Effect of Temperature on Dynamic Mechanical Properties of Biobased LDPE-Eucalyptus Fiber Composites

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Abstract: The design of manufacturing and construction parts and tools based on the combination of plant resources and thermoplastic polymers, with the aim of obtaining a durable, long-lasting product, has only recently taken off. For some, composite materials are needed to replace conventional materials made from carbon, glass, or aramid fibers, because of their manufacturing, recycling, and non-renewable costs. However, wood-based fibers have emerged as a competitor due to their economic, renewable, and recyclable advantages. The aim of this study was to investigate the properties of eucalyptus fiber composites, treated or not with sodium hydroxide (NaOH) solution in a low-density polyethylene (LDPE) matrix, using dynamic mechanical analysis (DMA) and mass loss due to temperature variation from 0°C to 700°C. The frequencies studied were 1 Hz and 20 Hz. Frequencies of 1 Hz and 20 Hz were used over a temperature range from 0°C to 120°C. Storage modulus E' and loss modulus E" as well as damping revealed the influence of eucalyptus fiber rate and treatment on these DMA components. The higher the fiber content, the better the E', and the same applies to processing. The transition temperature of the composites is analyzed and compared with that of the literature. This enables us to determine the range of applications for composite materials.

Keywords: composite, eucalyptus, storage modulus, loss modulus, loss weight

1. Introduction

Research into biobased composite materials is growing steadily, driven by strong demand in sectors such as automotive, aeronautics and construction. The interest in biobased materials is generally linked to their low production costs, light weight, availability, and low environmental impact, unlike conventional composites such as glass, aramid, carbon, and certain metals. In the literature, more than 95% of the work focuses on the formulation and measurement of the mechanical properties of biobased composites with LDPE, HDPE, PP and PLA matrices, reinforced with long and short fibers from birch, aspen, sisal, cotton, hemp, bamboo, and sawdust [1][2][3][4][5]. Theories and mechanisms have been developed to explain the phenomenon of adhesion between composite phases [6]. It turns out that fibrous reinforcements are subject to various forms of stress within polymer matrix materials, and the study of the viscoelasticity of these materials seems obvious [7] and a great

deal of work has been done on this viscoelastic property of fiber and particle composites [8][4][5]. For some, material performance can be determined from dynamic mechanical analysis, considering the unidirectional direction of fibers at different volume fractions in the composite [1], that the dynamic mechanical properties of the materials depend on the unidirectional orientation of the fibers [9][10]. In addition, Carwalho and Bretas [11] investigated the correlation between viscoelastic properties and interphase morphology of thermoplastic and carbon fiber composites. Thus, experimental determination of material parameters is highly desirable in order to design structural components for material model validation [12].

Knowing that the mechanical behavior of polymer-based composites is predominantly dominated by velocity- and time-dependent thermomechanical properties, Dynamic Mechanical Analysis (DMA) [13] is the most suitable technique for investigating the behavior of composites [12]. Thus, in this study, we used DMA to characterize the thermomechanical behavior of our composites of low-density polyethylene with eucalyptus fibers. Emphasis is also placed on related parameters such as dynamic viscosity as a function of temperature and time, and stiffness. For some authors [14] [15], studies have focused on the damage of carbon fiber-reinforced epoxy composites. Thomason's [16] work on glass-fiber-reinforced epoxy composites has elucidated thermomechanical and viscoelastic properties, considering stiffness and damping properties.

2. Materials and Methods
Materials
Three main components were used to formulate the composites. Low-density polyethylene (LDPE), the coupling agent maleic anhydride grafted polyethylene (MAPE), NaOH-treated eucalyptus fibers (ET) and untreated eucalyptus fibers (ENT). Table 1 illustrates the characteristics of the components.

