Hybridization Effect on Thermo-mechanical Behaviour of Epoxy/breadfruit Seed Shell Ash Particles and Momordica Angustisepala Fiber Composites for High-temperature Devices Application

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Abstract- ***Polymer general has a limitation in the area of high temperature which restricts, it used in devices application. To overcome this shortcoming that leads to the development of Epoxy/Breadfruit seed shell ash particles and Momordica Angustisepala fiber composites. Two sets of composites were produced epoxy/30wt%MAf-20wt%BFSAp and epoxy/30wt% functionalized MAf-20wt%BFSAp. The dynamic mechanical and thermal properties, scanning electron microscopy and X-ray diffractometer were determined. A 42.30% improvement in the storage modulus of the matrix was obtained at epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp hybrid composite. It established that combined effect of functionalized MAf and BFSAp can be used to improve the thermal behavior and dynamic mechanical of Epoxy- MAf-BFSAp hybrid composites which can be used for device production.***

Indexed Terms- ***Momordica Angustisepala fiber, Breadfruit seed shell, Microstructure, Thermal properties, and Storage Modulus.***

I. INTRODUCTION

The development of hybrid composites to overcome the limitation of polymer and single reinforcement of polymer composites for slightly high-temperature devices applications such as in the electronic and computer has been giving attention in recent years [1-3]. Among the recent work on hybrid composites is the combination of particular and fibers in the production of composites materials. The hybrid composites have several advantages such as high tensile, flexural strength, elastic, impact energy, thermal stability, abrasion and wear resistance [4]. Thermal properties of polymer composites play an important role in the study of the structure-property correlation of the thermal stability of the materials [5]. Thermogravimetric analysis (TGA) is an important method in determining the moisture and other volatile materials in the composites [6-8], while the dynamic mechanical analysis (DMA) studies the thermal-mechanical behavior of the composites as a function of temperature [9-10].

Heitor Luiz et al [11] reported on the dynamic mechanical and mechanical behavior of polymer/glass-curaua fiber hybrid composites. The impact strength, storage modulus, hardness values, flexural modulus, and strength increased with glass addition. Mohamed et al [12] used wt% of 40, 50 and 60 date palm fibers to enhance the flexural strength, dynamic mechanical and thermal properties. They observed that the flexural strength improved from 26.15 MPa to 32.64 MPa at 50wt% date palm fibers, also the thermal properties were improved with the composites having the higher weight retained than the epoxy. The storage modulus was enhanced upto 50wt% date palm fibers. Aisyah et al [13] presented the thermal properties of carbon-woven kenaf-epoxy composites. Thermogravimetric and differential scanning calorimeter was used for the thermal analysis. The carbon fiber composites displayed higher thermal stability. Jawaid and Khalil [14] presented improvement in the thermal properties of hybrid composites in comparison with single reinforcement, Atiqah et al [15] showed that there are higher thermal properties in reinforcing glass fibers with sugar palm fibers in a polyurethane matrix. Ridzuan et al [16] worked on the dynamic mechanical analysis and thermal properties of hybrid composites using epoxy/Pennisetum purpureum-glass fibers. The composites were produced using vacuum infusion

method. They observed enhanced thermal properties. From the literature above it appeared that the use of breadfruit shell ash particle (BFSAp) and Momordica angustisepala fiber (MAf) has not been reported in the improvement of thermal and dynamic mechanical analysis for devices application. Hence the research aims to explore the huge amount of waste BFS and MA in Africa regions for the development of epoxy composites with improves thermal and dynamic mechanical analysis

Breadfruit is a food plant planted in many Africa countries such as Nigeria, Angola, Togo, Ghana, Sudan, Senegal, Sierra Leone, and Cameroon, etc [17]. The breadfruit seed is cultivated and the shell is removed from the seed (see Figure 1). The seed is eaten as food while the shell is dumped to the ground or open burning causing environmental pollution [18]. Ezechukwu et al [18] stated about a developed Al-Si-Fe alloy composites reinforced with breadfruit seed shell ash particles. Breadfruit seed shell ash particles of particle size of 500nm were used in the production of composites. They concluded that the tensile strength and hardness values improved as the weight % breadfruit seed shell ash particles is increased in the formulation with slightly decreased in impact energy. Atuanya et al [19] developed composites materials using low-density polyethylene and breadfruit seed shell ash particles. The breadfruit seed shell ash particles were varied from 5-25wt% with 5wt% interval. There was a good distribution of breadfruit seed shell ash particles in the polymer and better mechanical properties obtained with a reduction in impact energy.

