Greener Journal of Science, Engineering and Technological Research
ISSN: 2276-7835
Vol. 14(1), pp. 18-27, 2025

https://gjournals.org/GJSETR
DOI: https://doi.org/10.15580/gjsetr.2025.1.091625136

The Effect of Rice Husk Ash and Pulverized Broken Bottle on Composite Material Developed from Gula Clay.

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ABSTRACT

This work focused on the influence of rice husk ash and pulverized broken bottle on composite material developed from Gula clay. Gula clay composite samples were prepared with varying proportions of broken bottles and rice husk after which they were subjected to test in order to ascertain their water absorption, compressive strength, linear shrinkage and thermal capacities. From the results as that were obtained from the work, it was found that the inclusion of broken bottles and rice husk significantly improves the thermal properties of Gula clay, with optimal reinforcement levels which include the enhancement of the strength of the composite material that resulted in the reduction of weight without compromising compressive strength, coupled with very low linear shrinkage as observed from all the samples which is an indication that composite materials can offer a sustainable alternative means to traditional building materials and can also offers good application in muffle furnace lining, coupled with its contribution in the waste reduction management that invariably offers good means of promoting environmental friendliness culminated by construction practices.

ARTICLE'S INFO

Article No.: *091625136*
Type: *Research*
Full Text: *PDF, PHP, HTML, EPUB, MP3*
DOI: ***10.15580/gjsetr.2025.1.091625136***

Accepted: *27/09/2025*
Published: *07/10/2025*

Keywords: *Gula clay, effect, Composite material, pulverized, broken bottles, rice husk, Thermogravimetry, compressive strength*

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Article's QR code

INTRODUCTION

Gula a village located in Shaffa, Hawul local government area of Borno State, with latitude 10˚N and longitude 12˚ E. this place is blessed with a lot of mineral resources. Clay is a product of geological weathering of the earth's surface, it has aluminum oxide (Al_2O_3) and silicon oxide (SiO_2) [15]. Clay has been very relevant in the modern material science, due to its ability to form composites with other materials, thereby forming a product with an improved functionality [9]. Clay in its raw form is porous which affects strength and density because higher porosity leads to reduced strength [2]. Clay is biodegradable and has low carbon footprint compared to synthetic materials [5]. Rice – husk composed mainly silica that can enhances strength and durability of clay composite. Rice husk is a byproduct of rice milling that is usually in abundance, give thermal insulation, and reduce environmental pollutant [11]. Broken bottles are typically made up from sand (silica) which is known for good strength, and hardness. Past studies have demonstrated potential of use of rice – husk as a reinforcement in many composite materials, including polymer, concrete and clay - based composites [13] and [9], Studied the thermal conductivity of clay bricks reinforced with rice – husk. Locally sourced Gula clay has natural plasticity and ease of molding, making it relevant for local use, but it requires modification to meet the modern usage requirement. Combining Gula clay with broken bottle and rice husk, this is intended to explore an innovative approach to create sustainable composite materials that align with global environmental and economic purposes. The use of these reinforcements did not only improve the materials properties but also addresses the challenges of waste management. Reducing porosity in bricks leads to reducing inter – particle distance leading to enhanced bond between particles [13], [4] posited that adding rice – husk ash to clay, increases its compressive strength by 20 – 30%. Fire – resistance properties of clay bricks was reported to improve by addition of rice – husk ash to clay brick [15]. According some the research work that have been carried on composite materials that involve clay, the understanding the specific properties of the clay soil and its suitability for different applications has been found to be crucial for successful usage in different industries is very important for national development [18]. And research has shown that the addition of reinforcements (rice husk ash) reduces shrinkage due to its ability to fill voids and prevent too much moisture loss [17], [16] using waste glass will not only reduce the volume of waste. Waste glass recycling enhances the durability of clay and enhances sustainable goals [14], and it has been proven that inclusion of quartz also helps to enhance the strength and longevity of structures [10] thus thereby contributing to its application in various areas suffice the it could also contribute in reducing environmental waste pollution cause by construction industry as it involves resource materials that contribute in environmental waste since it promotes the use of recycled materials, reducing the need for energy – intensive processes typically associated with the production of conventional building materials [12]. Waste glass powder and other additives generally contribute in reducing porosity in fired clay material due their incorporation as additives Waste glass powder addition into clay in particular was reported to reduce porosity, water absorption with the compressive strength increase proportionally to percentage of waste powdered bottle and rice –husk ash

In this research therefore focuses on influence of pulverized broken bottle and rice-husk ash on the Gula clay in order to ascertain how these additive could affect the properties of composite material developed by blending the Gula clay with waste glass powder and other additives with the duo in order to know if they will enhance the composite material amenability for application in refractory lining or other construction materials by improving the characteristic properties.

