Dynamic Mechanical Analysis of Ukam (Cochlosperum Planchonii) Fibre Reinforced Polyester Composites

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Abstract— Natural fibers show potential to replace glass fibers in thermoset and thermoplastic composites.. In this research Ukam/polyester composites were analyzed using Dynamic Mechanical Analysis (DMA). A three-point bend apparatus was used in the DMA testing. The samples were tested at 2 hertz, 5 hertz and 10 hertz at a displacement of 10 μm, and at room temperature. The fiber volume content of the Ukam was varied from 5%, 10%, 15%, 20% and 25%. The flexural storage modulus, the flexural loss modulus, and the loss factor were reported. Generally, as the fiber volume fraction of Ukam increased, the flexural storage and flexural loss modulus increased. The loss factor remained relatively constant with increasing fiber volume fraction. This enhancement of the properties can be attributed to improvement in the interfacial adhesion and compatibility between the ukam fibre and matrix.

Index Terms— damping factor, loss modulus, polyester resin, storage modulus, Ukam.

1 Introduction

In recent years, natural reinforcer (rice husk, bagasse, straw, banana and maiz stalk) in form of fiber or particulate has been widely used as reinforcement in thermoplastic composite materials. The natural reinforcers are lighter and cheaper and provide much higher strength per unit mass than most inorganic reinforcers. Besides ecological considerations, several technical aspects promote the renewed interest for the natural reinforcer as supplement or replacement for traditional reinforcer (e.g., glass fibers) in polymer composites [1-3]. Natural reinforcer from agricultural residues and forest products processing consist of lignocellulosic. As a resulted, they are subjected to thermal degradation during composite processing and application [1, 4]. It is of practical significance to understand and predict the thermal decomposition process of natural reinforcers and the knowledge will help better design of composite materials for thermal application. Previous research of thermal decomposition of natural reinforcers was primarily motivated by applications such as renewable biomass energy/natural fuels [5-7], forest fire propagation control [8]. Due to the complexity of thermal decomposition reactions of natural reinforcers, extensive researches has been done in determining individual behaviors of the main components or pseudo- components (e.g., pure cellulose, lignin, and hemicelluloses) [5-8]. Earlier works on the thermal decomposition of polymer reinforced with natural fibers shows that it has low thermal degradation of the polymer composites as a result of the lignocellulosic nature of natural fibers [4], which is not satisfactory. Hence there is need, to study the thermo-mechanical properties of ukam fibre polyester composites. It is in the light of the foregoing that the research on the investigation of the Dynamic Mechanical Analysis of Ukam Fibre Reinforced Polyester Composites was motivated. **Do not change the font sizes or line spacing to squeeze more text into a limited number of pages. Please be certain to follow all submission guidelines when formatting an article or it will be returned for reformatting.**

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2 Materials and Methods

2.1 Materials

Ukam fibres were sourced from Ikem in Isiuzo LGA of Enugu. The ukam plant fibre was obtained from the stem which consists of wood core with bast fibres. In this stem are a number of fibre bundles each containing individual fibre cells. The Ukam fibres was purchased from Juneng Nigeria Limited, Nsukka and Polyester resins, Polyvinyl alcohol (PVA), Cobalt octane, Methyl ethyl Ketone Peroxide (MEKP), Sodium hydroxide, acetic acid, silane, Benzoyl chloride and potassium permanganate (KMnO4) was supplied by Moore Chemicals, Zaria, Nigeria.

2.2 Fibre Surface Treatment

In this study, both untreated and chemically treated fibres were used for analysis and property characterization of the natural plant fibres. The fibres were chopped into 100 mm length before giving the treatment. The chopped strand fibre bundles were subjected to different surface treatments with alkali, silane solution, KMno4 and acetic anhydride to investigate the variation in the properties after treatment. The concentration of chemicals and soaking time are the key factor affecting treatment.

