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Mechanical Evaluation and Minerals Phases Identification of Fine and Coarse Okelele Block Clay Composites for Furnace Lining Application

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Abstract

The suitability of fine and coarse Okelele clays as refractory raw materials for furnace lining application was investigated. The clay samples were crushed and pounded with a mortar and pestle to a particle size of 20 microns. 230 g each of fine clay was mixed with 50 mls of water inside a bowl and stirred thoroughly to form homogenous plastic paste. 10 g, 15 g, 25 g, 35 g and 45 g of coarse clay were added respectively to the 230 g of homogenous fine clay paste in different container. The fine and coarse clays composites weighing 240 g, 245 g, 255 g, 265 g and 275 g were respectively put in a mold of dimension 3 x 5 x 6 cm and air dried for 7 days. The samples were fired at temperature of 1200 ^{o}C for five hours using Carbolite Furnace. After cooling, the fine and coarse clay composites of 240 g and 245g were broken by the heat and composites blocks 255 g, 265g and 275g were hardened and remove for compressive test analysis. The fine and coarse clays were characterized using X-ray Diffractometer PW 1830 for minerals phases' identification. The result of XRD shows that the clay was majorly composed of Quartz and Kaolinite with the traces of other minerals such as Smectile, Illite/Mica, Albite, Jarosite, Gypsum and Pyrite. The Kaolinite contains aluminum silicate ($Al_2O_3{\cdot}2SiO_2$) and Quartz has the silicon and oxygen atoms. The compressive strength test result judged the 275 g fire block of clays composite the best with the maximum force breaks of 7652 N with deflection of 3.734 mm and Young Modulus of 212 N/mm^2 for the time to failure of 22 seconds. The results proved that Okelele clays are suitable as refractory material for furnace lining application.

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Keywords: Okelele clays, Kaolinite, Quartz, Refractory materials

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1. Introduction

Nigeria is rich with abundant mineral resources but these resources have not been sufficiently explored and used. Clay is a naturally occurring material composed of layered structures of fine-grained minerals which reveal the property of plasticity at appropriate water content and permanently hard when fired [1]. Clay as a mineral that consist of silica (SiO_2), Alumina (Al_2O_3), water (H_2O), and other impurities are aluminosilicate, mostly answerable for its thermal property of refractoriness which applicable in the manufacturing of several refractory products. It is earthen and soil with intricate inorganic blend, whose structure diverges and generally depends on the environmental and geographical position [2].

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High demand for refractory materials for Furnace building and other related high temperature processes is enormous. Nigeria spends more than 2.27 billion naira yearly on the importation of refractories for industrial application [3]. The application of clay composites as a refractory material depends severally on its thermal property of refractoriness, chemical composition, mechanical and physical properties [2], [5-13]. Refractory materials are inorganic materials containing the mixtures of oxides obtained from naturally occurring minerals capable of withstanding very high temperature conditions without cracking, deforming, softening or change in composition [3]. The good characteristic of a refractory is to provide basic thermal properties, support winding (electric resistance) and be able to hold solid or liquid metals without entering into any undesirable chemical reaction with them. Thus refractory materials are characterized by the ability to withstand the heat, chemical attack, abrasion, impact, and shock caused by thermal stresses.

The clays used for furnace linings in metallurgical industries are classified as refractory clays. However, the degree of refractoriness and plasticity of any clay material is often influenced by the amount of the impurities contained in them [13]. The mechanical properties of different particle sizes of some impurities for some specific application had been investigated [14]. Chanchanga, Bida, Suleja and Zungeru clays deposits have better refractory and physical properties when compared with imported ones [3]. Some local clay deposits in other part of Nigeria have also been investigated with good results. Some of the clay deposits investigated for refractory application includes but not limited to Dukku clay deposit in Gombe State, Onibode, Ibamajo, Ijoko in Ogun State and Are in Ekiti State [15]. The characterization of Otukpo clay in Benue State was also reported [16].

