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MINERALOGY OF ILARO CLAY WITH MELON SHELL ASH AND RICE HUSK ASH ADDITIVES TOWARDS PRODUCTION OF REFRACTORY BRICKS

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Abstract. The major mineral constituents of clay are Silica (SiO_2), Alumina (Al_2O_3) and iron (Fe_2O_3). While the silica content impact strength, alumina content infuse refractoriness, and iron reduces refractoriness. The clay in and around Ilaro axis lack adequate mineralogical data. The study is aimed at investigating the mineralogical composition of clay deposits from three locations in Ilaro, Southwest Nigeria, namely; Lesli, Oke-ola and GRA with blend of melon shell ash (MSA) and rice husk ash (RHA) additives. The clay was harvested and characterized mineralogically using X-ray Fluorescence (XRF). The clay, MSA, and RHA mixes were used to produce ten batches of Refractory bricks using Taguchi Orthogonal array namely: BJ_{m0}, BJ_{m1}, BJ_{m2}, BJ_{m3}, BJ_{m4}, BJ_{m5}, BJ_{m6}, BJ_{m7}, BJ_{m8} and BJ_{m9}. The mineralogy, phase evolution and structural morphology of the produced refractory bricks were examined using XRF, X-ray Diffractometer (XRD) and Scanning Electron Microscopy (SEM). Results of the XRF for Lesli has alumina, silica and iron contents of 38.39%, 38.39%, and 13.3%; Oke-ola has alumina, silica and iron contents of, 40%, 53.5%, and 2.3%; while GRA has alumina, silica and iron contents of 44%, 48% and 2.2% respectively. Results for XRF of the refractory bricks produced from GRA clay deposit showed that values of alumina, silica and iron appreciated to; 45%, 56% and 1.28%, respectively. Results for XRD and SEM showed intense peak diffraction phases and scale-like grains of alumina and silica in the clay. The study found that the mineralogy of Ilaro clay without additives conform to acceptable refractory standards for low heat duty applications.

Keywords: Clay, mineralogy, refractoriness, additives, and low-heat duty

1.0 INTRODUCTION

Clay minerals typically form over long periods of time from the gradual chemical weathering of rocks, usually silicate-bearing, by low concentrations of carbonic acid and other diluted solvents. These solvents, which are often acidic, seep through the upper weathered layers before migrating through the weathering rock. Aside from the weathering process, hydrothermal activity also forms some clay minerals (Yagya et al., 2020). The two stages of a clay deposit are called primary and secondary deposits. While the secondary clays have been transported by water dissolution from their specific location and preserved in another sedimentary deposit, the primary clays are framed as a remnant in soil and captured at the arrangement site (Ameh & Obasi, 2021). Clay takes a long time to develop because of a process called chemical weathering of rocks, usually silicate-bearing ones, which occurs gradually due to low centralization of weak solvents like carbonic acid (Bordia & Camacho-Montes, 2022). Certain clay minerals are created during this aqueous action process, which sees the dissolveable drain the top layers of the enduring stone and then pass through it. They have different compositions depending on the earth's variations in both chemical and physical where clay deposits are located (Shuaib-Babata et al., 2024).

The main chemical make-up of clay are silica, alumina, and water; iron, alkalies, and alkali earth metals are frequently added in calculable proportions. Most clay minerals have atomic cross sections that correspond to two basic components. In order to place aluminum, iron, and magnesium iotas in an octahedral matrix so that they are equally spaced from six oxygen or hydroxls, one unit is made up of firmly pressed oxygen and hydroxls (Yagya et al., 2020). The next unit is composed of tetrahedrons of silica. The arrangement of the silica tetrahedrons results in a hexagonal system that is randomly reassembled to form the sheet $Si_4O_6(OH)_4$ (Osarenmwinda & Abel, 2023). Therefore, the application potential of any type of clay mineral found in nature depends on its composition, structure, and other inherent qualities (Kumar et al., 2020). Many techniques, such as warm differential examination, vital inquiry by nuclear retention examination, infrared spectrometry, and X-ray diffraction, are used to identify the specific clay minerals. A crucial step forward in establishing the concept of minerals is the study of substance. In this regard, clay minerals are classified into different groups as follows: Chorites, Illite, Vermiculite, Smectite, and Kaolinite (Umaru et al., 2021).

