Dynamic Mechanical Properties of Curaua Composites

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ABSTRACT: In this study, we aimed to evaluate the characteristics of curaua composites using dynamic mechanical analysis. Curaua composites with distinct fiber volume fractions (11–38%) were studied, and their storage and loss moduli were found to increase with reinforcement content. On the other hand, the activation energy of the relaxation process in the glass-transition region decreased with the curaua content; this conclusion was corroborated by the elastic chain concentration results. The Cole–Cole plots became flattened as the curaua content increased; this was indicative of a more homogeneous system, and the tan δ peak height and peak width at half-height decreased. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: composites; crosslinking; density; fibers; thermal properties

INTRODUCTION

The inclusion of fibers is known to improve the strength and stiffness of polymeric matrices. When natural fibers derived from renewable resources, rather than synthetic fibers, are employed as reinforcements, positive environmental benefits arise. The material can be considered to be more environmentally friendly, and the use of residues minimizes traditional disposal practices. Indeed, many studies have addressed the use of natural fibers to replace synthetic ones,[1,2] and low costs and acceptable specific mechanical properties[3] are commonly reported advantages.

Perhaps one of the most promising vegetable fibers currently available is curaua (*Ananas erectifolius*). The curaua plant is well known in the Amazon Basin, which is located in the North Region of Brazil. This fiber has similar costs compared to other vegetable fibers, but its strength is much higher than that of coir, sisal, and jute, and the fiber almost reaches the physical properties of the more expensive flax and even glass fibers.[4,5] Some studies in the literature have reported the mechanical, thermal, and dynamic mechanical properties of curaua composites[6] and also their potential use in the automobile sector.[4]

Because fiber-reinforced polymers are subjected to various types of dynamic stress during service, studies of their viscoelastic properties over a wide range of frequencies and temperatures are of great importance.[7] These studies can provide insight into various aspects of the structure of a material (e.g., its transition temperatures) that have a direct influence on its mechanical properties, such as the flexural and impact strengths,[8,9] and are of direct relevance to the vibration isolation or the dissipation of the vibration energy in engineering parts.

The mechanical properties of polymeric composite materials have been comprehensively studied for decades.[10–12] However, the same cannot be said of the dynamic mechanical properties, and thus, the composite properties are usually explained by consideration of the intrinsic properties of the fibers and fiber/matrix interface. In contrast, in this study, we aimed to analyze the traditional mechanical properties, considering in more detail the role of the matrix and the microstructural changes due to the presence of the fibers. The crosslinking density, concentration of elastic chains, Cole–Cole curves, and theoretical models were applied to help explain the observed phenomena.

EXPERIMENTAL

Materials

The commercial unsaturated isophthalic polyester resin Arazyn AZ 12.0 (supplied by AraAshland), with 0.5% of cobalt octonate, was used to prepare the composites. Commercial-grade benzoyl peroxide (a curing agent produced by Akzo Nobel) was also used. Curaua fiber was obtained from a native producer in the state of Para, Brazil.

Methods

The curaua fibers were chopped to the desired length (50 mm), washed in distilled water for 1 h, and then dried in an oven with air circulation (105°C, 50 min) before use. The fibers were then manually dispersed and randomly arranged with a rectangular frame of the required shape, and the resulting fiber mat was dried in an oven at 105°C for 30 min. To minimize the effect of any possible mat heterogeneity on the results, samples were extracted from distinct regions of each composite.

The polyester resin (100 mL) was mixed with 1 wt % of benzoyl peroxide and poured onto the mat previously placed in the mold. The composites were hot-compression-molded under the following conditions: 3 ton, 95°C, and 70 min. The volumetric fiber fractions studied were 11, 22, 32, and 38 vol %.

