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PROCESSING AND CHARACTERIZATION OF SELECTED CLAYS AND NON-PLASTIC MINERALS FOR ADVANCED STRUCTURAL APPLICATIONS

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Abstract: **Natural clays are readily available in many geographical locations in Nigeria and their use for advanced materials have limited by processing and properties assessments. This work was carried out to process and characterize some selected clays for structural applications. Ball clay and stoneware clay were obtained from Kogi State, Nigeria and were modified with some non-plastic minerals. The processed clays were achieved by ball milling with the addition of non-plastic minerals (feldspar, limestone and talc) and milled at 150 rpm for 30mins. The slurry was sieve with 100 µm sieve sizes and made to pass through a magnetic wet separator in other for the impurities to be reduced to a tolerable amount and spray dried at temperature of 220°C and the dried powder was calcine at 800°C for 30 minutes. The raw and processed clays were characterized with XRF, FTIR and SEM to determine the chemical compositions, the functional group as well as the microstructural features, respectively. The results of the characterization revealed that silicon (Si) followed by aluminum (Al) were the major elements in the particles while the spectral band suggested the presents of quarts and others minerals with Si-O stretching modes. The SEM micrographs revealed good proper mix and interaction between the clays and the solid minerals which suggested the adaptability of the materials for production of advanced materials structural components.**

Keywords: **Natural clay, solid minerals, structural components, advanced materials, processing, characterization.**

1. INTRODUCTION

Clay an earthy material that is plastic when moist but hard when fired composed mainly of fine particles of hydrous aluminum silicates and other minerals with soft, freely bound, fine grained natural rock having diameter less than 0.005 mm and composed essentially of particles. Natural clay refers to any naturally occurring soil-like substance that is abundant in aluminiumphyllosilicates. The composition of these phyllosilicates along with other compounds and the conditions of formation dictates the physical properties of clay. It is categorized into kaolin-type (including kaolin itself), smectite-type (such as montmorillonite), and illite-type (with illite being the primary example). However, a more practical classification involves grouping clays based on their intended use which indirectly relates to their chemical and physical compositions (Ochieng,2016; Adeyanju *et al.*, 2019). Clay minerals have been extensively used in preparing microsized dispersion on polymer matrix to bring about the desired enhanced properties (Sudhakar et al., 2021). However, sodium montmorillonite

(Na-MMt) a natural smectite clay (2:1 phyllosilicate) consisting of aluminosilicate layers with a high aspect ratio and a high surface area was subsequently used to synthesize polymer-clay nanocomposites (Adewumi *et al.*, 2023; Oladele *et al.*, 2024).

Among the clays, kaolin clay exhibits high levels of temperature maturity making it suitable for high-fire applications with firing temperatures reaching up to 1,800°C. Often, kaolin is blended with other clays to enhance workability and reduce firing temperatures. When fired, kaolin can become exceptionally hard and translucent with a smooth and glossy surface that eliminates the need for glazing. However, thin layers of kaolin are prone to brittleness and chipping at the edges which necessitate the need for blending kaolin with other clays to produce more durable items. Nevertheless, cured kaolin demonstrates good abrasion resistance (Ochieng, 2016; Neeraj and Chandra, 2021). Ball clay on the other hand is known for its high plasticity and minimal mineral impurities with varying colors depending on moisture levels. It typically appears dark grey when moist and light grey or buff after firing reaching maturity at a lower temperature of around 1,280°C.Due to their significant shrinkage during drying or firing, ball clays are often unsuitable for standalone use. However, when blended with other clays like porcelain, they offer enhanced workability. They are renowned for their rheological stability and light color after curing making them versatile for various applications (Praise, 2022). They can be mixed with stoneware clays to achieve a distinct finished appearance.

Stoneware clays are characterized by their high plasticity and are seldom found in pure form. When fired, they become hard and durable, resembling stone in texture and appearance. Stoneware clays have dense surfaces with flecked coloration and are typically fired to maturity at temperatures ranging from about 1,176°C to 1,237°C for mid-fire stoneware clay bodies, and approximately 1,204°C to 1,224°C for high-fire stoneware clays (Praise, 2022).Stoneware-type clays exhibit a diverse range of colors upon firing, spanning from light grey to chocolate brown or dark grey with the specific hues largely influenced by the firing temperature employed (Praise, 2022).These clays are highly favored for functional items such as dinnerware due to their robust and durable nature, making them the preferred choice for production potters. Stoneware interacts harmoniously with glazes and typically achieves leak-proof status when fired to maturity.It's crucial to acknowledge that stoneware clays exhibit considerable variation in composition, containing varying amounts of fire clay, kaolinite, quartz, mica, feldspar, and other minerals. The kaolinite content often displays significant disorder, while mica and quartz are generally found in fine particle sizes. Additionally, the presence of flint can vary across different samples.

