Effect of African Velvet Tamarind (Icheku) Shell and Melon Shell Additives on Clay Blends for High Temperature Application

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Abstract
The effect of agro-waste additives formulations African Velvet Tamarind (Icheku) and melon shells on Ehandiagu/Nkalaha clay blends was studied in this work. The clay samples collected from existing open mines, using digger and shovel, were cleaned, crushed using mortar and pestle and sieved with mechanized sieve arranged is descending order of fineness to obtain uniform particle sizes. The clay samples were soaked in a plastic container containing some quantities of de-ionized water. The water/clay mixture was vigorously stirred to ensure proper dissolution of the clay particles in water and the solution allowed settling for three days. The mixture was later filtered to remove unwanted materials and other debris in the mixture. The filtrate was allowed to settle for days and excess water decanted off. The clay obtained from this was sundried for three days and later oven dried at about 120^oC for six hours. The resulting dry processed clay samples were ground and sieved to obtain uniform particle sizes. The icheku and melon shells were sundried for three days, pulverized and sieved to obtain uniform particle sizes. The chemical compositions of the melon shell, the African Velvet Tamarind (Icheku) shell and the clay samples were determined using XRF. The Ehandiagu and Nkalaha clay samples were blended in the ratios: 20:80; 60:40; 50:50. The ground icheku and melon shells additives were incorporated into the clay blends at 5%, 10%, 15%, 20% and 25%. The samples were mixed enough water to make it plastic for molding. The samples were molded into different shapes using wooden mould for refractoriness, modulus of rupture, thermal shock and linear shrinkage tests. The results obtained showed improvement in refractoriness by 4.61%, about 23.08% in linear shrinkage and about 3.32% in modulus of rupture.

Introduction

Nigeria is vastly endowed with huge clay deposits. Though, most of these clay deposits remain unidentified, however, some of the identified ones remain poorly improved to meet international standard for high temperature industrial applications. Clay minerals are considered as indispensable industrial minerals Aramide et al, (2014). Application of these important natural deposits Aramide et al, (2014) further explained borders on its structure and chemical composition. Mathew and Owoeye (2016) envisaged huge demand for refractory clays in Nigerian industries.
This was corroborated by Borode et al. (2000) who put the refractory bricks requirements of Ajaokuta Steel Complex at about 36,000 tons to complete its furnace lining. Adondua (1998) further argued that when fully operational, Ajaokuta Steel Complex and Delta Steel are expected

to consume an estimate of 43,503 and 2500 tons/year of fire clay refractories respectively for their operations. Buttressing the need and urgency to improve the quality of Nigerian clay deposits to enhance its applicability in the industries, Obadinma (2000) put the annual importation of refractories in Nigeria at 27 million metric tons in 1987; hence, making the country to spend huge foreign exchange in refractory clay importation, despite the huge deposits which can be conveniently harnessed and developed to meet our local demand and possibly some for exports to earn foreign exchange. Although, Aneke (1982) attested that many works have been done towards identification of numerous clay deposits in Nigeria and their possible applications however, Nwajagu (1995) was of the opinion that most of the Nigerian deposits remain unidentified and that even some of those identified have not been systematically studied. Chima (2018) reported that the use of agro-waste materials as additives in bricks is gaining increased research attention due to their effective role in decreasing the total energy needs of industrial furnaces. Safeer et al, (2017) observed that these additives leave pores upon burning, thereby, causing decreased thermal conductivity which in effect, affects the mechanical properties of the bricks. Chima (2018) further argued that increasing industrial standards and requirements has continued to place huge demand for clay brick with higher insulation ability and that the insulation capacity of brick can be increased by increasing its porosity. Therefore, Demir, (2008), concluded that combustible organic pore forming materials are commonly employed in this regards. Single clay is mainly characterized by inadequate properties; hence, limiting its use for refractory production. Considering industrial demands for refractory products with adequate properties, it becomes necessary to blend different clays, so as to exploit the good properties of the different deposits. It also becomes imperative to select clays based on the physical, chemical and thermal properties from different deposits and blend them with some agro wastes to enhance their performance in service. Therefore, the need to use local agro-waste materials to improve the quality of clay deposits in some south eastern states of Nigeria to develop high quality refractory products becomes imperative.

