

EVALUATION OF PHYSICAL AND DYNAMIC MECHANICAL PROPERTIES OF COCONUT HUSK ASH (CHA) REINFORCED POLYESTER COMPOSITES

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Abstract: The evaluation of physical and dynamic mechanical analysis (DMA) properties was carried out on a developed Coconut Husk Ash Reinforced Polymer Composite. Sieve analysis of pretreated coconut husk ash was done to obtain 75 μm , 150 μm and 300 μm particles sizes. These particles were used at varying compositions of 5%, 10%, 15%, 20% and 25% as reinforcements for polyester composites. The catalyst and accelerator used were Methyl Ethyl Ketone Peroxide and Cobalt Naphthenate respectively. The densities of the evaluated composites made with 150 μm particles were found to be less dense with values ranging from 0.9792 g/cm^3 to 1.2561 g/cm^3 than those made with 75 μm and 300 μm . The results also show that the percentage water absorbed by samples increased, ranging from 0 to over 2000 E'/MPa for all percentage reinforcements of coconut husk ash, with an increase in the duration of immersion of the samples in distilled water. However, 25% reinforcement had better results for all particle sizes. There were obvious variations of storage modulus, loss modulus and mechanical loss factor with percentage weight of reinforcement, temperature and frequency. The composite with 15% reinforcement displayed better results. The composite shows promising results as a material for interior components in aerospace and automobile industries.

Keywords: dynamic mechanical analysis, sieve analysis, storage modulus, loss modulus, mechanical loss factor

1. INTRODUCTION

Composite materials are made up of a microscopic or macroscopic combination of two or more distinct materials with a recognizable interface between them. In composite materials, the reinforcements do not dissolve or merge completely in the matrix; usually, they can be physically identified with interfaces between them. Matrices can either be metallic, ceramic or polymeric. Their purpose is to bind the reinforcements (which can be natural or synthetic) together using their cohesive and adhesive characteristics for load transfer to the reinforcements and to protect the reinforcements from the environment and handling. The matrix is often considered the weak link in a composite when viewed from a structural perspective [1].

Environmental regulations and ethical concerns have triggered increased research for eco-friendly

reinforcement materials that are without adversely affecting the required composite properties. As a consequence, this responsiveness is pushing governments toward more stringent legislations, which promote the energy preservation and protection of the quality of the environment for future generations [2].

Bio-based products such as shells of various dry fruits, rice husks, wheat husk, straws and hemp fibres have been used to prepare bio-based reinforced polymer composites [2-7]. There are great advantages in developing new bio-based products which include, among many others, biodegradability, lightweight, abundance, cost-effectiveness, non-toxicity, relative strength, renewability and their consideration to be virtuous products; they also have good formability, thermal insulation properties and acoustic properties [8].

Coconut fruits are produced by coconut palm trees; they comprise an outer epicarp, a mesocarp, and an inner endocarp. The epicarp (which is the outer skin of

the fruit) and mesocarp (which is heavy, fibrous, and tanned when dry) has many industrial uses. The endocarp is the hard dark core that is made up of solid white with varied thickness, depending on the age of the fruit. It has a consistent oily pulp and liquid called coconut water that is sweet, slightly acidic and thick [9].

Figure 1 shows a sectional view of the coconut fruit. Annually, approximately 33 billion coconuts are harvested worldwide with only 15% of these coconuts being utilized for fibres and chips [10]. The Coconut husk is made up of polymeric structures of 28% cellulose, 38% hemicellulose and 32.8% lignin [11]. This study is aimed at producing a coconut husk ash-reinforced polyester composite and evaluating its physical properties and dynamic mechanical analysis.

The studies of physical properties of composites such as density and water absorption characteristics by researchers are prompted by the quest to get engineering materials that have less weight for use in industries like aerospace and automobile and to check the reliability of these materials in humid environments. Dynamic mechanical analysis is used to indicate the molecular relaxation dynamics of material [12]. The objective of this study is to evaluate the density, water absorption characteristics and dynamic mechanical analysis of polyester when reinforced with varying quantities of coconut husk ash.



Fig. 1. Sectional view of a coconut fruit [28]

2. MATERIALS AND METHODS

Coconut husks were locally sourced from Makurdi, Benue State, Nigeria. Unsaturated Polyester Resin (matrix material), Methyl Ethyl Ketone Peroxide (catalyst) and Cobalt Naphthenate (accelerator) were obtained from Ojota Chemical Market, Lagos, Nigeria. The following equipment and materials were used for the tests: Electronic weighing machine, laboratory sieves (JINLING BS40 Series SHANG CHINA),

disposable cups, distilled water, Dynamic Mechanical Analyser (Model: NETZSCH DMA 242E).

Light-weighted materials with less-humid and dynamic-loading used for engineering applications such as interior components in aerospace and automobile industries come with a high cost. The search for alternative materials that are locally available with less cost has become eminent for use as composite reinforcement with competitive properties with the high-cost materials [5].