Ceramics powder with chemical formula $Al_2Si_2O_5(OH)_4$, SiO_2, $KAlSi_3O_8$ comprises of clay particles, modifiers, and additives aimed at enhancing the ease of handling the powder during component fabrication. Among these additives are a binding agent that helps maintain the powder's cohesion after compaction and a release agent which facilitates the easy of removal of a compacted component from die. Ceramics powders are primarily composed of clay, silica and filler materials that are usually non-plastics in nature and serves as reinforcement to the clays as well as the modifier (Zerbo *et al.*, 2019; Hammas *et al.*, 2020). Although ceramics are typically known for their brittleness, they possess various properties that can be exploited to render them suitable for diverse applications. These include automotive engines, aerospace technology, electronics for insulator components, and biomedical applications such as bone and tooth replacements. Additionally, ceramics find use in construction materials and cutting tools, among other areas (Dolores *et al.*, 2019). Hence, this work process clays from different locations by mixing them with mineral rocks and characterize to evaluate their compositions and properties for effective adaptation in advanced materials applications.

2. MATERIALS AND METHOD

The materials that were used are; Caustic Soda (NaOH), Frit (Na_2O, Al_2O_3Si), limestone($CaCO_3$), ball clay, stoneware clay, feldspar, alumina balls and talc which were sourced from Kogi, Edo and Niger States, Nigeria.

2.1 Preparation of the Processed Clay Particles

The processed clay particles were developed by adding plastics materials(ball clay and stoneware clay) and non-plastics materials (feldspar, limestone and talc) with additives (NaOH and frit). Figure 1(a-b) showed the images of the plastic clays; ball clay and stoneware clay, respectively whileFigure 2 (a-c) are the images of the non-plastic minerals; feldspar, talc and limestone, respectively. Figure 3(a-b) are the images of the caustic soda and the frit (activators). Table 1 present the formulation of the processed clays