Although, south eastern states of Nigeria are endowed with numerous clay deposits, some of them are of low quality and falls short of international standard for application as a high refractory material. Utilization of industrial or agricultural wastes as a source of raw materials for industry to develop new products or enhance the quality of existing natural materials has been a major concern for researchers for sometimes now. Nigeria in general and South-Eastern Nigeria in particular has enormous clay deposits. Unfortunately, most of these clay deposits lack adequate properties to meet the high standard demand of modern industries. This has in recent times, raised the curiosity of researchers in developing alternative means of improving the quality of some of these materials to compete and measure favorably with international standards and specifications. Nweke and Ugwu (2007) reported that coconut fibers, laterites, and other useful materials have been mixed in different proportions to produce roofing sheets and bricks as building materials. Hassan (1985) however, studied the effect of additives like saw dust, graphite and asbestos on Kankara clay and concluded that addition of such additives (about 15%) resulted in clay brick with good thermal insulating property.

African Velvet Tamarind **(**icheku) and melon are commonly and randomly consumed in the south eastern states of Nigeria and this in effect liters the environment with their shells which has no known second hand value. Therefore, to mitigate the inherent environmental nuisance occasioned by poor disposal of African Velvet Tamarind icheku and melon shells by incorporating them into poor refractory clays like Eha-Ndiagu and Nkalaha, this work seeks to improve the refractory quality of these clay deposits for industrial potentials and achieve a cleaner environment by adequate disposal and utilization of African Velvet Tamarind icheku and melon shells.

2. Materials and Methods

2.1 Materials

The following materials were used in this research work: Eha-Ndiagu and Nkalaha clay samples, icheku shell, melon shell, water, mortar, pestle, digger, sieve, shovel, plastic bag.

2.2 Method.

The collected Eha-Ndiagu and Nkalaha clay samples were aggregated and air dried for days. The dried samples were later ground manually using a mortar and a pestle to homogenize the particle sizes. The samples were soaked in a plastic container containing some quantities of de-ionized water. The water/clay mixture was vigorously stirred to ensure proper dissolution of the clay particles in water and the solution allowed settling for days. The mixture was later filtered to remove unwanted materials and other debris in the mixture. The filtrate was allowed to settle for days and excess water decanted off. The clay obtained from this was sundried for three days and later oven dried at about 120^{o}C for six hours. The resulting dry processed clay was ground and sieved to obtain uniform particle size. The icheku and melon shells were sundried for three days, pulverized and sieved to obtain uniform particle sizes. The chemical compositions of the melon shell, the African Velvet Tamarind (Icheku) shell and the clay samples were determined using XRF. Eha-Ndiagu and Nkalaha clay samples were blended in the ratios: 20:80; 60:40; 50:50. The ground icheku and melon shells additives were incorporated into the clay blends at 5%, 10%, 15%, 20% and 25%. The samples were mixed enough water to make it plastic for molding. The samples were molded into different shapes using wooden mould for various tests.

Determination of the Chemical Composition the Clay Samples using XRF

The clay samples and the additives were analyzed to establish their chemical compositions at National Steel Raw Materials Exploration Agency (NSRMEA), Kaduna, and using X-ray Fluorescence (XRF) analysis technique**.**

2.3 Refractoriness Test

Pyrometric cone equivalence (PCE) method in accordance with ASTM C24-79 was used to determine the refractoriness of the clay blends. The test cones were prepared by mixing the clay blends and additives with appropriate quantity of water to make it plastic. The test samples were dried and fired to a temperature of about 900^{o}C in electric furnace. The test samples were mounted on a refractory plaque alongside with the standard cones designed to deform at temperatures above or below that of the test samples. The plague was placed in an electric furnace and heated at the rate of about 10^{o}C per minute to the temperature at which the tips of the test cones had bent over the level with the base. The temperature at which each cone bent over was taken to be the refractoriness of the clay blends. The test cones were then compared with the standard cones and the test materials were assigned the pyrometric cone equivalent (PCE) of the standard cone that it resembled most in bending behavior.