The economic circumstance in Nigeria as at today has necessitated for the inward sourcing of locally available raw materials across the country for domestic and industrial applications. Due to the aforementioned economic needs and the fact that the Okelele clay deposit in Ilorin, Kwara State is only used for local pottery by old women living around the area and building bricks by local bricklayer. The minerals phases' identification and refractory properties of this particular clay deposit needs to be investigated.

2. Materials and Method

The fine and coarse clay samples were collected from a deposit in Okelele, Ilorin East local government area of Kwara state. The Molding iron bar, Mortar and pistol, Electronic weighing balance, HT 4/28 Carbolite Gero Muffle Furnace Machine located at Geology Department, University of Ilorin, (0-3000 °C), XFS300 Testometric compression test machine located at Agricultural Biotechnology Laboratory, Department of Biotechnology engineering, University of Ilorin and PW 1830 X-ray Diffractometer located at the Department of Geology, University of Ibadan were used in this work. The clay samples were crushed and pounded with a mortar and pestle to a particle size of 20 microns. 230 g each of fine clay was mixed with 50 mls of water inside a bowl and stirred thoroughly to form homogenous plastic paste. 10 g, 15 g, 25 g, 35 g and 45 g of coarse clay were added respectively to the 230 g of homogenous fine clay paste. The fine and coarse clays composites weighing 240 g, 245 g, 255 g, 265 g and 275 g were respectively put in a mold of dimension 3 x 5 x 6 cm and air dried for 7 days. The samples were fired at temperature of 1200 ^{o}C for 12 hours using HT 4/28 Carbolite Gero Muffle Furnace (0-3000 °C). After cooling, the fine and coarse clay composites of 240 g and 245g were broken by the heat and composites 255 g, 265g and 275g were hardened and sound like a glass when tapped. Figure 1 shows the fabricated broken and unbroken fired block of clays after firing.

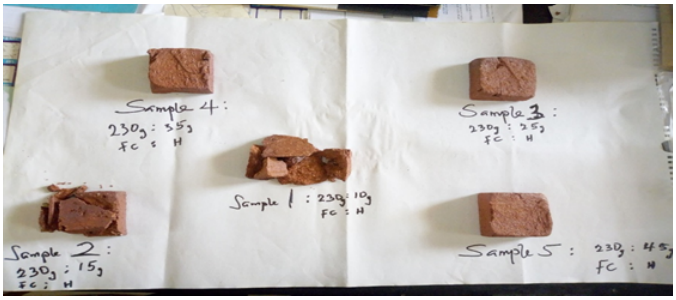

Figure 1. Shows the broken and unbroken Fired Block of Clays after firing

2.1. X-ray Diffractometer (XRD) Analysis

XRD was used to identify the phase of minerals constituents of the clays. The fine and coarse clays were separately crushed and milled to fine particles and put in test tubes. The samples were subjected to X-ray using the Philips PW 1830 X-ray diffractometer with a cu-anode at the "University of Ibadan" Ibadan, Oyo State. After the X-ray characterization of the samples, mineral peaks were identified using XPert High Score plus Software. The background and peak positions were identified and based on the peak positions and intensities; a search-match routine was performed.

2.2. Compression Test

The unbroken fired block of clays (255g, 265g and 275g) were subjected to mechanical compression test at the Civil Engineering Laboratory of the University of Ilorin, Ilorin Kwara State, to show how these materials deform (elongate, compress, twist) or break as a function of applied load, time, temperature and other conditions. The mechanical test was performed using XFS300 Testometric compression test machine. The capacity of this machine is 10,000 pounds (tension and compression). The samples of the given clay material took a rectangular shape which is unreformed (with no permanent strain or residual stress), or original shape.