Plate 1. Cut Stems of Cochlospermum Planchonii Fibers

Plate 2. A Sample of Ukam woven fibre

2.3 Sample Preparation

Ukam fiber was used as reinforcement and general purpose polyester resin as a matrix for the composite material of the laminated specimens. The steps of manufacturing the composite using the hand lay-up process are described below (Mohammed et. al. 2010). The hand lay-up process is an open molding technique. The surface was thoroughly cleaned for use by removing any dust and dirt from it. After the mold surface has been cleaned, the release agent was applied and the mold surface was coated with free wax using a smooth cloth. Then a film of polyvinyl alcohol (PVA) is applied over the wax using sponge. PVA is a water-soluble material and 15% solution in water is used. When water evaporates a thin film of PVA is formed on the mold surface. PVA film was dried completely before the application of resin coat. This is very important because it helps in the removal of the laminates from the mould. The matrix material was prepared using general purpose polyester. Cobalt octane (0.35% by volume of resin) is added to act as accelerator and Methyl Ethyl Ketone peroxide (MEKP) (1% by volume) is added to act as catalyst. Resin, accelerator and catalyst were thoroughly mixed. The use of accelerator is necessary because resin does not cure properly. After adding the accelerator and catalyst to the polyester resin, it is left for some time so that bubbles formed during stirring may not die out. The amount of added accelerator and catalyst is not high because a high percentage reduces gel time of polyester resin and may adversely affect impregnation. A unidirectional ukam fiber with thickness (0.3 mm) was used as a reinforcement, which was cut in layers of required dimensions for each specimen. To prepare the laminate, the Ukam fiber is laid in the mould, the resin is spread uniformly over the fiber by means of a brush until fibers are immersed in the resin. External pressure plate is used to apply pressure while curing to enhance uniform thickness reached. The casting was carried out at room temperature for 24 hours and firmly removed from the mold to get a fine finished composite plate.

2.4 Characterization

DMA (dynamic mechanical was carried out using DMA 242E machine in strength of materials laboratory of mechanical engineering, A.B.U. Zaria. The test parameters were first configured via the proteus software using the computer (PC). Parameters such as evaluation functions (storage modulus, tan d and deflection), instruments set up (sample holder, furnace temperature and furnace thermocouple) and measurement mode (temperature, static and dynamic loads) were configured. Sample specimen of dimension of 60x12x5mm was produced for each test. The sample was loaded on to the machine using three-point-bending sample holder and subsequently locked into the furnace. The test was then carried out for the respective DMA

3 RESULTS AND DISCUSSION

Dynamic mechanical analysis (DMA) is dynamic non-destructive material testing. DMA inputs a sinusoidal force and measures the deflection of the material response to the force. DMA uses the inputted force and material displacement to calculate bulk material properties. The material properties interested in for this research are the flexural storage modulus, flexural loss modulus, and loss factor (tan δ). The storage modulus is a measurement of materials stiffness, similar to Young's modulus. The loss modulus represents the viscous response of the material. This can be defined as the energy lost from the material. The loss factor is simply a ratio of the loss modulus and the storage modulus. The loss factor is also known as tan δ, where δ is the phase angle from the sinusoidal input and the material response. The loss factor is also known as damping. DMA has the unique capability of being able to vary the frequency throughout a test. This makes it very simple to compare how materials are affected by different frequencies. The temperature can also be changed throughout a test like the frequency. The glass transition temperature can be measured by dynamically changing the temperature and observing the storage modulus, loss modulus, and loss factor. There are three ways to detect the glass transition temperature. The temperature where a spike in the loss factor is found, the point where the loss modulus has a sudden increase before it begins a slow decrease, and at the point where the storage modulus begins to decline [9].

3.1 Storage Modulus

Figures 2-6 showed the variations in storage modulus of untreated and treated Polyester/ Ukam fibers composites as a function of temperature. From Figures 2-6 showed high storage modulus at room temperature followed as a function of temperature. The storage moduli (E') of the untreated and treated composites were found to be: 1750, 1800, 1750, 1650, 200MPa for the polyester at 60 °C for the untreated, Alkali, KMNO4, Silane and Acetylation treatment respectively. These values increased to 2750, 5250, 2800, 3100, 4100MPa at 60 °C for the untreated (15%fibers), Alkali (20% fibres), KMNO4 (25% fibres), Silane (10% fibres) and Acetylation (25% fibres) treatment respectively. The positive effect of the addition of fibers and the treatment was evidence (see Figures 2-6). All the samples show high storage modulus at room temperature followed by significant drops in the temperature range 25-200 □C. The higher E' values of the treated composites may due to greater interfacial adhesion and bond strength between matrix resin and fibre as reported in previous studies(Daiane Romanzini et al, 2013). Figures 2-6 has the typical curve of the storage modulus: a plateau firstly until the glass transition temperature, accompanied by a sharp decrease. At high temperatures, the treated fibers composites has a relatively higher storage modulus than that of neat polyester, which is attributed to the high stiffness and strength of Ukam fibers and also the increased crystallinity of polyester by Ukam fibers and chemical treatment as confirmed by the SEM analysis. Therefore, the combination of Ukam fibers reinforcement and the enhanced crystallization of the polyester matrix lead to the better mechanical properties of the composite.