Figure 1: Photograph of breadfruit seed and the shell [19].

Momordica angustisepala fiber (MAF) is one of the promising natural fibers for the production of polymer composites [20]. The fibers can be obtained in many Africa countries such as Nigeria, Cameroon, Ghana, Benin, and Cote Divoire e.tc [21]. The fibers are obtained from MA stem by pounding and washing of the fibers to remove impurities [22]. It can be cultivated in commercial quantities. The MA fibers are used in Africa as a local sponge for bathing [22]. Achigan-Dako [23] used MA as local medicine in Nigeria and Atuanya et al [21] characterized the MAf for possible use for polymer composites production. Their findings show tensile strength of 35-57.93MPa and tensile modulus 2-4.4GPa. MAf is like other natural fibers that usually have weak interfacial bonding when used in the production of polymer composites.

II. MATERIALS AND METHOD

The fibers were cleaned with acetone for 48hrs and oven-dried at 75oC for 5hrs. The cleaned fibers bundle was kept in glass containers. The cleaned fibers were soaked in 80ml sodium hydroxide (OH) solution and were heated to the temperature of 105oC for 3hrs after then the fibers were washed with 5%HCl solutions and distilled water and dried in the oven at 75oC to obtain hydroxylated fibers [18]. The silane fictionalization was done by preparing 2ml of Aminopropyltriethoxysilane (APS) in 100ml ethanol at a temperature of 150oC at a time of 2hours [24] (see Figure 2).

Figure 2: Chemical treatment of MA fibers

Before the breadfruit seed shell was used in the production of the composites. The breadfruit seed shell was sun-dried for three days and pulverized into a fine powder. The pulverized powder was packed in a crucible made of graphite and placed in a muffle electric furnace. The furnace was heated to a temperature of 1000oC and hold for 5hours. The particle size of the powder sample after heating was done and size of 63µm was used in the research [24]. X-ray fluorescence (XRF) model X-MET 8000 was used in determining the composition of the BFSAp.

A white Epoxy resin LY556 (HERENBA BRAND) and Hardener (HY951) was used in the production of the composites. The hand-laying method was used in the production of the hybrid composites using Epoxy-30wt%MAf-20wt%BFSAp and Epoxy-30wt% treated MAf-20wt%BFSAp. The produced composites are displayed in Figure 3.

Figure 3: Photograph of produced samples

SEM model JEOL JSM840A was used to determine the microstructure of the composites. X-ray diffractometer was used to determine the crystallinity index of the composites. The thermogravimetric machine model(STA 6000) was used for the thermal analysis. The test was conducted as per ASTM E1131 with a heating rate of 10oC/min. The dynamic mechanical test was done with a dynamic mechanical machine model (Perkin Elmer 8000). The test was conducted as per ASTM D4065 using a heating rate of 5oC/min and 1Hz.

III. RESULTS AND DISCUSSION

3.1 Composition of the Fibres

The treated and untreated MA fibers were immersed in the toluene/ethanol solution (2:1%v/v) before there composition was determined [24]. The results obtained are displayed in Figure 4. However, the cellulose content of the untreated fibers increased after treatment. It was clear that OH-APS treatment of MAf decreases the amount of wax, hemicelluloses, and impurities. This decrease obtained could be attributed to the fact that the treatment of the fibers with OH-APS activates the O-H and hydrolyzable alkoxy group to formed silanols. This action changes the surface of the MAf and decreases the amount of wax, hemicelluloses, and impurities. A similar observation was obtained in the work of [25-26].

