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Thermogravimetric and Thermal Differential Analysis Instrumental Study of Nigerian Building and Road Research Institute Compressed Stabilized Earth Block Decomposition on Temperature Basis

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ABSTRACT

The Nigeria Building and Road Research Institute (NBRRI) had over the years keyed into the production of compressed stabilized earth Blocks using not more than 95% laterite and not less than 5% cement stabilization as binder compacted with 3KN compaction pressure. The interlocking compressed stabilized earth blocks (ICSEBs) had been characterized for compressive test, fire resistance, and thermal conductivity among others. Nevertheless, these blocks required further test such as thermal decomposition and it's kinetic to ascertain the extent to which these blocks could be used. In this study, thermogravimetric and differential thermal analysis instrumental tests were carried out on the ICSEB via TGA 4000 PerkinElmer model and degas in Nitrogen atmosphere with a purge rate of 20 mL/minute. The heating rate was at 10 ^{o}C/min, 15 ^{o}C/min and 20 ^{o}C/min in the temperature range of 30 ^{o}C – 950 ^{o}C based on the ASTM D standard. The results revealed that devolatilization and oxidation stability of the ICSEB occurred in the temperature range of 246.03℃ - 553.34℃ with greater mass loss of 93.75% at 10℃/min heating rate, 85.20% at 15 ^{o}C/min heating rate and 86.69% at 20 ^{o}C/min heating rate respectively devoid of water of hydroxylation at the first phase of the decomposition. The differential thermal analysis revealed that the ICSEB as depicted on the curves decomposed at the peak of 351 ^{o}C for 10 ^{o}C/min heating rate, 380 ^{o}C for 15 ^{o}C/min heating rate and 400 ^{o}C for 20 ^{o}C/min heating rate respectively. Thus; the determined thermal stability of NBRRI-ICSEB is considered low heat bearing material that could not withstand heating up to 600℃.

Keywords: Interlocking, Devolatilization, Degas in Nitrogen, Heating rate, Low bearing Oxidation stability, Water of hydroxylation

1. INTRODUCTION

The desire of everyone to own a befitting and comfortable shelter is sacrosanct in national development. Housing is vital for the survival and day-to-day socioeconomic activities, without which life becomes miserable to humanity. According to [1], "Nigeria's housing deficit was estimated at 7 million in 1991, it rose to 12 million in 2007; 14 million in 2010; 20 million in 2018, and 28 million in 2023". However; to own a house for low-income earners has been the major constrained especially in developing countries such as Nigeria, due to high cost of building materials amidst the economic hardship. Thus; the search for alternative building materials either as a partial replacement or total replacement of cement has become a subject of intense research in the recent using earth soil.

A compressed stabilized block produced from earth soil has been in use for century known as compressed stabilized earth block (CSEB). Several studies were carried out on the CSEB to determine its suitability as an alternative to conventional building material, which includes those of authors [2; 3] on acoustical properties, authors [4; 5] studies on the durability of CSEB on the assumption of using masonry standards (Eurocode, BS, SANS, ASTM) ranging between 1.2 and 2.1 MPa)). Authors [6] studied the durability in terms of exposure to wetting-drying (WD) cycles and high temperature and, [7] on the effect of sand on the properties of compressed soil-cement stabilized block. All studies were to establish the economic importance and efficiency of the use of CSEB as reported that the quality depends on the mechanical and physical properties of the soil test.

Compressed Stabilized Earth Block is primarily made up of laterite soil, cement and water. Laterite soil is formed as a result of leaching, leaving behind a residue resulting from natural process of laterization in tropical and sub-tropical regions [8]. Laterite soil can be cohesive (silt and clay particle size) in its behavior and it can be non-cohesive (sand particle size) in its behavior [9]. Cement binder is used as a stabilizer for unfired laterite bricks. According to [10], the performance of unfired bricks improves with increase in the binder incorporation.