The value of storage modulus decreases at higher temperature due to loss in stiffness of the Ukam fibers. The increase in the storage modulus was attributed to the reinforcement effect in the polyester composites imparted by Ukam fibers allow better stress transfer from the polyester matrix to the Ukam fibers. These effects are related to the improvement of the thermo-mechanical stability in polyester composites.

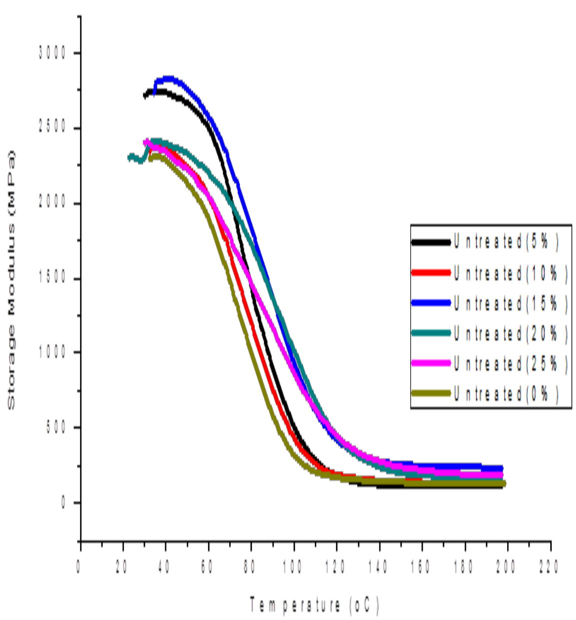

Figure 2: Variation of Storage Modulus with Temperatures for the untreated Fibres Composites

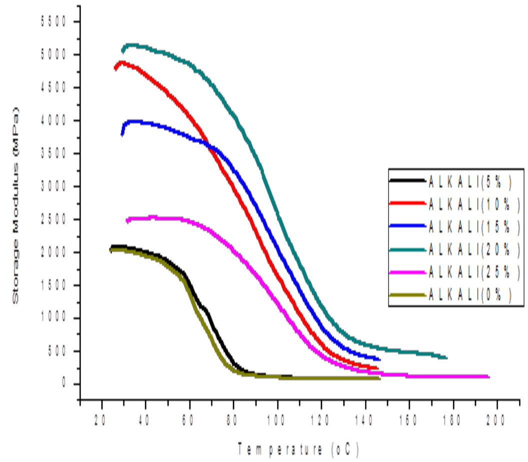

Figure 3: Variation of Storage Modulus with Temperatures for the Alkali-treated Fibres Composites

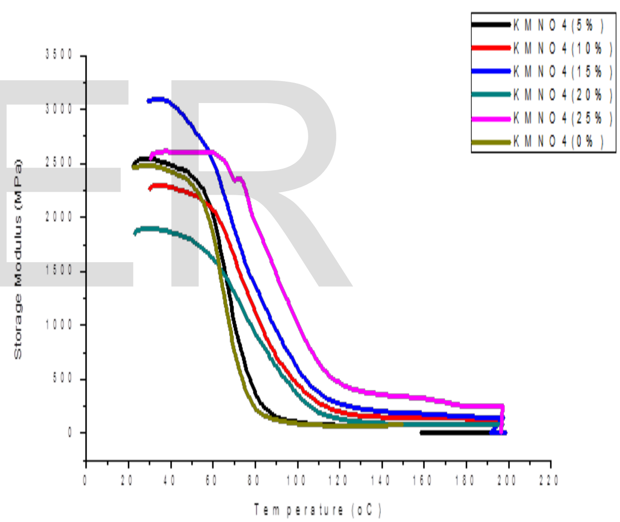

Figure 4: Variation of Storage Modulus with Temperatures for the KMNO4-treated fibres Composites