MATERIALS AND METHODS

Collection of materials and sample Preparation

Materials used are Gula clay, rice-husk and waste broken bottles. The clay was shoveled from the ground surface from Gula in Shafa, Hawu L.G.A of Southern Borno, Borno State, Nigeria. water was added to the clay, stirred and left unperturbed for three (3) days. The water was poured off leaving behind the clay. 1mole of oxalic acid solution was poured on the washed clay, stirred properly, allowed to stay for 1hour, decanted, and washed with water. The dried clay lumps were crushed and milled in mortar while waste glass bottles were packed from shops, washed, sun-dried crushed, milled and sieved. The rice – husk collected from rice – mill was pulverized.

Washed Gula clay with varying amount of 5%,10%,15%,20% and 25%, at constant quantity of 10% powdered waste broken bottles was mixed with water. The powdered waste broken bottles, pulverized rice – husk in mechanical mixer, the slurry was molded into cylindrical shape using compression molding machine at 10MPa. Water was added to the mixture at water to clay ratio of 7: 20. The green compacts produced were left undisturbed for 24hours, then oven dried for 12 hours at a temperature of 110℃, in order to remove moisture and other volatile matters. The oven – dried compacts were then fired in an electric muffle furnace at 7℃/min until temperature of 1150℃was attained. This temperature was maintained for four (4) before permitting the samples to cool to room temperature in the furnace. The compacts are of dimension Ø 30mm by 40mm.

Compressive Strength

This test was carried on the selected samples to determine its load bearing capacity in line with Adeolu et al., 2021 procedure, using universal testing machine. Where the samples were placed one after the other on the horizontal between the plates of the machine and a load of 10 kg / mins was applied. the compressive strength, was calculated, maximum load at failure was recorded.

Thermogravimetric Analysis of Modified Clay

Thermogravimetric analysis of the produced clay samples was carried – out with the thermal analysis machine. Powdered sample was placed in an alumina crucible. A constant heating rate of 5℃ / mins was applied from 20℃ to 1000℃ under nitrogen atmosphere with a flow rate of 50 mL/ mins.

Recording the temperature dependence of mass loss (TG), the first – order differential curves of mass loss to temperature (DTG) were determined. The decomposition temperature (Td) was taken at the maximum of the DTG peaks. The mass loss was calculated from the area of the peaks exhibited by the DTG curves. The standard deviation for Td and residual mass are ± 2℃ and $\pm 3\%$ respectively.

Preliminary Test on Materials Used

These were carried – out to examine the porosity, linear shrinkage and volume shrinkage. Chemical composition of the materials was analyzed [2]. Element composition of clay was determined using the x – ray fluorescence (XRF) and the mineralogical phases of the samples were also determined using x – ray diffractometry (XRD).

Linear Shrinkage (%): the difference between the initial heights of the samples taken and the final heights after sintering expressed in % is the linear shrinkage.

$$\text{Linear shrinkage} = \frac{initial\ height - Final\ height}{initial\ height} \times 100$$

Volume shrinkage (%): the volumes of the cylindrical samples were calculated, using the initial diameter and final diameter of the cylindrical samples after sintering.

$$\text{Volume shrinkage} = \frac{initial\ volume - final\ volume}{initial\ volume} \times 100$$

Volume of cylinder = $\pi r^2 h$

Water Absorption (%): this measured the amount of water or liquid the refractory materials can absorbed under a certain condition or the measurement of the % water required or necessary to be added to aggregate to get a saturated surface.

Cold Compression Strength: the compressive strength analysis was carried out to determine the material strength under a certain load to failure. The tests were performed on Instron 1195 at a fixed crosshead speed of 10mm minutes. The sample was prepared according to ASTM C862 – 93 and cold crushing strength and modulus of elasticity were calculated using equation below

$$\text{Cold compression strength} = \frac{Load\ of\ Fracture}{Surface\ area\ of\ sample}$$

X – Ray Diffraction Analysis (XRD): The samples were prepared for XRD analysis using a back loading preparation method (Degamos et al., 2013). The selected samples were analyzed using a PANalytical X'Pert Pro powder diffractometer with X'Celerator detector and variable divergence and receiving slits with Fe filtered Co – Ka radiation. The phases were identified using X'Pert Highscore plus software. The receiving slit was placed at 0.040°. the counting area was from 5 - 70° on a 20 scale. The count time was 1.5s. Anton paar HTK 16 heating chamber with Pt heating strip was used to obtain the temperature – scanned XRD data. [8], [6], [3]. While ARL 9800XP spectrometer was used for the analysis of the elemental composition of the samples was determined. The clay samples were milled and mixed with lithium tetra borate for chemical analysis. The measurement of ignition loss was done using calcination method.

RESULTS AND DISCUSSIONS

The elemental composition of clay samples was determined using X-ray fluorescence with spectrometer. The clay samples were leached with chemical solutions of oxalic acid and sodium diathonide respectively, with the aim of measuring iron reduction in the clay, and understanding the percentage of element that causes higher refractoriness in the clay, which is more of our concern about the clay.

The X-Ray fluorescence result is shown Table 1 for the pure clay and the clay leached with two different chemicals.

Table 1: Show the X-Ray fluorescence results for the pure clay and the clay leached with two different chemicals.