Clay has been used for agriculture, industry, and building since the prehistoric era. It is utilized as a building material and can be fired or sun-dried into bricks. Clay is also very important to industry. For instance, it is used to make porcelain, china, and earthenware, as well as pipes for sewage and drainage, and wall and floor tiles. In foundry work, highly absorbent bentonite is frequently used to prepare the moulding sands for metal casting as well as to face the molds (Yagya et al., 2020). The oil industry mostly uses the less absorbent bentonite as deodorizing and filtration agents during the refining process of petroleum. For the paper, ceramics, plastics, and rubber industries, clay is a useful raw material. Drilling muds made of clay combined with other ingredients are used to protect the cutting bit during drilling (Yagya et al., 2020). To recognize that the quality and strength of the material will play a major role in the nation's enhanced modern use of clay minerals, thorough assessments of this resource are necessary. Currently, Nigerian neighborhood networks rely on traditional knowledge to create certain clay products whose quality is debatable and does not meet fair criteria (Nurudeen, 2020). Despite the enormous potential, clay minerals are still terribly underdeveloped, and the few pockets of clay-based businesses that do exist have essentially addressed the crude for the production of fired products and auxiliary items. If properly handled, these clay deposits may prove to be a substantial source of unknown wage labor. Accordingly, it is crucial to consider the microstructure morphology and determine the mineralogical, fundamental, and utilitarian gathering arrangement in such clay deposits in order to determine the profitability of employing clay from a particular deposit for any application (Anumata, 2021).

According to Ugheoke et al. (2025) deposits of clay raw materials are widely dispersed throughout Nigeria's six geopolitical zones and have been widely studied. In Orin, Igbara Odo, Ikere and Ado Ekiti in Ekiti State, South-Wastern Nigeria, samples of clay were harvested and examined by Waing et al. (2022) from the result it sufficed that kaolinite is the predominant clay mineral of the examined clay deposits with aluminum phosphate, quartz, and potassium iron oxide minerals. From Ikenanzizi Obowo Local Government region, Imo State, South Eastern Nigeria, clay samples were randomly harvested and examined by Anumata (2021), from the results the main constituent of the clay were kaolinite with high quartz and average SiO_2 composition of 58.30±19.32 %, pH range from 1.91-2.89, specific gravity range from 2.68 – 2.96. The authors, however, concluded that clay is a good fit for manufacture of better refractory materials for intense temperature applications owing to their high content of kaolinite. More so, clay samples were harvested from Yauri, Dukku, and Jega rivers banks, Kebbi State, North West, Nigeria, and analysed by Zaidu (2023) from the results the clay minerals were mainly kaolinite, illite, bentonite, quartz, gibbsite, and feldspars. The authors, therefore, maintained that clay minerals from Dukku and Yauri possess the requirements for use in refractory application and foundry work, whereas, Jega clay mineral can be used for commercial purposes after reduction of its impurities like iron.

Ogun State is among the states enriched with clay. These clays are yet to be properly characterized. It cannot be said precisely if regional distribution of clays in Nigeria play an important role in the mineralogy, elemental composition and performance of these clay, since not all deposits of clay have been studied (Anumata, 2021). The mineralogy of these clay deposits could be tied to the geology of the environment from which they are obtained. The quality of these clay found determine its application and sustainability for ceramic products such as in bricks, ceramic wares, and refractory. In Ilaro environ of Yewa South Local Government Area of Ogun State, clay minerals in the area are used by the lay men in the construction of mud houses. The determination of the composition of thses clay necessary so as to determine its probable areas of application. Futhermore, the findings of the study will also provide documentation of relevant information on clay reserves, and the economic significance of the mineral. This study is focus on determining the mineralogy of clay and produced bricks with blend of additives from Ilaro in Yewa South Local Government Area of Ogun State.

2.0 METHOD

2.1 Study Location

Ilaro and its environ is located in Ogun State, Southwestern Nigeria, and it is the headquarter of Yewa South Local Government Area. Ilaro is geographically located on longitude 3^{o}1’36.2 E to 3^{o}0’42.4 E, and latitude 006^{o}52’9.6 N to 006^{o}54’49 N, and falls within the Dahomey basin (Oluwaseun et al., 2019).