The viscoelastic properties of the composites were determined in an Anton Paar Physica MCR 101 analyzer, Germany with $50 \times 10 \times 3$ mm^3 specimens. The specimens were heated from room temperature to 180°C at a heating rate of 3°C/min, and the experiment was carried out in torsion mode at a strain amplitude of 0.08% with distinct frequencies: 1, 3, 10, or 30 Hz.
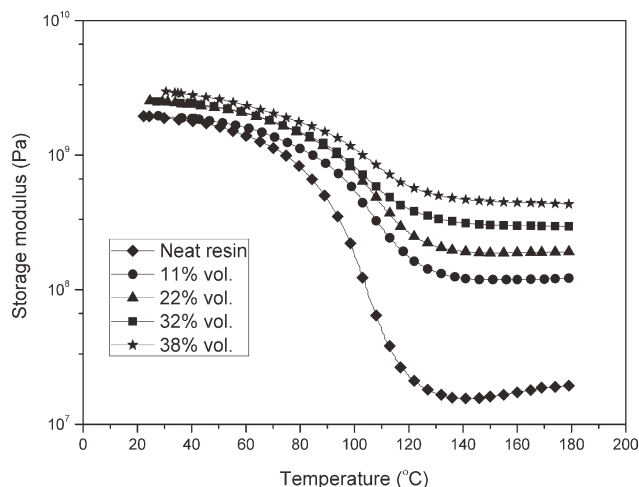

Figure 1 Variation in the storage modulus with temperature for all of the samples.

TABLE I
Storage Modulus Retention (at 1 Hz) for the Neat Resin and the Curaua Composites

Sample	$E'_{60}{}^{a}/E'_{30} \times 100$	$E'_{90}/E'_{30} \times 100$	$E'_{120}/E'_{30} \times 100$
Neat resin	72.8	25.2	1.3
11 vol %	81.6	44.1	9.0
22 vol %	82.2	46.1	10.4
32 vol %	87.5	49.4	16.3
38 vol %	79.6	79.4	20.2

^{a} Temperature where the storage modulus were collected.

RESULTS AND DISCUSSION

Storage modulus

As shown in Figure 1, the storage modulus decreased with temperature and increased with curaua content, especially above the glass-transition region. The latter may have been directly related to the inherent stiffness of curaua. Table I shows the modulus retention at three distinct temperature levels, and it can be seen that the drop in the modulus was attenuated with the incorporation of more fibers; that is, for higher fiber contents, the drop became less drastic.

Furthermore, the neat polyester, as well as the composites, showed a sharp fall in the storage modulus (E') values above the glass-transition temperature (T_g) because of the increased molecular mobility of the polymer chains.[13] This drop in modulus was considerably reduced for the composites; this indicated the reinforcing effect of the curaua fiber, especially above T_g. In addition, the modulus in the glassy region was not influenced to the same extent as it was in the elastomeric region.[14] A prominent increase in the rigidity of the matrix in the elastomeric region was expected because of the reinforcing effect imparted by the fibers,[8] which led to a comparatively higher elastic modulus.[13,15]

The effectiveness of the fiber in increasing the modulus of the material could be better demonstrated by a study of the C[16] coefficient, which is used to evaluate the decrease in modulus when a material passes through its glass transition. It is important to bear in mind that the matrix modulus in the glassy state is primarily determined by the strength of the intermolecular forces and the way the polymer chains are packed.[15]

Table II shows a tendency toward lower Effectiveness of reinforcement (C) values with the addition of more fibers, that is, the drop in modulus for the reinforced systems decreased with increasing curaua content. Also, the C values became higher as the frequency increased; this indicated that the reinforcement was less effective because of a late response of the material under the applied stress.[17] This occurred even though higher frequencies tended to

TABLE II
Effectiveness of Fiber Addition to the Curaua Composites Determined at Different Frequencies

Sample	Coefficient C (1 Hz)	Coefficient C (3 Hz)	Coefficient C (10 Hz)	Coefficient C (30 Hz)
11 vol %	0.14	0.17	0.19	0.23
22 vol %	0.12	0.11	0.16	0.19
32 vol %	0.08	0.07	0.09	0.14
38 vol %	0.06	0.07	0.08	0.10

shift the storage modulus to higher values, mainly in the elastomeric region.

Crosslinking density

The determination of the variation in storage modulus with temperature allowed us to estimate the crosslinking density through the selection of a modulus value in the rubbery region at a preset temperature (in this study, 160°C). This temperature was selected in the temperature range in which the reinforcement effect was most evident, that is, the rubbery region. According to the theory of ideal rubbers,[18] storage modulus in the elastomeric region (E'_r) increases linearly with temperature in this region (which can be better visualized for the neat resin). A value of front factor, which takes into account the factors that distinguish thermosets from ideal rubbers (e.g. the shorter length of the elastic chains) (ϕ) = 1 was used, as recommended by Pellice et al.,[19] because of the good prediction in terms of order of magnitude, which allowed the calculation of crosslinking density (v_e) with the E'_r values shown in Table III.