	Pure Gula Clay	**Clay Dissolved in Oxalic Acid**	**Clay Dissolved in Sodium Diathonide**
SiO_2	83.685	79.136	84.449
Al_2O_3	16.322	14.406	15.410
Fe_2O_3	3.006	2.640	2.893
TiO_2	Not Present	Not Present	Not Present
% FeO_2 Reduction	0.00%	12.18%	3.65%

XRF results of the as-received Gula clay, Gula clay leached with oxalic acid and sodium diathonide solution. From Table 1, oxalic acid was found to be more effective in reducing Fe_2O_3 (12.18% reduction of Fe_2O_3) compared to sodium diahonite (3.65% reduction of Fe_2O_3). Based this preliminary result obtained, oxalic acid was chosen in treating the Gula clay used in this research. By reducing the iron content in clay helps to improve the warping resistance of the clay during firing, improves colour stability and its compressive strength [3].

XRD Results

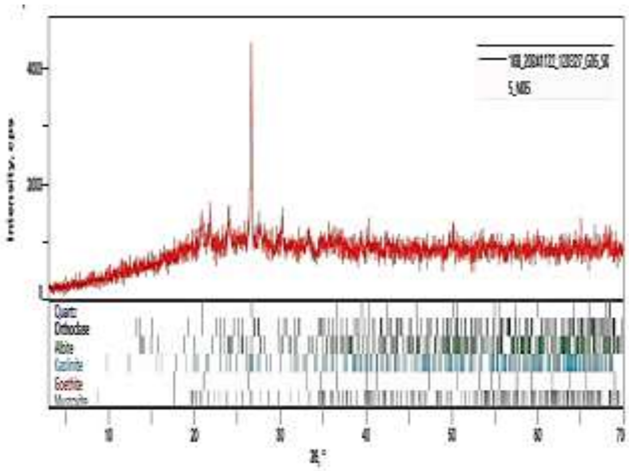

Fig1a: Broken **bottles 10%, rice husk 25%)**

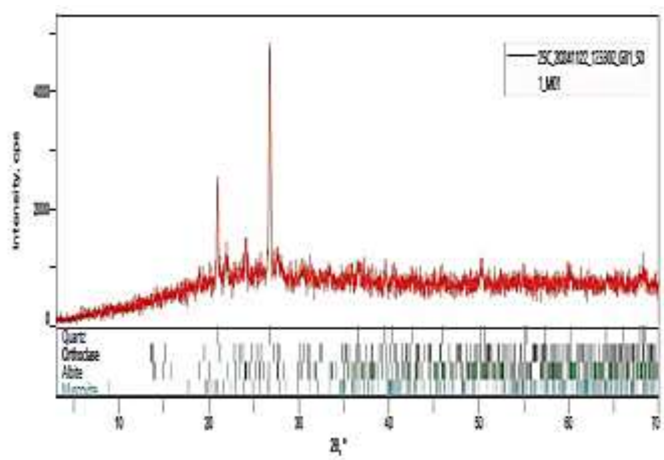

Fig.1b: Broken bottles 10%, rice husk 10%)

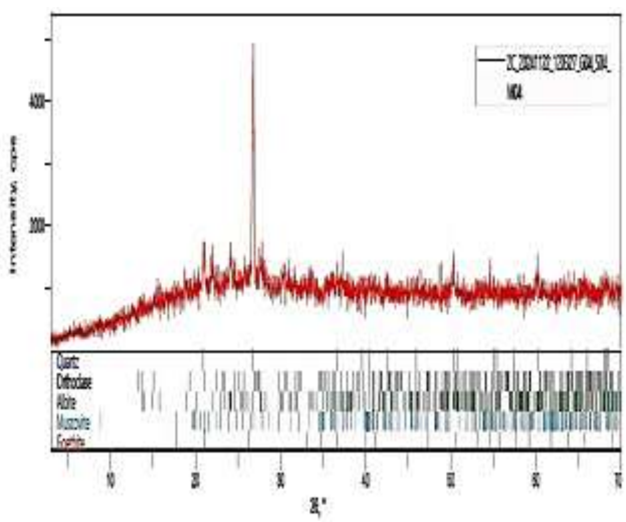

Fig.1c: Clay 90%, broken bottles 10%)

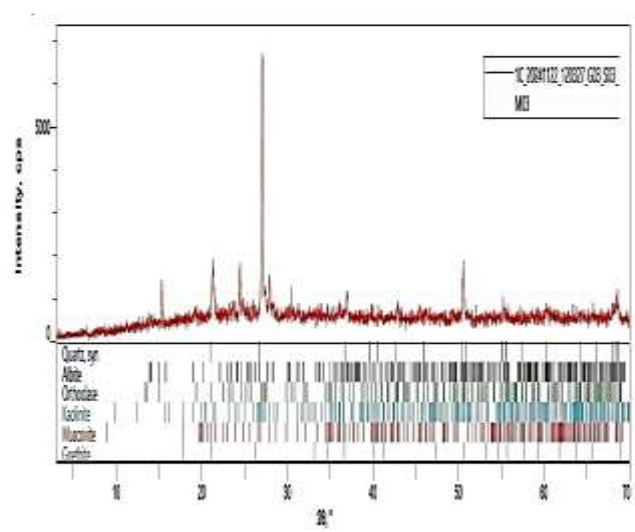

Fig.1d: Clay 100%)

