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THERMOGRAVIMETRIC AND DIFFERENTIAL THERMAL ANALYSIS STUDY OF INTERLOCKING COMPRESSED STABILIZED EARTH BLOCK AND ITS MICROSCOPIC CHARACTERIZATION

***Hamidu, L. A. J[1]. and Adamu, A. Y[1].**
[1]*Nigerian Building and Road Research Institute,*
10 NBRRI Way/I.T. Igbani Street, off Awolowo Way, Jabi, Abuja, Nigeria
Corresponding author: lucadohamidu@yahoo.com, kiddogimba@yahoo.com*

ABSTRACT

The search for alternative building and road construction material in a strained economic situation is critical for socio-economic activities to thrive. The Nigerian Building and Road Research Institute (NBRRI) has keyed into the technology of making interlocking compressed stabilized earth block (ICSEB), as an alternative building materials. Nevertheless, parameters such as decomposition process and its microscopic properties have not been studied for the user comfort as friendly building material. This paper, examine "the time dependency thermogravimetric and differential thermal analysis (TGA-DTA), scanning electron microscope (SEM) and Fourier transform infrared (FTIR) of the already made ICSEB" based on the 95% laterite and not less than 5% cementing stabilization, with compaction pressure of 3 KN/mm^2. Results revealed that, the ICSEB TGA takes 41.45 minutes, 33.33 minutes and 29.58 minutes to decompose at 10 ^{o}C/min, 15 ^{o}C/min and 20 ^{o}C/min heating rates and the combined TGA-DTA is endothermic process decomposition. SEM morphology showed heterogeneous phase formation with visible cracks, EDX detected 15 elements at 537 µm, having 41.37% carbon, 24.06% silicon, 21.35% aluminium, 5.14% Iron, 3.04% calcium, while others are in traces. These results imply that low aluminium and silicon content with greater carbon was responsible for early decomposition of the block in less than 1 hour heating. This therefore, suggests that the block has low-bearing capacity which requires optimization for its sustainability being an affordable and economical material within the reach of low-income earners.

KEYWORDS: Breakthrough, Mechanism, Optimization, Stabilization, Time Dependency

1. INTRODUCTION

The search for alternative building materials either as a partial replacement or total replacement of Portland cement has become a subject of intense research in the recent times, which has led to the exploration of earth soil potentials. Earth soil over the years has been the major component used in the building and road construction, it is therefore desirous that everyone should own a befitting and comfortable shelter for successful running of day-to-day activities for a living. However; owning a house had been a major challenge to the low income earners especially in developing country such as Nigeria, due to high cost of building materials amidst the economic hardship occasion by divers' factors (rural-urban migration, security, social amenities, good roads, health care services e.t.c). These factors have caused a high demand for research into locally sourced, eco-friendly and affordable building materials. According to (Hamidu and Adamu, 2024), "the high cost of building materials is worrisome to low-income earners which are not only applicable to developing countries, but to also the developed countries".

Some research works carried out on the Nigerian Building and Road Research Institute compressed stabilized earth blocks (NBRRI-CSEBs) for the user comfort, includes thermal conductivity (Bakam et al, 2020a), fire resistance (Bakam et al, 2020b), and thermal decomposition as a function of temperature (Hamidu and Adamu, 2024), all reported that the blocks could not withstand temperature above 500 ^{o}C of firing, thus; making it a low-bearing material that requires surface coverage for longevity of the block. Similarly, studies on CSEBs by other authors on the acoustical properties which analyzed the effect of compaction pressure, water hyacinth ash and lime found out that the properties of CSEBs can be steered using binder concentration and compaction in a controlled manner (Ouma et al, 2023). Acoustics of compressed earth blocks bound using sugarcane bagasse ash and water hyacinth were also studied (Ongwen and Alruqi, 2023).

In related development, (Lyambo, 2012) study on the durability of CSEB reported that the durability is controlled by three factors: block strength, deterioration mechanism and the design of the building. These mechanisms were based on the assumption using masonry standards (Eurocode, BS, SANS, ASTM) which is ranging between 1.2 and 2.1 MPa (Delgado and Guerrero, 2007). The durability in terms of exposure to wetting-drying (WD) cycles and high temperature was carried out (Nshimiyimana et al, 2022), their finding revealed that the stabilization of CEBs with lime-rich binder is more resilient to the WD cycles than cement, which also shows that the cement stabilized CEBs would at least retain their strength after exposure to high temperature. (Nurul et al, 2020) did a study on the compressive strength of cement-stabilized CEBs; they reported that the CEBs gained the highest compressive strength of 3.4 N/mm^2 when stabilized with 10% of cement content over a curing period of 28 days. (Muhwezi and Achanit, 2019) conducted a study on the effect of sand on the properties of compressed soil-cement stabilized block reported the quality depends on the mechanical and physical properties of the soil test.