Figure 2. Testometric Compression Test Machine used

Figure 3. Cracked Block of Clay during Compression Test

3. Results and Discussion

Figure 4 and 5 show the XRD patterns of fine and coarse clays. The Debye Scherer equation was employed for the estimation of grain sizes of fine and coarse clays.

$$\text{Grain size } g = \frac{k\lambda}{\beta\cos\theta} \tag{1}$$

Where k is the Debye Scherer constant (0.94)

$\lambda = 1.56 \text{ x}10^{-10}\text{m} = 0.156\text{nm}$

β = (FWHM) Full width at half maximum (radians)

θ = Peak positions (radians)

The estimated grain sizes and mineral constituents of fine and coarse clays are shown in Table 1 and Table 2.

Tables 1 and 2 give the results for the minerals phase identification for the fine and coarse Okelele clays. The Kaolinite and Quartz are dominance in the mineral phase identifications for the fine and coarse Okelele clays composites. Kaolinite which is also called China clay, is the best refractory clay type and will not soften below 1750 °C. Kaolinite clays possessed little plasticity due to their large clay particles. The Kaolinite contains $Al_2O_3{\cdot}2SiO_2$. The pure kaolinite can be found at the site of its parent rock (primary clay) and when it has not been mixed with impurities, its refractoriness is great. The Quartz is a very hard crystalline mineral mostly found in nature contained the silicon and oxygen atoms. Quartz is the most conventional source of silica to be used for refractory production. The refractory made from Silica (Silica refractory bricks) possesses excellent thermal shock resistance at specific temperature range.

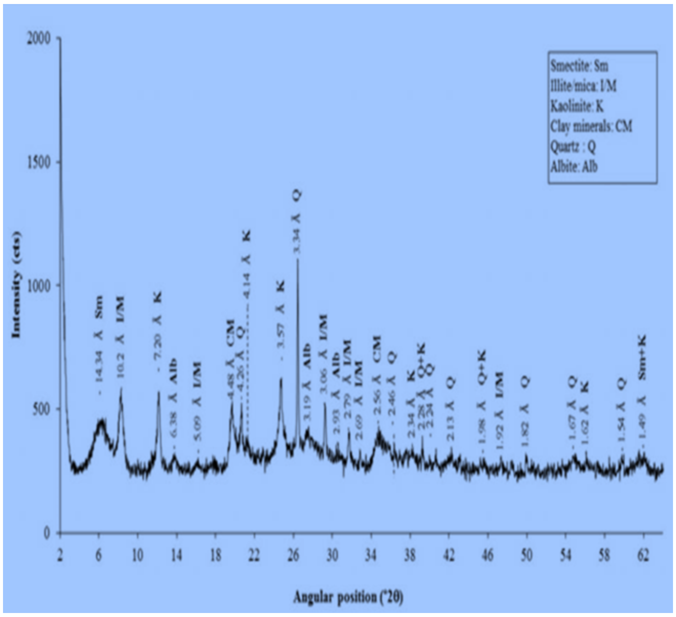

Figure 4. XRD Pattern of Fine Clay

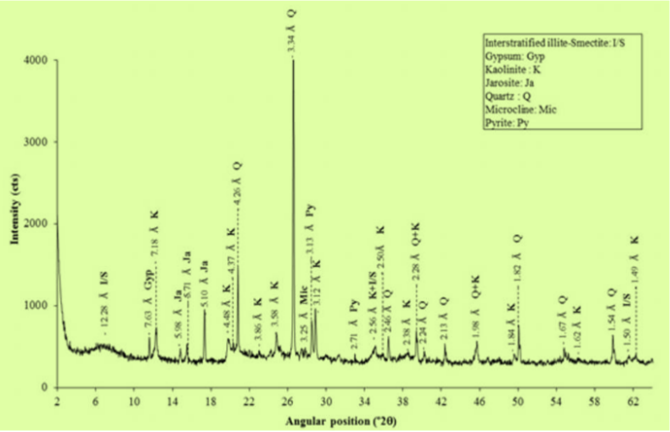

Figure 5. XRD Pattern of coarse Clay

The compressive strength test on 255 g, 265 g and 275 g fire blocks of clay composites were carried out to investigate the load carrying capacity of the fire blocks under compression using compression testing machine. This is important to determine the compressive strength of fire blocks for its suitability as furnace lining. The materials behaviours under a load were determined. The maximum stress a material can withstand over a period under a load (constant or progressive) was determined to a break (rupture) or to a limit. These results are shown in Table 3, 4 and 5.