XRD was employed to assess the crystalline structure of the composite. The XRD patterns revealed the following:

Table 2: Shows the Phases present in the Samples

Phase composition (%)	100% clay	10% broken bottle + 90% clay	10%Rice-husk +10% broken bottle	25%Rice-husk +10% broken bottle
Quartz	62	57	55	61.5
Orthoclase	10	18	26	17.9
Albite	16	2	1.34	10
Muscovite	1	14	3.2	10.5

The results indicate that Quartz is the predominant mineral in all samples, with its weight fraction ranging from 55% to 62%. This suggests high silicate content, which is typical for many geological formations. Sample 100% clay has a significant amount of Albite and Orthoclase, indicating the presence of feldspar minerals, while quartz maintains the highest content. The presence of Kaolinite and Muscovite suggests some degree of weathering and alteration. Sample 10% broken bottle shows a higher proportion of Orthoclase and Muscovite, with a notable amount of Goethite, which could mean iron oxide presence and potential oxidative conditions. Sample 10% rice - husk has the highest Orthoclase content among the samples, with a moderate amount of Kaolinite and Goethite, suggesting a different mineralogical environment or alteration process compared to the other samples. Sample 25% is similar to Sample 100% clay in terms of Quartz and Albite content but has a higher. Four phases were found in all the selected samples. In the selected samples analyzed, 10%BB, 10%RH, 25%RH and 100%GC, the orthoclase was increased from 10% - 26% (160%). For sample with 10%RH, (10 – 17.9) for sample with 25RH, 10 – 18.2%, for samples with 10% BB. It was observed that orthoclase phase which increase compressive strength of clay is found in the clay with broken bottles, and rice – husk. The optimum amount of orthoclase is produced sample with 10% RH + 10% BB. From the XRD result obtained, as the quantity of rice – husk increased to 25%, the value reduced drastically.

The SEM Analysis

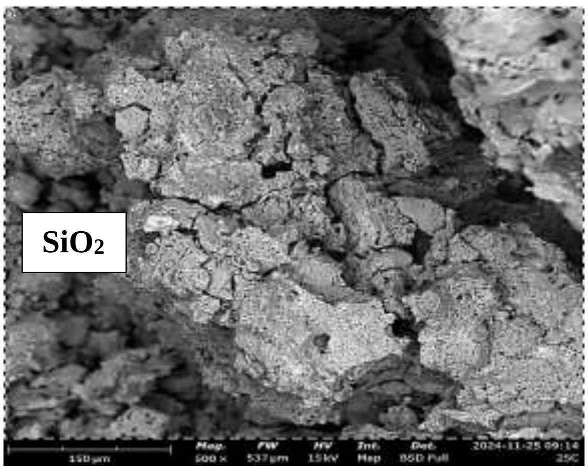

Plate1a: Broken Bottles 10%, Rice Husk 25%

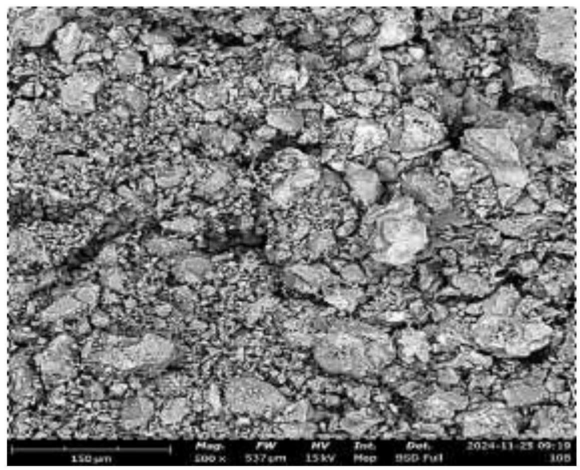

Plate**1b**: Broken Bottles 10%, Rice Husk 10%)

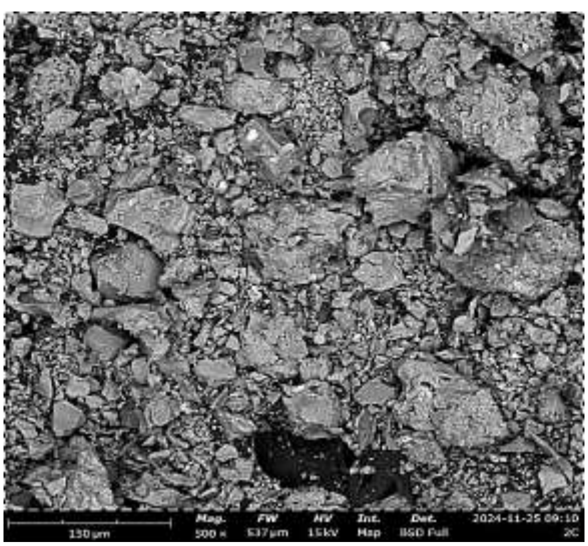

Plate1c: Clay 90%, Broken Bottles 10%)