Building collapses has been a reoccurring phenomenon in Nigeria in the recent which had led to losses of human lives and materials. The Nigerian Building and Road Research Institute

(NBRRI) had keyed into the production of compressed stabilized earth block (CSEB) for use in building, where thermal conductivity [11], fire resistance [12] and effect of firing at high temperature were carried out and recommended that the produced ICSEB had low bearing fire resistance.

However, thermal decomposition of these CSEB has not been ascertained to understand the durability of the in-use life of the blocks. It is therefore imperative to determine critical mechanisms such as the rate of decomposition and kinetic of the ICSEB produced by NBRRI to understand the durability of such vital material used for building. Decomposition rate and kinetic study which is seldom investigated are usually carried out using specialized processes such as thermogravimetric analysis (TGA) or thermogravimetric (TG), differential thermal analysis (DTA) and differential scanning calorimeter (DSC).

The TGA is a powerful technique that measures mass changes of materials with temperature or time usually carried out at different heating rates which involves the information about thermal stability, reactivity, reaction mechanism, decomposition kinetics among others [13]. “TGA measurements is primarily used to determine the composition of materials and to predict their thermal stability up to elevated temperatures” [14]. Other works includes [15] on thermal analysis of calcium oxalate monohydrate. Authors [15; 16] described the pattern of the TGA curves given to specific materials and chemical compounds as reaction occur over specific temperature ranges and heating rates such as removal of water of hydroxylation within the ranges of ambient temperature and 105 ^oC, while from 200 ^oC to 450 ^oC could be removal of carbonic materials (CO and CO_2) and above 600 ^oC is the pure material composition. TGA has been extensively used to study material characterization to identify the key component existing [17]. This study therefore is focusing on the use of Thermogravimetric (TGA) and differential thermal analysis (DTA) instrumentation to investigate the decomposition rate mechanism on NBRRI ICSEBs based on ASTM standards.

2. MATERIALS AND METHODS

2.1 Materials

The material used in this work, is the already produced 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.

2.2 Methods

The ICSEB sample was crushed and ground using laboratory mortar and pestle to smaller sizes of 1 mm. this was followed by the TGA-DTA experimentation. The quantity of 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 ^{o}C. It was further connected to the desk top computer to obtain all information about the sample and degas in Nitrogen atmosphere with a purge rate of 20 mL/minute using TGA 4000 PerkinElmer model. The heating began at 30 ^{o}C – 950 ^{o}C for each sample at 10 ^{o}C/min, 15 ^{o}C/min and 20 ^{o}C/min respectively. At the end of each run, data were generated.

2.3 Parametric Determination of the NBRRI-ICSEB

The thermal decomposition and kinetics of the NBRRI-ICSEB were determined using the established standard procedure adopted from authors [18; 19].

$$\mathrm{A\ (solid\ block)} \rightarrow \mathrm{B\ (solid)} + \mathrm{C\ (gaseous)} \tag{1}$$

Where; A is the solid block of ICSEB, B is the solid phase of thermal decomposition and C is the combustible gaseous substances during heating. This is physical properties that change in thermogravimetric studies, as well as the change in mass relation to the extent of conversion (a) while "a" using Equation 2 [19; 20].

$$a = \frac{W_0 - W}{W_0 - W_\infty} \tag{2}$$

Where; W is the weight of ICSEB in mg; W_0 is the weight of ICSEB at initial stage in mg and W_∞ is the weight of ICSEB at the final decomposition stage in mg.

3. RESULTS AND DISCUSSION

The results from the TGA and DTA heating at different flowrate of 10, 15 and 20 ^{o}C/min are displayed on the abscissa (X-axis) as temperature and on the ordinate (Y-axis) are the weight percentage (%). Figure 1, presents the TGA curves for the heating rates at 10, 15 and 20 ^{o}C/min.