Building collapses has been a reoccurring phenomenon in Nigeria, which had led to loss of human lives, properties and materials. It is therefore imperative to determine critical mechanisms such as the rate of decomposition and kinetic of the ICSEBs produced by NBRRI to understand the durability of such vital building material from the abundant natural resources. (Fernandez-Caliani et al, 2004) reported on the use of TGA/DTA usage for determining the pattern of energy transition in clays and clay material properties. Decomposition rate and kinetic study which is seldom investigated is usually carried out using specialized process such as thermogravimetric analysis (TGA) or thermogravimetric (TG) and differential thermal analysis (DTA) (Al-Gawari, 2022). While phase formations of the matrices is analyzed via: scanning electron microscope (SEM) in conjunction with energy dispersive X-ray (EDX) for their morphological and elemental compositions (Hamidu et al, 2019). Furthermore; the vibrational frequencies of the active functional group for the elements responsible for the chemical and physical properties of material is best studied using Fourier transform infrared (FTIR) (Tinti et al, 2015). This was also reported by (Kamwa et al, 2023) on their study on “effect of Curing Temperature on Properties of Compressed Lateritic Earth Bricks Stabilized with Natural Pozzolan-Based Geopolymer Binders Synthesized in Acidic and Alkaline Media” using both the FTIR and SEM/EDX for the analysis where curing temperatures above 45 °C for the acidic-stabilized CEBs and 55 °C for the alkaline-stabilized CEBs, were studied. In this study, the use of thermogravimetric (TGA) and differential thermal analysis (DTA) instrumentation study to investigate the decomposition of NBRRI-ICSEB based on American Society for Testing and Materials (ASTM) standards as function of time and to further examine the morphological composition via scanning electron microscope (SEM-EDX) and Fourier transfer infrared (FTIR) spectroscope for proper understanding of the elemental composition is sort.

2. MATERIALS AND METHODS

2.1 Materials

The material used in this work, was the already made interlocking compressed stabilized earth blocks (ICSEBs) by the Nigerian Building and Road Research Institute (NBRRI), which had composition of not less than 5% cement to not more than 95% laterite stabilization compacted at 3 KN/mm^2 (Bakam et al, 2020a). The ICSEB characterization was performed using combination of several methods which includes; thermogravimetric and differential thermal analyses, scanning electron microscope/energy dispersive X-ray spectroscope (SEM/EDX) and Fourier transform infrared spectroscope (FTIR).

2.2 Methods

The ICSEB sample was crushed and ground using laboratory mortar and pestle to smaller sizes of 1 mm (Hamidu and Adamu, 2024). This was followed by the TGA-DTA experimentation, where 50 mg of the sample was weighed and put into the sample holder, and placed in the analysis chamber, covered on the cheallar and allowed to cool to 15 ºC. It was further connected to the desk top computer to obtain all information about the sample degas in a static Nitrogen atmosphere with a purge rate of 20 mL/minute using TGA 4000 PerkinElmer model. The heating began at 0 minute to 90 minutes for each sample at 10 ºC/min, 15 ºC/min and 20 ºC/min heating rates until stabilization. At the end of each run, data with TGA and combine TGA-DTA curves were generated.

The SEM study was carried out in conjunction with energy dispersive X-ray spectroscopy (EDX) on the NBRRI-ICSEB for morphological and elemental compositions, using SEM machine model sputter coater Q150T by Quorum UK. The sample was first placed on a stub; thereafter it was placed in a sputter coater followed by deposition into the SEM machine column for analysis at different magnifications, 30, 80, 100 µm and full count detection at 537 µm. while the quantitative elemental compositions were obtained by linking the EDX analyzer coupled to the microscope for the periodic table. Fourier transform infrared model FT-IR-8400S, Schmazdu and Sputter Coater Q150T UK model was used for the FTIR spectroscopic was recorded in the range of 400 – 4000 cm^{-1} wavelength using the pressed pellets containing 5 mg of ICSEB and 95 mg Kbr (Hamidu et al, 2019).

3. RESULTS AND DISCUSSION

3.1 Thermogravimetric Analysis of NBRRI-ICSEB

The results from the TGA heating at different heating rates of 10, 15 and 20 ºC/min is presented on Figure 1, with time (min) on the abscissa (X-axis) and weight percentage (%) on the ordinate (Y-axis).