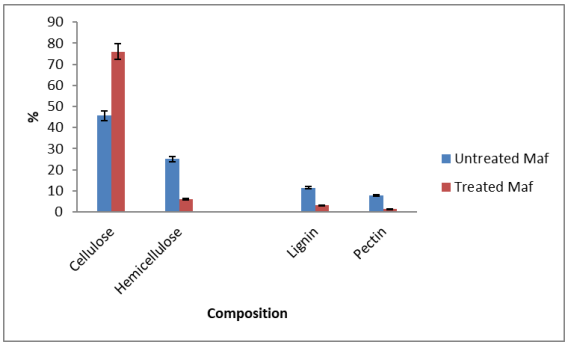

Figure 4: Composition of the Fiber

3.2 Composition of the BFSAp

X-ray fluorescence (XRF) analysis was used to determine the chemical composition of the BFSAp. The result of the XRF is displayed in Table 1. From Table 1 it was seen clearly that the major components of BFSAp are: K2O(30.5%), CaO(30.6%), Fe2O3(17.8%), SiO2(8.5%) and P2O5 (6.01%). The result obtained is in par with the BFSAp earlier analyzed by Atuanya et al [21] and used in the production of composites.

Table 1: Composition of BFSAp

Element	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3	BaO	EuO_3
%	8.5	6.01	2.0	30.5	30.6	0.25	0.09	17.8	0.3	3.7

3.3 Microstructure of the MAf and BFSAp

Figures 5-6 displayed the SEM/EDS of the treated and untreated MAf. The SEM images show that fibers are longitudinal and straight in shapes. However, there is quite a difference between the SEM image of the treated and untreated MAf. It was seen clearly in Figure 6 that there are cleaned and straight fibers without grooves. The chemical treatments help to remove the waxy epidermal tissue, hemicelluloses in the MAf. Thinner diameter of the MAf was obtained from the treatment this help to increase the strength. This is in par with the work of [27-28]. In the EDS of Figure 6, the presence of Si in the OH-ASP treated MAf shows that the hydrolyzable alkoxy group formed silanols [28].