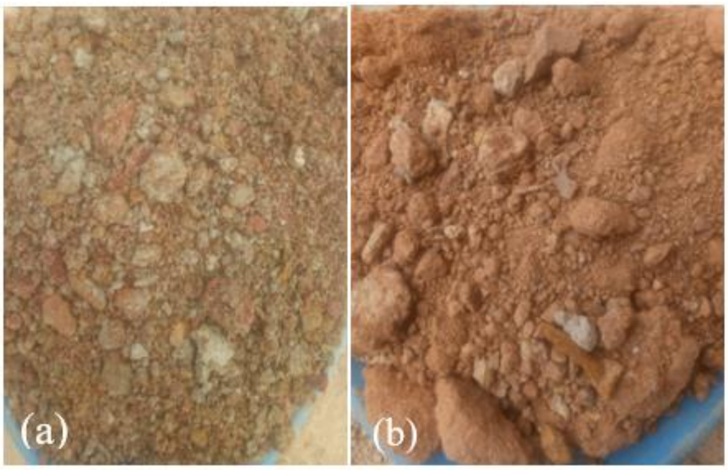

Figure 1: Images of (a) Ball Clay and (b) Stoneware Clay

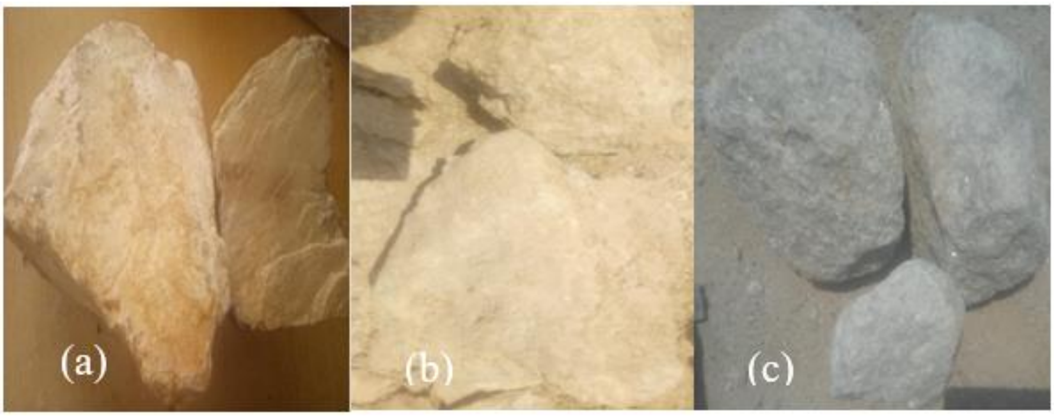

Figure 2: Images of (a) Feldspar Rock, (b) Talc Rock, and (c) Limestone

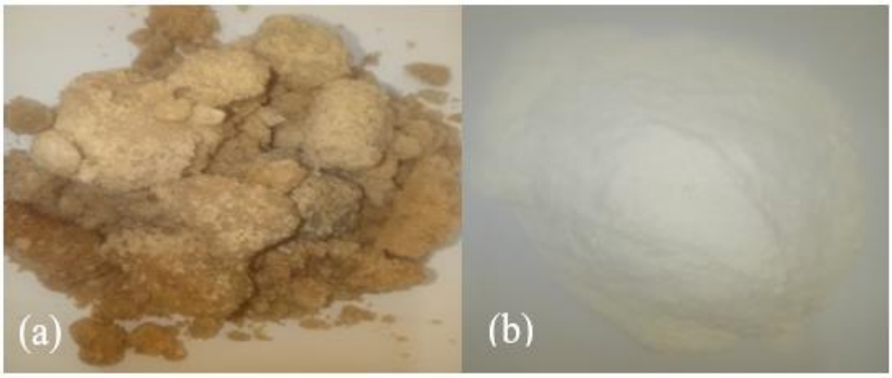

Figure 3: Images of (a) Caustic Soda and (b) Frit

Table 1: Formulation of the Processed Clay

Mineral Materials	Amount (%, g, ml)
Ball Clay	41.2700
Stoneware Clay	25.8000
Talc	5.6500
Limestone	0.9400
Feldspars	24.4000
NaOH	0.3240
Frit	0.0434
Alumina Balls	600.0000g
Distilled Water	150.0000ml

Milling Operation

The non-plastics materials were mixed and milled with the plastics materials using alumina ball and waterin the ball mill at 150 rpm for 30mins for thorough milling. The blend was sieved with 100 µm sieve sizes and made to pass through a magnetic wet separator as shown in Figure 4 in other for the impurities such as ironpresent to be reduced to the tolerable amount.

Figure 4: Magnetic Liquid Trap Set-Up images

Spray Drying Operation

The slurry was spray dried at temperature of 220°C according to Liu and Zhou, (2015) where the principle of atomization of the fluid into a dry powder with the aid of a hot gas stream was adopted and the clay particles were then discharged from the bottom of the equipment as shown in Figure 5and the dried powder was calcine in the laboratory kiln at800°C for 30 minutes.The dried clay particles was sieved thoroughly to screen out the over size.