Following the above background, this study has developed a polyester composite that is reinforced with coconut husk ash, having competitive physical and mechanical dynamic properties with the high-cost materials.

2.1. Methods

Preparation of reinforcement material

Coconut husk ash was used as the reinforcement material. Dry coconut husks were soaked in clean water for 48 hours to soften them, after which they were retted to coconut husk fibres. The retted fibres were thoroughly washed in clean water to remove dirt; they were dried and thereafter pretreated. Chemical pretreatment of the coconut husk fibres was achieved by soaking the dried coconut husk fibres in a solution of sodium hydroxide (NaOH) for 24 hours [13]. The fibers were removed and washed thoroughly with clean water; they were dried and burnt with a sufficient supply of oxygen (in the open air) to obtain the ash particles. Three particle sizes (75 μm , 150 μm and 300 μm) were characterized from the ash using standard laboratory sieves.

Composite materials formulation

The weight percentages of the matrix (Polyester resin) and reinforcement (Coconut husk ash) were weighed into a mixing container as required by the values in Table 1 and stirred continuously at room temperature for about three minutes until a homogenous mix was observed; the same procedure was repeated for other matrix and reinforcement amounts needed for the other particulate mixtures. Each mixture was placed on an electronic weighing machine and the catalyst (Methyl Ethyl Ketone Peroxide, MEKP) was added in drops until the required weight was obtained (1%wt of Polyester resin); the mixture was thoroughly stirred for three more minutes. Thereafter, the accelerator (Cobalt Naphthenate) was also added to the required weight (0.5%wt of Polyester resin) [14].

Composite production

Manual mixing in a plastic container and hand lay-up techniques were used for the composite's production. The composites were prepared using 5%, 10%, 15%, 20%, and 25%, reinforcement of coconut husk ash for each particle size.

A spirit level was used to check the level of the table upon which the wooden moulds were to be placed. The

mixtures, obtained as explained above, were gently poured into the wooden mould which had been coated with paraffin wax (as a releasing agent) to ease the removal after curing. While pouring the mixture, the mould was gently shaken continuously so that the mixture spreads evenly. The ceiling fans were switched off to prevent bubble formation. The casts were allowed to cure, at room temperature. The cured plates were removed from the mould with the overflow flakes cut off. Finally, the composite plates were cut into different dimensions for mechanical and physical property tests as shown in Figure 2 below. This part should contain sufficient detail so that all procedures can be repeated. It can be divided into subsections if several methods are described. Composite materials formulation is shown in Table 1.

Tab. 1. Composite materials formulation

Materials	Particle size	Weight percentage (wt%)				
		5	10	15	20	25
Coconut husk ash	75 μm	5	10	15	20	25
Polyester resin		95	90	85	80	75
Coconut husk ash	150 μm	5	10	15	20	25
Polyester resin		95	90	85	80	75
Coconut husk ash	300 μm	5	10	15	20	25
Polyester resin		95	90	85	80	75



Fig. 2. Picture of samples produced

Density test

The density test was done according to Archimedes' principle. A sample with known mass was immersed in a graduated beaker containing a known volume of distilled water. The difference in the volume of water was recorded as the volume of the sample. The density of samples was obtained using equation 1.

$$\rho = \frac{m}{v} = \frac{m}{v_2 - v_1}, \quad (1)$$

where: ρ = density, g/cm^3 ; m = mass of substance, g; v = volume of substance, cm^3 ; v_1 = volume of liquid before immersion of substance, cm^3 ; v_2 = volume of liquid after immersion of substance, cm^3 .

Water adsorption test

The water absorption test of the composites was carried out according to American Standard Testing and Measurement Method (ASTM) D570. Samples were weighed and thereafter placed in a disposable cup filled with distilled water at room temperature. The samples were removed from the water, cleaned with a neat piece of cloth, and reweighed at an interval of 24 hours for 168 hours. The water uptake (weight gained) was measured by subtracting the weight of the dry sample from the weight of the wet sample. The percentage of water absorption was determined by using equation 2 [10].

$$\text{Percentage of water absorption} = \frac{w_2 - w_1}{w_2} \times 100\%, \quad (2)$$

where: w_2 = weight of the wet sample, g; w_1 = weight of the dry sample, g.

Dynamical Mechanical Analysis

Dynamic mechanical properties of the samples were determined using Dynamic Mechanical Analyzer, using NETZSCH Proteus software. Samples made from 150 μm reinforcements with the dimensions $40 \times 12 \times 5$ mm were used for the test. The following conditions were observed: measurements made in the open air, and dual measurement method using cantilever, loading frequency of 2 Hz, 5 Hz and 10 Hz, temperature interval

was between 30°C and 150°C, at a heating speed of 3°C/minute, following Bastiurea *et al.* [15].