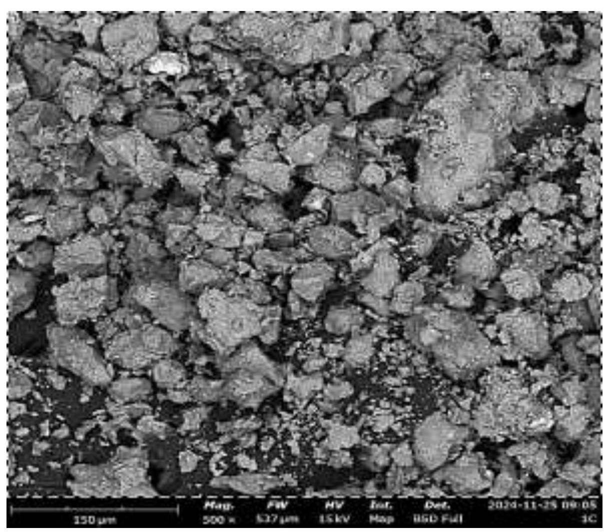

Plate1d: Control Sample (Clay 100%)

This reveals a predominantly clay-based matrix with significant concentrations of Silicon (Si, 48.57%) and Aluminum (Al, 20.96%), which are the primary constituents of clay minerals. Iron (Fe, 18.48%) and smaller amounts of Potassium (K, 4.75%) and Titanium (Ti, 2.38%) contribute to the composition, indicating a typical structure of raw clay. The surface morphology is homogeneous with minimal inclusions, reflecting the absence of reinforcements.

The inclusion of 10% broken bottles results in an increased Silicon content (Si, 52.92%), highlighting the contribution of the silicate-rich glass. Aluminum content (Al, 18.58%) decreases slightly, while Sodium (Na, 5.17%) and Calcium (Ca, 4.15%) show increased levels, due to the glass composition. The SEM images exhibit a more compact structure with visible glass inclusions, which enhance the matrix's structural integrity.

Adding 10% rice husk introduces organic and ash-derived components, leading to higher concentrations of Iron (Fe, 21.44%) and Potassium (K, 5.46%). Silicon (Si, 49.61%) remains high due to the combined contributions from clay and broken bottles. The SEM morphology reveals rice husk-derived pores or voids, providing insight into increased surface roughness and potential void formation due to organic combustion during processing.

The 25% rice husk content results in the most pronounced variation, with Silicon (Si, 46.51%) and Aluminum (Al, 20.66%) slightly reduced compared to previous samples. Increased Sodium (Na, 4.07%) and Magnesium (Mg, 2.69%) indicate ash contributions from the rice husk. The SEM analysis would reveal a more porous structure with visible husk fragments, reflecting the higher organic content and potential challenges in achieving homogeneity. The addition of broken bottles enhances compactness and structural stability, while rice husk contributes to porosity and surface irregularities. Higher rice husk content may reduce overall cohesion within the composite, suggesting a tradeoff between weight reduction and mechanical integrity.

Thermogravimetry Analysis of the Samples

The thermal stability of the clay samples was evaluated using Thermogravimetric Analysis (TGA). The TGA curves reveal the weight loss behavior of the samples as a function of temperature, providing critical insights into their decomposition characteristics.

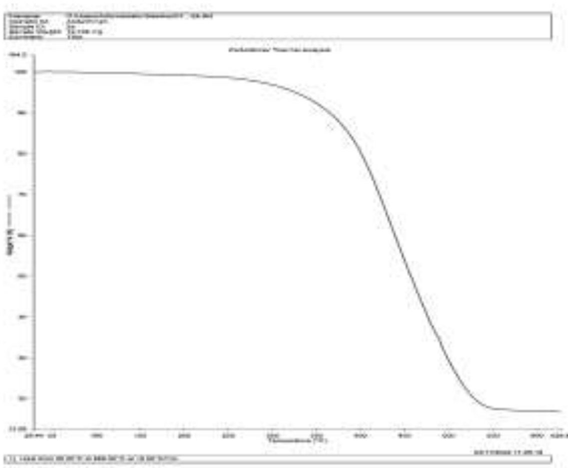

Fig2a: Control Sample (Clay 100%)

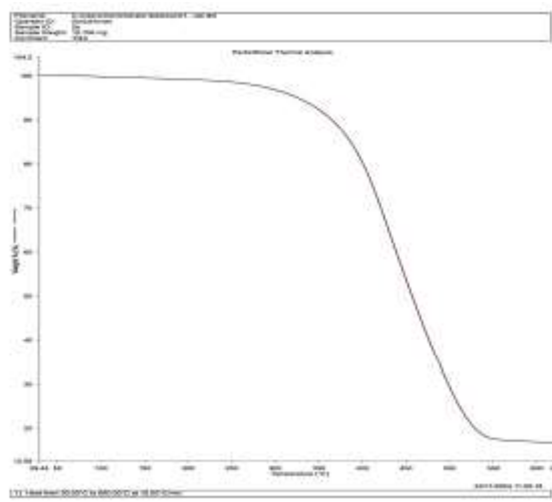

Fig.2b: Sample 90%, Broken Bottles 10%):