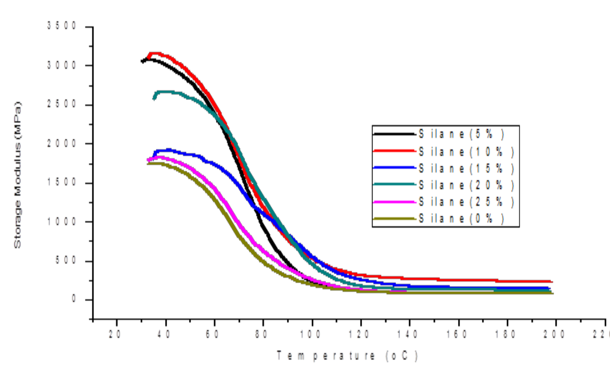

Figure 5: Variation of Storage Modulus with Temperatures for

the Silane-treated Fibres Composites

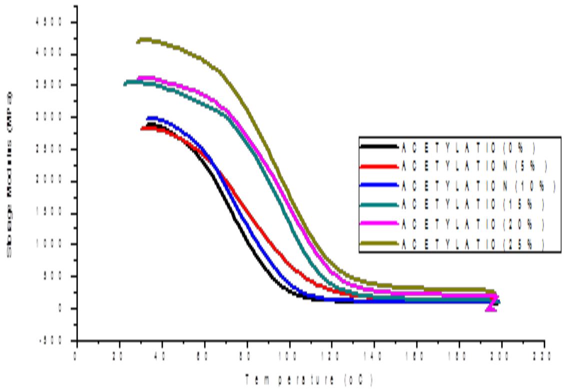

Figure 6: Variation of Storage Modulus with Temperatures for the Acetylation-treated Fibres Composites

Alkali treated Ukam fiber/polyester composite has highest storage modulus (5250MPa), when compared to rest of the composites sample. The highest storage modulus of Alkali treated composites may be attributed the strong fiber matrix interface between the fiber and matrix, which leads to efficient stress transfer from the matrix to fiber and enhancement in storage modulus. Followed by Acetylation treated and Silane treated fibre reinforced composite also gives higher values of storage moduli (4100, 3100MPa) in comparison to untreated fiber composite (2750MPa).

Therefore, with the obtained DMA test result, it will be appropriate to say that the addition of chemically treated fiber increases the storage modulus which indicates that the load bearing capacity of Ukam fiber reinforced composite is increased. Therefore, it can be said that, treated Ukam fiber reinforcement helps in increasing the storage modulus of polymer composite. The chemical treatment improves the wetting behavior of fibers with resin as result of which storage modulus increases.

3.2 Loss Modulus

Similarly, Figures 7-11 shows the variation in loss modulus of the untreated and treated composites with temperature. The untreated composites show the loss modulus peak at 80-100 □C, these values shifted to a higher temperature for the treatment fibres. These values obtained which could be attributed to the mobility of resin molecules.

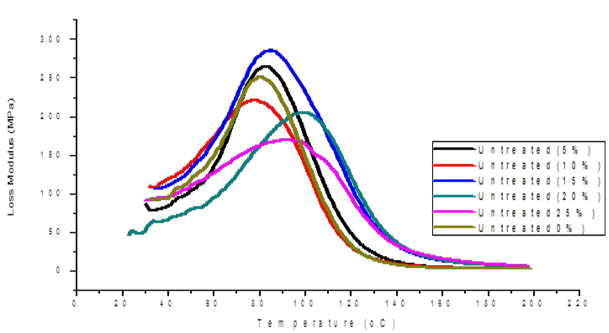

Figure 7: Variation of Loss Modulus with Temperatures for the Untreated fibres Composites