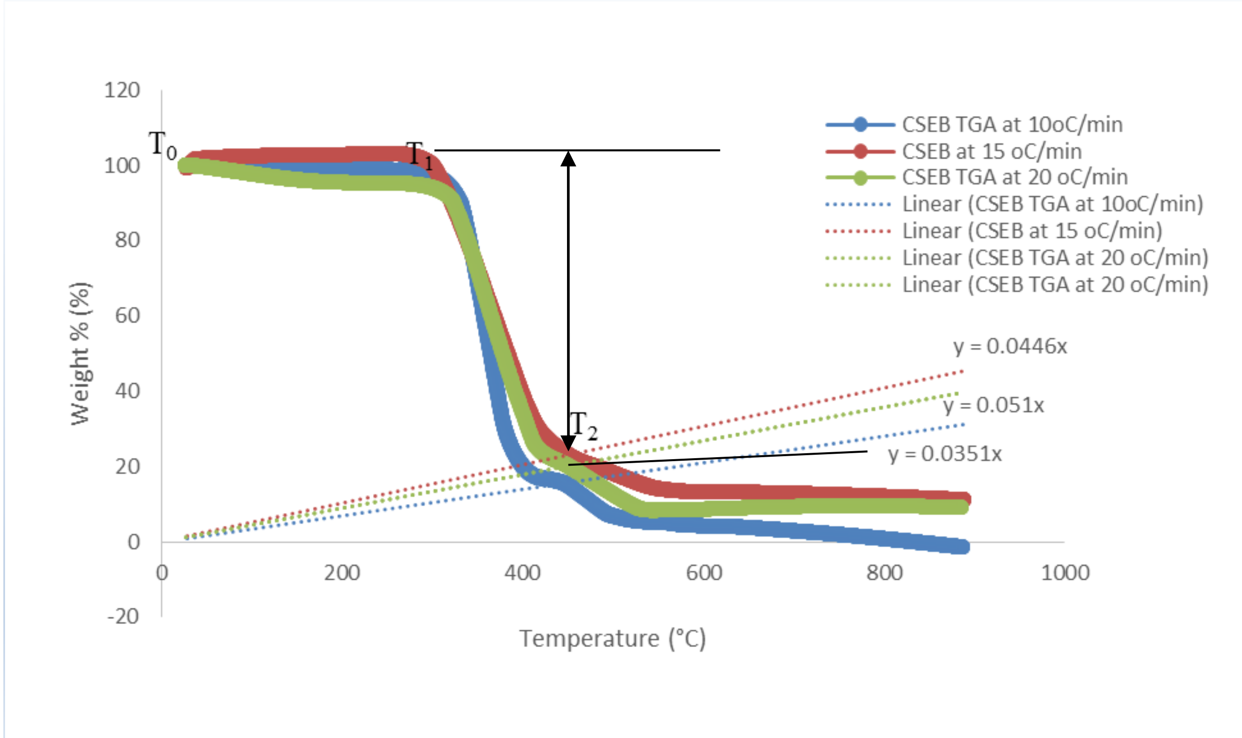

Figure 1: NBRRI Interlocking Compressed Stabilized Earth Block Thermogravimetric Curves

The preceding Figures 2, 3 and 4 revealed the actual TGA and DTA curves from the thermogravimetric instrument for the NBRRI-ICSEB decompositions at the rates under study. The individual single stage curves were based on the temperature and weight percentage mass loss at which the removal of water of hydroxylation and pyrolysis of the ICSEB occur according to [21] using dynamic thermogravimetric as it is linearly heated.

Figure 2 (a and b) is the decomposition of ICSEB at 10 °C/min heating rate TGA and Combined TGA/DTA curves.