To express the crosslinking density in terms of unit mass (Y), it was necessary to estimate the density values at 160°C (ρ_r) with the volumetric expansion coefficients of the neat polyester in the glassy state (α_g) and in the rubbery state (α_r) state. In this study, the following values were used: $\alpha_g = 3 \times 10^{-4}$ K^{-1} and $\alpha_r = 6 \times 10^{-4}$ K^{-1}.[18]

Table III shows the v_e measured density (at 20°C), calculated density (at 160°C), and Y values for all of the studied samples. An increase in v_e and the crosslinking density per unit mass was noted with the addition of curaua; it was perhaps due to an increase in the number of physical interactions (fiber/matrix). It is important to add that the reinforcement effect promoted by the fibers was qualitatively equivalent to the increase in the crosslinking density per unit mass observed in Table III. Because physical interactions are weaker than chemical ones, it is reasonable to assume that an increase in the concentration of elastic chains occurred. However, perhaps a more plausible explanation was the presence of richer crosslinking regions, with a greater concentration of elastic chains per area/volume.

Figure 2 provides a schematic diagram of the polyester resin upon incorporation of the curaua fiber and possible rearrangements. Figure 2(a) shows a composite configuration with the presence of strong (chemical) and weak (physical) interactions. When curaua was incorporated [Fig. 2(b)], there was a consequent decrease in strong interactions (chemical), which restricted the number of crosslinkings due to the physical presence of the fiber, which was analogous to steric hindrance. Furthermore, the crosslinkings may have concentrated in a more specific area. A distinct result could have been found if there was a strong chemical interaction between the fiber and the matrix; this was not expected for the untreated vegetable fiber used.

According to Table III, an increase in the density occurred when curaua was incorporated, which was expected because the density of the curaua fiber used was 1.37 g/cm^3 and the density of the neat resin was 1.16 g/cm^3. In addition, the density was found to decrease at higher temperatures (i.e., 160°C) because of the consequent increase in internal energy, which gave mobility to the polymer chains and promoted thermal expansion.

Loss modulus

As shown in Figure 3, it was clear that the incorporation of curaua caused a broadening of the loss modulus peak. This may have been due to the inhibition of the relaxation process within the composites.[8] Also,

TABLE III
Crosslink Densities of the Curaua Composite Networks

Sample	$E_r{}^b$ (160°C, MPa)	v_e (mol/cm^3)	ρ^c (20°C, g/cm^3)	ρ_r (160°C, g/cm^3)	Y (mol/g)
Neat resin	17.6	1.62×10^{-3}	1.160^a	1.096	1.48×10^{-3}
11 vol %	118.8	1.10×10^{-2}	1.179	1.116	9.86×10^{-3}
22 vol %	187.8	1.74×10^{-2}	1.187	1.124	1.55×10^{-2}
32 vol %	296.9	2.75×10^{-2}	1.204	1.140	2.41×10^{-2}
38 vol %	440.2	4.08×10^{-2}	1.208	1.145	3.56×10^{-2}

