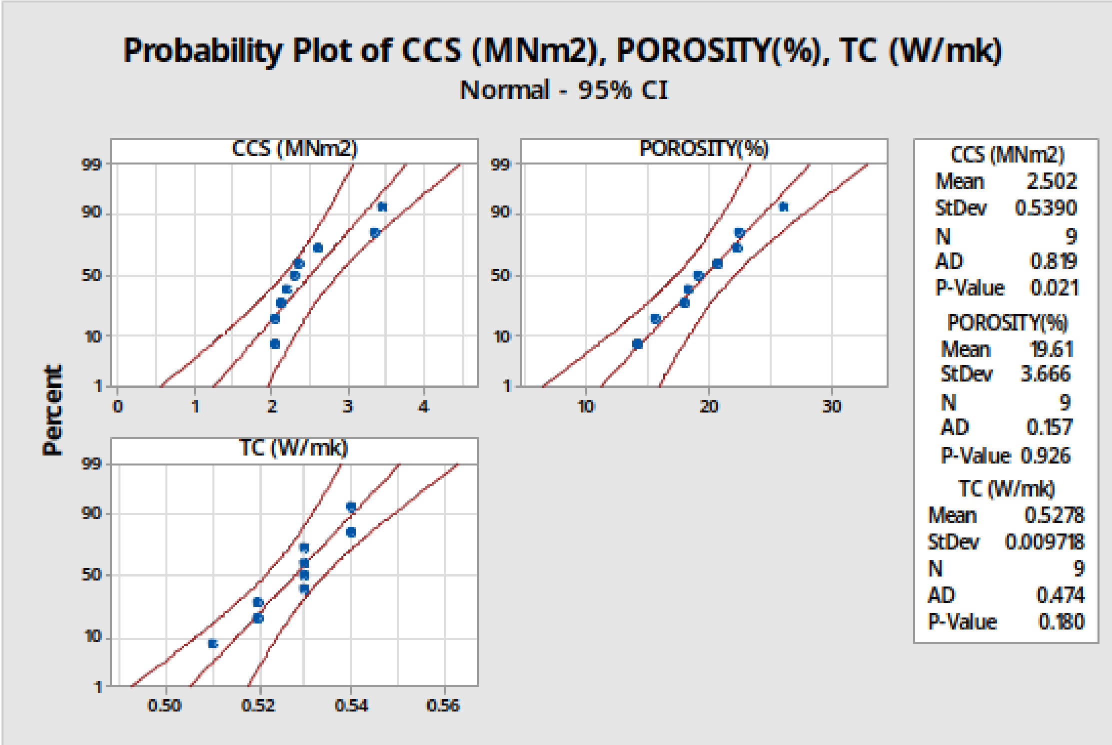

Figure 6: Probability plot of CCS (MNm^2), porosity (%), TC (W/mk).

the samples hinder thermal flow, which results in low thermal conductivity of the bricks. This agrees with Ref. [14] in their research that shows that the thermal conductivity reduces with an increase in melon seed husk [14].

3.4. Result of Taguchi analysis

From the Taguchi-based plots (Figures 2-5), the cold crushing graph shows that the optimal composition for achieving better strength was at 90% clay content, 15% mixture of rice husk and waste paper at a temperature of 1000 °C. The response tables (Tables 5-6) for larger is a better shows mean ranking on the order of the most significant factors that mostly impart on the cold crushing strength of the insulation bricks, as follows Clay, temperature and mixture of rice husk and waste paper, respectively. The thermal conductivity graphs show that the optimum composition that exhibits better insulation properties was at 80% clay, 20% mixture of rice husk and waste paper at temperature of 950 °C. The response tables (Tables 7-8) for smaller is better indicate the ranking of the order of the most significant factor that mostly contributes to improving the thermal conductivity of the insulation bricks. The order is a mixture of rice husk and waste paper, temperature, and then clay. Likewise, the probability plot (Figure 6) displays that the data follows a normal distribution with a 95% confidence interval.

4. Conclusion

In conclusion, this research work shows an improvement in the thermal insulation properties of Tamiya clay bricks from 1.759531 to 0.51 W/mK. The Taguchi experiments identified Clay and Temperature as significant factors affecting response variable Cold crushing strength. While mixture of additives and temperature as significant factors affecting the response variable thermal conductivity. It was found from the Taguchi experimental trials that the porosity of refractory insulating bricks increases with increase in additives and increase in temperature. Therefore, if mild compromise is made in porosity and thermal conductivity, an efficient brick could be produced at production parameters of 90% clay, 10% RH&PW and firing temperature of 850°C. Notwithstanding, a blend of the Tamiya clay deposit with alumina-rich clay deposit to improve the refractoriness of the insulation bricks can be recommended in the future research.

Data availability

Data will be made available upon reasonable request from the corresponding author.

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