Table 1: Characteristics of the components

Characteristics	Components			
			Eucalyptus Fiber	
	LDPE	MAPE	ET	ENT
Density (g/m^3)	0,916	-	0,74 ± 0,011	0,68 ± 0,010
Melting temperature (°C)	113	111	-	
Residual moisture (%)		-	0,32 ± 0,0013	0,87 ± 0,0022

LDPE = Pure low-density polyethylene
LDPE15ENT = LDPE with 15% untreated fibers
LDPE15ET = LDPE with 15% treated fibers
LDPE25ENT = LDPE with 25% untreated fibers
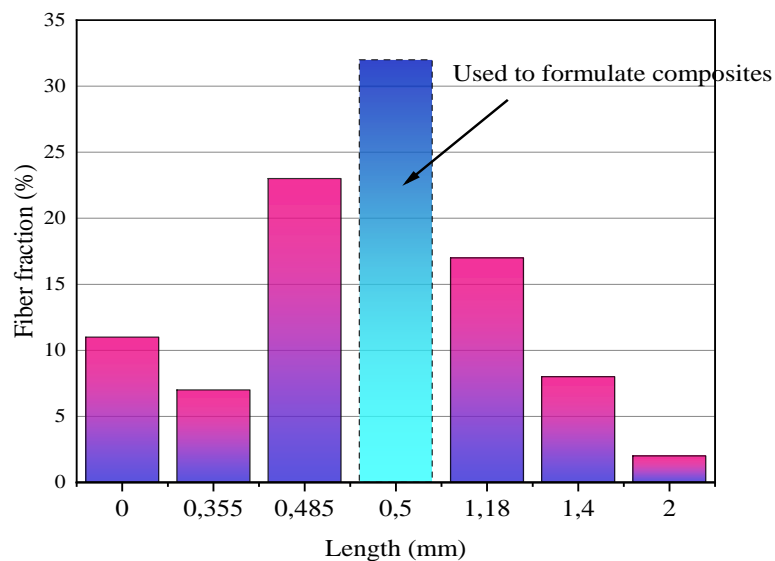LDPE25ET = LDPE with 25% treated fibers

Figure 1: Fraction length distribution by particle size

Methods

Specimen formulation

The materials used in this study are formulated on I2E3's ZERES ZE900 injection molding machine. The flexural specimen is used for the DMA test. Injection parameters are set for an injection pressure of 14820 PSI, an injection speed of 2 inch/s, an injection temperature of 380°C and a molding time of 15 sec. The specimens thus formulated are reinforced with treated or untreated fibers at concentrations of 0%, 15% and 25% of the total composite mass. MAPE contains 1.5% maleic anhydride and represents 3% of the total composite mass. According to the literature, an increase in MAPE coupling agent leads to chemical bond entanglement in the composite. Figure 2 shows an image of the DMA test samples.

Figure 2: Sample images for thermomechanical testing

Thermal stability test used

Considering that the material is homogeneous in all directions, and that all parasitic effects such as moisture pick-up, pressure and surrounding temperature are negligible during the test. The samples were cut into squares with a mass of 20 ± 1mg and stored in a chamber at room temperature before use the following day. However, seven samples of the same size were taken for each test and subjected to fixed temperatures of 100°C, 200°C, 300°C, 400°C, 500°C, 600°C and 700°C in a Fisher Scientific ash oven. The samples are removed and placed in a desiccator until cooled, after which the mass is evaluated on the 1 mg precision analytical balance.

Figure 3: Samples cut to monitor the evolution of composite residual mass as a function of temperature.

Dynamic mechanical analysis (DMA) testing

For DMA testing, the DMA Q800 V21.3 Build 96 instrument was used, with settings for the DMA multi-frequency-stress module. Samples were cut to a rectangular shape 35×12.58×3.18 mm with analysis frequencies of 1 Hz and 20Hz, and temperatures over the range 0-140°C. The storage modulus E' and loss modulus E",

which are the real and imaginary components of the complex modulus E* shown in equation 1, were analyzed and discussed.

$$E^* = E' + iE''$$
(Eq. 1)

The damping factor tanδ a is presented and discussed, as well as the proportionality factors of the average values of the instantaneous storage modules with respect to the base storage module according to equation 2.

$$P(T_i) \frac{E'^{(T_i)}}{E'^{(T_{base})}}$$
(Eq. 2)