Table 1. Estimated grain sizes and mineral constituents of fine Clay

Peak no.	2theta (rad)	FWHM	Grain Size (nm)	Constituents
1	4.43	2.187	3.844597803	Smectile
2	8.46	5.038	1.672251355	Illite/mica
3	12.24	7.12	1.186799367	Kaolinite
4	13.56	5.097	1.660000572	Albite
5	15.38	3.167	2.677013045	Illite/mica
6	19.47	5.272	1.616958333	Clay mineral
7	20.43	2.187	3.90360038	Quartz
8	23.46	5.038	1.703266297	Kaolinite
9	26.24	7.12	1.211663863	Kaolinite
10	27.56	5.097	1.697242735	Quartz
11	28.38	3.167	2.736431091	Albite
12	30.47	5.272	1.651722605	Illite/mica
13	31.32	2.348	3.716247528	Albite
14	32.12	7.257	1.204777898	Illite/mica
15	34.04	4.328	2.030195653	Illite/mica
16	36.58	2.039	4.339821991	Clay mineral
17	38.433	2.147	4.144207588	Quartz
18	40.465	5.035	1.778423867	Kaolinite
19	42.245	7.123	1.264497493	Quartz plus Kaolinite
20	46.567	5.027	1.819527192	Quartz
21	48.382	3.164	2.91109195	Quartz
22	50.473	5.278	1.75982879	Quartz plus Kaolinite
23	54.436	2.157	4.380159907	Illite/mica
24	55.467	5.034	1.885640471	Quartz
25	60.245	7.125	1.363317463	Quartz
26	62.562	5.077	1.936374021	Kaolinite

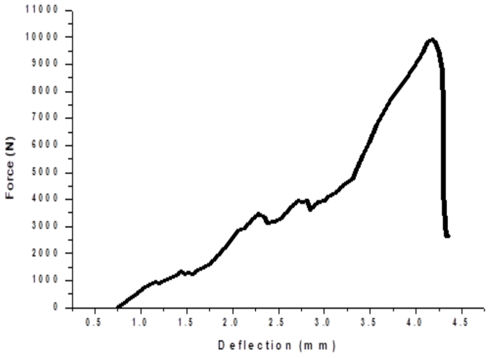

Figure 6. Force (N) against Deflection (mm) of 255 g fine and coarse fire block clay composites

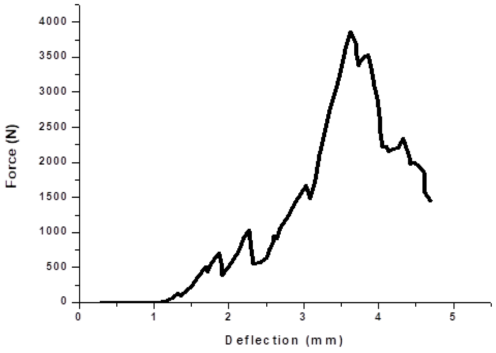

Figure 7. Force (N) against Deflection (mm) of 265g fine and coarse fire block of clay composites

The 255 g block has the force break of 2632 N and deflection break at 4.343 mm. The time to failure is 26.133 seconds for the Young Modulus of 174.476 N/mm^2 among other parameters (Table 3). The 265 g block has the force break of 1439 N and deflection breaks at 4.671 mm. The time to failure is 28.1 seconds for the Young Modulus of 94 N/mm^2 (Table 4) while the 275 g block has the force break of 7652 N and deflection breaks at 3.734 mm. The time to failure is 22 seconds for the maximum Young Modulus of 212 N/mm^2 (Table 5).

Generally, the 275 g block of fire clays composites requires the maximum break force and has the maximum Young Modulus relatives to blocks 255 g and 265 g of clays composites under study. The 275 g block of fire clay composites will be better for furnace lining application than the 255 g and 265 g blocks.