3. RESULTS AND DISCUSSION

3.1. Density

The results of the density of various percentages and particle sizes of composites samples are shown in Figure 3. The densities are in the range of 0.72 g/cm³ to 1.29 g/cm³. The results showed that the density of composites made with 150 μm particles was less dense than the composites made with 75 μm and 300 μm (except for 10% reinforcement where 75 μm is less), with the least value been 0.98 g/cm³ and the highest been 1.26 g/cm³. This result reflects similar research results of other organic particle reinforced polyester composites [16, 17]. It was also observed that the density of these composites was not higher than the density values (1.15 g/cm³ and 1.6 g/cm³) reported for unreinforced polyester by Shehu *et al.* and Oluremi *et al.* [16,18] showing that the density of composite formulated by the substitution of coconut husk ash particles was not increased above that of unreinforced polyester.

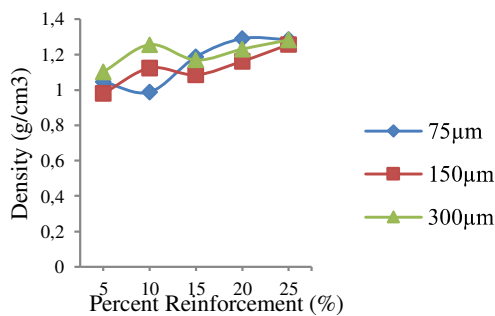


Fig. 3. Effect of reinforcement on density of coconut husk ash reinforced polyester composite

3.2. Water absorption

The results of the water absorption of various percentages and particle sizes of composites samples are shown in Figures 4, 5 and 6. The figures display the rate of water absorption daily for 168 hours. The results showed that the percentage water absorbed by samples increased, for all percentage reinforcements of coconut husk ash, with an increase in the duration of immersion of the samples in distilled water. As a lignocellulose material, coconut husk ash is hydrophilic, hence would absorb moisture when placed in a humid environment [17]. The percentage of water absorbed, however, decreased with an increase in the percent reinforcement of coconut husk ash, with 25% reinforcement having the best values for all particle sizes; it was also found to decrease as the particle size of reinforcement increased, but for a few exceptions with 5% reinforcement which would possibly be because of void formation. The result is similar to the

findings of Shehu *et al.* and Dagwa *et al.* [16, 19], who reported that the finer the reinforcement particles used, the more water will be absorbed.

Alkali treatments have been reported to reduce the hydroxyl group in the cell wall of the biodegradable material molecule, thus decreasing the water absorption of composites [20]. This is a possible reason why the composite displayed the obtained results.

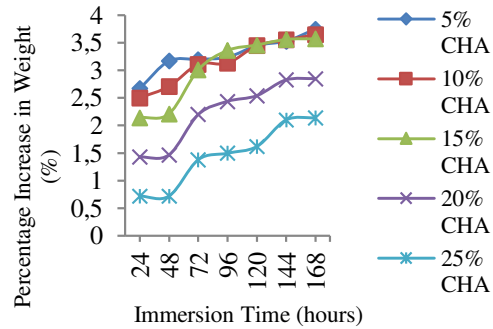


Fig. 4. Water absorption characteristics of 75 μm coconut husk ash reinforced polyester composite

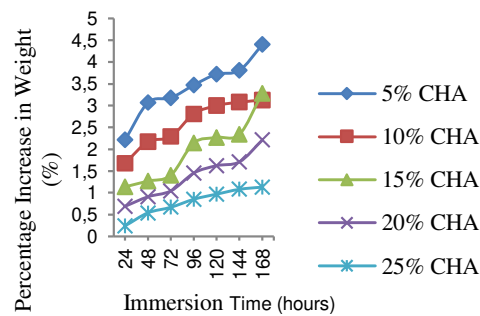


Fig. 5. Water absorption characteristics of 150 μm coconut husk ash reinforced polyester composite

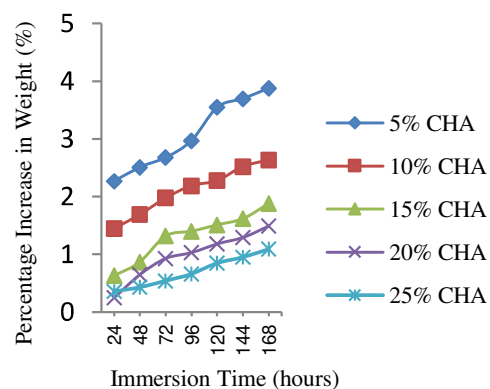


Fig. 6. Water absorption characteristics of 300 μm coconut husk ash reinforced polyester composite

3.3. Dynamic mechanical analysis

The results of the water absorption of various percentages and particle sizes of composites samples

are shown in Figures 7, 8, 9, 10 and 11. The DMA results provide information about the mechanical properties of the smaller specimen, usually sinusoidal as a function of time and temperature by subjecting it to

a small sinusoidal force. This information is often presented as storage modulus, loss modulus and mechanical loss factor (damping factor).