3. Results and Discussion

Monitoring mass variation of materials

Residual mass monitoring of the composite and fiber samples in Figure 4 revealed specific temperature-sensitive zones. The residual mass variation curves can be divided into four parts, each providing information on the degradation of both the fibers and the LDPE polymer. Between 0 and 100°C, there is a loss of around 5%, linked to the loss of moisture and extractables, for all samples except LDPE. The work of Groenli and al [17] confirms this, locating this loss below 200°C. Furthermore, the first constituent the main biopolymer to degrade is hemicellulose, in the range 180°C to 330°C [18][19], with a residual part degrading up to 700°C. Figure 4 shows the same evolution. And according to Chen et al [19], who have worked on the thermal degradation of eucalyptus, we note a similarity in the degradation results for our two types of eucalyptus fiber. Virtually all cellulose degradation is observed above 300°C [20][21][22]. The work of Yang et al. [23] attributed this cellulose degradation to a specific range between 315°C and 400°C, with a maximum peak in mass loss rate at 355°C. Lignin degrades from 250°C [24]and this degradation is spread over a wide temperature range, unlike holocellulose. It is practically consumed by 500°C [25]. However, the chemical structure of wood biopolymers and the nature of the wood's plant species influence the thermodegradability of these constituents. Hemicellulose, which is rich in polysaccharides, has an amorphous character, which forces it to detach from the main chain at low temperatures. In the case of lignin, the aromatic groups make up the majority of the constituents, and the reactivity of their chemical bonds is spread over a wide range, which explains their extensive degradation at temperatures of up to 500°C.
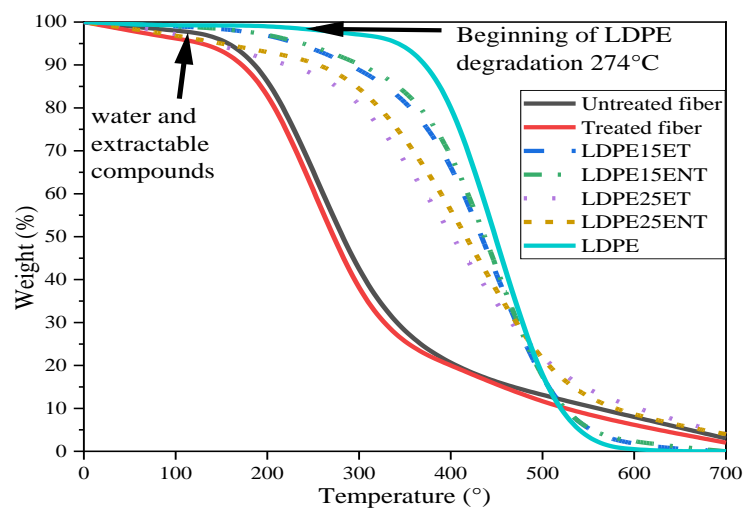

Figure 4: Residual mass evolution of fibers, LDPE and composites

However, sample degradation is more severe between 200°C and 500°C, but with sample-dependent mass loss. No significant variation in mass loss between treated and untreated fibers. The same observation was made between LDPE15ENT and LDPE15ET composites, then between LDPE25ENT and LDPE25ET. On the other hand, there was a significant variation between treated and untreated fibers and composites. The evolution of the mass loss of the composites is like that of virgin LDPE, i.e. between the fibers and the LDPE. There is therefore a combination of properties of the constituents (fibers and LDPE) which improves the thermal properties when it comes to the mass loss of the composites.

In addition, the fiber content has an influence on the mass loss of the composites. For example, in the 300°C to 400°C range, LDPE25ET composites lost 36% of their mass, while LDPE15ET composites lost 15%. This difference of around 42% for LDPE25ET is significant for mass loss. This means that the higher the mass fraction of fibers in the matrix, the higher the mass loss of the resulting composite. This is linked to the biopolymers, which are not in equal proportion to the fiber content.

As for the influence of fiber treatment, the variation in mass loss between treated and untreated fiber composites remains low. Still between 300°C and 400°C, for example, PEBD25ENT has a mass loss of 31%, while PEBD25ET has 36%. This represents 0.86% of the mass loss of PEBD25ET, a rate of difference that remains insignificant, allowing us to conclude that fiber treatment has virtually no influence on the mass loss of composites.