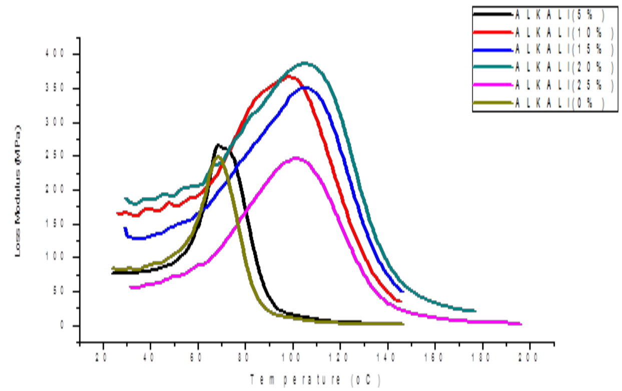

Figure 8: Variation of Loss Modulus with Temperatures for the Alkali-treated Fibres Composites

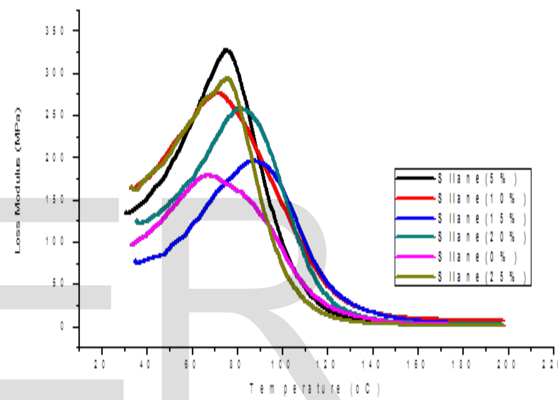

Figure 9: Variation of Loss Modulus with Temperatures for the Silane-treated Fibres Composites

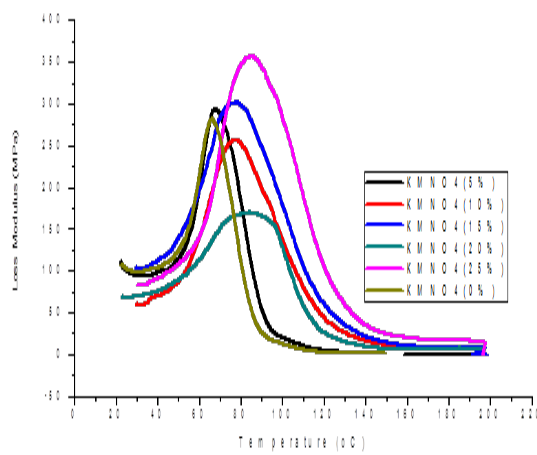

Figure 10: Variation of Loss Modulus with Temperatures for the KMNO4-treated Fibres Composites

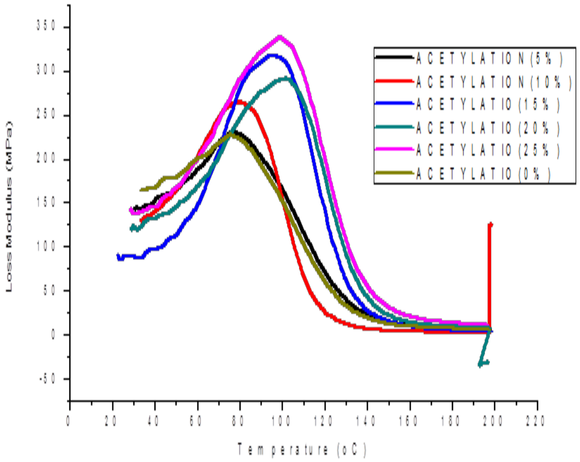

Figure 11: Variation of Loss Modulus with Temperatures for the Acetylation-treated Fibres Composites