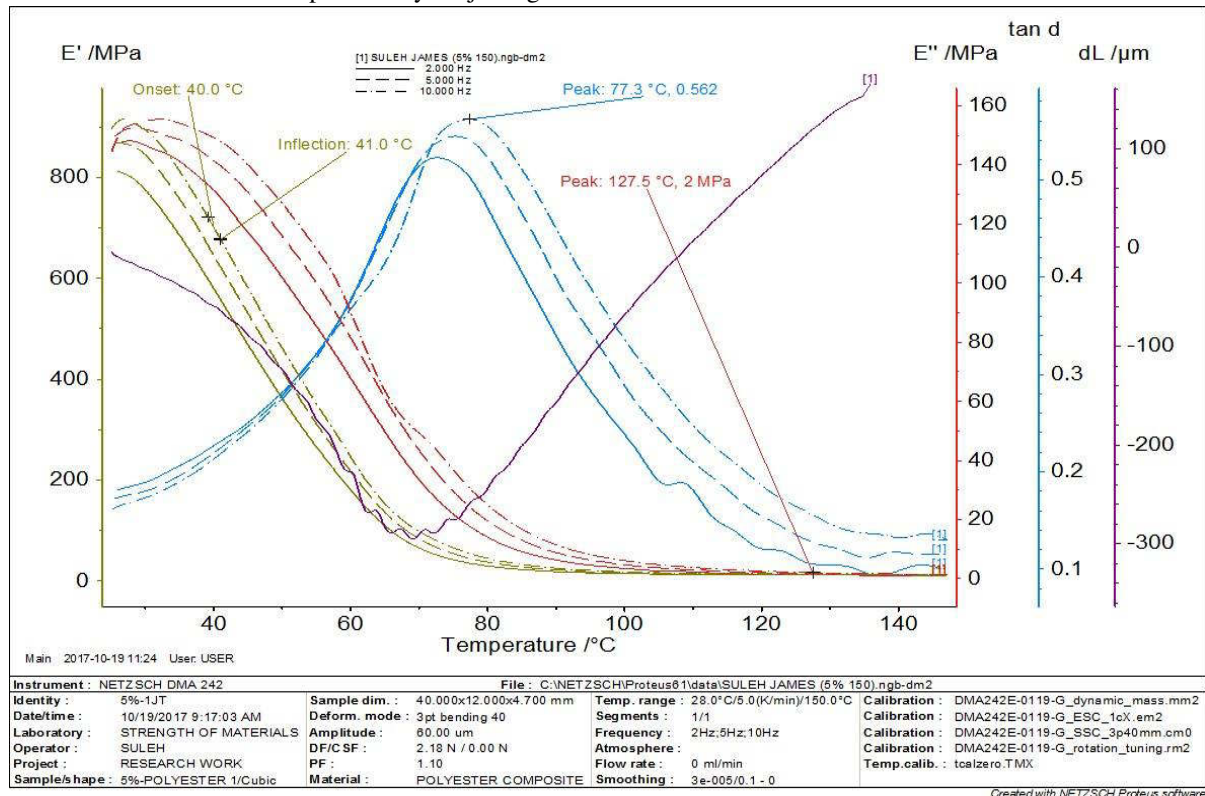


Fig. 7. Storage modulus (E'), loss modulus (E'') loss factor ($Tan \delta$) and change in length (dL) of 5% coconut husk ash reinforced polyester composite