2.2 Sample Preparation

The clay sample was randomly collected in lumps from three deposits site namely; Lesli, Oke-ola and GRA at Ilaro in Yewa South, Ogun State (Figure 1). The clay lump materials were crushed, sieved with 250 micron sieve and dispersed in a wide stainless bowl container. It was stirred vigorously to ensure proper dissolution and left for five days. The dissolved clay was washed and filtrated using the same sieve size in order to remove impurities. The filtrate obtained was allowed to settle before decanting excess water to obtain slurry. This slurry was sun dried for two weeks and oven dried at temperature of 100^{o}C for 5 hours, after which it was pulverized and sieved to obtained clay particle size of 75 micron. Melon shell and rice husk were sourced from Sayedero and Oja Odan market in Ilaro. The sample with sun dried for five days, thereafter, incinerated to form ash. Among the tools needed for this are sieve, bowl, mortar and pestle.

2.3 Production of Refractory Brick

To create a homogeneous material mixes, the clay was next combined with Melon shell ash (MSA) and later with Rice Husk Ash (RHA) additives using Taguchi orthogonal array formulations, as represented in Table 1. The basic materials were mixed manually. The homogeneous material mixes was then supplemented with 15% by mass of Ordinary Portland Cement (OPC) of grade 42.5N as a chemical stabilizer until the proper mixing is achieved. Refractory bricks produced

(Figure 2) was subjected to mineralogical, SEM (Scanning electron microscope) analysis and XRD (X-ray Diffraction) analysis.

Table 1: Percentage Proportion of Materials Mixes

Experimental Run	Kaolin + cement (wt%)	MSA (wt%)	RHA (wt%)	Sample Identity
1.	100.0	0.0	0.0	BJ_{m0}
2.	75.0	12.5	12.5	BJ_{m1}
3.	50.0	25.0	25.0	BJ_{m2}
4.	25.0	37.5	37.5	BJ_{m3}
5.	75.0	17.5	7.5	BJ_{m4}
6.	75.0	7.5	17.5	BJ_{m5}
7.	50.0	35.0	15.0	BJ_{m6}
8.	50.0	15.0	35.0	BJ_{m7}
9.	25.0	50.0	25.0	BJ_{m8}
10.	25.0	25.0	50.0	BJ_{m9}

Figure 1: Clay Sample harvested at Ilaro **Figure 2: Produced Bricks**

2.4 Mineralogy

The mineralogy of the clay sample, additives and produced refractory bricks were done using X-ray Fluorescence, Ragaku Machine at Center of Excellence in Agricultural Development and Sustenance Environment (CEADESE) Laboratory, Federal University of Agriculture, Abeokuta (FUNAAB). Meanwhile, the Phase evolution and morphology of the bricks samples were done using X-ray Diffraction (XRD) machine, and Scanning Electron Microscope at Mechanical Engineering Laboratory, Covenant University, Ota, Ogun State.

3.0 RESULT AND DISCUSSION

The results obtained from the method as highlighted in section 2 are presented in this part of the paper, which conform to the set objectives of this research. This borders on the mineralogy of clay deposit and additives, morphology of the produced bricks and face evaluation of the produced bricks.

3.1 Mineralogy of the Clay Deposit and Additives

The results for the mineralogy of GRA clay deposit and additives are presented in Table 2 and the mineralogy of the produced refractory bricks are presented in Table 3. According to the results, Ilaro Clay had 44.0% silica and 48.0% alumina compositions before the addition of the additives. Conversely, Table 2 shows that the silica compositions of rice husk ash and melon shell ash are 78.35 percent and 74.8%, respectively. When the kaolin was combined with additives to make refractory bricks, the results increased to 45% and 56% of silica and alumina, respectively (Table 3).