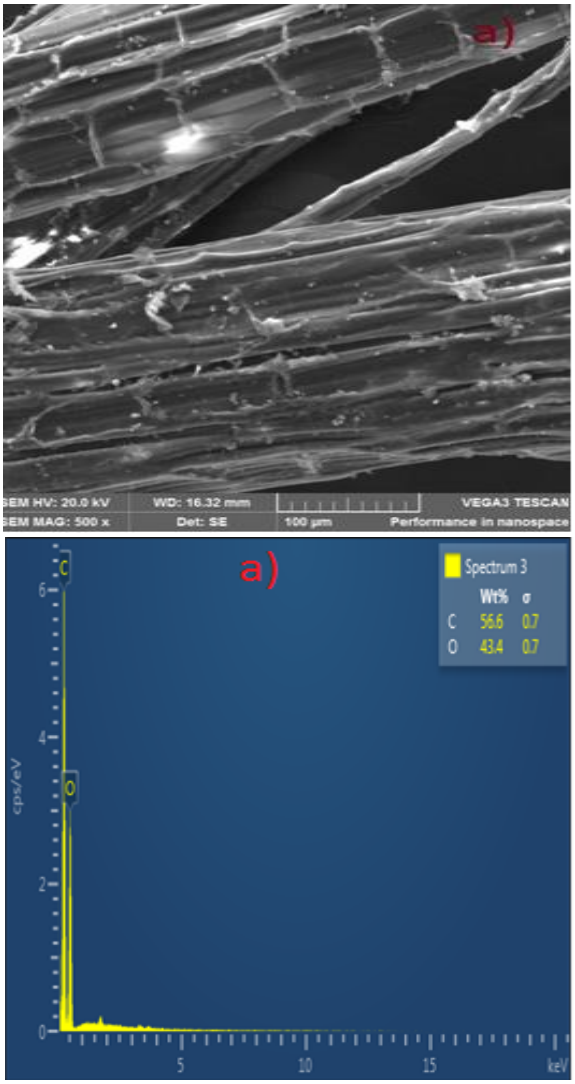

Figure 5: SEM/EDS of the untreated MA Fiber

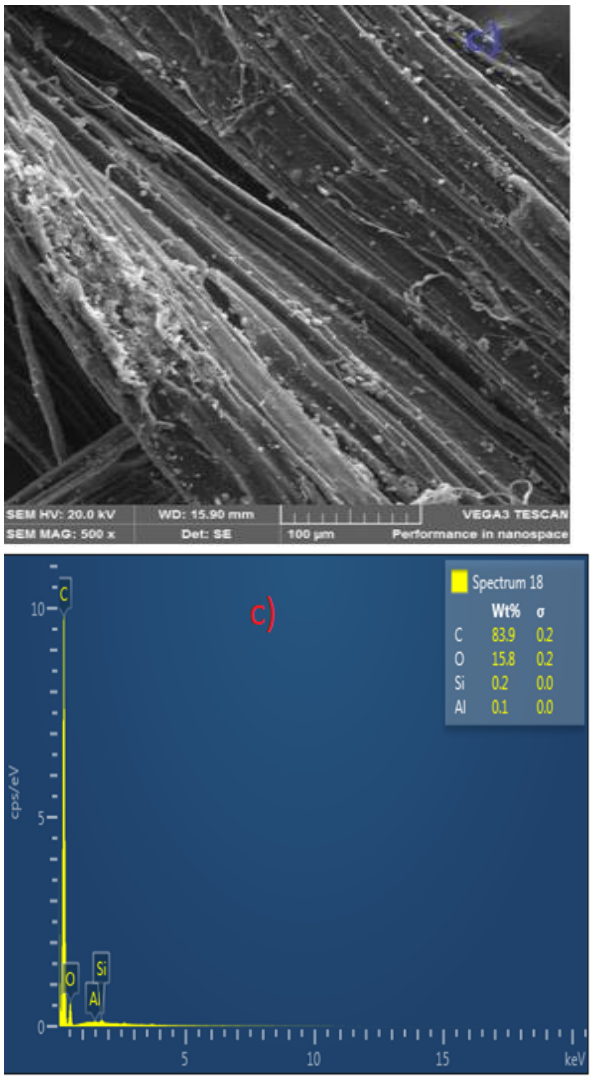

Figure 6: SEM/EDS of the treated OH-APS MA Fiber

Figure 7 displayed the SEM/EDS of the BFSAp. The microstructure of the BFSAp shows that the particles are round and spherical in shapes and randomly distributed. In Figure 7 it was observed that the particle size varies from fine, medium, and coarse. These particles help to close the interlocking chains of the polymer, and hence increase the strength of the composites. The EDS has a high peak of K and Ca this is in par with the result of the XRF discussed above that BFSAp contain a high amount of K2O and CaO

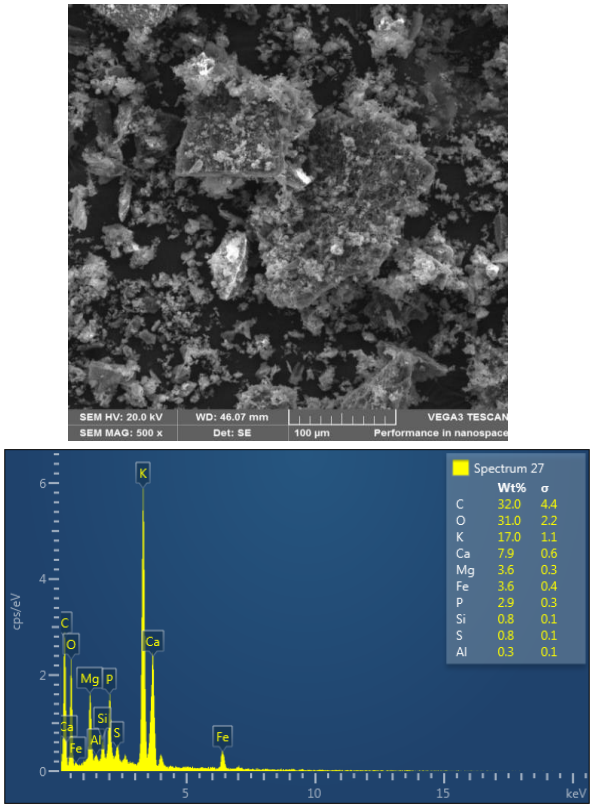

Figure 7: SEM/EDS of the BFSAp

3. 4 Crystallinity Index

The X-ray diffraction patterns of composites are given in Figure 8. The crystallinity index (CrI) of the composites were calculated according to the empirical method shown in the following equation (1)

$$\%CI = \frac{I_{002} - I_{am}}{I_{002}} X100 \quad (1)$$

where I002 and Iam are the peak intensities of crystalline and amorphous materials, respectively.