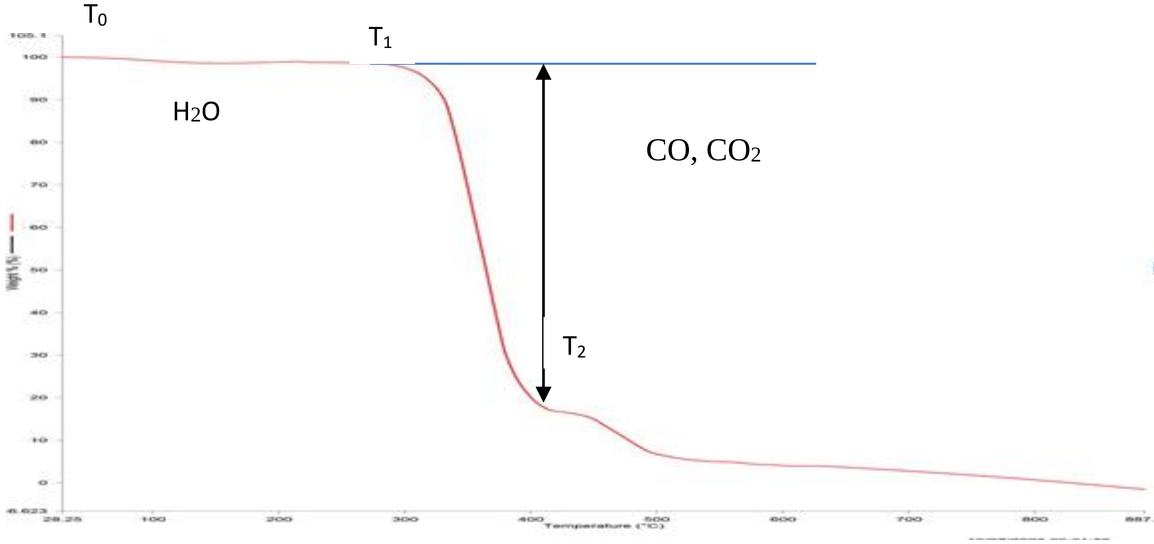

Figure 2a: Thermogravimetric Analysis Curve of Interlocking Compressed Stabilized Earth Blocks at 10°C/min

From Figure 2a, at the heating rate of 10 °C/min, the first phase change which occurred between the ambient temperatures (T_0) (30.01 °C)) and (T_1) (246.03 °C)), for the ICSEB is the loss of moisture and the weight loss at this initial phase was 1.26%, within this range the mass loss in horizontal region was low and became stable showing no change in mass of the ICSEB implying it is thermally stable [21]. The second phase of the decomposition began the temperature (T_1) (246.03 °C)) progressively and became stabilized at the temperature (T_2) (548.26 °C)). In this region, bulk of the NBRRI-ICSEB mass was lost, which is attributed to removal of carbonic compound and organic matters taking about 93.75% of the total initial weight, as visibly seen, while the residual which is 4.99% of the material is the weight of the pure component used in the production of ICSEB which is supported by [16] report. The entire process of the decomposition of the ICSEB was taken from the contribution of laterite and the stabilizer cement ended at less than 600 °C.

In Figure 2b, the combined TGA and DTA curve had shown the starting point of heating to the peak of decomposition, when the heat absorption occurred.