Figures 6, 7 and 8 shows the plots of Force (N) against De-

Table 2. Estimated grain sizes and mineral constituents of coarse Clay

Peak no	2theta (rad)	FWHM	Grain Size (nm)	Constituents
1	12.501	0.025	338.0839019	Interstratified illite- Smectile
2	17.45	0.037	229.7356597	Gypsum
3	19.86	0.128	66.63776651	Kaolinite
4	20.941	0.136	62.82444633	Jarosite
5	21.165	0.164	52.11724848	Jarosite
6	21.464	0.0172	497.1759627	Jarosite
7	23.622	0.141	60.87648083	Kaolinite
8	24.901	0.137	62.8043888	Kaolinite
9	24.02	0.12	71.58228586	Quartz
10	26.242	0.113	76.34585664	Kaolinite
11	28.501	0.026	333.40756	Kaolinite
12	34.45	0.038	231.4836125	Microcline
13	35.86	0.124	71.21558276	Pyrite
14	36.941	0.133	66.60274151	Kaolinite
15	37.165	0.162	54.71585907	Pyrite
16	38.464	0.017	523.4384171	Kaolinite plus Interstratified illite- Smectile
17	39.529	0.022	405.8083969	Kaolinite
18	40.43	0.033	271.3138386	Quartz
19	41.821	0.122	73.72312076	Kaolinite
20	42.937	0.134	67.37494247	Quartz plus kaolinite
21	45.178	0.161	56.52152987	Quartz
22	48.426	0. 170	54.14907336	Quartz
23	50.643	0.144	64.5478197	Quartz plus kaolinite
24	51.936	0.13	71.88749106	Quartz
25	55.04	0.134	70.70014627	Kaolinite
26	56.228	0.12	79.38153044	Quartz
27	60.52	0.022	442.1458637	Interstratified illite- Smectile
28	62.439	0.03	327.4856536	Kaolinite

Table 3. Compressibility Analysis of 255g Block of Clay

Test No	Def. @ Break (mm)	Def. @ L.O.P. (mm)	Def. @ Peak (mm)	Def. @ Yield (mm)	Force @ Break (N)	Force @ L.O.P. (N)	Force @ Peak (N)
1	4.343	2.087	4.186	2.303	2631.700	2911.200	9924.000

Test No	Force @ Yield (N)	Strain @ Break (%)	Strain @ L.O.P. (%)	Strain @ Peak (%)	Strain @ Yield (%)	Stress @ Break (N/mm^2)	Stress @ L.O.P. (N/mm^2)
1	3469.000	7.896	3.795	7.611	4.187	2.056	2.274

Test No	Stress @ Peak (N/mm^2)	Stress @ Yield (N/mm^2)	Time to Failure (Secs)	Time to Peak (Secs)	Youngs Modulus (N/mm^2)	Tangential Modulus @ 0.000 N/mm^2 (N/mm^2)	Secant Modulus 0.000 to 0.000 N/mm^2 (N/mm^2)
1	7.753	2.710	26.133	25.187	174.476	4.727	

flection (mm) for the 255 g, 265 g and 275 g fire blocks of clay composites respectively. Figure 9 compares the behaviours of the three fire blocks together. The plot reveals the maximum load of the fire blocks at respective deflection (mm). Our interest in these plots is to investigate and compares the maximum load fire block clays composites can withstand. The 275 g block has the maximum compressive strength and Young Modulus of 7652 N and 212 N/mm^2 respectively making it better than the 255 g and 265 g blocks for furnace lining application.