The major peaks observed for samples at 2θ diffraction angles were around 10.15 and 20.17o. The first peaks at 10.15o are the low angle reflection representing I(am) of amorphous material with an intensity of counts 1856, 2083, 2436 for the epoxy, epoxy/30wt%MAf-20wt%BFSAp and epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp respectively. While the reflection at 20.17o had higher intensity, and it represented I(002) of crystalline material in with the intensity of 2150, 3761, 6772 counts for the epoxy, epoxy/30wt%MAf-20wt%BFSAp and epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp respectively(see Figure 8). Values of 13.67, 44.61, and

64.03% were obtained for the crystallinity index for the epoxy, epoxy/30wt%MAf-20wt%BFSAp, and epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp respectively. It was seen that the degree of crystallinity index of the treated composite is higher than the untreated composite and epoxy. This might be because of the removal of hemicellulose which results in close packing of the cellulosic chain [29]. The higher crystallinity index for the functionalized OH-APS treated MAf help to strengthen the composites and increase the interfacial bonding between the reinforcement and the epoxy matrix. A similar observation of an increase in the crystallinity index of composite has been reported in the literature [30-31].

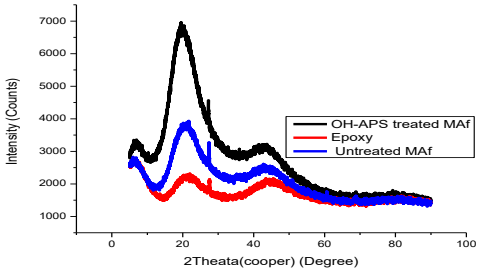

Figure 8: X-ray diffraction analysis of the composites

3.5 Thermal Analysis

Figure 9 and Table 2 presented the TGA analysis of the samples under investigation. It can be evident in Figure 9 that the BFSAp had higher thermal stability and lower weight loss of the entire sample. This observation could be attributed to the higher temperature treatment of the BFSAp and also the chemical makeup in the BFSAp: K2O, CaO, Fe2O3, SiO2 and P2O5, these presences of hard oxides help to slow down the rate of degradation and burning of the BFSAp, a similar observation was obtained in the work of [21]. In Table 2 it was seen clearly that the retained it 81.89% weight at a temperature around 1000oC. The MAf has thermal behavior opposite that of BFSAp. In Table 2 it can be observed that the MAf had lower thermal stability. The MAf has a residue weight of 2.19 with maximum thermal decomposition temperature of 355.25oC, this is in par with the other natural fibers [21, 23].

From Figure 9 it was observed that the composites have higher thermal stability than the epoxy matrix. The initial weight loss observed at a temperature between 50-100oC is as a result of the vaporization of the moisture in the materials. The MAf has the lower thermal properties this could be attributed to the fact that chemical makeup of the natural fibers like cellulose, hemicelluloses, pectin, etc could result in higher weight loss due to thermal decomposition. Worked has shown that there is the absorption of water in the interfacial between the epoxy and the reinforcement which has been recorded in much work [32].

From Figure 9, it was evident that composites have two degradation peaks. The first peak is as a result of the water molecule in the materials and the second peaks shifted to higher temperature resulting in higher thermal stability when compared with the epoxy matrix. The derivative thermogravimetric weight loss was also analyzed to determine the derivative mass loss of the samples under investigation. The higher derivative peak in Figure 9b corresponded to the maximum rate of decomposition and degradation. However the composites have a lower rate of derivative mass loss (see Figure 9b), degradation of epoxy occurs in two-level chain scission and dehydration [33].