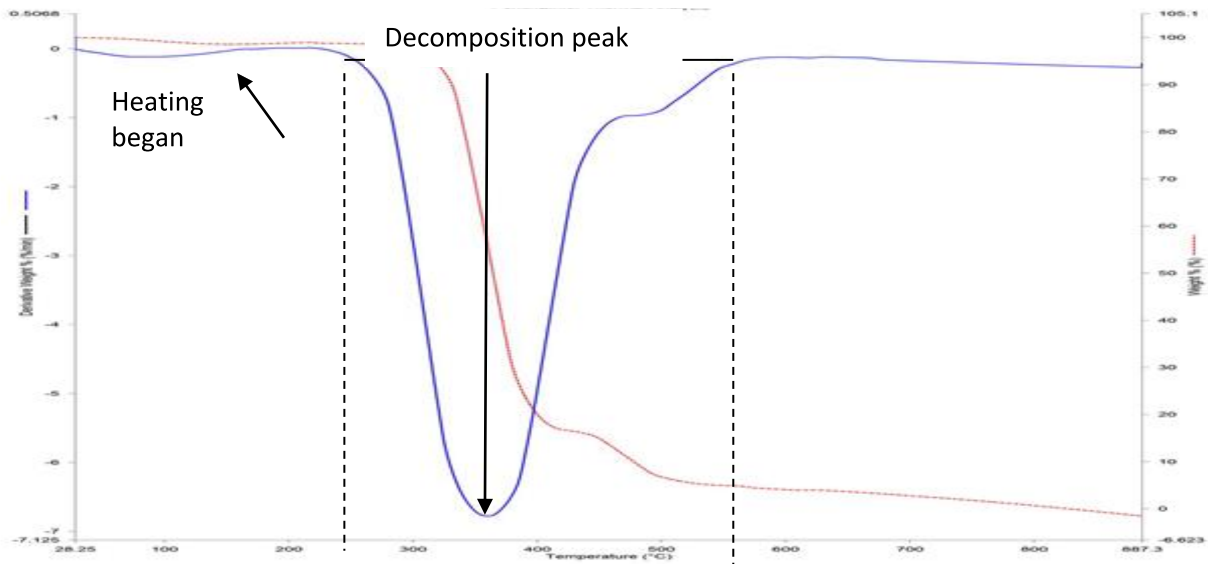

Figure 2b: Thermogravimetric and Differential Thermal Analysis Curve of Interlocking Compressed Stabilized Earth Blocks at 10 ^{o}C/min

In Figure 2b, the DTA curve associated with mass loss was peak at 352 ^{o}C for 10 ℃/min heating rate which may be attributed to predominant effect of removal of organic and volatile matters in the ICSEB produced blocks. The phase change was due to the decomposition of carbonates and release of CO_2 with more heat being absorbed in the region as supported by [22; 23].

Figure 3 presents the NBRRI-ICSEB decomposition at 15 ^{o}C/min heating rate TGA and combine TGA/DTA curves.

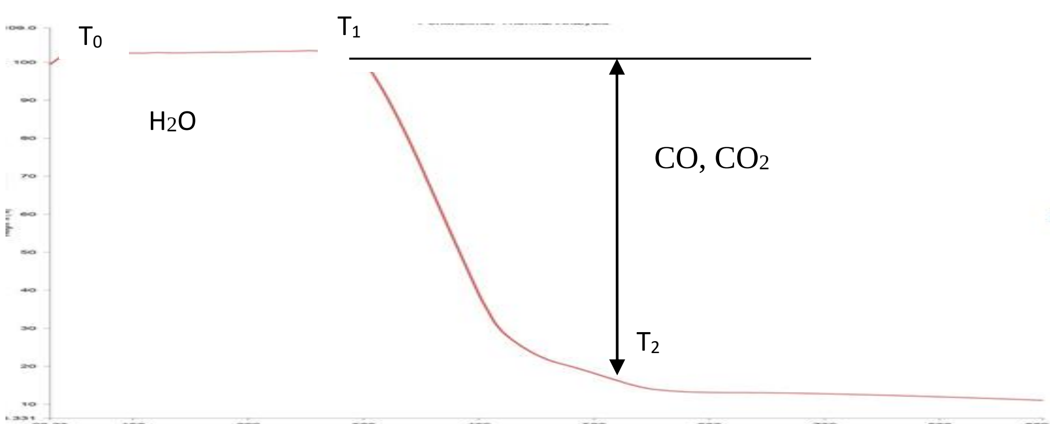

Figure 3a: Thermogravimetric Analysis Curve of Interlocking Compressed Stabilized Earth Blocks at 15 ^{o}C/min

At the heating rate of 15 ^{o}C/min, the heating began at ambient temperature (T_0) (30.15 ^{o}C)) until the process became stable at the temperature (T_1) (303.26 ^{o}C)). In this transition phase, the NBRRI-ICSEB mass loss was 0.77%, this negligible mass loss which was insignificant hence the dormant change. As the temperature progress to high temperature region from (T_1) (303.26 ^oC)) to (T_2) (553.34 ^oC)) progressively in the second phase, bulk of the ICSEB mass was lost as it stabilizes with mass loss of 85.20% being volatile carbon and organic matters in the ICSEB devoid of moisture known as water of hydroxylation removed in the first phase [21; 22] and was visibly seen, while the remaining residual of 14.03% being the pure component material used in the production of ICSEB.

Figure 3b is the combine TGA/DTA thermogravimetric curve for ICSEB at a heating rate of 15 ^oC/min.