Table 2: Mineralogy of Ilaro Clay and Additives

Compounds	Clay	RHA	MSA
Al_2O_3	44.40	0.18	2.84
SiO_2	48.20	78.35	74.80
Fe_2O_3	2.20	0.23	1.13
CaO	0.05	2.72	2.31
MgO	ND	ND	0.40
K_2O	1.60	4.55	4.66
TiO_2	2.40	ND	ND
Na_2O	ND	ND	0.62
SO_3	ND	ND	0.72
P_2O_5	ND	2.88	10.01
V_2O_5	0.50	ND	0.01
MnO	0.14	ND	0.37
Cr_2O_3	0.27	ND	ND
BaO	0.20	ND	0.09
CuO	0.06	ND	ND
Cl	0.12	ND	ND
ZnO	ND	0.04	0.05

Christopher (2016) opined that these values are adequate to show that the improved Ilaro clay falls within the category of refractory fireclay. In comparison to Kankara, Mokwa, Auchi, Ijero Ekiti, Nsu, Warri, Maraba-Rido, Malete, and Eket, which had alumina contents of 31.73%, 28.7%, 27.11%, 30.31%, 31.24%, 31.12%, 33.14%, 29.6%, and 27%, respectively, improved Ilaro Clay's alumina content was favorable (Sutas et al., 2022).

The clay is between 45% and 65% Al_2O_3, which is the typical range for fireclays. Nonetheless, its silica concentration fell within the globally recognized range of 25–45%, with a small excess over the maximum limit of 45% (ASTM, 2013).

Table 3: Mineralogy of the Produced Refractory Bricks

Compound	BJ_{m0}	BJ_{m1}	BJ_{m2}	BJ_{m3}	BJ_{m4}	BJ_{m5}	BJ_{m6}	BJ_{m7}	BJ_{m8}	BJ_{m9}
Al_2O_3	43.40	42.30	41.80	40.60	45.00	42.80	43.70	42.10	38.90	39.40
SiO_2	48.70	50.10	53.80	51.40	56.00	52.10	51.50	50.70	53.70	52.50
Fe_2O_3	1.80	1.69	1.33	1.31	1.28	1.27	1.25	1.23	1.22	1.21
CaO	0.05	2.30	1.80	1.60	1.45	1.34	1.22	1.24	1.18	1.16
MnO	0.14	0.31	0.20	0.18	0.16	0.22	0.24	0.25	0.23	0.21
MgO	ND	0.28	0.26	0.25	0.22	0.20	0.18	0.16	0.14	0.12
SO_3	ND	0.52	0.34	0.29	0.27	0.25	0.22	0.20	0.18	0.15
K_2O	2.10	0.36	0.28	0.22	0.20	0.18	0.14	0.12	0.10	0.09
P_2O_5	ND	5.70	3.45	3.20	2.90	3.00	3.20	3.40	3.42	3.38
Na_2O	ND	0.40	0.24	0.21	0.18	0.16	0.14	0.12	0.10	0.08
ZnO	ND	0.34	0.19	0.15	0.13	0.11	0.08	0.06	0.05	0.03
Cl	0.16	0.09	0.07	0.10	0.12	0.14	0.16	0.18	0.20	0.22
BaO	0.20	0.18	0.16	0.17	0.18	0.15	0.13	0.11	0.09	0.10
Cr_2O_3	0.20	0.26	0.22	0.20	0.18	0.15	0.14	0.12	0.10	0.05
CuO	0.50	0.05	0.03	0.07	0.10	0.14	0.17	2.01	2.30	2.32
V_2O_5	0.70	0.46	0.30	0.60	0.11	0.21	0.23	0.41	0.64	0.81

3.2 Morphology of the Produced Bricks

The result for the morphology of BJ_{m0}, BJ_{m3}, BJ_{m4} and BJ_{m8} produced refractory bricks are presented in Figure 3 – 6. The results revealed a scale-like grains of silica and alumina in the bricks, which conformed to what was reported by Mokwa & Salihi (2011).