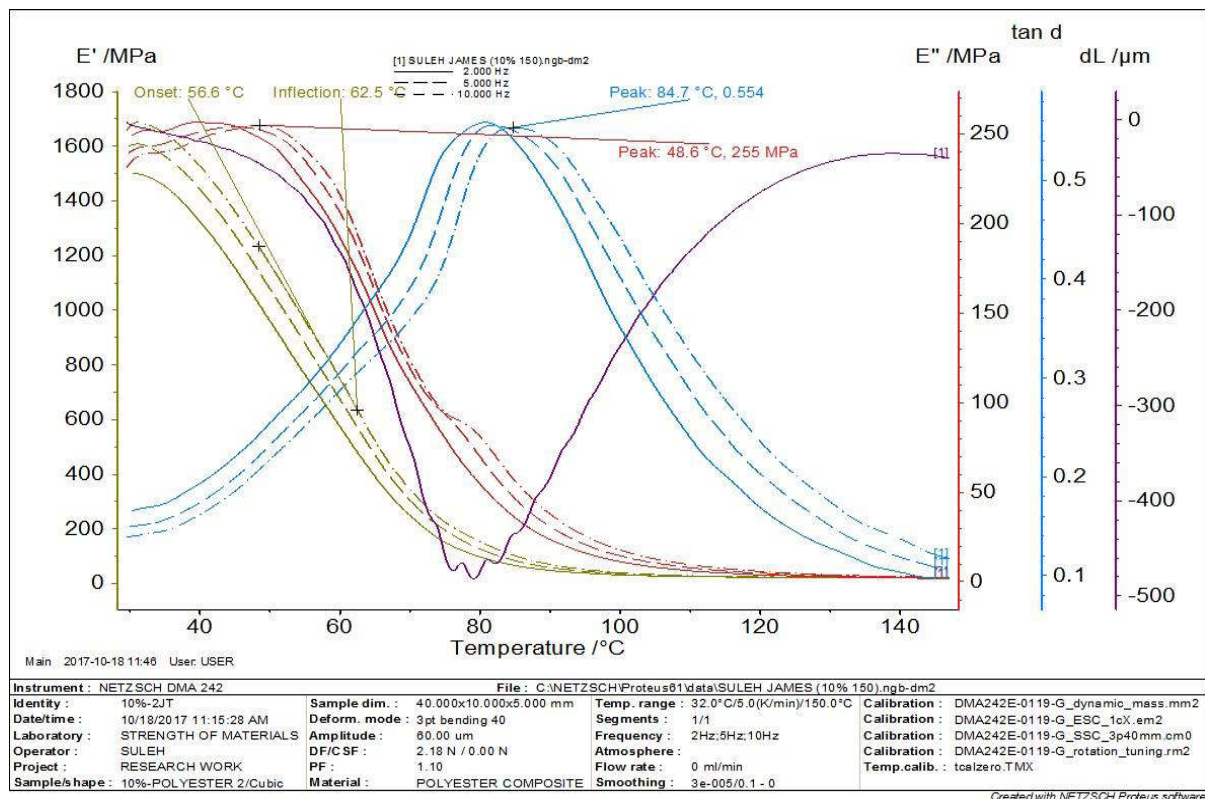


Fig. 8. Storage modulus (E'), loss modulus (E'') loss factor ($Tan \delta$) and change in length (dL) of 10% coconut husk ash reinforced polyester composite

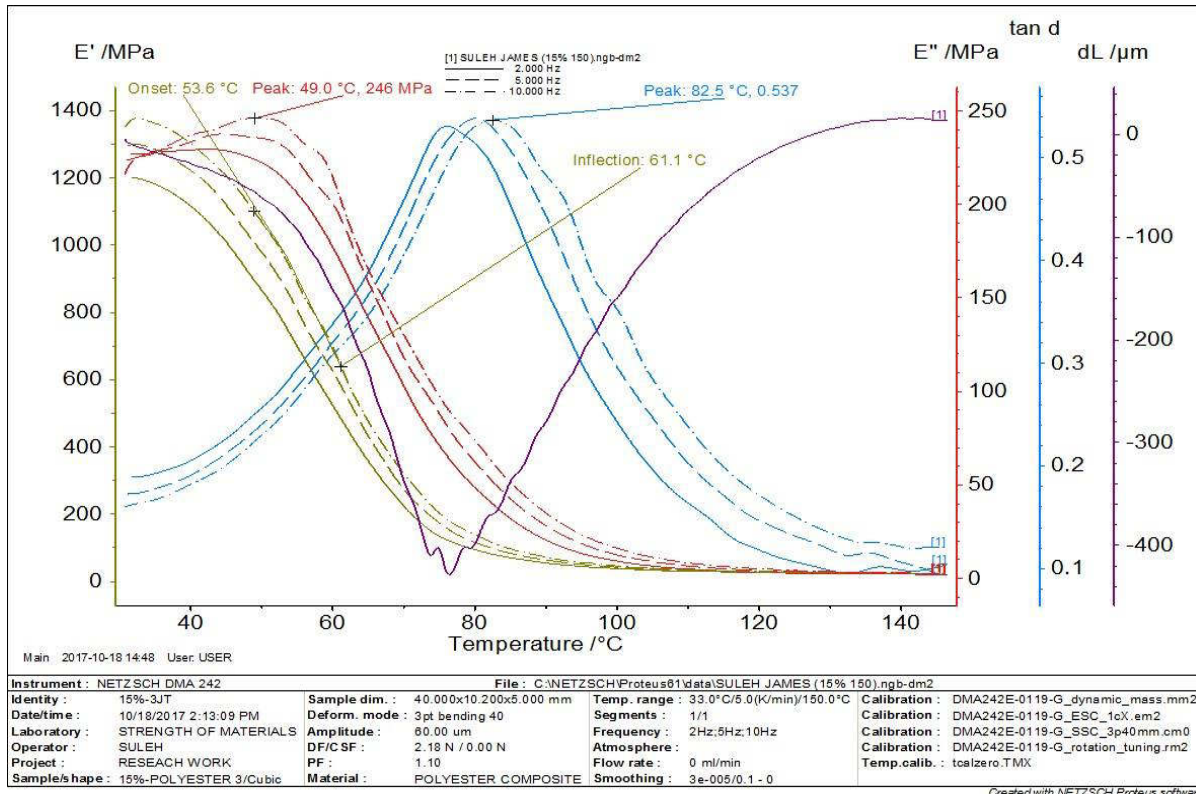


Fig. 9. Storage modulus (E'), loss modulus (E'') loss factor ($Tan \delta$) and change in length (dL) of 15% coconut husk ash reinforced polyester composite