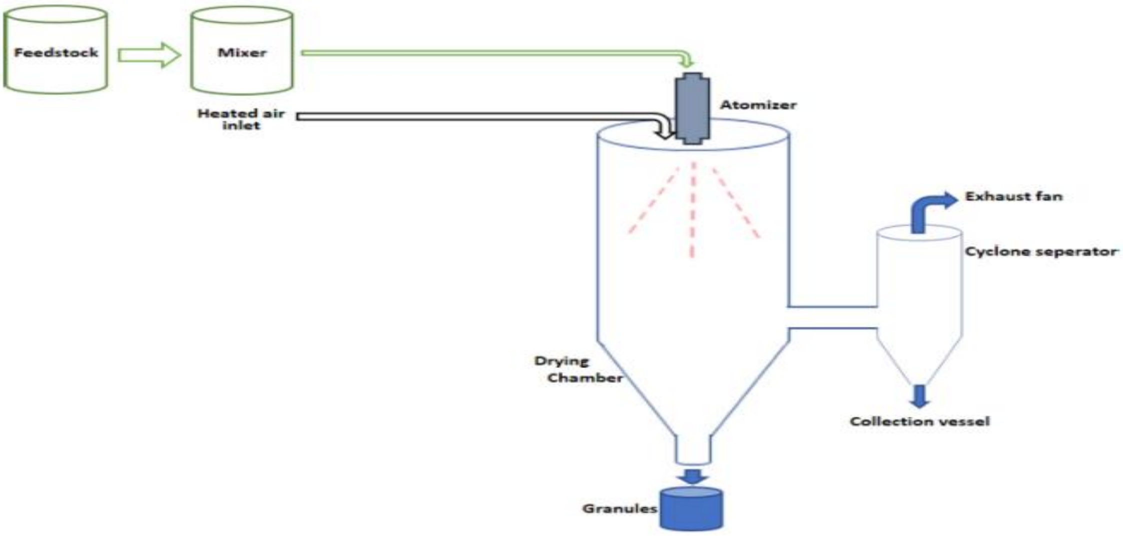

Figure 5: Centrifugal Spray Drying Process

Characterization of the Clay Particles

Scanning Electron Microscopy

The surface morphology characterization of the clay particles were carried out using Tescan VEGA 3 with Oxford instrument detector. All samples were gold coated before subjected to the examination to improve the conductivity of the samples at a voltage of 20 kV.

XRF of Clay Particles

X-Ray Fluorescent (XRF) spectrometry is highly beneficial due to its capability to swiftly offer a detailed evaluation of the relative differences in elemental compositions of various Earth materials. Operating on the wavelength-dispersive principle, XRF spectrometry relies on the emission of X-ray photons by individual atoms, each possessing characteristic energy or wavelength features that can be measured. The samples underwent scanning under ambient conditions, with a scanning rate of 0.02o/min across a scattering 2ϴ angle range. The crystallinity index value was then calculated to assess the degree of crystallinity present in the samples.

FTIR Analysis

A Fourier transform infrared (FTIR) Bruker TENSOR 27 spectrophotometer was employed to identify the functional groups present in both modified and raw clay particulate samples.Powderedclays were mixed with potassium Bromide (KBr) to make the samples transparent. FTIR was operated at 32 scans per minute scanning rate using 2 per cm signal-to-noise ratio resolution in 4000-600 cm^{-1} mode of transmission range at ambient temperature and relative humidity of 25 and 65%.

3. RESULTS AND DISCUSSION

Figure 6showed the morphology of stoneware clay where the presence of powdered particles in mud forms alongside larger grain pieces was observed. The image reveals numerous aggregates, creating pores within the clay structure, hindering proper grain flow. Also, Figure 7 presents the morphology of ball clays showcasing predominantly powdered particles and numerous aggregates with a few larger particles. This observation suggests a mixture of larger and powdered grains within the clay composition which may not encourage proper flow during processing, hence, the need for modification. However, Figure 8 presents the morphology of the processed clay particles where granular skeleton comprising quartz and feldspar grains overlaid by fine clay particles were seen. The physical modification of the clays alongside the processing of the materials actually encourages good interaction and reaction between the clays and the additive used in processing the material which decreased the pores between the particles. This process will enhance the flow ability of the particles and aid proper dispersion during processing.The plastic material that is the clay minerals which were the based materials produce plasticity and binding characteristics to the mass, good rheological flow properties, good density level and enhance mechanical properties while the non-plastics materials function as fluxing agents, impacting the needed strength, toughness and cementing the crystalline phase of other additives, function as modifier as well as fluxing agents in agreement with the observations of previous researchers (Zerbo *et al.*, 2019; Hammas *et al.*, 2020; Oliveira and Santos, 2020; Fuertes *et al.*, 2022).