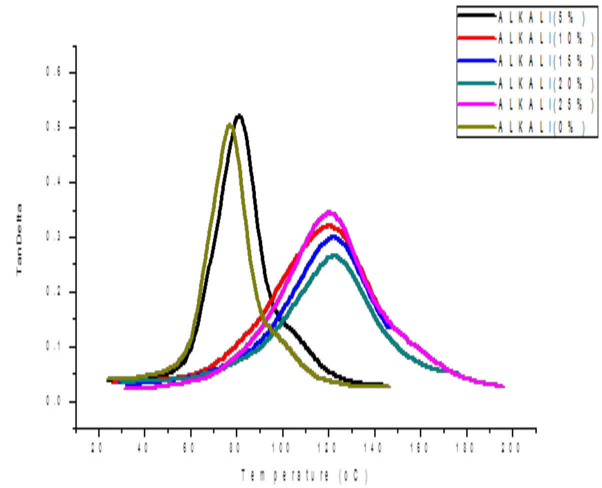

Figure 13: Variation of TanDelta with Temperatures for the Alkali-treated Fibres Composites

3.3 Damping Factor (tanδ)

The ratio of these parameters is denoted as loss factor (tan δ), which is used to determine the glass transition temperature. Figures 12-16 shows the variation in tanδ with temperature for untreated and treated composites. The higher tanδ value untreated composite as compared with the treated fibres. It was also observed that the tanδ values shifted to higher temperature with treatment and addition of fibers to polyester (Daiane Romanzini et al, 2013). It is reasonable to expect a higher tanδ temperature for the treated composite when compared to the other composites. This is due to the enhanced interfacial adhesion between untreated fabric and matrix. The Alkali treated fibers has the higher temperature. These could be attributed that the Alkali treated fibers has good load bearing capacity, high interfacial adhesion and improved stress transfer than others treatment (compared Figure 12 with Figure 13)

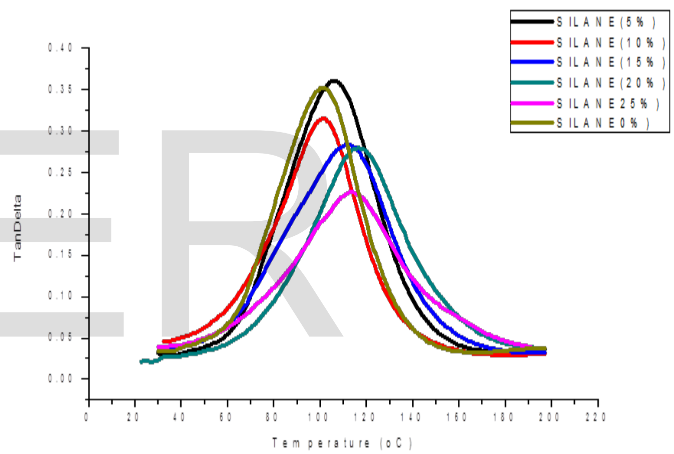

Figure 14: Variation of TanDelta with Temperatures for the Silane-treated Fibres Composites

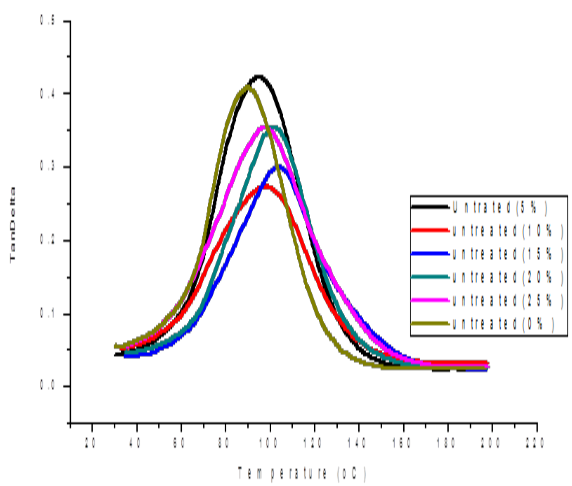

Figure 12: Variation of TanDelta with Temperatures for the Untreated Fibres Composites

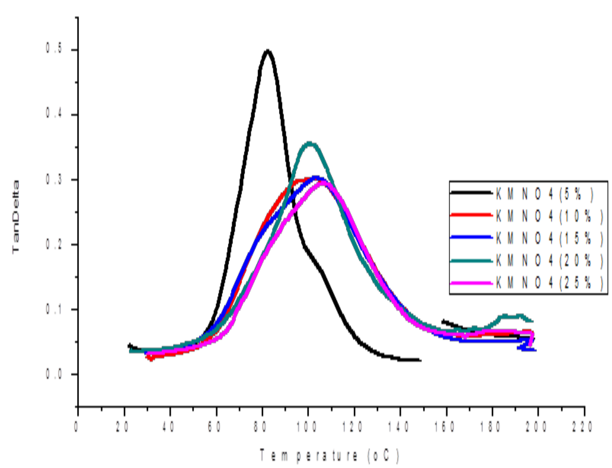

Figure 15: Variation of TanDelta with Temperatures for the KMNO4-treated Fibres Composites