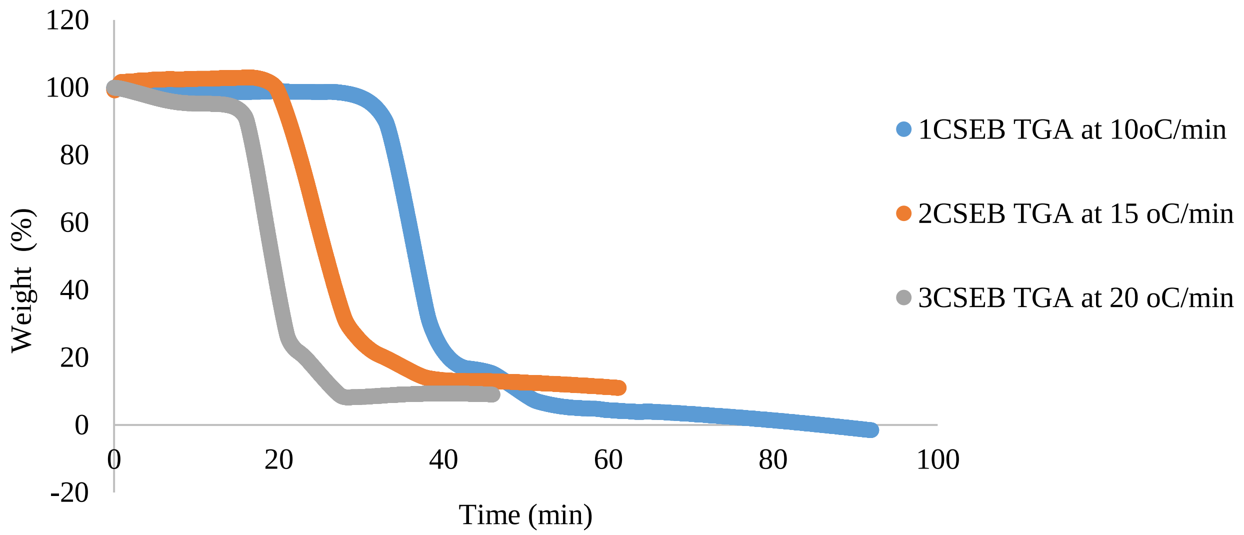

Figure 1: NBRRI Interlocking Compressed Stabilized Earth Block Thermogravimetric Curves

From Figure 1, the blue line (1CSEB TGA at 10 ºC/min) shows the time dependency at 10 ºC/minute decomposition process. The horizontal region became stable showing no change in mass of the ICSEB between 0.54 min and 31.15 min with a weight loss of 4.504 %. This implies the ICSEB thermal stability and removal of water of hydroxylation. At the second phase, the mass loss begins from (31.15 min) 95.458 weight (%) and stabilized in between (41.35 min) 18.464 weight (%) implying the removal of carbonic compound and organic matters as the main stage taking about 81.53% of the total initial weight, while the residual 18.47% being the pure component of the materials in the ICSEB. According to Dilnesa (2020), the removal of water of hydroxylation using TGA occurs in the range of 400 to 500 ºC, while above 600 ºC is the decarbonization of the material. This short time loss in the properties of the compressed stabilized earth block produced by NBRRI at 10 ºC/min heating rate supports the findings of (Hamidu and Adamu, 2024) and (Bakam et al, 2020a) that the NBRRI-ICSEB cannot withstand heating at temperature above 500ºC (41.35 minutes). The second stage investigation of the heating rate at 15 ºC/min is indicated by the brown line (2CSEB TGA at 15 ºC/min), the heating began at (0.8 min) until the process became stable at 18.83 min. The second phase of the decomposition of organic material and other volatile components began at 18.83 min progressively to 34.48 min taking 82.28% of the material ICSEB, being the volatile carbon and organic matters present in the ICSEB, while the residual being the pure component material used in the production of ICSEB is 17.72%. And the third phase of the TGA was the investigation of the heating rate at 20 ºC/min as indicated by the gray line (3CSEB TGA at 20 ºC/min). The removal of moisture began 0.6 min until stabilization stage on the horizontal region of the curve at 15.08 min. While complete decomposition reached steady state at 29.92

min taking 91.70% weight of the material being carbon content and other volatile organic matters, while the 8.30% residual being the pure component that makes up the ICSEB. The thermal process of the phase transition is described by the combined thermograph curves for both TGA-DTA which outline the process of energy pattern by the NBRRI-ICSEB and its stability.

3.2 Combined Thermogravimetric and Differential Thermal Analysis of NBRRI-ICSEB

The combined TGA/DTA decomposition process shown in Figure 2 illustrates the thermal decomposition of a typical material.

Figures (2a, b and c) presents and describe the complete process of time dependency of the ICSEB's removal of water of hydroxylation and transition (phase change) to final decarbonization of the material resulting to the residual as the pure material component that is present the matrices.