[a] Data provided by the supplier.
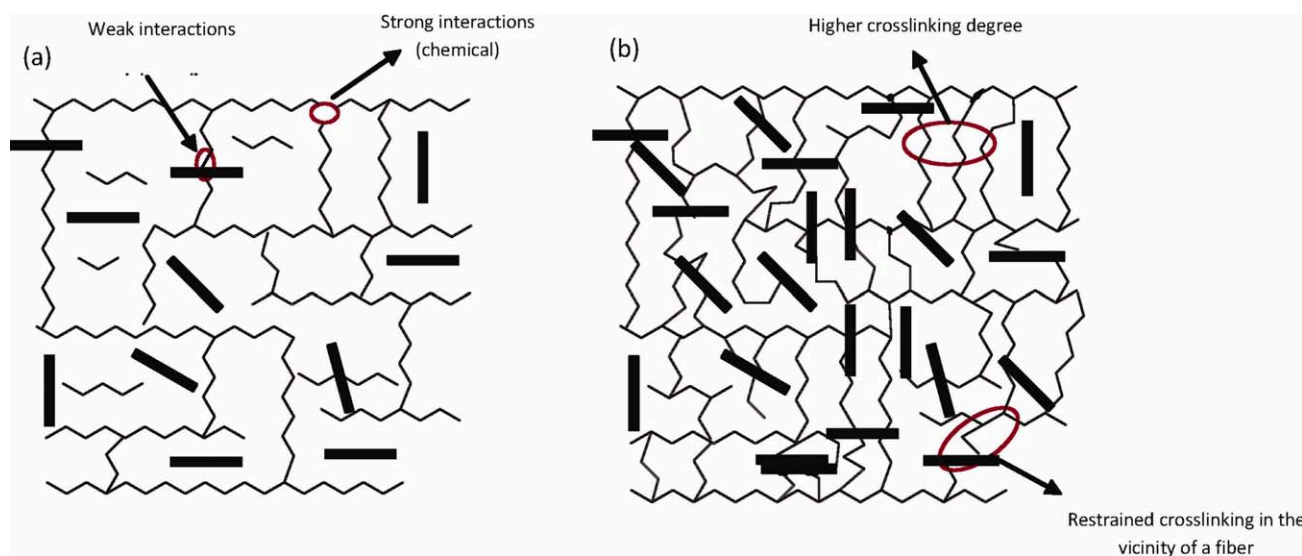[b] Storage modulus in the elastomeric region.
[c] Density.

Figure 2 Schematic representation of the polyester resin with the incorporation of curaua fiber: (a) interactions in the curaua composite and (b) rearrangements in the neat resin structure due to the presence of curaua. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

in the glass-transition region, there was an increase in the loss modulus as the curaua content increased; this may have been due to an increase in the interfacial area because there were more fibers per unit volume; this increased the internal friction and led to greater energy dissipation.[20] Figure 4 shows the loss modulus values in the 20–60°C range, and at lower temperatures, the same trend as that observed for impact strength was noted.

Cole–Cole interactions

For the single-relaxation-time model, the Cole–Cole plot (obtained by the plotting of log loss modulus (E'') vs log E') is a semicircle. However, this model is inadequate for polymers and the Cole–Cole plots are not semicircles.[14] Indeed, it can be seen that the semi-

circle for the neat resin (Fig. 5) showed pronounced asymmetry at the high-frequency end (low values of E' and E''). This behavior is typical of most amorphous polymers in their primary relaxation regions.

As the curaua content increased, this semicircle became more flattened, showing greater symmetry; this suggested perhaps greater adhesion between the components. Because the quality of the chemical or mechanical adhesion between the fiber and the matrix did not depend on the curaua content, it may have been that the overall structure responded more homogeneously as the curaua was incorporated. The heterogeneity of the neat resin may have been due to the presence of macroscopic heterogeneities, such as hot spots formed by an excess of initiator, localized areas of higher crosslinking degree, or even voids. With decreasing curaua content, there was a decrease
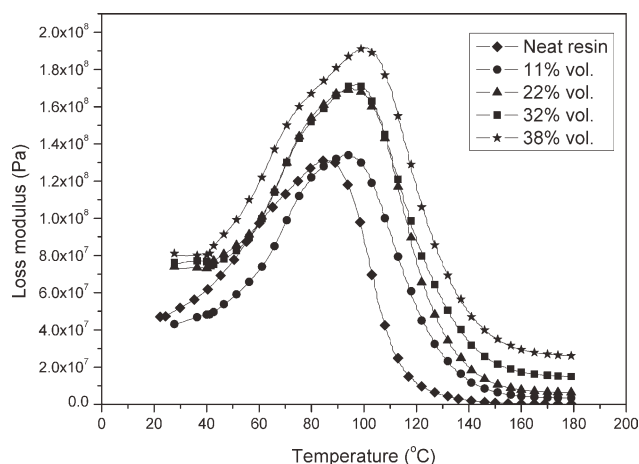