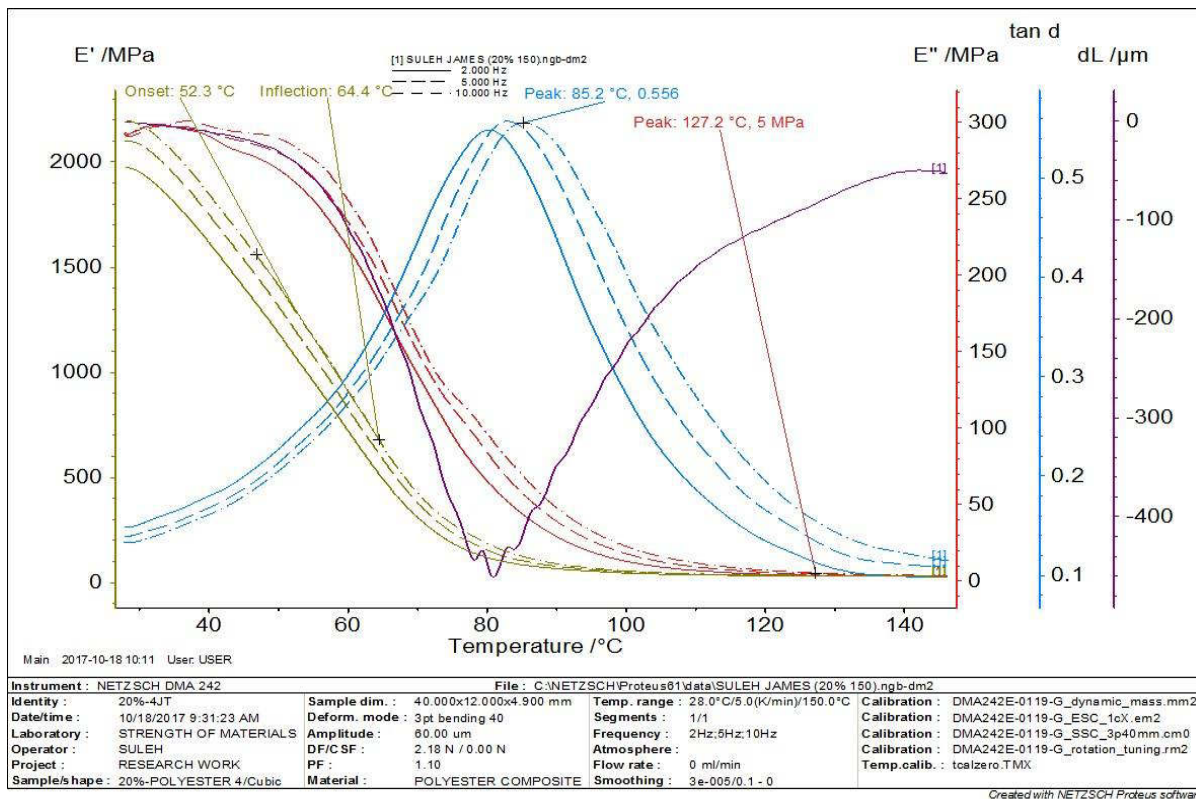


Fig. 10. Storage modulus (E'), loss modulus (E'') loss factor ($Tan \delta$) and change in length (dL) of 20% coconut husk ash reinforced polyester composite