2.4 Thermal Shock Resistance Test

The prism spalling test method (ASTM C-484) was used for this test. The test was carried out in an electric furnace heated at the rate of 10^0C/min. The spalling resistance was measured by the number of repeated thermal cycles (heating, cooling and testing for failure) the clay sample will withstand before breaking Lawal et al. (2005). The test samples (50x50x10) mm were heated in an electric furnace to a temperature of about 1200°C at the rate of about 10^0C/min and allowed to be in the furnace for about ten minutes. The samples were removed one after the other from the furnace using a pair of tongs and cooled in air (outside the furnace) for about 10 minutes, and observed for cracks. With no cracks observed, the samples were put back into the furnace and reheated for more 10 minutes and then cooled for another 10 minutes. The process was continued until cracks were observed on the test samples. The number of cycles of heating and cooling before cracking for each specimen was recorded as its thermal shock resistance.

2.5 Determination of Modulus of Rupture

This is a mechanical parameter which quantifies the ability of a ceramic material to resist deformation under load in observance of ASTM C133 – 97. Five long rectangular test pieces were made and air dried for 7 days after which they were oven dried at 105°C until a constant weight was obtained. Four of the pieces were fired to their respective temperatures of 900oC, 1000oC, 1100oC and 1200oC in an electric kiln (Fulham Pottery). A 3-point loading or bending jig was used where a test tile 10x4x1cm was tested until it failed. The electrical transversal strength machine was used to determine the breaking load, P (Kg). A vernier caliper was used to determine the distance between supports L (cm) of the transversal machine. The height, H (cm) and the width, B (cm) of the broken pieces were determined and the average value obtained from the two broken parts was recorded.

The pressure at which the test piece failed was recorded and the M.O.R was evaluated using the expression below;

M.O.R = …………………………………..2.1 Kefas et al., (2007):

Where F=force

L=length between knife edges.

B=breadth of brick.

H=height of brick.

2.6 Determination of Linear shrinkage

The shrinkage was determined considering both the dried and fired shrinkage. The drying shrinkage is an index of the body to withstand cracking or retain shape and size after firing. It also indicates the degree of plasticity of the mixture. After molding the rectangular test pieces, a vernier caliper was used to insert a 10 cm mark on each of them and was recorded as the original length Lo (cm). The test samples were air dried for 10 days and oven (cabinet) at 105 0C until a constant weight was obtained. The shrinkage from the 10 cm mark was determined and recorded as the dried length, Ld (cm). The dried samples were fired to their respective temperatures of 900oC, 1000°C, 1100°C and 1200°C with each temperature corresponding to a particular test piece. The shrinkage of the test pieces from the 10 cm mark were then determined and recorded as the fired length, Lf (cm).

Dry Shrinkage (%) = 100[Lo - Ld]/Lo …………… 2.2

Linear Shrinkage (%) = 100[Ld - Lf]/ Ld ………….2.3
Total Shrinkage (%) = 100[Lo – Lf]/ Lo …………. 2.4

3. Results and Discussion

Table 3.1 shows the chemical composition of Ehandiagu and Nkalaha clay samples respectively while Tables 3.2 and 3.3 show the chemical compositions of melon shell and African Velvet Tamarind (Icheku) shell.

From Table 3.1 it could be observed that silica and alumina are the major constituents of the clays. The alumina content of Ehandiagu and Nkalaha clay samples are 21.304% and 23.975% respectively. The percentage composition of alumina in clay is an indication of its refractoriness Abubakar et al., (2014). The alumina content of Ehandiagu and Nkalaha clays may be regarded as low and consequently classified as high melting clays Nnuka and Agbo (2000) opined. The silica content of the two clays (53-73%) also satisfied their classification as high melting clays Chester (1973). Silica is considered as filler in clay. It enabled the brick to retain its shape and imparted durability quality, prevented shrinkage and warping.

The presence of alkali metal oxides like CaO, K_2O and Na_2O in reasonable amounts in Ehandiagu and Nkalaha clays show good thermal ability during firing at low temperatures, hence, reducing the vitrification temperature and refractoriness of the clay as Maiti and Kumar (1992) observed. Also, the presence of these oxides in clay Maiti and Kumar (1992) noted acts as mild fluxes, as they combine with the oxides of silica and alumina on firing to form eutectics and so reduce the vitrificaion temperature and refractoriness of the clay. The calcium oxide CaO (lime) content of Nkalaha clay is 2.499% while that of Ehandiagu clay is 0.763%. Altayework, (2013) reported the lime content of clay to normally constitute less than 10 per cent of clay. The lime content of clay reduces its shrinkage on drying. Expectedly, Nkalaha clay had lower shrinkage value compared to Ehandiagu clay.