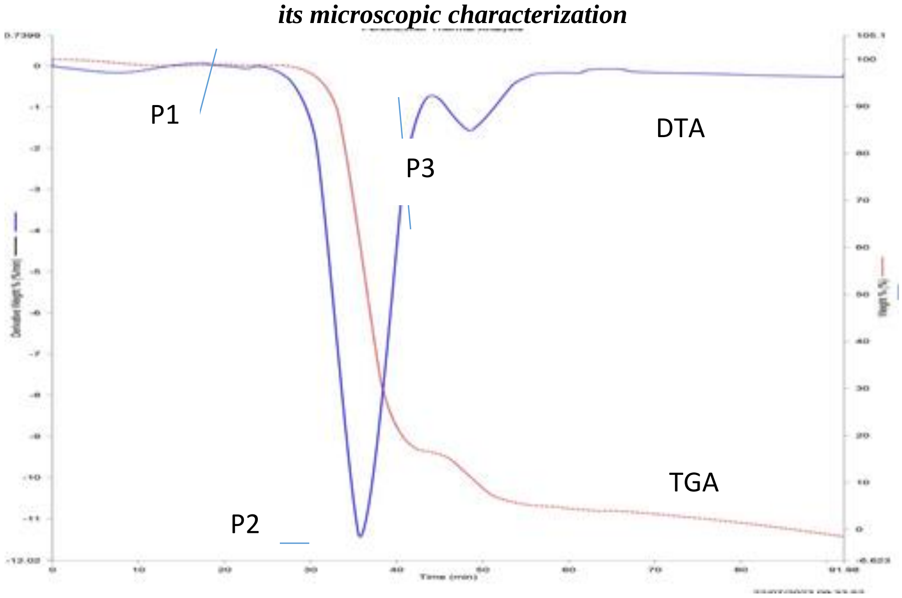

Figure 2a: NBRRI-ICSEB TGA-DTA at 10 ºC/min

Figure 2a, describe the transition stages (P1, P2 and P3) for NBRRI-ICSEB curve at 10 ºC/min. P1 is attributed to removal of water of hydroxylation adsorbed in the ICSEB, between P1 and P2, endothermic process was observed in 35 minutes, depicting the oxidization and removal of volatile compound containing in the ICSEB. As the heating process continued from P2 to P3, all carbonaceous materials were removed and complete decomposition attained in 54 minutes with endothermic process taking place, as the ICSEB absorbed more heat. This process could be related to the similar work by Fernandez-Caliani et al. (2004) reported in the journal of clays and clay materials.

Figure 2b; present the combined TGA-DTA of NBRRI-ICSEB decomposition at 15 ºC/min heating rate.

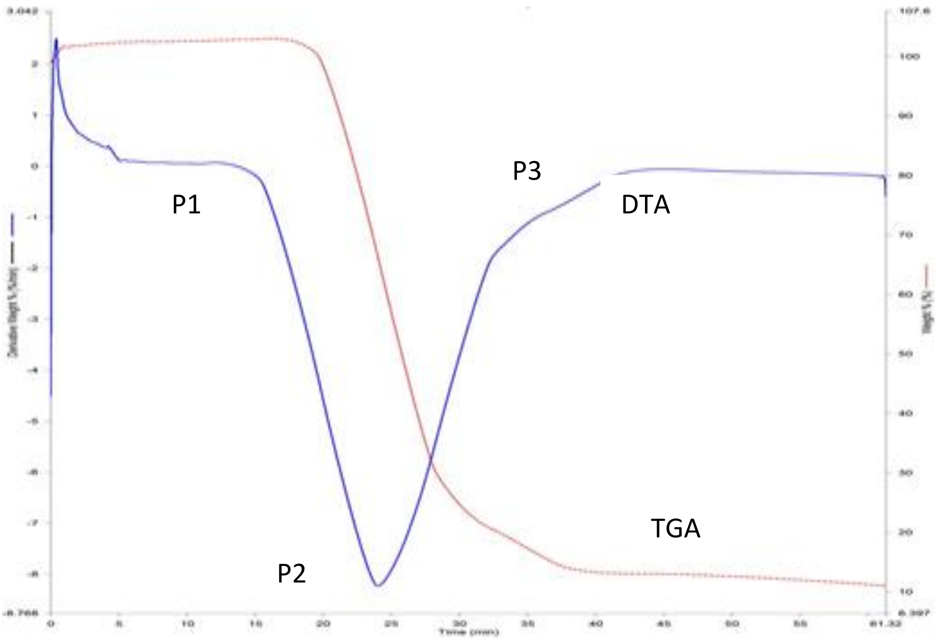

Figure 2b: NBRRI-ICSEB Combine TGA-DTA at 15ºC/min

This Figure shows the thermal decomposition of the ICSEB at heating rate of 15 ºC/min. Point P1 is attributed to the removal of adsorbed water in ICSEB known as the water of hydroxylation within 16 minutes of heating time (Hamidu and Adamu, 2024). Between P1 and P2 as heating progressed, endothermic process was observed with heat being absorbed by the ICSEB (25 minute), thereafter, from P2 to P3 is the complete decomposition process removing all carbonaceous material with the residual as pure components that sustained the ICSEB in 43 minutes. This process is similar to the work reported by (Hamidu and Adamu, 2024) and (Fernandez-Caliani et al, 2004) on TGA-DTA processes in material characterization.