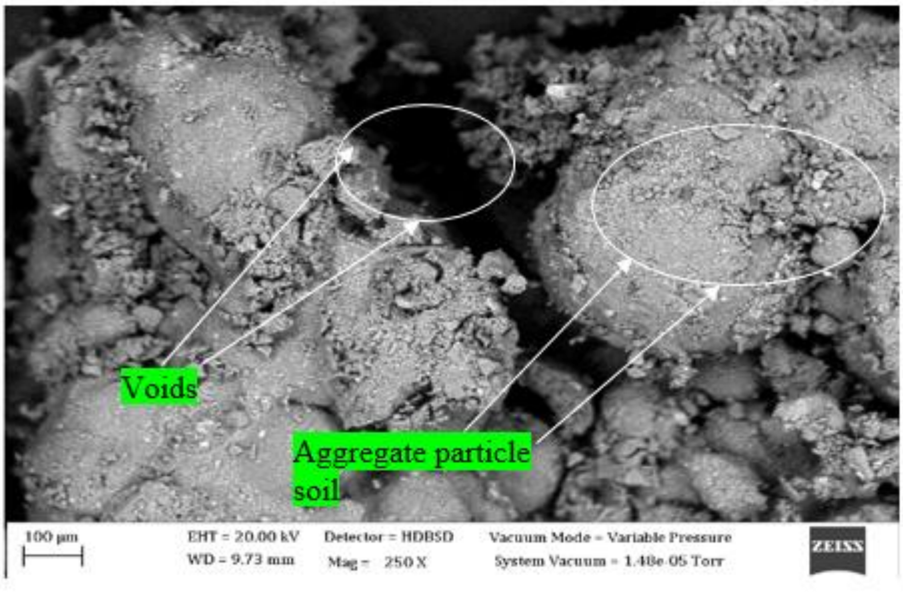

Figure 6: SEM Image of Stoneware Clay

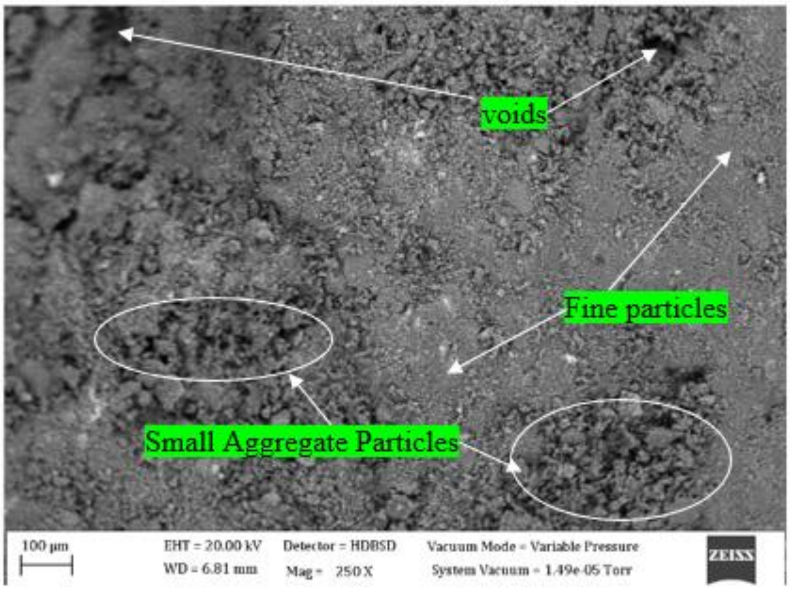

Figure 7: SEM Image of Ball Clay

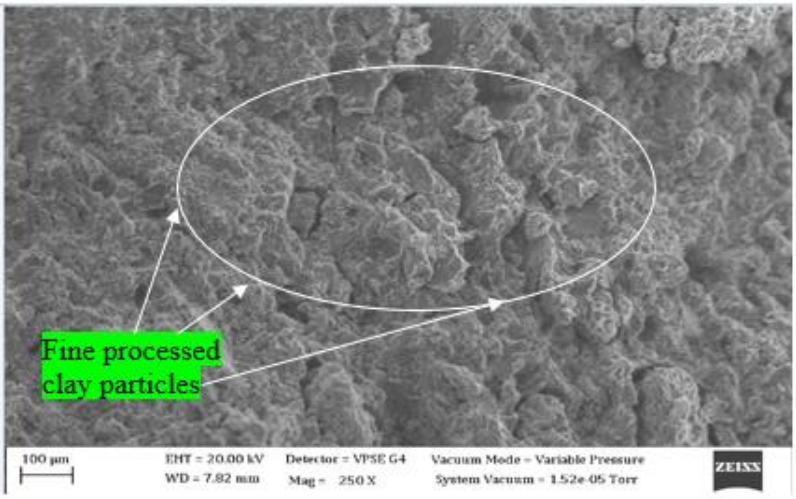

Figure 8: SEM Image of Processed Clay Particles

X-Ray Fluorescence

Chemical compositions analysis of the clay materials and the processed clay materials werecarried out using XRF and the results were presented in Tables 2-4.It was revealedfrom the analysis that, the processed clay have the amount of the trace elements (Fe, S, P, Cu, Zn, Ni and the hazardous elements Sr, Zr, Y and Nb), metals and their oxidesreduced while there were enhancements in the main elements (Si, Al, Ti, K, Na, Mg and Ca) that are desirable. This will aid enhancement in the properties of the material during processing.