Table 4. Compressibility Analysis of 265 g Block of Clay

Test No	Def. @ Break (mm)	Def. @ L.O.P. (mm)	Def. @ Peak (mm)	Def. @ Yield (mm)	Force @ Break (N)	Force @ L.O.P. (N)	Force @ Peak (N)
1	4.671	2.164	3.628	2.273	1438.900	847.400	3851.000

Test No	Force @ Yield (N)	Strain @ Break (%)	Strain @ L.O.P. (%)	Strain @ Peak (%)	Strain @ Yield (%)	Stress @ Break (N/mm^2)	Stress @ L.O.P. (N/mm^2)
1	1038.600	8.493	3.935	6.596	4.133	1.022	0.602

Test No	Stress @ Peak (N/mm^2)	Stress @ Yield (N/mm^2)	Time to Failure (Secs)	Time to Peak (Secs)	Youngs Modulus (N/mm^2)	Tangential Modulus @ 0.000 N/mm^2 (N/mm^2)	Secant Modulus 0.000 to 0.000 N/mm^2 (N/mm^2)
1	2.735	0.738	28.100	21.845	93.892	21.094	

Table 5. Compressibility Analysis of 275 g Block of Clay

Test No	Def. @ Break (mm)	Def. @ L.O.P. (mm)	Def. @ Peak (mm)	Def. @ Yield (mm)	Force @ Break (N)	Force @ L.O.P. (N)	Force @ Peak (N)
1	3.734	2.349	3.132	3.132	7652.000	2964.300	9658.000

Test No	Force @ Yield (N)	Strain @ Break (%)	Strain @ L.O.P. (%)	Strain @ Peak (%)	Strain @ Yield (%)	Stress @ Break (N/mm^2)	Stress @ L.O.P. (N/mm^2)
1	9658.000	6.789	4.271	5.695	5.695	4.270	1.654

Test No	Stress @ Peak (N/mm^2)	Stress @ Yield (N/mm^2)	Time to Failure (Secs)	Time to Peak (Secs)	Youngs Modulus (N/mm^2)	Tangential Modulus @ 0.000 N/mm^2 (N/mm^2)	Secant Modulus 0.000 to 0.000 N/mm^2 (N/mm^2)
1	5.390	5.390	22.443	18.842	212.102	6.752	

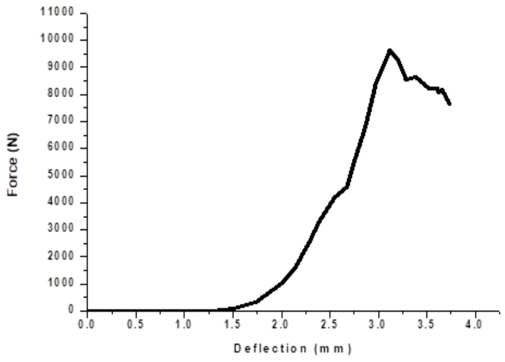

Figure 8. Force (N) against Deflection (mm) of: 275g fine and coarse fire block of clay composites

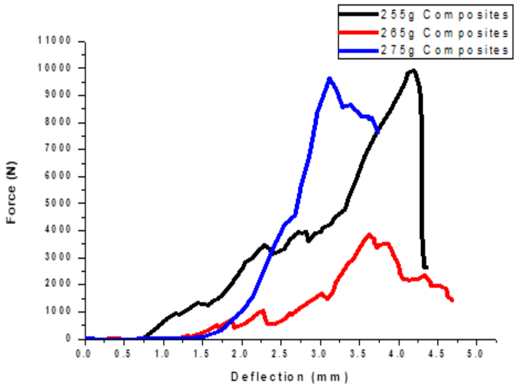

Figure 9. Force (N) against Deflection (mm) of different fabricated fine and coarse fire blocks of clay composites

4. Conclusion

In this research work, Okelele fine and coarse clays have been characterized to establish their potentials for furnace lining application. The maximum compressive strength and Young Modulus as demonstrated by 275 g block clay are 7652 N and 212 N/mm^2 at firing temperature of 1200 ^{o}C. The results of compressive strength analysis, mineral phase's identification and ability to withstand higher firing temperature of 1200 ^{o}C proved that, Okelele fine and coarse fire block of clays meet the needed criteria for use as refractory raw materials.

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