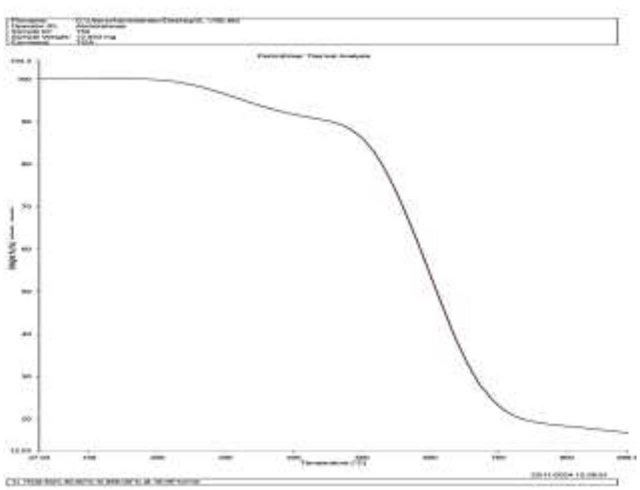

Fig.2c: Clay Broken Bottles 10%, Rice Husk 15%

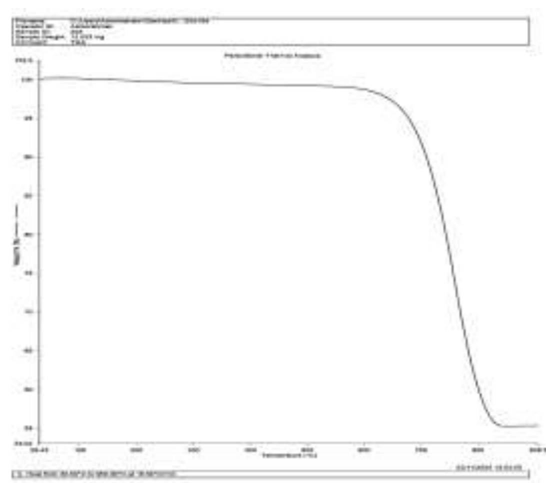

Fig.2d: result sample 25% Rice - husk

The thermal stability and decomposition profile of the pure clay serve as the baseline for comparison. The control sample is expected to show characteristic weight loss associated with the removal of absorbed water, dihydroxylation of the clay minerals, and possible organic content decomposition. The addition of 10% broken bottles enhances thermal stability due to the inorganic and heat -resistant nature of the glass particles. The TGA curve demonstrate reduced weight loss compared to the control, especially in high-temperature ranges, as glass components are stable under heat. Introducing 15% rice husk, a lignocellulosic material, contributes organic content. The TGA show additional weight loss peaks corresponding to the thermal degradation of cellulose, hemicellulose, and lignin. The combination of rice husk with broken bottles results in a composite with moderate thermal stability improvements and distinct decomposition stages. At 25% rice husk, the organic content increases significantly, which might lead to more pronounced weight loss in the TGA at lower and mid-temperature ranges due to the pyrolysis of rice husk components. However, the stability of the broken bottles offset the impact, depending on the overall matrix interactions. Addition of 10% BB improves the thermal to weight loss ability of Gula clay from 627 ˚C to 650 ˚C. Addition of Rice – Husk from 5% - 25% show a consistent increase in thermal stability of Gula clay. With 15% RH, the thermal stability increased from 627 ˚C to 850 ˚C, with further increase in amount of rice-husk from 15% to 25%, the thermal strength improved highly from 627 ˚C to 920 ˚C.

THERMAL SHOCK

Table 3: Shows the Thermal Shock Results for the Samples

Sample Name	Temperature ℃	Number of Cycles	Time inside Furnace (minutes)	Time outside in Furnace (minutes)	Status
100% Treated Gula Clay (TGC)	1100	8	15	10	Safe
10% pulverized broken bottles (PBB)+ 90% TGC	1100	8	15	10	Safe
5% Rice Husk (RH) + 10% PBB + 85% TGC	1100	8	15	10	Safe
10% (RH) + 10% PBB + 80% TGC	1100	8	15	10	Safe
15% (RH) + 10% PBB + 75% TGC	1100	8	15	10	Safe
20% (RH) + 10% PBB + 70% TGC	1100	8	15	10	Safe
25% (RH) + 10% PBB +65% TGC	1100	8	15	10	Safe

As the result shown from the table below it can be discovered that all the samples have been able to withstand the thermal shock of 10 cycles with no any failure. This indicate that the reduction of iron II content with oxalic acid has improved the clay thermal property, the chemical improves the failure strength at higher temperature with a steady load act upon it, also the rice husk and the broken bottles. This improvement in thermal property indicates the promising nature of this clay for muffle furnace use [19]. All the samples can safely resist thermal shock at the temperature of 1100 ˚C. This implies that these samples can be used to lining muffle furnace for melting and heat treatment operations for non – ferrous metals.