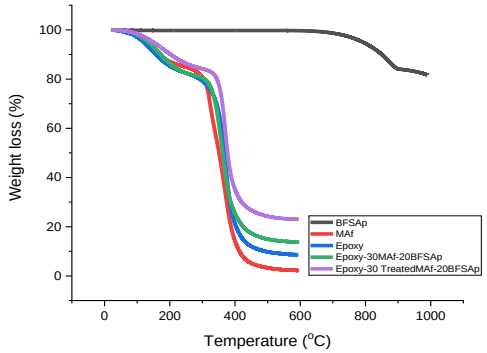

Figure 9a: Variation of Weight loss with Temperature

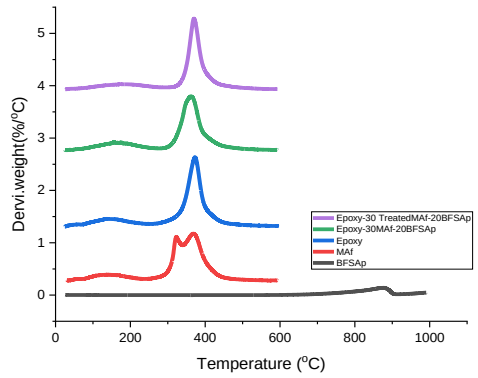

Figure 9a: Variation of derivative of weight with Temperature

Table 2: TGA analysis of the Samples

Samples	$T_{5\%\ loss}(^oC)$	$T2_{5\%\ loss}(^oC)$	$T_{50\%\ loss}(^oC)$	$T_{max}(^oC)$	Weight left(g)
BFSAp	790.61	N/A	N/A	885.00	81.89
MAf	119.81	317.21	351.02	355.25	2.19
Epoxy	121.58	327.17	357.99	367.8	8.6
Epoxy-30wt%MAf-20wt%BFSAp	137.09	330.09	362.20	400.25	13.82
Epoxy-treated 30wt%MAf-20wt%BFSAp	148.57	355.37	377.27	423.12	23.00

Figure 9 it can be seen clearly that the hybrid composites have higher thermal stability than the polymer matrix for example 50% weight loss was recorded at 357.99, 362.20 and 377.27oC and temperature of maximum thermal decomposition of 367.80, 400.25 and 423.12oC with residue weight of 8.60, 13.82 and 23.00g for the epoxy, epoxy/30wt%MAf-20wt%BFSAp, and epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp respectively. The enhancement of the thermal behavior can be farfetched from the high thermal stability of the BFSAp which delayed the rate of degradation of the composites. However, the functionalized MAf had higher thermal stability than the untreated MAf. The high enhancement in the thermal behavior of the treated MAf was attributed to O-H and hydrolyzable alkoxy groups that formed silanols. This active silanols change the surface of the MAf and decrease the amount of wax, hemicelluloses, and impurities and improving the thermal behavior. It can be recorded in this new work that the combined effect of functionalized MAf and BFSAp can be used to improve the thermal behavior of epoxy- 30wt%MAf-20wt%BFSAp hybrid composites which can be used for devices production.

3.6 Dynamic Mechanical Analysis

3.6.1 Storage Modulus

Figure 10a presented the results of the storage modulus. The storage modulus displayed the information such as the interfacial bonding, elasticity of the materials, and stiffness with temperature. It displayed the glassy, transition, and rubbery regions in the materials. It can be seen that there was an increase in the storage modulus at the frozen stage of the glassy temperature. It was observed that the decrease in the values of the storage modulus observed above the glassy transition temperatures was as a result of the ease of movement of the polymer chain as temperature increases [33].

There was a great increment in the values of storage modulus in the elastomeric and glassy ranges of the samples, this phenomenon can be explained in that fact that in the strength obtained in this region higher depend on the package of the polymer chain and intermolecular forces that hold the polymer chain together [34]. The reduction in the values of the storage modulus observed in all the samples as temperature increases were attributed to the movement of the polymer chain in a microbrwnian level [35]. However the reduction in the storage modulus was lower in the composites as compared with the epoxy matrix, the hydrodynamic factor was the major for the enhancement of the storage modulus of the composites as results in the reduction in the mobility and deformability of the polymer [36].

The composites have higher storage modulus as compared with the matrix this means higher stiffness and good particles-fibers distribution were obtained in the matrix. for e.g at 140oC a storage modulus of 355.35, 401.23, and 505.67MPa were obtained at epoxy, epoxy/30wt%MAf-20wt%BFSAp and epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp respectively, which corresponded to 42.30% improvement in the storage modulus of the matrix at epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp hybrid composite. The increase in the storage modulus of the composites could be attributed to good interfacial bonding between the BFSAp, MAf and

epoxy matrix which results to transfer of load from the reinforcing phases to the matrix and blocking the interlocking molecular chains of the polymer, this reduced the ease mobility of the polymer chains under load as temperature increases [34-36].