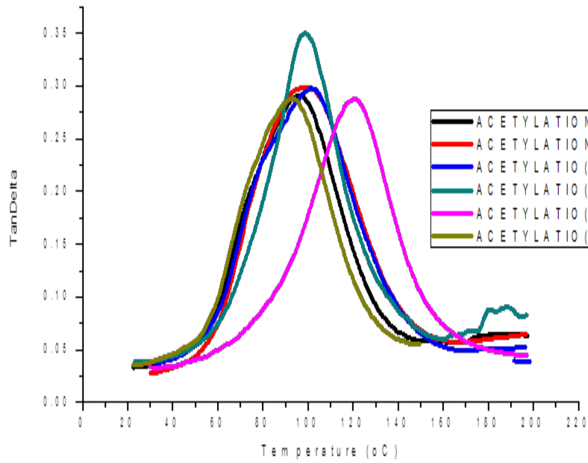

Figure 16: Variation of TanDelta with Temperatures for the Acetylation-treated Fibres Composites

Also in Figures 12-16, it can be observed that the addition of Ukam fibers decreases damping factor (tan *δ*) in the glass transition region because molecular mobility of the composites decreased and the mechanical loss to overcome interfriction between molecular chains is reduced. Hence that addition of Ukam fibers to polyester improves adhesion as composites show lower tan *δ* than polyester. This decrease is attributed to the softening of the polymer due to the increase in the chain mobility of the polymer matrix at high temperatures. This means that the segmental mobility of polymer chains increases with temperature rise and at glass transition temperature, the state changes from glassy to rubbery state.

Therefore the load bearing capacity is drastically reduced at elevated temperature. This increase is attributed to the increase in viscoelastic property of the polymer material at higher temperature. The contribution of viscous part to the dynamic response is relatively high in higher temperature region. This response is high up to a specific temperature, which is called as glass transition temperature (Tg). Beyond Tg, the tan δ value decreases, and the decay in modulus levels off, indicating that a more elastic region is reached, the so-called rubber plateau. In this rubber elastic region, the polymer chains have full mobility and properties are determined by the entangled network. This rubber elastic region is found till the minimum of tan δ().

The chemical treatment and fibers shifted the glass transition temperatures of the polyester to a higher temperature; this could be due to the immobility of polymer molecules near the surface fibers due to various molecular interactions, which increases the Tg of the composite. Upon further heating the polymer chain starts to disentangle and the dynamic modulus decays further out of the measurable range and the material become the liquid like. This is due to wetting behavior of fiber with resin as a result of which storage modulus is increased and tan δ (loss factor) decreases.

Untreated composites exhibit a higher tan δ peak value as compared to treated composites. Higher tan δ peak value for untreated composite may be attributed to more energy dissipation by internal friction at the weaker interface between untreated fiber and matrix. Among the treated composites, Alkali treated composites showed a lower tan δ peak values. Since the tan δ peak value is related to fiber matrix adhesion, lower tan δ peak value of Alkali treated composite corresponds to better adhesion and compatibility between Alkalis treated Ukam fibers and resin than that of the other treatments. Acetylation and Silane treated fibre reinforced composite also exhibits similar tan δ value as compared to untreated composite.

4 Conclusion

The dynamic mechanical analysis of ukam fibre reinforced polyester composite has been investigated. The DMA results show high storage modulus at room temperature followed by significant drops in the temperature range of 25-200 □C while the untreated composites showed the loss modulus peak at 80-100 oC, these values shifted to a higher temperature for the treatment fibres. It was also observed that the damping factor shifted to higher temperature with treatment and addition of fibers to polyester. Furthermore, the DMA study indicates that the increased modulus, together with the positive shift in tan delta peak position is attributed to the physical interaction between the polymer and ukam fibres.

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