Table 2: XRF of the Ball Clay

Metals	Amount
Mg	0.0654
Al	25.3178
Si	59.8465
P	0.4529
S	0.1299
K	1.0495
Ca	0.5663
Ti	5.6952
V	0.0000

Mn	0.0779
Fe	6.0682
Ni	0.0472
Cu	0.0650
Zn	0.0457
Ga	0.0288
Rb	0.0245
Sr	0.0588
Y	0.0129
Zr	0.3739
Nb	0.0409
Pb	0.0326

Table 3: XRF of the Stoneware Clay

Metals	Amount
Mg	0.0258
Al	20.5951
Si	56.6510
P	0.0523
S	0.1594
K	4.7388
Ca	4.0341
Ti	4.1191
Cr	1.0836
Mn	1.0439
Fe	6.9376
Ni	0.0706
Zn	0.0292
Ga	0.0182
As	0.0124
Rb	0.0152
Sr	0.0798
Y	0.0134
Zr	0.2805
Nb	0.0404

Table 4: XRF of the Processed Clay

Metals	Amount
Na	0.2981
Mg	0.8204
Al	16.2930
Si	64.1672
P	0.0514
S	0.0400
K	5.5417
Ca	4.3641
Ti	4.2814
Cr	0.1910
Mn	0.0791
Fe	2.6402
Ni	0.1794
Cu	0.0173
Zn	0.0285
Rb	0.0242
Sr	0.3904
Y	0.0026
Zr	0.2338
Nb	0.0152
Ba	0.3410

FTIR Analysis on Raw Clays and Processed Ceramics Particulate

A total of 34 peaks were identified through direct spectra analysis, as depicted in Figures 9-11. The average spectrum exhibits characteristic absorption bands of quartz within the spectral range of 1200 to 900 cm-1, notably featuring peaks at 790.2 and 670.9 cm-1 in agreement with Müller *et al.*(2014a).The bands observed around 1000 cm-1 were attributed to Si-O stretching modes. Notably, between 3500-3000 cm-1 (O-H stretching), the intensity approached zero for some samples due to calcination and spray drying processes, which effectively eliminated molecular water and surface hydroxyl groups. Nonetheless, the average spectrum generally showcases characteristic absorption bands of quartz within the spectral range of 1114.5 to 900 cm-1, including the double peak at 790.2 and 670.9 cm-1 in agreement with Müller *et al.*(2014). Additional bands can be correlated with microcline (potassium feldspar) and plagioclase feldspar, as evidenced by peaks ranging from 793.9 to 749.2 cm-1 and 674.6 to 670.9 cm-1, respectively, in agreement with prior research (Vahur*et al.*, 2016). Vibrational bands associated with albite (plagioclase/sodium feldspar) and orthoclase (potassium feldspar) minerals are identifiable within the ranges of 1114.5–909.5 and 793.9–670.9 cm-1, respectively (Müller *et al.*, 2014a).Furthermore, significant vibrations of carbonate minerals, likely calcite, are detected within bands at 1423.8 and 793.9 cm-1. This was in agreement with the results of previous researchers (Müller *et al.*,2014; Cantisani*et al.*, 2012). It's important to note that a reduction in band intensity does not signify a weakening of the bond but rather a decrease in the concentration of the "bonds" responsible for such a band. The bond strength influences the position of the band at lower or higher wave numbers: the energy of a band (cm-1) roughly correlates with the "bond order" of the bond responsible for the band and is inversely proportional to the reduced mass of the atoms involved in the vibration.The results of the FT-IR have further validated the results of the X-RF of both the raw clays, and the processed ceramics particulate where silicon dioxide (silica) inform of quartz and aluminium oxide in form of alumina appeared the dominance minerals in the particulate after processing the clays into the ceramic and also, the reduction in the iron and other ferromagnetic elements in the processed ceramics particulates.