Potassium oxide K_2O has values of 0.076% and 0.097% respectively in Ehandiagu and Nkalaha clays. Potassium oxide K_2O is a low temperature fusing agent which melts at low firing temperature to fuse into the structure of the clay body thereby helped to bind the clay material and also reduced the sintering temperature.

Although, the Fe_2O_3 content of Nkalaha clay 4.331%, is higher than that of Ehandiagu clay 0.724%, however, both clays can be said to be low in iron. High iron oxide content in clay, Nnuka and Agbo, (2000) reported affects high temperature characteristics of the clay material.

Table 3.1 Results of the Chemical analysis by (XRF) of Ehandiagu and Nkalaha clay samples.

Oxides	**Ehandiagu**	**Nkalaha**
SiO_2	**70.147**	**62.264**
Al_2O_3	**21.304**	**23.975**
CaO	**0.763**	**2.499**
MgO	**1.501**	**0.713**
Na_2O	**0.581**	**0.380**
Fe_2O	**0.724**	**4.331**
K_2O	**0.076**	**0.097**
MnO	**0.003**	**0.893**

TiO_2	**1.703**	**0.100**

Table 3.2 shows that SiO_2, P_2O_5, K_2O, Al_2AO_3, Fe_2O_3 and CaO as the dominant oxides in melon shell. From Table 3.3, it could be observed that CaO, SiO_2, K_2O, MnO, Fe_2O_3, MgO, ZnO, P_2O_5 and Na_2O are the dominant oxides in African Velvet Tamarind (icheku) shell. The high alumina content of the melon shell increased the alumina content of the clay blend, thereby, increased the refractoriness of the clay blend. Although, both additives contained SiO_2, however, the melon shell had higher percentage of SiO_2 at 73.4%. SiO_2 functioned as filler in the clay blend and this helped inter particle binding when fused. The presence of silicon oxide in the additives was useful in reducing the drying shrinkage of the clay blend. Therefore, the high silica content of the additives helped to reduce excessive shrinkage in the clay blend which would have occurred after firing. The percentage compositions of CaO in the additives were 1.88% and 23.48% in melon shell and African Velvet Tamarind (Icheku) shell respectively. Nnuka et al. (1992) reported that beyond 0.3% composition, calcium oxide enhances mullite formation in fire clay thereby improving high temperature characteristics like sintering temperature.

Phosphorus oxide, P_2O_5, was found in the two additives, although, melon shell has higher percentage composition of about 7.94%. Phosphorus oxide is regarded a useful oxide in refractory brick, Chima (2018). Phosphorus and silicon, Iloabachie et al. (2022) opined are known to react with alumina (Al_2O_3) to form aluminum-phosphate/silicate bond which Decker (2003), observed has appreciable strength above 350^0C. The alumina phosphate bond acts as a binder which enhances the brick's refractory properties like thermal shock resistance, refractoriness and strength. The bond improves non wetting effect of molten metal on the material and also increases the resistance of the material to carbon II oxide attack. The carbon II oxide is a byproduct of the furnace which causes cracking and destruction of the refractory lining Decker, (2003).

Table 3.2 Chemical Composition of Melon Shell by XRF

Metallic Oxides	Composition (%)
SiO_2	73.4
K_2O	3.62
CaO	1.88
MnO	0.47
TiO_2	0.21
Fe_2O_3	2.65
MgO	1.08
BaO	0.053
Al_2O_3	7.42
ZnO	0.398
Na_2O	0.39
P_2O_5	7.94

Table 3.3 Chemical Composition of African Velvet Tamarind (Icheku) Shell by XRF

Metallic Oxides	Composition (%)
SiO_2	19.40
K_2O	8.96
CaO	23.48
MnO	10.75
TiO_2	0.17
Fe_2O_3	6.82
MgO	9.04
BaO	0.15
ZnO	5.53
Na_2O	5.21
CuO	2.27
P_2O_5	4.13

Table 3.4 Refractory Properties of Ehandiagu/Nkalaha Clay Blends with Combination of Melon Shell/ AVT Shell Additives