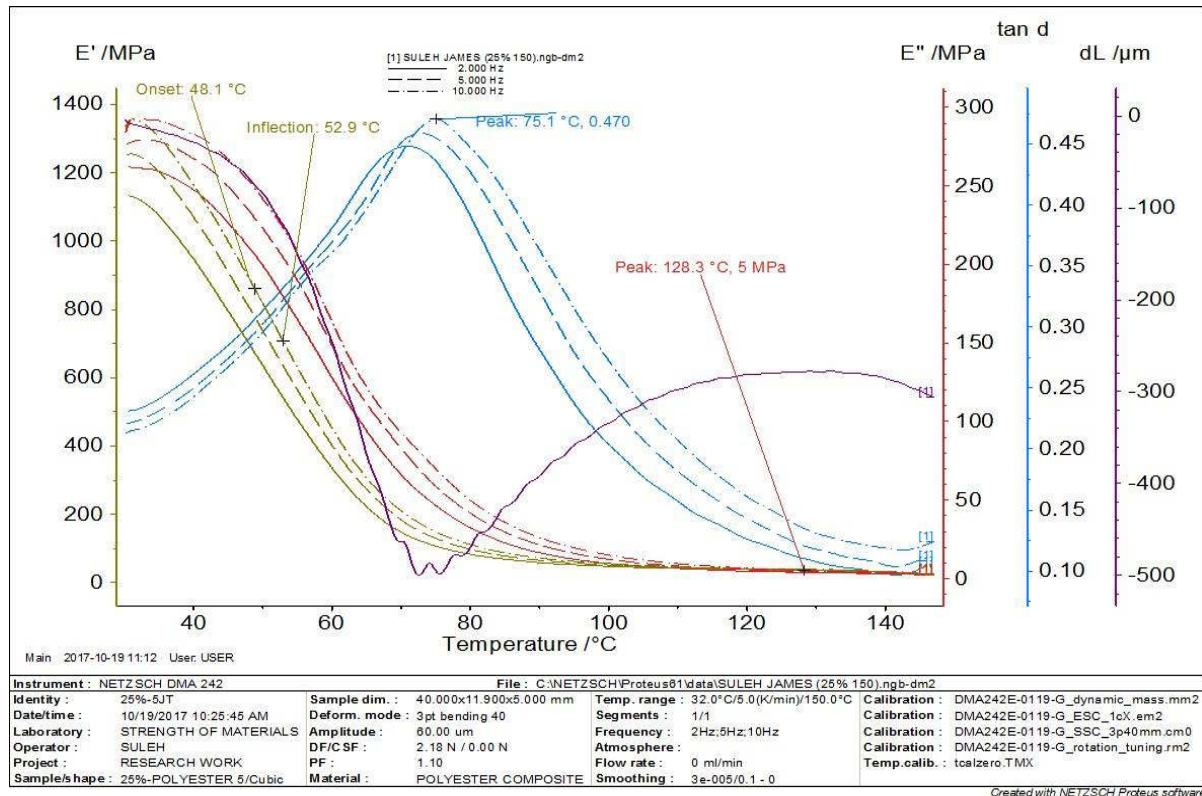


Fig. 11. Storage modulus (E'), loss modulus (E'') loss factor ($\tan \delta$) and change in length (dL) of 25% coconut husk ash reinforced polyester composite

Storage modulus

Storage modulus is the in-phase component of the complex modulus that measures the elastic response (stored energy) of a viscoelastic material [21]. The variation in storage modulus as a function of weight percent of reinforcement, temperature and frequency for the studied composites can be seen in Figures 7, 8, 9, 10 and 11.

The results show that as the weight percent of CHA increases in the composite, the values of storage modulus also begin to increase. The E' of the polymer has been reported to increase in the elastomeric region due to strong reinforcement/matrix interface and addition of reinforcing materials that impart higher stiffness [21]; this was the result that was obtained when CHA, being a high stiffness material, was added to the polyester matrix: E' increased as the percentage reinforcement of CHA increased to a maximum value at 20 wt% CHA; while the increase in weight percent of a high-stiffness material can help to increase the E' , when the weight percent is so high that there is poor reinforcement/matrix interface or there is a presence of void in the composite, the reinforcing effect of the material will not be maximized [22]. The decrease in E' values at 15 wt% CHA and 25 wt% CHA can be attributed to void formation and agglomeration resulting in weak reinforcement/matrix interface.

Figures 8, 7, 9, 10 and 11 show that as the process temperature increases within the transition region, the value of E' decreased steadily for all weight percent of

the composite; At lower temperature, E' is higher because the thermal energy is not enough to reach the threshold energy to reorganize the molecular chain segment of the composites; but as the temperature increases, the thermal energy reaches the threshold energy and the molecular chain segment is reorganized; hence, there was a loss of coherent structure as the material changed from glassy to rubbery state because of molecular mobility of the polymer molecules as the temperature was raised [23]. The study of temperature influence is important because materials with the same composition can fail at different E' values when they are subjected to environments of different temperatures. The samples are observed to have better E' values below 40 $^{\circ}\text{C}$; Ornaghi *et al.* have also reported a similar result [24].

The results also show that, for all composite reinforcements, E' increased with an increase in loading frequency, 10 Hz having the highest E' values for all reinforcements. Polymers, being viscoelastic materials, are by definition time-dependent. This means that the response of viscoelastic material to an imposed deformation will depend on how fast or slow the deformation was applied to the sample [25]. Since frequency is the inverse of time, high frequencies are analogous to shorter times while low frequencies are analogous to longer times. Hence, at a higher frequency, the molecular chain segment has a shorter time to respond to the applied stress and lacks the time to reorganize, resulting in higher E' ; as the frequency

decreases, there is enough time for the molecular chain segment to reorganize, which can relax the local strain into a lower energy conformation resulting into lower E' [25].

Loss modulus

The Loss modulus of a composite is the maximum energy released by composite materials during deformation. It presents the viscous response of the materials which depends on the motion of polymeric molecules in the composites [22]. The variation in loss modulus as a function of weight percent of reinforcement, temperature and frequency for the studied composites can be seen in Figures 7, 8, 8, 10 and 11.