Figure 2c; present the combined TGA-DTA of NBRRI-ICSEB thermal decomposition at 20 ºC/min heating rate.

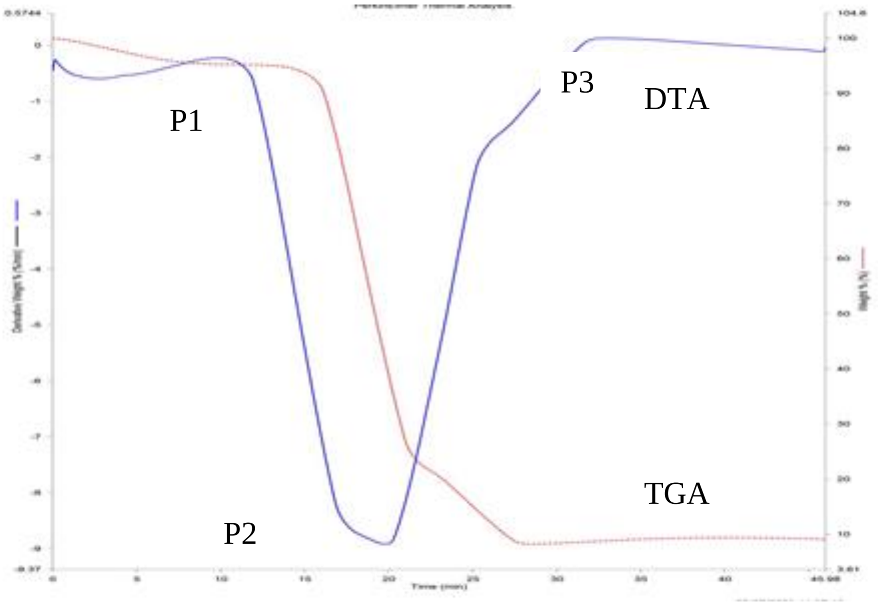

Figure 2c: NBRRI-ICSEB Combine TGA-DTA at 20 ºC/min

Point P1 in Figure 2c is attributed to the removal of adsorbed water in the ICSEB known as dihydroxylation process which is removed within 12 minutes of heating, as heating progressed, between P1 and P2, an endothermic process of decomposition where observed with heat being absorbed by the NBRRI-ICSEB and complete removal and oxidization of volatile compounds in 20 minutes of heating. Between P2 and P3 is the removal of the carbonaceous material in the NBRRI-ICSEB within 32 minutes of heating time and the residual is pure material sustaining the ICSEB as also reported in temperature dependency thermogravimetric and differential thermal analysis of NBRRI-ICSEB (Hamidu and Adamu, 2024).

The entire decomposition processes from the TGA results in Figure 1 and combined TGA-DTA in Figure 2 results, revealed that the NBRRI-ICSEB made from laterite of not more than 95% and not less than 5% cement stabilization has shown it absorb more heat which is referred to as endothermic process of decomposition. Based on results from this finding, it implies that the NBRRI-ICSEB is a low bearing material and has short duration of heating time of less than 1 hour. This fast decomposition of the NBRRI-ICSEB could be due to the high content of the combustible materials in the earth soil that forms major component in making the interlocking compressed stabilized earth block.

3.3 Scanning Electron Microscope and Energy Dispersive Spectroscope of NBRRI-ICSEB

The scanning electron microscope (SEM) and energy dispersive x-ray (EDX) spectroscope shows the

morphological and elemental compositions of NBRRI-ICSEB. The results for SEM-EDX is shown on Figure 3 (a to d), and Table 1 for the elemental compositions. The SEM images were taken at 30, 80 and 100 µm to study the visibilities of the phase's formation, while complete image detection alongside with the EDX was obtained at 537 µm running at 15 keV.

Figure 3a show the SEM image at 30 µm, with visible cracks within the matrix. In this image, silver like shining silicates and alumina are heterogeneously appearing with pore at the center implies non-homogeneity in the matrix.

Figure 3a: NBRRI-ICSEB Scanning Electron Microscopy Image at 30µm

Figure 3b is the SEM image at 80 µm detection.