Refractory Property	Control Sample	Clay Blend + Melon Shell and AVT Shell Additives				
		5%	**10%**	**15%**	**20%**	**25%**
Thermal Shock Resistance	31	29	27	29	28	30
Modulus of Rupture (MPa)	32.25	33.32	29.42	28.74	27.5	26.3
Linear Shrinkage	7.02	8.25	8.31	8.38	8.52	8.64
Refractoriness (°C)	1520	1590				

From Table 3.4, it could be observed that the control sample had a higher value of 31 cycles for thermal shock resistance. Fig.3.1 revealed fluctuations in thermal shock resistance values with incorporation of the combined additives in the clay blends. These fluctuations in thermal shock values at various percentage compositions of the combined additives were all lower than that of the control sample. The 25% combined additive combination had the lowest percentage fluctuation of about 3.33%. This result indicated that though, the combination of melon shell and African Velvet Tamarind (Icheku) shell did not improve the thermal property of the clay blends, however, the result of this test may be useful in applications where low fluctuations in thermal shock is required. Similar trend had been reported by Chima (2018) where combination of two additives (groundnut shell and rice husk) deteriorated the thermal shock resistance of Nguzu and Amaiyi clay blends.

The modulus of rupture result, Table 3.4 showed that the control sample recorded 32.25 MPa value. From Fig. 3.4 an increase in modulus of rupture was observed at 5% additive combination representing an improvement of about 3.32%. Beyond the 5% additive combination, the modulus of rupture continued to decrease as the percentage of additive combination increased. The observed decrease in the values of modulus of rupture may be likely due to increase in porosity obtained with increase in composition of the combined additives. The increase in the quantity of the combined additive in effect, affected the strength of the clay blend adversely. Furthermore, evaporation of gases through the clay body produced due to burning of additives created a highly porous clay body which reflected negatively on the transverse strength. Therefore, this means that the amount of the additives must be controlled to avoid adverse effects.

Therefore, it could be deduced from Fig. 3.4 that enhancement of modulus of rupture depends on the quantity of additives combination and this agrees with the work of Safeer et al, (2017).

Table 3.4 showed that the linear shrinkage of the control sample of the clay blend was 7.02. Ugwuoke and Amalu (2017) stated that linear shrinkage represents the firing efficiency of clay samples. Fig. 3.3 showed that the linear shrinkage of the clay blend with additives combination increased as the quantity of additives combinations increased. An enhancement of about 23.08% in linear shrinkage was achieved between the control sample and the clay blend with additives combination composition of about 25%. Although Omowumi (2001) quoted a recommended shrinkage value range of about 4-10%, Chester (1973) however, recommended linear shrinkage range of 7-10% for refractory clays. The linear shrinkage of this study satisfied this. The observed increase in linear shrinkage value may be attributed to possible rearrangement of the particles of the clay blend when the combined additives were burnt off. This resulted to more compact structure of the clay blend with higher shrinkage value.

The refractoriness of the control sample of the clay blend was found to be 1520°C and that of the clay blend with additives composition was 1590°C as was observed in Table 3.4. From the results of Table 3.4, it could also be observed that an improvement of about 4.61 % was achieved by incorporating the combined additives in the clay blend. Generally, the refractoriness of a clay body is affected by its oxide composition. Specifically, alumina, Al_2O_3 of a clay material determines refractoriness. One of the additives, melon shell has alumina composition of about 7.42%. Also, fro Tables 3.2 and 3.3, melon shell and African Velvet Tamarind (Icheku) shell have P_2O_5 contents of 7.94 and 4.13 respectively. The observed enhancement in the value of refractoriness of the clay blend with the combined additives may be likely due to the presence of these oxides.

4. Conclusion

The following conclusions were made in this work:

- Chemical composition of melon shell and African Velvet Tamarind (Icheku) shell showed that they can be used as combined additives in clay blend to enhance the refractoriness of the clay blend.

- The degree of enhancement of modulus of rupture by incorporation of combined additives in clay blend depends on the quantity of the combined additives.
- Combination of melon shell and African Velvet Tamarind (Icheku) shell greatly improved the linear shrinkage of Ehandiagu/Nkalaha clay blends by 23.08% at about 25% combined additives compositions.