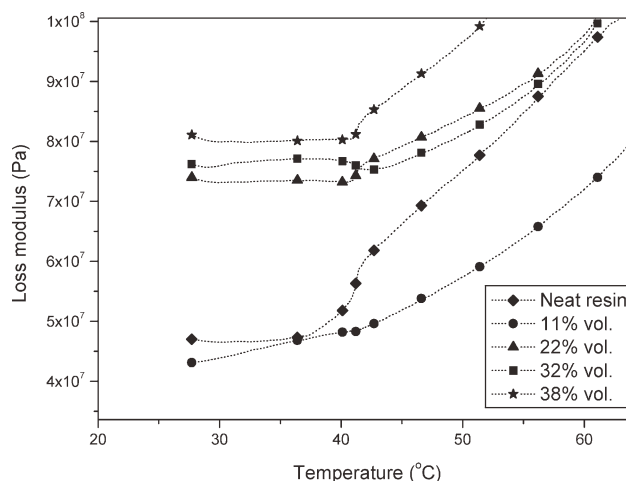**Figure 3** Variation in the loss modulus with temperature for all samples.

Figure 4 Loss modulus obtained in the range 20–60°C for the neat resin and the curaua composites.
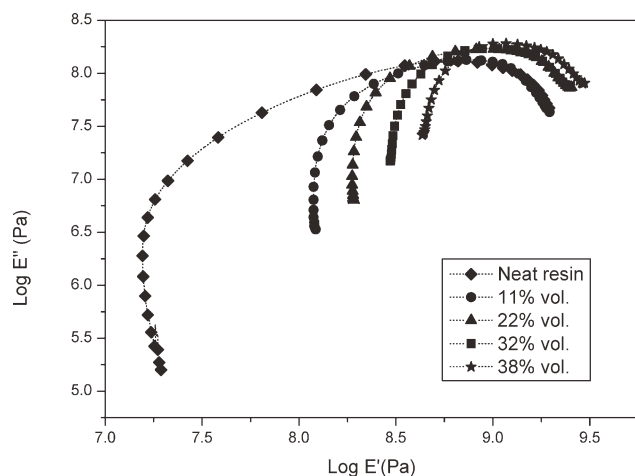
Figure 5 Cole–Cole plot for the neat resin and the curaua composites.

in the reinforcement effect and a greater concentration of regions with a distinct degree of reinforcement; that is, the fiber reinforcement effect was counterbalanced by the crosslinking reinforcement effect.

Damping factor

The ratio between the loss modulus and the storage modulus is called the *mechanical loss factor,* or tan δ. In composites, damping is influenced by the incorporation, type, and distribution of fibers, the fiber/matrix interaction, and the void content.[17,21] The tan δ results were used to extract the T_g values of the samples, which are shown in Table IV.

The literature[8] suggests that composites with higher fiber contents, that is, more restrained systems, present higher T_g values because the cooperative molecular motion would require greater energy. However, the transition temperature did not show a significant change with the curaua content in this study. This may have been a consequence of the low degree of reinforcement due to the poor adhesion between the matrix and the untreated vegetable fiber used in this study. When the reinforcement degree was low,[15] the values tended to remain constant. It may have been that T_g (in the composite materials) was influenced by the fiber/matrix interface to a greater extent than the other factors cited previously. Because the strength of the fiber/matrix interface did not change with the incorporation of curaua (even though there was a greater interface/unit volume), changes in the T_g were not evident.

The neat resin displayed lower T_g values, even though its network may have presented a higher crosslinking degree; this indicated a greater level of restraint and a lower number of structural units between crosslinkings.[22] Considering these results, we suggest that the fiber led to stronger bonding than the crosslinking.

The Arrhenius[23] equation enables the calculation of the activation energy based on the peak of maximum energy dissipation when dynamic mechanical analysis is carried out at different frequencies. Table IV shows that the activation energy decreased when curaua was added. In general, a higher degree of reinforcement led to a higher activation energy because of the restraints imposed on the resin. However, a higher reinforcement content did not necessarily indicate a lower mobility of the matrix if the crosslinking degree was affected. In other words, in a system where the crosslinking degree was high (e.g., neat resin), the crosslinking density decreased with higher curaua content because the fibers were between the crosslinkings. Therefore, it was necessary to determine how the fibers and the crosslinkings separately contributed to the material reinforcement.