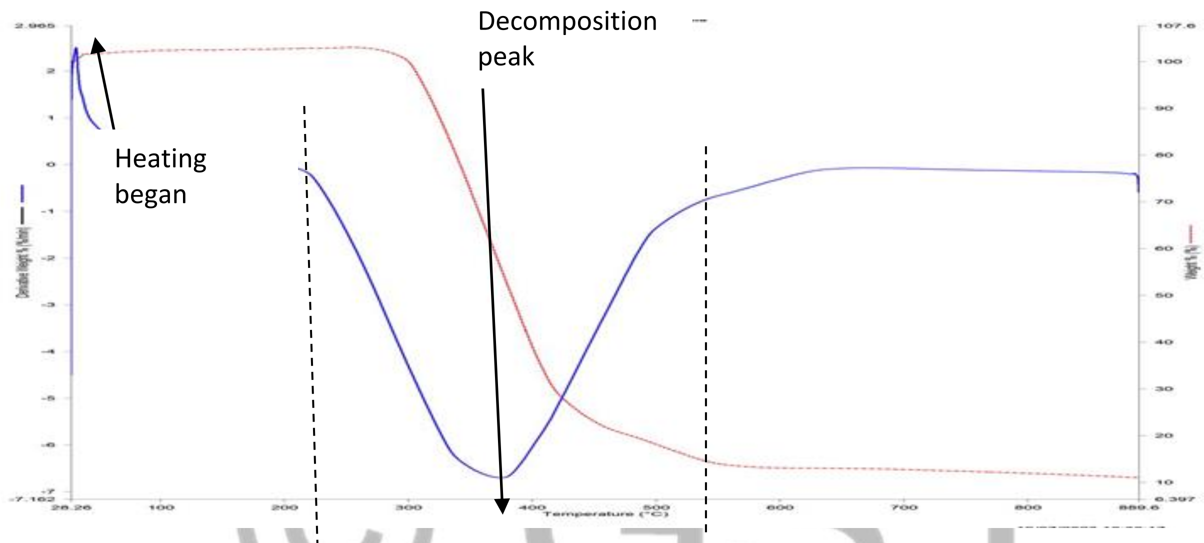

Figure 3b: Thermogravimetric and Differential Thermal Analysis Curve of Interlocking Compressed Stabilized Earth Blocks at 15 ^oC/min

In Figure 3b, the mass loss associated with a decomposition peak on DTA curve was at 380 ^oC for 15 ℃/min heating rate which is the removal of organic and devolatilization matters in the ICSEB produced blocks. The phase change was due to the decomposition of carbonates, release of CO_2 and other volatile matters in the ICSEB [23] with more heat being absorbed in the region.