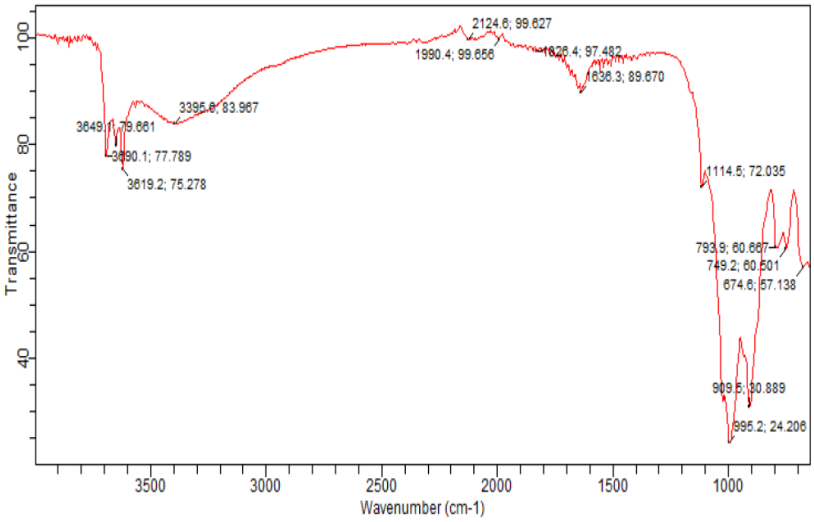

Figure 9: Infrared spectra of raw ball clay

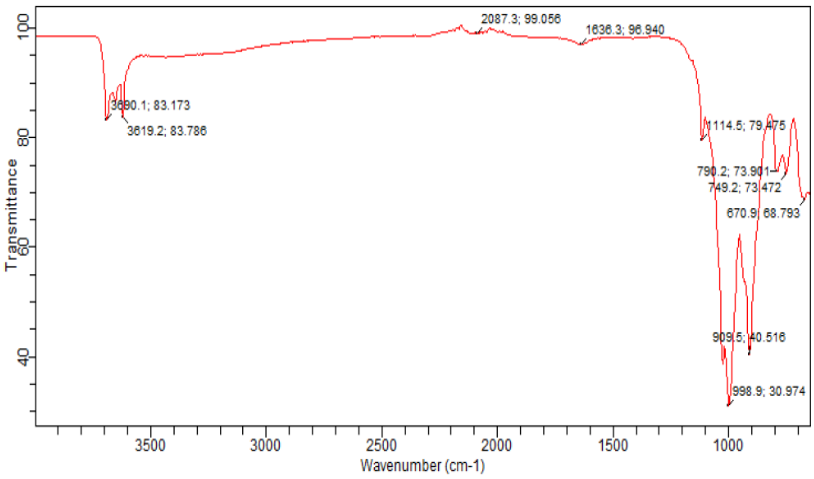

Figure 10: Infrared spectra of stoneware clay

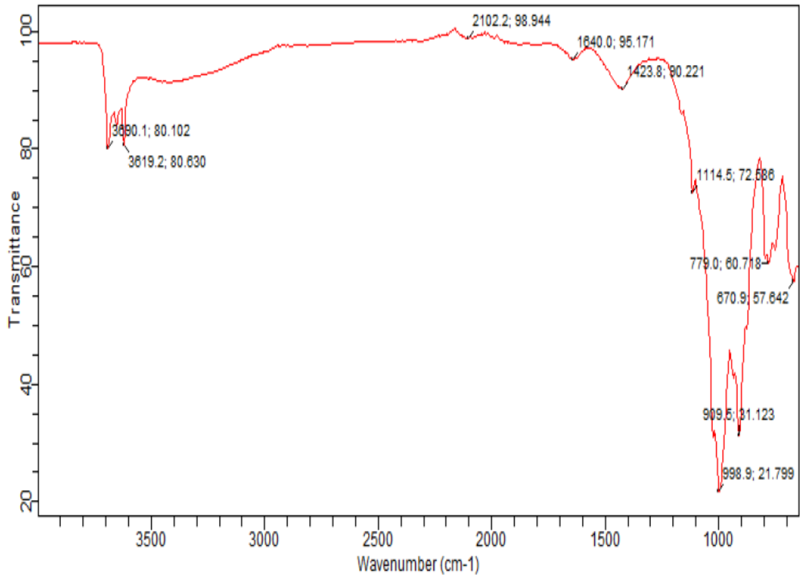

Figure 11: Infrared spectra of the processed ceramics particulate

4. CONCLUSION

The work considers the possibility processing and modifying the composition and properties of ball clay and stoneware clay for advanced structural applications. From the results, it was seen that by adding the non-plastic minerals to mixture of ball and stoneware clays through milling operation, spray drying and magnetic separator, the amount of deleterious elements are reduced while the quantity needed elemental compositions are improved. Thus, leading the desirable functional bands and surface morphology that support flow and easy processing, thereby, enhancing the quality of the product from the materials.

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