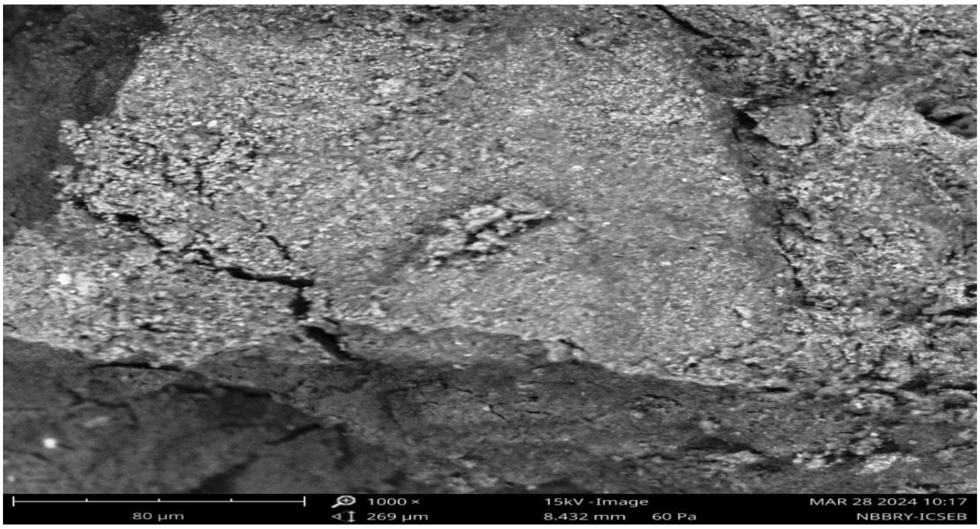

Figure 3b: NBRRI-ICSEB Scanning Electron Microscopy Image at 80 µm

At 80 µm, all the phases are separated by visible cracks and pore formations with colour black implying the presence of carbon as dominant elements, with embedded silicates and aluminum materials. This separation is an indication of the quality of materials used in NBRRI-ICSEB production. These cracks could create permeability for water penetration into the block leading to its deterioration. The carbon dominance could be the reason for early decomposition as reported (Hamidu and Adamu, 2024), (Bakam et al, 2020a) on the thermal stability of NBRRI-ICSEB.

Figure 3c is the NBRRI-ICSEB SEM image at 100 µm detection.

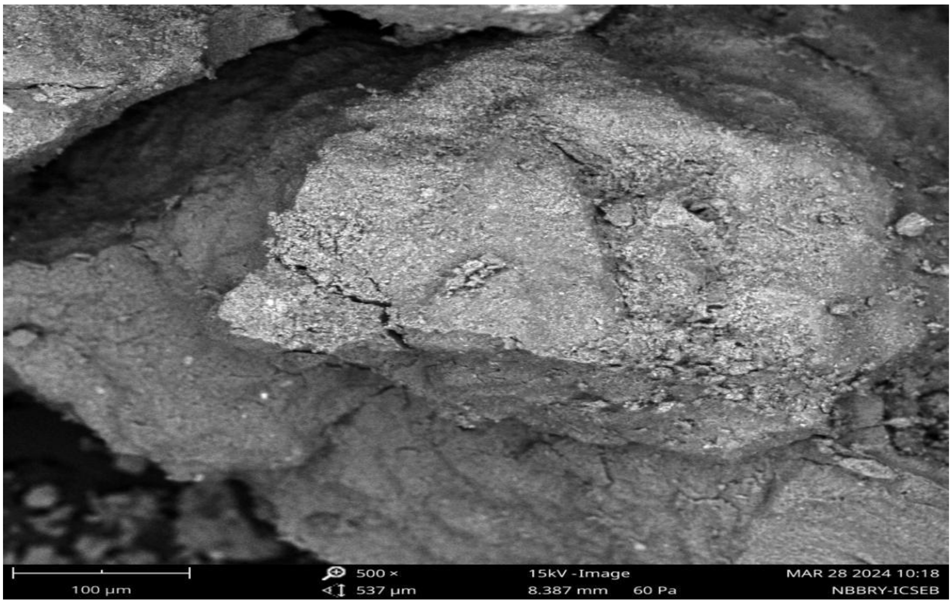

Figure 3c: NBRRI-ICSEB Scanning Electron Microscopy Image at 100 µm

At 100 µm, the Carbon, Silicon, Aluminium and Silver phases are clearly visible with cracks within the regions. The result also indicates that, the matrix is not homogenous and thus creating visible phase's separations. At full image detection shown on Figure 3d all elements were detected.

Figure 3d: NBRRI-ICSEB Full Detection Scanning Electron Microscopy Image at 537 µm

The EDX analysis of NBRRI-ICSEB (Table 1) revealed the elemental compositions present in the block, the presence of Si, Al, Fe, Ca, and Na above 1% with others in traces of less than 1% confirms the formation alumino-silicon-carbonates binder in the matrix.