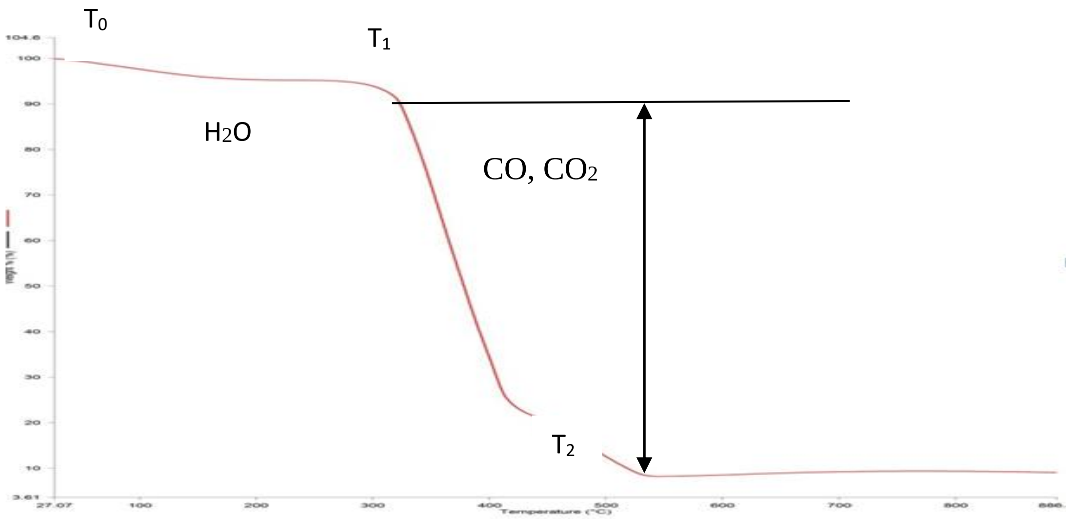

Figure 4a: Thermogravimetric Analysis Curve of Interlocking Compressed Stabilized Earth Blocks at 20 ^oC/min

The third phase of the TGA test was the investigation at the heating rate of 20 ^oC/min. The mass loss within the range of ambient temperature (T_0) (30.03 ^oC)) and (T_1) (273.06 ^oC)) was visible but with low significance as the mass loss of 4.91% was recorded. As the temperature progress from (T_1) (273.06 ^oC)) being the stabilization stage on the horizontal region of the curve, the decomposition began until it reached steady state at (T_2) 538.09 ^oC when the mass loss was visibly seen due to removal of moisture content (water of hydroxylation) was with mass loss of 86.69% weight of the material being carbon content and other volatile organic matters [16; 21], while the residual of 8.40% is the pure component that makes up the ICSEB.

Figure 4b is the combine TGA/DTA curve for ICSEB decomposition at a heating rate of 20 ^oC/min.

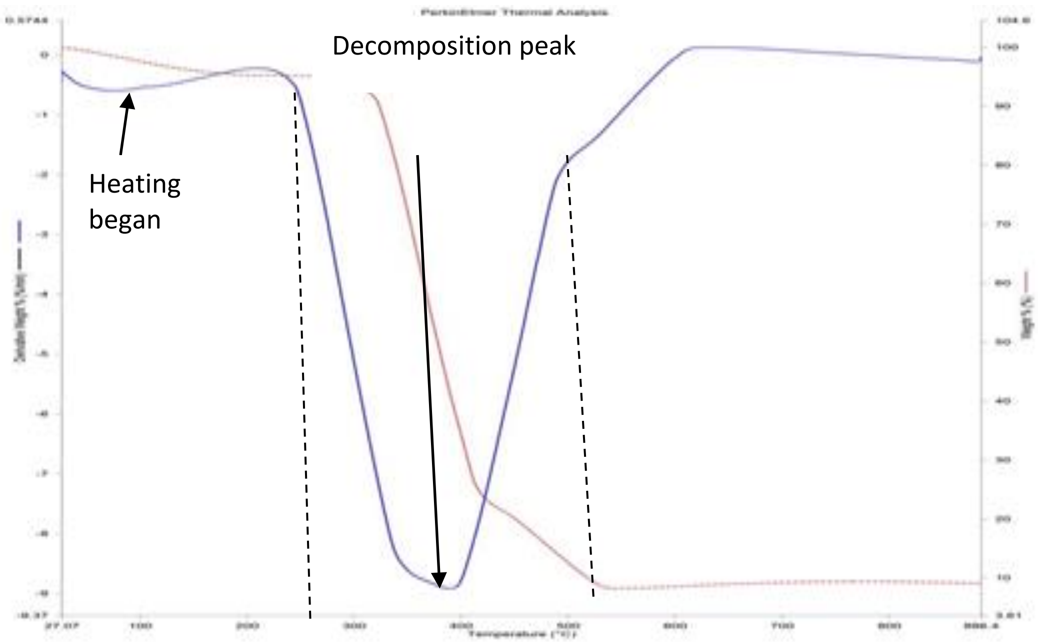

Figure 4b: Thermogravimetric and Differential Thermal Analysis Curve of Interlocking Compressed Stabilized Earth Blocks at 20 ^{o}C/min

In Figure 4b, the mass loss associated with a decomposition peak on DTA curve was at 400 ^{o}C for 20 °C/min heating rate which is the removal of organic and volatile matters in the ICSEB produced blocks. The phase change was due to the decomposition of carbonates, release of CO_2 and other volatile matters in the ICSEB [23] with more heat being absorbed in the region.