To illustrate this phenomenon, it was of interest to investigate the effect on the neat resin when, for instance, the styrene content was increased. In this system, the modulus in the elastomeric region increased because the average crosslinking density increased. As a result, the number of polyester structural units between crosslinkings decreased because there were more styrene segments between the polymeric chains. In this case, the higher storage modulus of the resin observed for higher styrene content would not imply that the system was more restrained from a kinetic mobility point of view. The number of cooperative rearrangement regions would increase because the polyester resin was more restrained, but at the same time, the energy necessary to move a single monomeric unity would be lower.[22] In an analogous way, a higher curaua content increased the storage modulus due to the restraints imposed on the system by the fibers, although at the same time, it may have provided more mobility to specific regions containing fiber and led to lower activation energy values.

Peak height and peak width at half-height

The height and width of the tan δ peak are indicative of the structural characteristics and the properties of a particular composite material. In this study,

TABLE IV
T_g Values (at 1 Hz) and Activation Energy of the Neat Resin and the Curaua Composites

Sample	T_g	Activation energy in α relaxation (kJ/mol)
Neat resin	110.9	331.4 ($R^2 = 0.919^a$)
11 vol %	113.2	310.4 ($R^2 = 0.997^a$)
22 vol %	113.4	307.4 ($R^2 = 0.993^a$)
32 vol %	110.2	293.9 ($R^2 = 0.999^a$)
38 vol %	112.1	278.9 ($R^2 = 0.996^a$)

[a] Coefficient of determination obtained.

TABLE V
Peak Height, Peak Width at Half-Height, and Area under the Peak for the Neat Resin and the Curaua Composites (at 1 Hz)

Sample	Peak height	Peak width at half-height	Area under the peak
Neat resin	0.64	12.4	24.8
11 vol %	0.30	6.6	13.2
22 vol %	0.28	6.0	12.1
32 vol %	0.21	4.9	9.8
38 vol %	0.17	4.0	8.1

TABLE VI
Theoretical Prediction of the Storage Modulus (GPa) for the Curaua Composites

Sample	Experimental	Eq. (1)	Eq. (2)	Eq. (3)
11 vol %	1.88	2.01	1.96	2.56
22 vol %	2.41	2.26	2.16	3.95
32 vol %	2.42	2.48	2.34	5.74
38 vol %	2.83	2.61	2.44	7.06

Experimental storage modulus of the neat resin = 1.77 GPa.

a frequency of 1 Hz was chosen because this frequency was most commonly used in dynamic mechanical analysis. The peak height and peak width at half-height results are summarized in Table V, and a linear decrease (Fig. 6) was observed for higher curaua contents.

It has been reported that the peak height becomes larger for composites containing fewer fibers and when adhesion is poor.[24] The matrix content varies inversely with the fiber content and, therefore, more energy will be transferred to the fibers through the interfacial area when more fibers are available.[9] The neat matrix dissipates more energy than the composites because the fibers carry a greater amount of the load; this allows only a small part of it to strain the interface.[7] Thus, composites show considerably less damping in the transition region compared to neat resins. In addition, a stronger interface allows less dissipation because it restricts the movement of the polymer molecules.[25–27]

The width of the tan δ peak (shown in Table V) was found to become smaller upon the addition of fiber; this was indicative of a greater homogeneity and was consistent with the findings of the Cole–Cole plots. The area under the tan δ peak became smaller with increasing curaua loading (Table V); this was expected because both the peak height and peak width at half-height decreased for the composites.
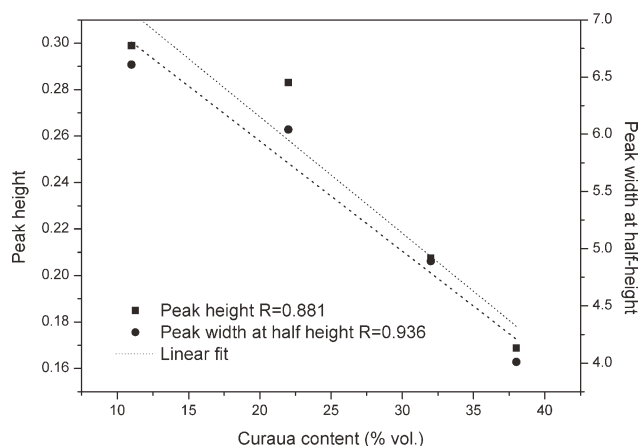
Theoretical prediction of the viscoelastic properties