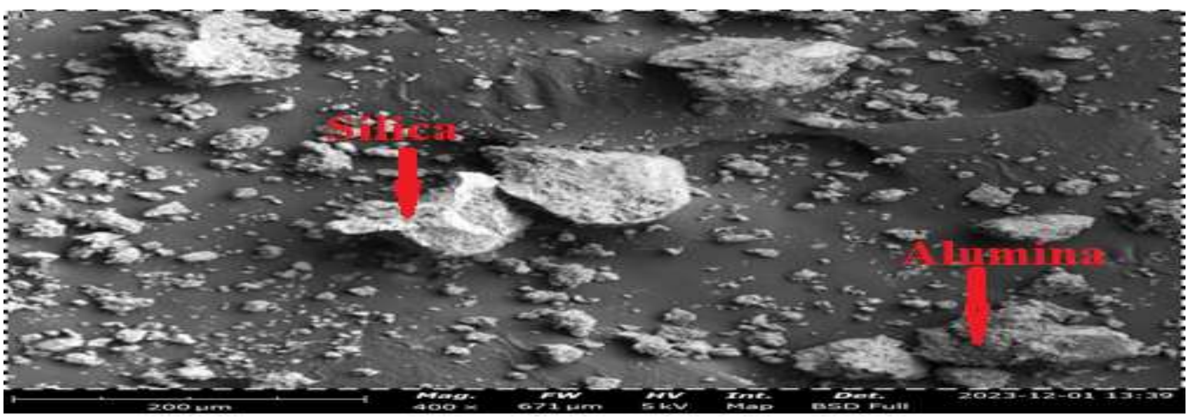

Figure 3: SEM result for BJ_{m0} produced bricks

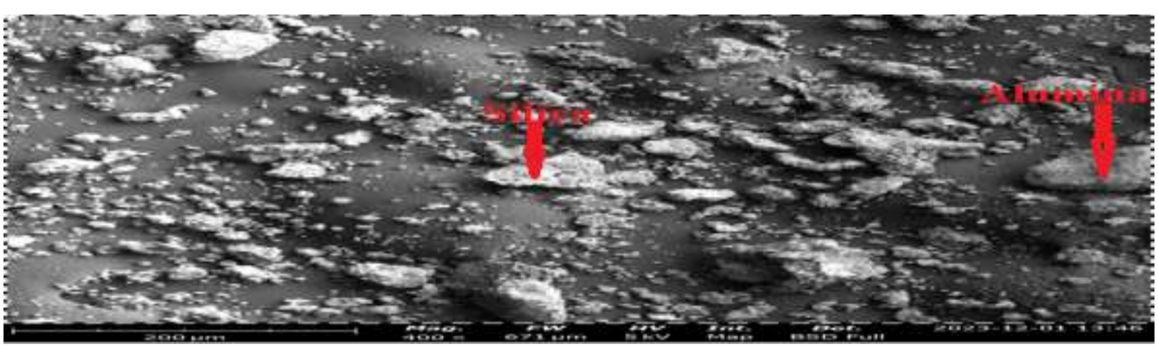

Figure 4: SEM result for BJ_{m3} produced brick

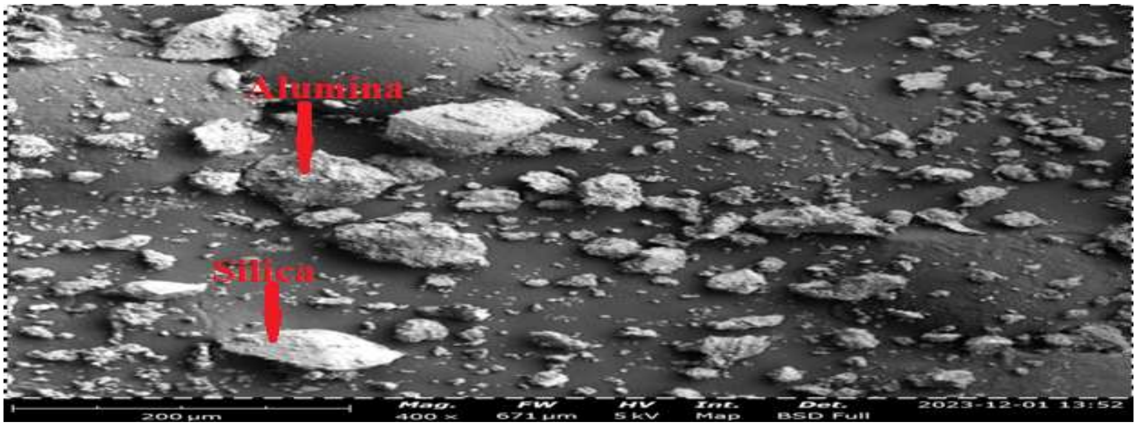

Figure 5: SEM result for BJ_{m4} produced brick

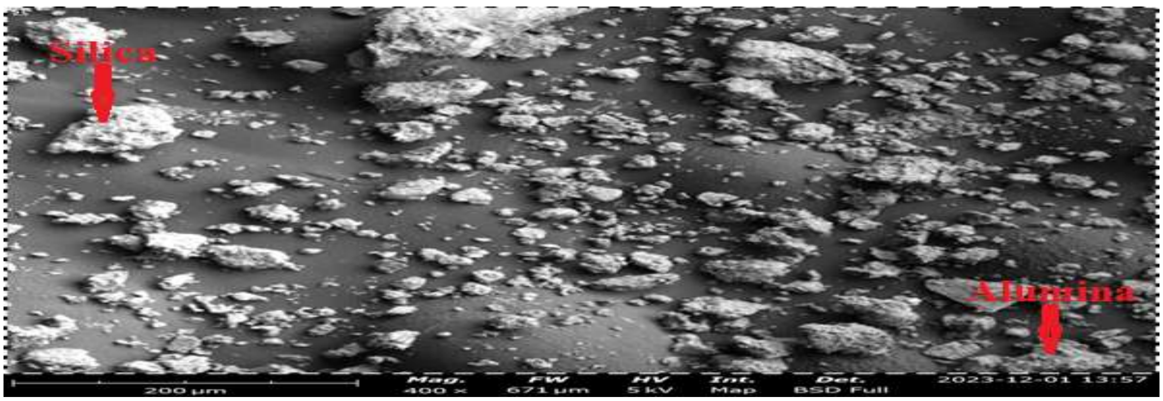

Figure 6: SEM result for BJ_{m8} produced brick

3.3 Phase Evolution of the Produced Bricks

The results for the phase evolution of the BJ_{m0}, BJ_{m3}, BJ_{m4} and BJ_{m8} produced bricks are presented in Figure 7 – 10. It can be seen from the results that Quartz and kaolinite are the dominant minerals present in the produced bricks sample. These results are in tandem with what Yagya et al. (2021) reported on clay mineralogy in Southwestern, Nigeria.