Water Absorption

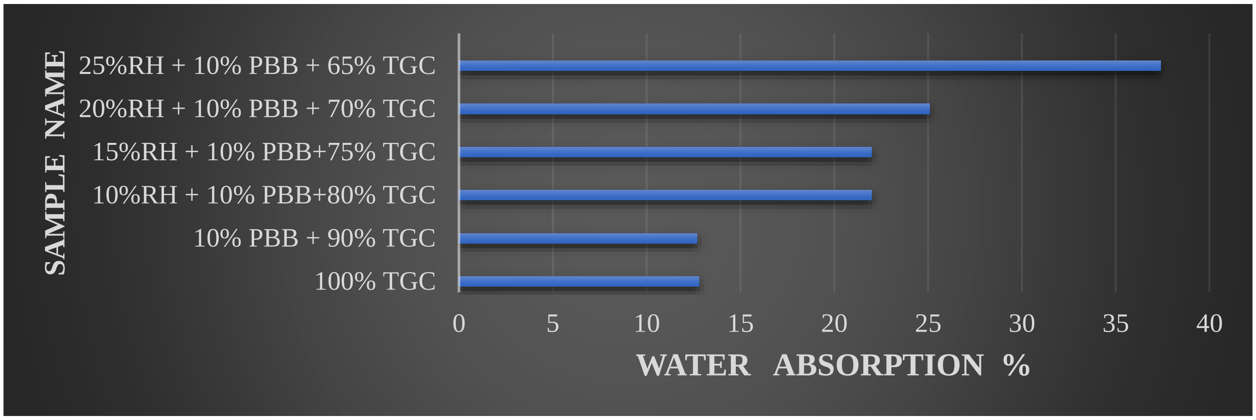

Fig.3: Shows the graph of Samples against %Water Absorption.

The water absorption percentage increases with the addition of rice husk, indicating that rice husk introduces more porosity or hydrophilic properties to the composite. The combination of broken bottles (10%) and rice husk affects the composite's water absorption differently, depending on the rice husk content. **Clay (100%)**: The pure clay sample exhibits a water absorption rate of **12.79%**, serving as the baseline. **Addition of Broken Bottles (10%)**: The inclusion of broken bottles slightly decreases water absorption to **12.69%**, suggesting the bottles contribute to reducing porosity or hydrophilicity. At **10% rice husk**, water absorption increases significantly to **21.99%**, indicating the hydrophilic nature of rice husk. At **15% rice husk**, the absorption remains steady at **21.99%**, showing no immediate change with this proportion. At **25% rice husk**, water absorption peaks at **37.41%**, demonstrating a clear correlation between higher rice husk content and increased moisture uptake. The trend suggests that while the addition of broken bottles slightly improves water resistance, rice husk significantly increases water absorption due to its porous and hydrophilic nature. This finding highlights the need to balance these reinforcements based on the composite's intended application.