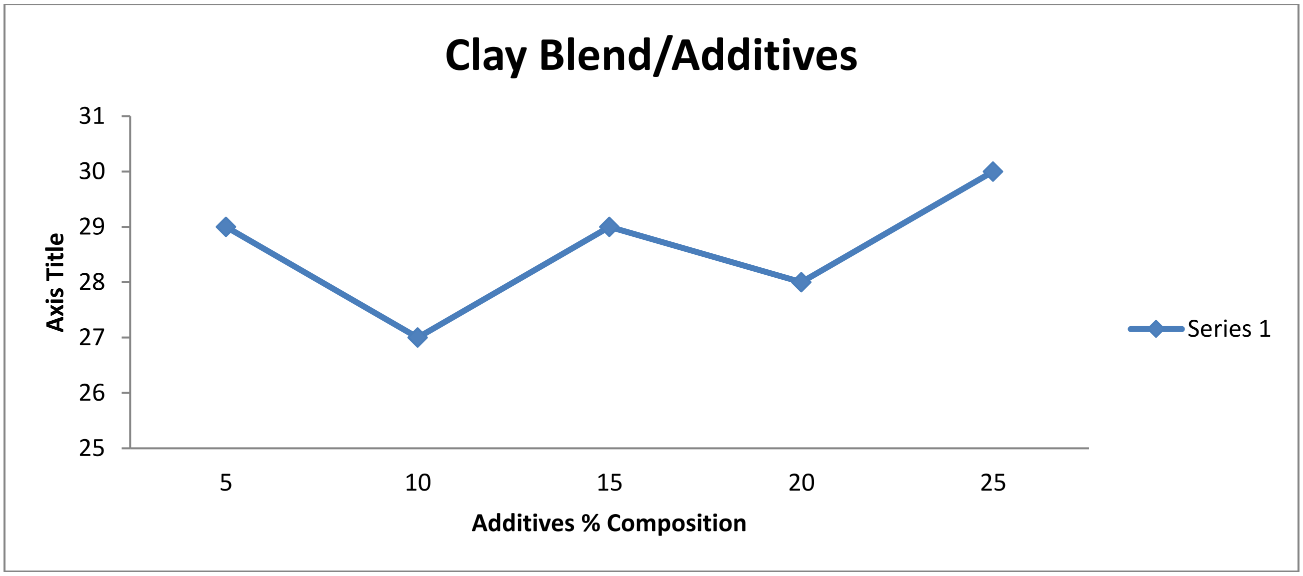

Fig. 3.1: Effect of Melon Shell/ African Velvet Tamarind (Icheku) Shell Composition on the Thermal Shock Resistance of Ehandiagu/Nkalaha Clay Blends.