Table 1: EDX Analysis of NBRRI-ICSEB Compositions

Element Number	Element Symbol	Element Name	Atomic Conc.
14	Si	Silicon	24.06
13	Al	Aluminium	21.35
6	C	Carbon	41.37
26	Fe	Iron	5.14
20	Ca	Calcium	3.04
47	Ag	Silver	0.40
30	Zn	Zinc	0.55
11	Na	Sodium	1.06
12	Mg	Magnesium	0.85
15	P	Phosphorus	0.58
17	Cl	Chlorine	0.46
22	Ti	Titanium	0.34
16	S	Sulfur	0.47
19	K	Potassium	0.35
23	V	Vanadium	0.00
			100.00

From the EDX (Table 1), the weight percentage shows that carbonaceous material is predominant followed by silicone and Aluminium. The carbon dominance in the EDX was responsible to the rapid decomposition of the NBRRI-ICSEB as shown in Figure 1 and continued reaction (combustion) leads to more energy is absorbed (endothermic process) and weakening the properties of the materials sustaining the block.

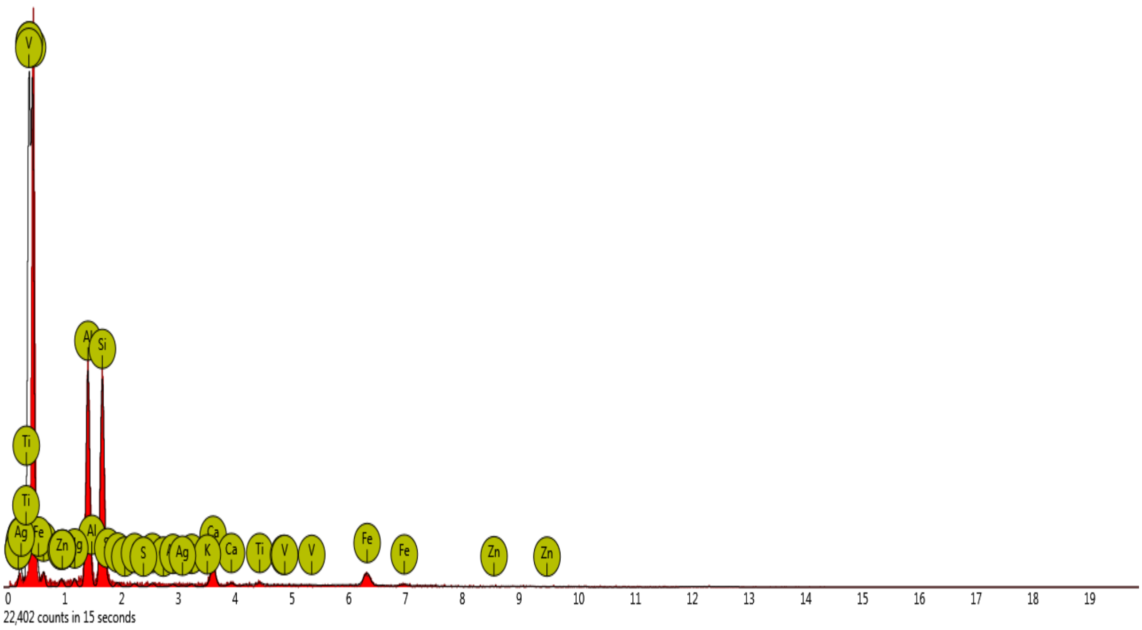

Figure 4: NBRRI-ICSEB Energy Dispersive X-ray Spectra

3.4 Vibrational Frequencies of NBRRI-ICSEB

The vibrational frequencies of the Nigerian Building and Road Research Institute-Interlocking Compressed Stabilized Earth Block (NBRRI-ICSEB) showing the key components influencing the chemical and physical properties of the material is presented in Figure 5.