The curves for the variation of loss modulus with percent reinforcement of CHA are similar to those of storage modulus. The loss modulus in the transition region is higher for composites with higher CHA content, which may be due to an increase in internal friction, prompting energy dissipation [24]. It can also be observed that as the CHA content increases, the loss modulus curves spread over wide distributions and show a higher peak. This effect can be a consequence of the inhibition of the relaxation process of the chain segments in the composites due to an increase in the rigidity of chain segments, increasing material heterogeneity [24]. Higher peaks of loss modulus have also been attributed to an increase in the mobility of the polymer chains [24].

The variation of loss modulus as a function of temperature can be seen in Figures 7, 8, 9, 10 and 11. It can be observed that as the temperature increases, the loss modulus also increases to a maximum point for all percent reinforcements of the composite before a decline. The point of inflection is sometimes used to locate the glass transition temperature. These bell-shaped curves have been reported by other researchers too [20, 21, 23]. However, whereas the increase in loss modulus as temperature increases can be easily seen in the curves of these works, the curves of this research may not be seen; this is because of the temperature range of the experiment. Oboh *et al.* reported that the best way to interpret these curves is to view them as force versus energy dissipation rather than modulus versus temperature, thus upon initial heating, though the material will be less stiff, more force will be dissipated as heat, increasing the loss modulus; as the temperature continues to increase above a critical point as a result of further heating, there will be a reduction in molecular friction and hence less energy will be dissipated leading to a decrease in loss modulus [12].

It can be observed that the loss modulus increases with an increase in frequency, with 10 Hz having the highest values. Similar to the explanation of storage modulus, the molecular chain segment has a shorter time to respond to the applied stress at a higher frequency and lacks the time to reorganize, resulting in

a higher loss modulus; as the frequency decreases, the time is enough for molecular chain segment to reorganize hence lower storage modulus [26].

Mechanical loss factor

The Mechanical loss factor of polymer composites is a measure of the energy they dissipate; it tells how good a material will be at absorbing energy. It is also known as Damping Factor or Tan Delta and is the ratio of loss modulus to the storage modulus. Delta should range between 0° and 90° ; as delta approaches 0° , the material tends to a purely elastic behaviour, while as delta approaches 90° , the material tends to a purely viscous behaviour. The variation in mechanical loss factor as a function of weight percent of reinforcement, temperature and frequency for the studied composites can be seen in Figures 7, 8, 9, 10 and 11.

The results of mechanical loss factor variation with temperature show that as the temperature increases, the damping factor also increases to a maximum value and thereafter there is a gradual decrease, for all percent reinforcement of the composite. The peak region is the material's transition region from the glassy region to a rubbery region, and the glass transition temperature, T_g is located in this region. Below T_g , damping is low because the deformations in this region are primarily elastic, the molecular slips resulting in the viscous flow are low since the chain segments are in a frozen state. Similarly, the chain segments are quite free to move in the rubbery region, hence damping is low. The molecular chains begin to move in the transition region and every time a frozen segment moves, its excess energy is dissipated as heat [24]. The position and height of the damping peak are indicative of the structure and properties of a particular composite material [27].

The variation of damping plots with the temperature at frequencies show that the damping plots peak heights increase with frequency. It can be seen that for all percent reinforcements, 10 Hz has the highest damping plot peaks. This is explained by the fact that any material subjected to constant stress over some time, undergoes a decrease in its elastic modulus due to its molecular rearrangement which minimizes the localized stresses, hence when it is performed over a short time (high frequency), higher damping plots are obtained; also, T_g will be shifted towards higher temperatures [27].

4. CONCLUSIONS

In this research work, the physical properties and dynamic mechanical analysis of coconut husk ash reinforced polyester composite were studied. The following conclusions were drawn:

1. This study has developed a polyester composite that is reinforced with coconut husk ash, having

- competitive physical and mechanical dynamic properties with the high-cost materials.
- The densities of the evaluated composites made with 150 μm particles were found to be less dense with values ranging from 0.9792 g/cm^3 to 1.2561 g/cm^3 than those made with 75 μm and 300 μm .
 - The percentage water absorbed by samples increased, ranging from 0 to over 2000 E'/MPa for all percentage reinforcements of coconut husk ash, with an increase in the duration of immersion of the samples in distilled water. However, 25% reinforcement had better results for all particle sizes.
 - The composite showed a minimal increase in density and moisture absorption when placed in a humid environment. It also displayed remarkable dynamic mechanical properties. The composite reinforced with 15% coconut husk ash had the best dynamic mechanical properties.
 - Based on the results obtained, coconut husk ash reinforced polyester composite is a promising engineering material for many light-weighted, less-humid and dynamic-loading engineering applications such as interior components in aerospace and automobile industries.

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