Linear Shrinkage

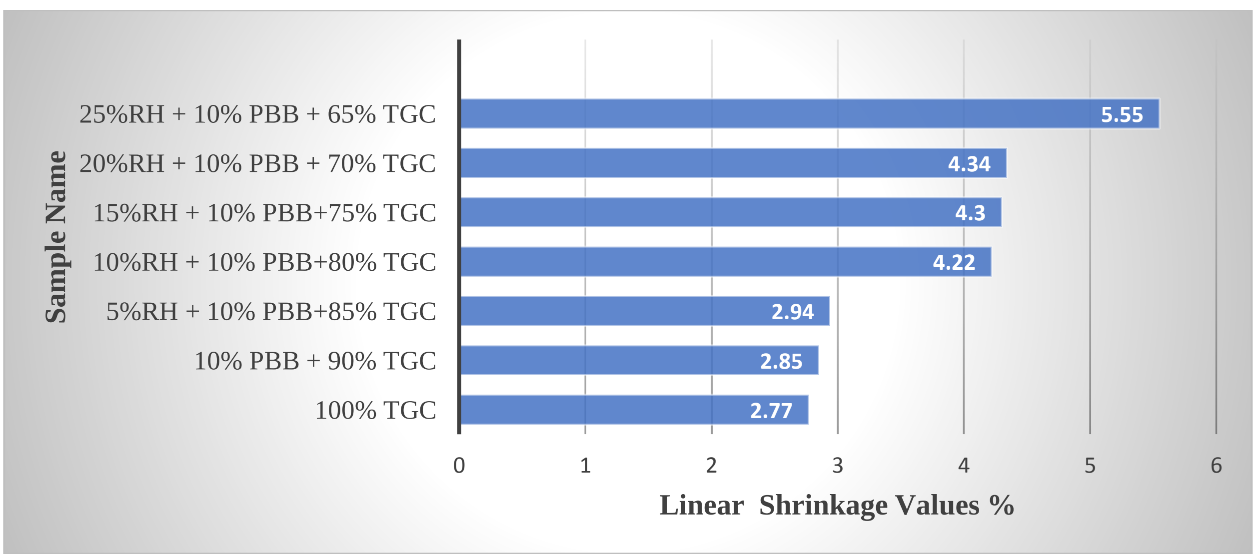

Fig. 4: Graph of %Linear Shrinkage Values against Samples

The linear shrinkage of the control sample is 2.77, while sample with 10%BB is 2.85. There is corresponding increase in linear shrinkage in the clay with increased amount of rice-husk from 5 – 25% RH for 2.94 – 5.55. in all, the linear shrinkage is below 10%, showing that the clay samples produced have low shrinkage. This implies that this samples when used in the furnace will maintain their structural integrity. Thereby reducing the possibility of cracks in the furnace lining.

Compressive Strength

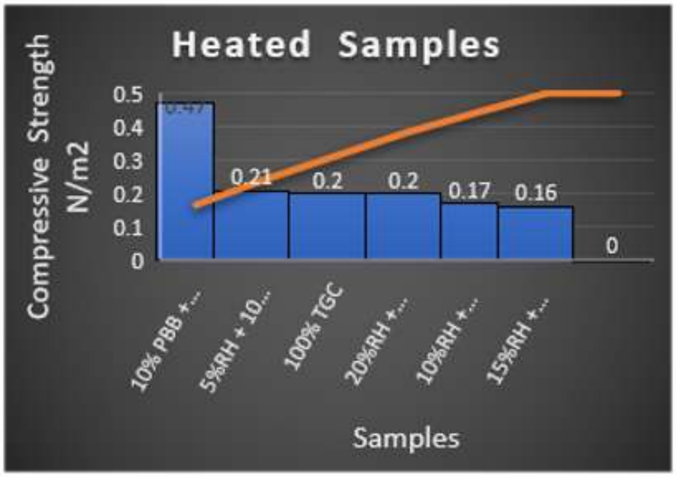

Fig.5a: Compressive Strength of Heated Samples

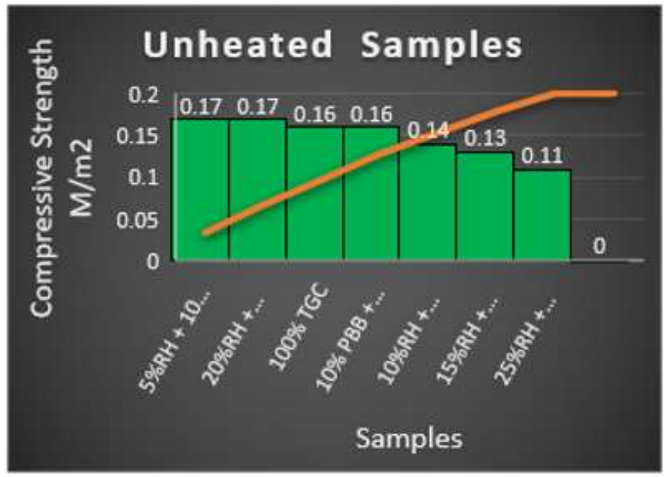

Fig.5b: Compressive of Unheated Samples

For the heated samples, sample of 10% granulated broken bottle has the best compressive strength of 0.47N/mm^2. The addition of rice - husk of 5 – 20% did not adversely lead to reduction in compressive strength when compared with the control sample (100% Gula clay) (0.16 - 0.20). But as the rice – husk amount increases above 20%, the compressive strength drastically decreased to 0.05N/mm2. This implies that the presence of rice-husk in an amount less than 25%, helped in creating pores in the clay therefore weight of the bricks for furnace application without compromising their compressive strength.

For optimum result, sample with 20% RH + 10%BB +70%GC is the best. The unheated samples, the compressive strength is generally low, except sample 20%RH + 10%BB+70%GC. Whose compressive strength is 0.17N/mm2, while that of control sample is 0.16N/mm2.

CONCLUSION

Composite Performance**:** The interplay between organic (rice husk) and inorganic (broken bottles) reinforcements influences the degradation pathway, suggesting a trade-off between thermal stability and organic material inclusion. These results, derived from advanced analytical techniques, provide a comprehensive understanding of the thermal behavior of the composite materials, essential for their potential applications in various high-performance industries. Lower rice husk content composites (Sample 5% and 10%) may be better suited for applications requiring structural strength. Higher rice husk content (Sample 25%) could be explored for lightweight or insulating materials. This analysis highlights the effects of broken bottles and rice husk on the clay composite's elemental composition and structural morphology, offering insights into optimizing material properties for specific applications.

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Cite this Article: Barnabas, AA; Nathan, C; Emmanuel, IU; Rufai, AM; Zuberu, M; Sadisu, A; Ibrahim, U (2025). The Effect of Rice- Husk Ash and Pulverized Broken Bottle on Composite Material Developed from Gula Clay*. Greener Journal of Science, Engineering and Technological Research*, 14(1): 18-27, **https://doi.org/10.15580/gjsetr.2025.1.091625136.**