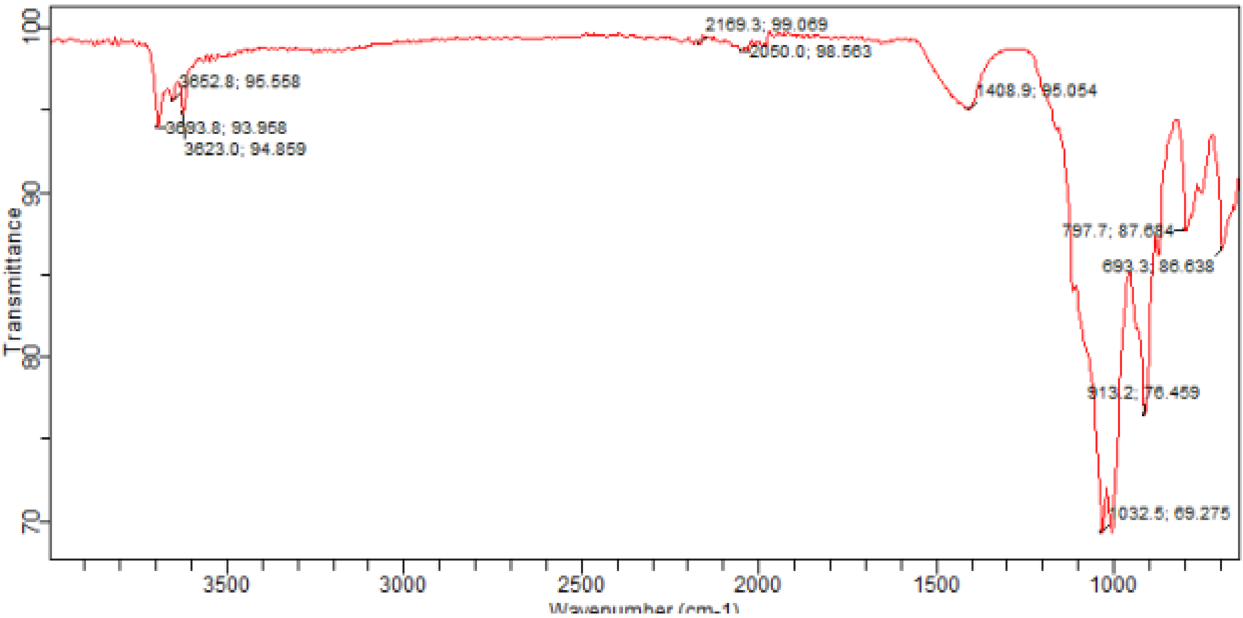

Figure 5: NBRRI-ICSEB Fourier Transform Infrared Spectrum

The structure is characterized by broad spectra and overlapping bands of Si-O-H vibration assigned to clays, kaolinite and iron oxides at 3693.8 cm^{-1} are attributed to minerals presence for the NBRRI-ICSEB (Kaze et al, 2018). Accordingly, the hydroxyl (O-H) stretching which occurs between 3719 – 3685 cm^{-1} and Si-O bending at 1032.5 cm^{-1} stretching between 1136 and 1070 cm^{-1} show silicates containing double layers of $O - Si - O$, implies that the material is a composition of hydroxyl silicate principally identified as phyllosilicate (Encyclopedia, 2017). Furthermore; the silicate layer vibrational frequency occurring in the absorbance range 3682 cm^{-1} – 3577 cm^{-1} reflects the internal OH groups between the tetrahedral and octahedral sheets at 3652.8 cm^{-1} depicting internal H-bonding between the octahedral surface. In the ranges of 1478 – 1408 cm^{-1} vibrational frequency is assigned to carbonates, in this range, the absorbance peak occurred at 1408.9 cm^{-1} could be due to the component of cements material used in the stabilization of the NBRRI-ICSEB. The FTIR results revealed that most component of ICSEB produced had silicates and carbonates as dominating the functional group which also supported the EDX elemental composition in Table 1 where carbon had the highest percentage followed by silicon and Aluminium. According to (Bruckman and Wriessnig, 2013) and (Grinard et al, 2012), carbonate is a key component influencing both chemical and physical soil properties as accurately established using FTIR. The carbonaceous material is a material that support combustion when burning, thus results into more heat accumulation (absorb in by the block) thereby weakening the residual properties of the components that made up the NBRRI-ICSEB. This also supports the combined TGA/DTA results in 3.2 as endothermic process of decomposition.

4.0 CONCLUSION

Time dependency thermogravimetric and differential thermal analysis, scanning electron microscope and Fourier transform infrared studies of the Nigerian Building and Road Research Institute Interlocking Compressed Stabilized Earth Block (NBRRI-ICSEB) was examined. The following conclusions are drawn; that the NBRRI-ICSEB has low bearing capacity to withstand heating more than 1 hour at 10 ^oC/min, 15 ^oC/min and 20 ^oC/min heating rates to decompose. The combined TGA/DTA results shows the NBRRI-ICSEB absorbs in heat energy which could be described as endothermic process of decomposition as carbonaceous materials continue to combust. The SEM and EDX micrograph, revealed the inhomogeneity formation (heterogeneous formation) with visible cracks in the regions of 30 µm, 80 µm and 100 µm. The EDX revealed 15 elements existing in the formulated NBRRI-ICSEB with carbon dominance, which is responsible to influencing the chemical and physical properties of the produced block. The FTIR spectrum revealed the presence of carbonaceous material as the major functional group, which could also aid combustibility of the ICSEB. We therefore recommend NBRRI-ICSEB for optimization to meet up with the demand for usage as environmentally friendly material for user comfort in building.

Declaration of competing interest

The authors declare that there is no competing financial interest or personal relationships that could have influenced the work reported in this paper.

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Data Availability

All data generated or analyzed during this study are included in this article for the SEM-EDX, and while TGA-DTA can be made available on request from the corresponding author.

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