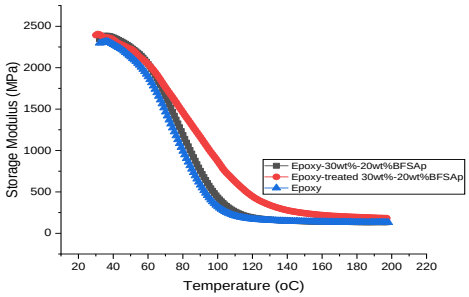

Figure 10a: Variation of Storage modulus with Temperature

3.6.2 Loss Modulus

Figure 10b displayed the results of the loss modulus. Loss modulus measured the amount of energy dissipated during deformation in the viscoelastic region of the materials. From Figure10b it evident the composites have a higher improvement in the values of loss modulus. The addition of BFSAp and MAf help to improve the loss modulus which means that the maximum dissipation of mechanical energy per heat cycle at the maximum and reduced at a higher temperature, as a result, the movement of the epoxy chain become easing [32]. The broadening peaks observed for the loss modulus of the composites in comparison with the narrow in the epoxy means that the reinforcement used in this study was able to improve the polymer chain segment. The functionalized MAf has a higher peak resulting in higher energy dissipation as compared with the composite without functionalized MAf and epoxy.

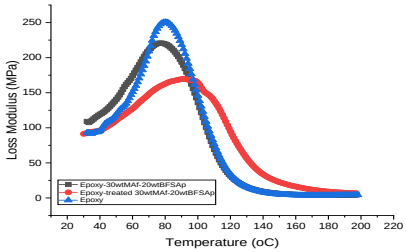

Figure 10b: Variation of Loss modulus with Temperature

3.6.3 Damping Factor

The damping factor was affected by the addition of the BFSAp and MAf to the formulation. There was an increase in the damping factor in the transition temperature ranges to a maximum and decreased in the rubbery region. As compared with the matrix, the reinforcement reduced the viscoelastic of the materials and hence increased in the mobility of the molecular of the polymer chain. The functionalized MAf have the smaller damping factor among the samples under investigation and the broadening and wider of the damping as a result of improving the density of crosslinking of the epoxy polymer [36]

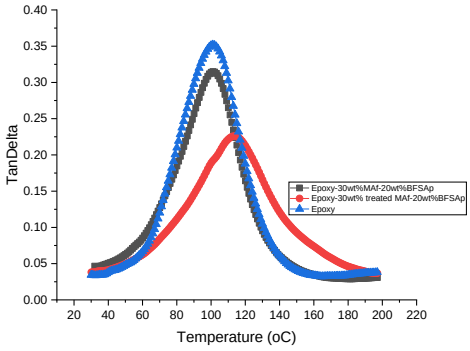

Figure 10c: Variation damping factor with Temperature

3.6.4 Cole-Cole plot

The relationship existing between the storage and the loss modulus was analyzed using the Cole-Cole Wicket plot. Figure 10d presented the Cole-Cole curves of the composites under investigation. The Cole-Cole was used to discuss the changes that occurred in the cross-linked in the polymer as a result of the addition of BFSAp and MAf. It can be seen in Figure 10d that the Cole-Cole is higher in the epoxy polymer than the composites. The semicircles observed in the polymer are attributed to the amorphous behavior of the polymer in the relaxation region[31]. The addition of the BFSAp and MAf to the formulation increased the heterogeneity in the materials that accompanied the relaxation time as can be seen clearly in Figure 10d. It was evident from the imperfect semicircular shape as displayed in Figure 10d.

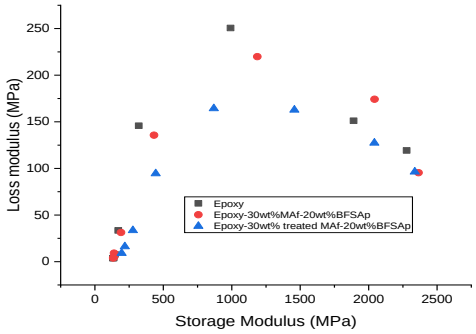

Figure 10d: Displayed the Cole-Cole plot

CONCLUSION

New findings were achieved in this present work. The higher crystallinity index obtained for functionalized MAf help to strengthen the composites and increase the interfacial bonding between the reinforcement and the epoxy matrix. A 42.30% improvement in the storage modulus of the matrix was obtained at epoxy/OH-APS treated 30wt%MAf-20wt%BFSAp hybrid composite. The TGA curves shifted to higher temperatures resulting in higher thermal stability of the composites. It can be concluded in this work that the combined effect of functionalized MAf and BFSAp can be used to improve the thermal behavior and dynamic mechanical of Epoxy- MAf-BFSAp hybrid composites which can be used for devices production.

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