Storage modulus

For practical reasons, it is very important to be able to theoretically predict how materials will perform under certain circumstances, and different models have been proposed to describe the reinforcement effect upon inclusion of the reinforcement,[7] such as those shown in eqs. (1)–(3):

$$E_c = E_m(1 + 1.25V_f) \tag{1}$$

$$E_c = E_m(1 + V_f) \tag{2}$$

$$E_c = E_m(1 + 2.5V_f + 14.1V_f^2) \tag{3}$$

Where E_c is Composite modulus, E_m is Matrix modulus and V_f is volume fraction of reinforcement.

When the theoretical predictions were compared with the experimental values of this study (Table VI), it was clear that eqs. (1) and (2) showed the best agreement with the experimental values for 22 vol % reinforcement. For lower fiber contents, the reinforcement appeared to be less effective than expected, and with Guth's equation, the values were found to be much higher than those obtained experimentally.

Tan δ height

The level of damping can be predicted with the simple rule of mixtures[7] [eq. (4)]. However, when rigid inclusions are studied, the V_f tan δ_f term can be neglected (because there is no significant damping for solid elastic materials), and a simplified equation [eq. (5)] can be used. In addition, one can modify eq. (5) by taking into account the composite and matrix moduli; this leads to eq. (6). This is considered a more realistic approach because the reinforcement also affects this property:

$$\tan\delta_c = V_m \tan\delta_m + V_f \tan\delta_f \tag{4}$$

$$\tan\delta_c = V_m \tan\delta_m \tag{5}$$

$$\tan\delta_c = V_m\left(\frac{E'_m}{E'_c}\right)\tan\delta_m \tag{6}$$

Figure 6 Peak height and peak width at half-height of the curaua composites.

TABLE VII
Comparison between the Experimental and Theoretical Values of the Tan δ Peak Heights for the Curaua Composites at Different Temperatures

Sample	Experimental	Eq. (5)	Eq. (6) (at T_g)
11 vol %	0.298	0.57	0.54
22 vol %	0.283	0.50	0.37
32 vol %	0.207	0.44	0.32
38 vol %	0.169	0.40	0.25

Experimental tan δ height for the neat resin = 0.642.

Where δ_c is tan δ of the composite, δ_m is tan δ of the matrix, V_m is volume fraction of the matrix, E'_m is storage modulus of the matrix and E'_c is storage modulus of the composite.

The theoretical tan δ height values obtained with eqs. (5) and (6) are presented in Table VII and could be compared with the experimental results. Equation (5) gives theoretical values, which were higher than those of the measured data. This may have occurred because only two variables were considered. When more variables were considered in the formula [eq. (6)], the theoretical values approximated the experimental ones. This could be attributed to the more realistic approach, with more variables, capturing the more complex particularities of the composite material.

CONCLUSIONS

In this study, composites with distinct curaua fiber volume fractions (11, 22, 32, and 38 vol %) were produced, and their dynamic mechanical properties were evaluated. The storage modulus did not show significant variation in the glassy region for any sample studied. However, as the temperature increased, especially above the glass-transition region, a reinforcement effect was noted. In addition, the loss modulus increased with fiber loading; this showed greater energy dissipation. T_g obtained from the tan δ peak did not show any particular trend for the samples studied, but the activation energy decreased with the amount of curaua incorporated.

Cole–Cole plots were obtained, and their semicircle became more flattened with the addition of curaua fiber. This was considered indicative of a more homogeneous system and corroborated the results for the tan δ peak width at half-height. The parameters obtained with the theory of ideal rubbers indicated an increase in the concentration of elastic chains; this suggested more regions under the influence of physical interactions and restrained cross-linking in the vicinity of the fibers. Finally, some of the theoretical models used to predict the storage modulus and tan δ height showed good agreement with the experimental data.

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