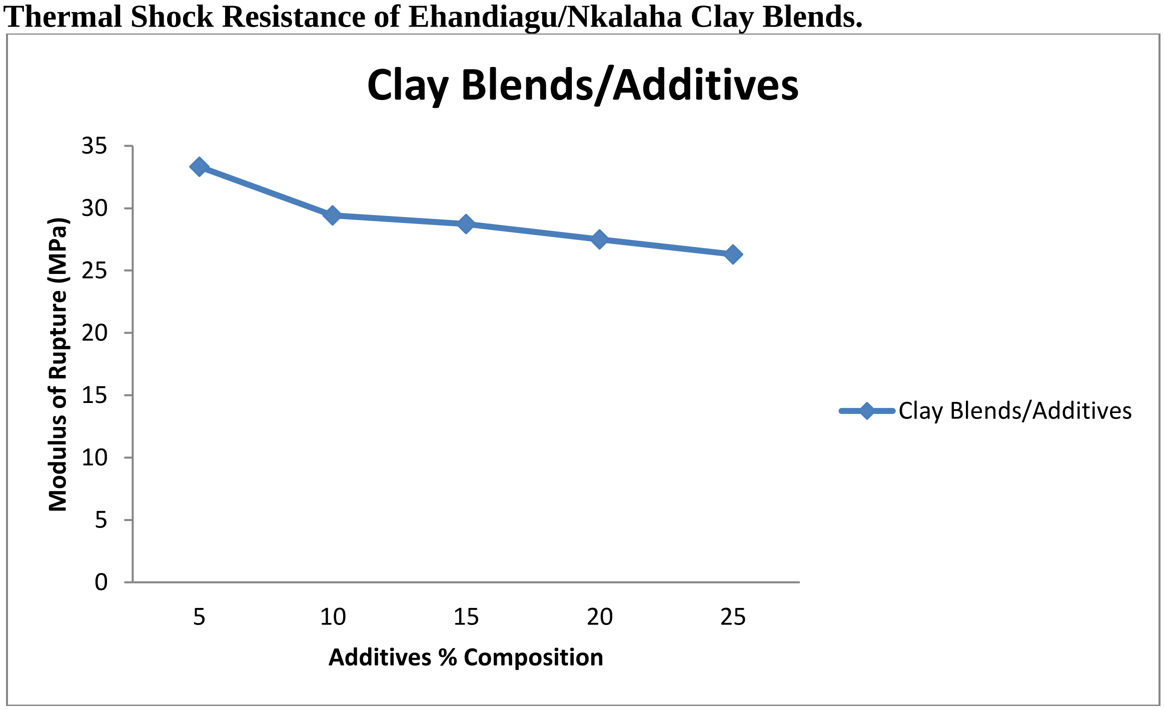

Fig. 3.2: Effect of Melon Shell/ African Velvet Tamarind (Icheku) Shell Composition on the Modulus of Rupture of Ehandiagu/Nkalaha Clay Blends.

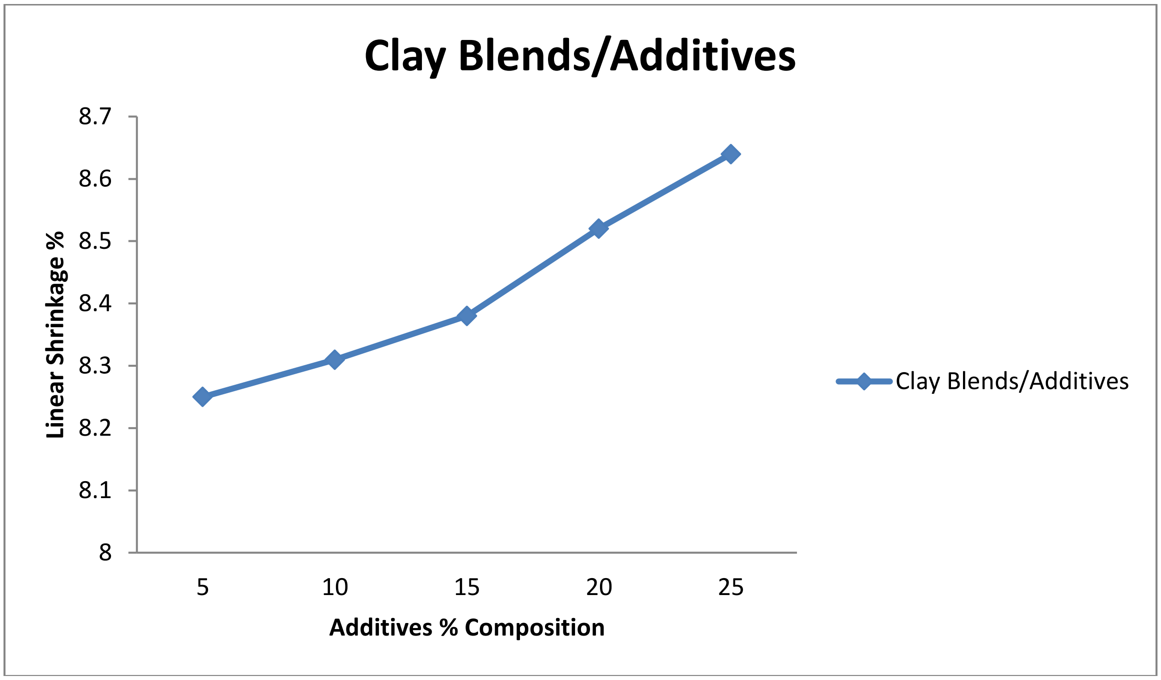

Fig. 3.3: Effect of Melon Shell/ African Velvet Tamarind (Icheku) Shell Composition on the Linear Shrinkage of Ehandiagu/Nkalaha Clay Blends.

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Icheku Seed and Shell

Icheku Shell

Melon Shell