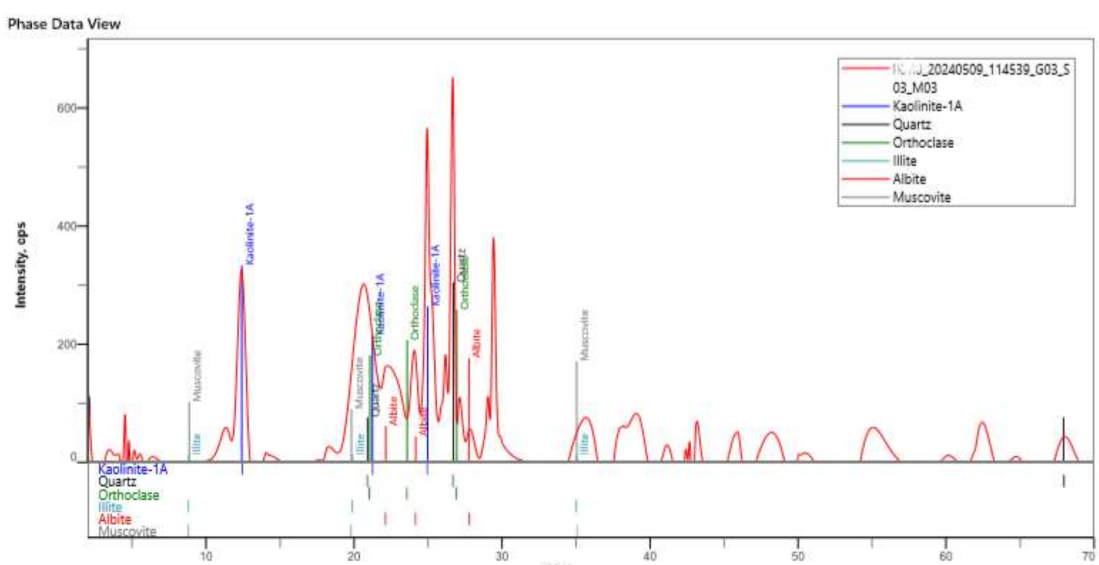

Figure 7: XRD Result for BJ_{m0} Produced Brick

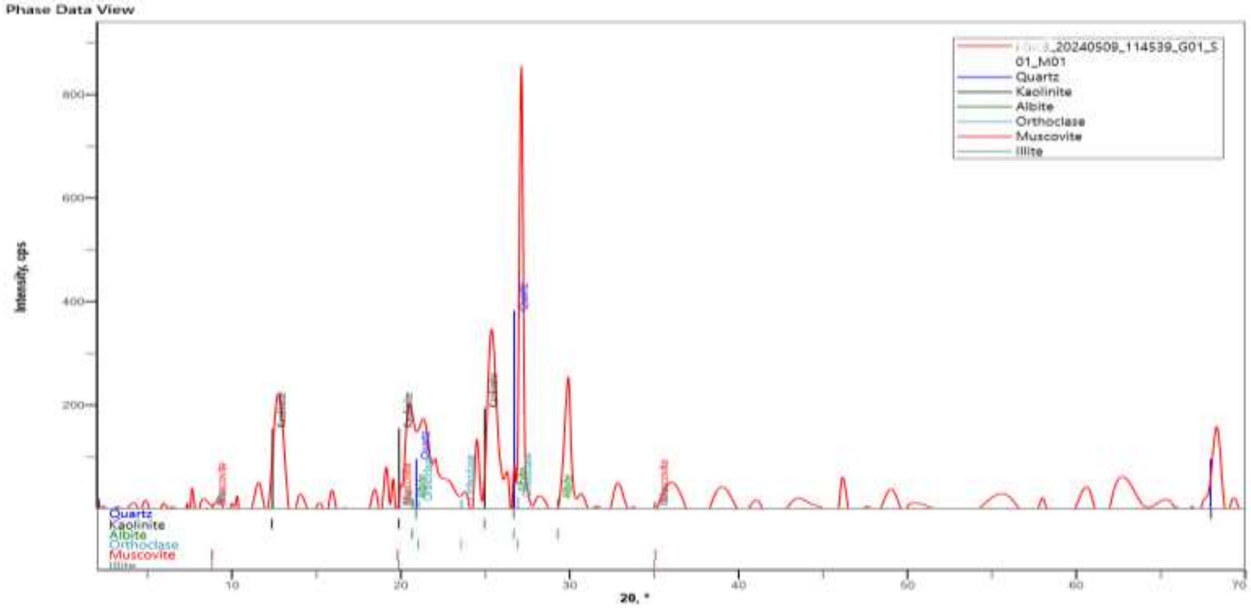

Figure 8: XRD Result for BJ_{m3} produced Brick

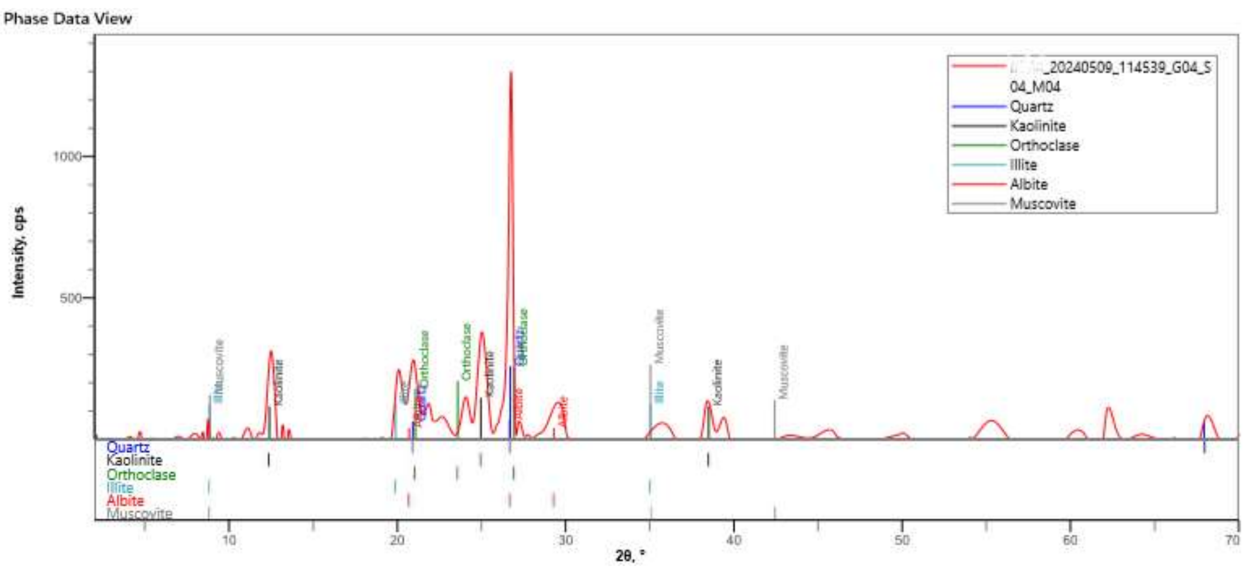

Figure 9: XRD Result for BJ_{m4} Produced Brick

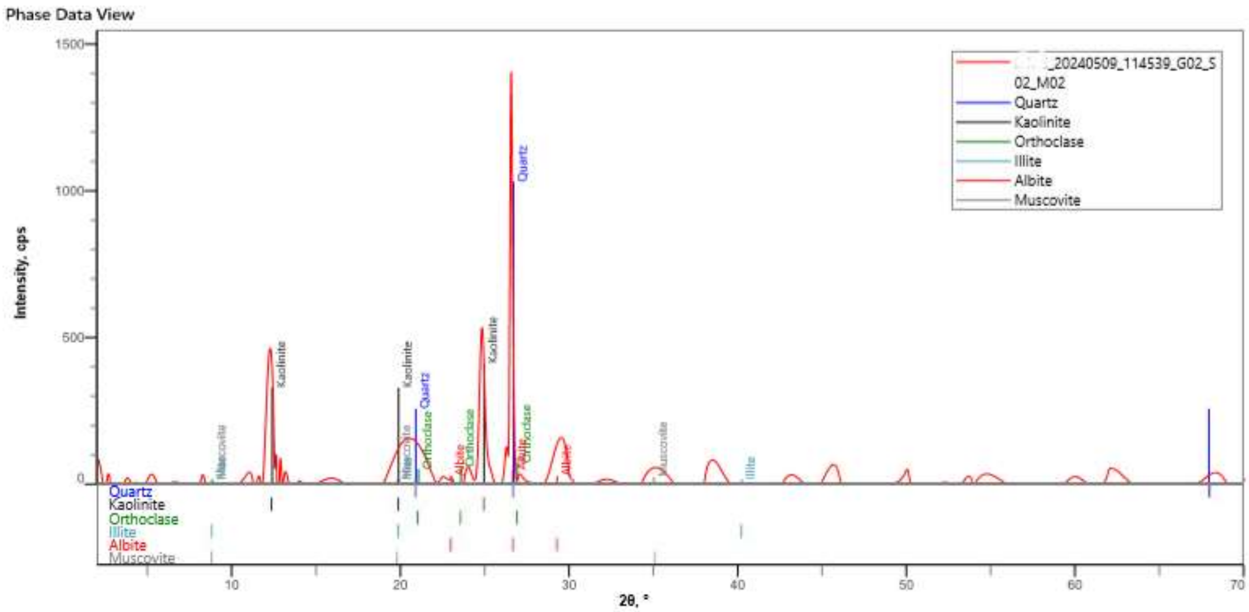

Figure 10: XRD Result for BJ_{m8} Produced Brick

4.0 CONCLUSION

The mineralogical compositions were successfully determined for the clay deposit and additives and the materials were used to produce the refractory bricks. Some 48% of the minerals in the clay were silica, and 44% were alumina. These values increased to 45% alumina and 56% silica in the refractory bricks that were produced. The silica and alumina concentrations of rice husk ash and melon shell ash are 78.35%, 0.18%, and 74.8%, 2.84%, respectively. The samples' chemical compositions fell within the range for the standard clay and additives. The morphological results demonstrated that all of the refractory bricks contained silica and alumina grains that resembled scales, which is why their characteristics compared favorably to those of the standard. On the other hand, the phase evolution data indicated that the predominant minerals in the bricks are alumina and silica.

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