3.1 Thermal decomposition Characteristic

The results obtained from the heating rates at 10, 15 and 20 ^{o}C/min from the TGA and DTA curves for NBRRI-ICSEB as presented in Figures 2a, 3a and 4a. The thermal decomposition of each of the NBRRI-ICSEB which shows the removal of water of hydroxylation within the range of ambient temperature (30 ^{o}C) to the stabilization region of 246.03 ^{o}C was negligible. Material thermal decomposition attributed to removal of water of hydroxylation is within this range as supported by [24]. This was also represented in all the DTA curves in Figures 2b, 3b and 4b where the peaks at which decomposition temperature range reached. The main devolatilization occurred within the second phase temperatures, ranging from 246.03 ^{o}C to 553.34 ^{o}C where bulk of masses of the ICSEB was lost, mainly due to carbonaceous material in the NBRRI-ICSEB devoid of moisture as already removed in the first phase of pyrolysis. The phase change is similar to those of [13] study on the use of thermogravimetric analysis. This implies that the

NBRRI-ICSEB cannot withstand high temperature as most decomposition occur below 600 °C in the range of oxides of carbon and other devolatilized matter based on the principles of TGA [21].

The Figures 2b, 3b and 4b of the differential thermal analysis curves of NBRRI-ICSEB showing the peaks at which decompositions occurred, were endothermic. Thermal decomposition is usually a chemical decomposition process caused by heat at which the substances chemically decomposed, and such reaction is usually described as heat absorption by the material (endothermic) being heat the required to break the bonds binding the ICSEB as undergoing the decomposition.

Table 1 present the thermogravimetric analysis at 10, 15 and 20 °C/min heating rates for the Nigerian Building and Road Research Institute Interlocking compressed stabilized earth block (NBRRI-ICSEB) as determined.

Table 1: Thermogravimetric Analysis at 10, 15 and 20 °C/min heating rates

Heating rate (℃/min)	Stage 1		Stage 2		Residual
	Temperature range (℃)	Weightlessness rate (%)	Temperature range (℃)	Weightlessness rate (%)	%
10	30.11 – 246.03	1.255	246.03 – 548.26	93.748	4.997
15	30.15 – 303.26	0.772	303.26 – 553.34	85.202	14.026
20	30.03 – 273.06	4.910	273.06 – 538.09	86.688	8.422

From Table 1, it could be deduced that greater mass losses was between 246.03 and 548.26 ℃ at a heating rate of 10 ℃/min, between 303.26 and 553.34 ℃ at a heating rate of 15 ℃/min and between 273.06 and 538.09 ℃ at a heating rate of 20 ℃/min respectively. According authors [23], these losses were associated with endothermic peak due to decomposition of calcite materials.

4 CONCLUSION

An instrumental characterization of the Nigerian Building and Road Research Institute-interlocking Compressed Stabilized Earth Block (NBRRI-ICSEB) was studied in this work via TGA/DTA instrumentation. The decomposition process was set between 30 ℃ to 950 ℃ for the heating rates at 10, 15 and 20 ℃/min respectively. The influence of temperature change was observed on the ICSEB material where greater mass loss was at second phase of the decomposition which was 93.75% mass loss at 10 ℃/min, 85.20% mass loss at 15 ℃/min and

86.67% mass loss at 20 ℃/min TGA analysis. While the corresponding TGA/DTA analysis revealed that the decomposition peak was attained at temperatures of 351℃ for 10 ℃/min, 380℃ for 15 ℃/min and 400℃ for 20 ℃/min heating rates respectively. The thermal stability is therefore determined based on the oxidation stability at stated heating rates. Thus; the finding has determined that the ICSEB decomposition temperature which is less than 600 ℃ as low bearing material. It is therefore recommended for further work on the life in-use and characterization of active components that make-up the NBRRI-ICSEB for optimization.

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CONFLICT OF INTEREST

The authors declared that there is no conflict of interest in this research, as it has not received any grant to do this work.

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