Storage and loss modules, amortization

Figures 5 and 6 show the variation curves for the storage and loss moduli, as well as the damping factor at frequencies of 1Hz and 20 Hz respectively. Damping has a frequency-dependent character due to the viscous behavior of the LDPE thermoplastic. This is confirmed by the work of Ropers and al [26]. However, for both frequencies and irrespective of the rate of fiber treatment or not, the storage modulus decreases as a function of temperature. This means that the material loses its strength as the temperature becomes exponential. As we explained earlier in the section on temperature-dependent mass loss in composites, the mobility of molecular chains is also strongly temperature-dependent in the case of thermoplastic matrix composites. Many studies have demonstrated this behavior of composites [27][28] [29].
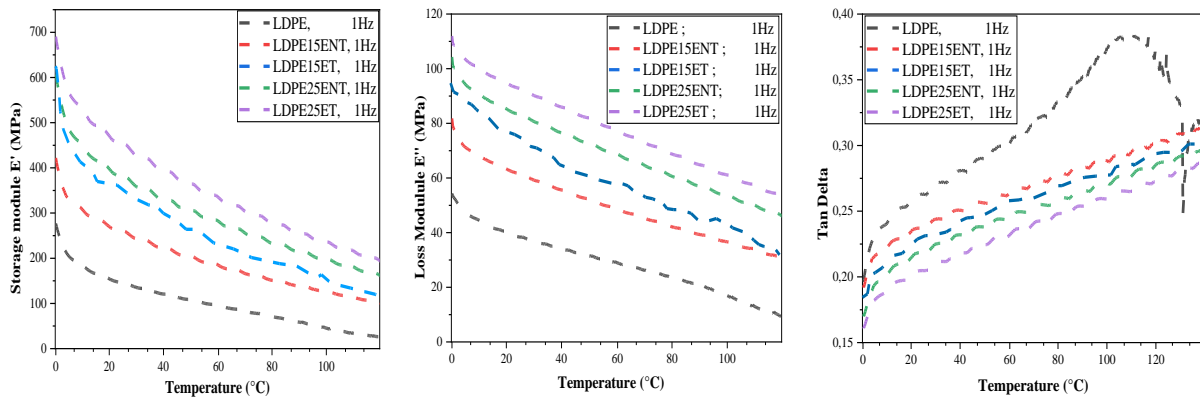

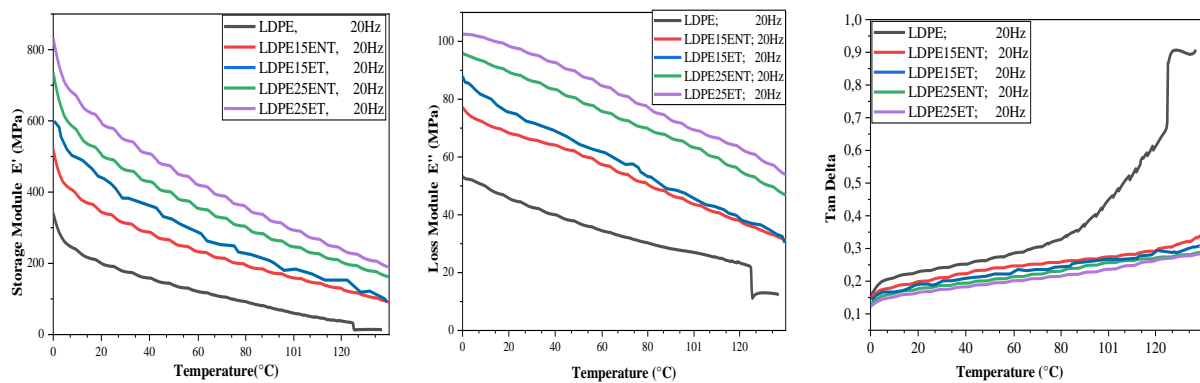*Figure 5: Storage modulus E', loss modulus E'' and damping factor tanδ of DMA at 1 Hz*

Figure 6: Storage modulus E', loss modulus E'' and damping factor tanδ of DMA at 20 Hz

Considering both frequencies, we note an increase in the storage modulus E' of composites compared with virgin LDPE, and this becomes significant as the fiber content increases. A difference in E' modulus of between 50MPa and 100MPa is noted for all samples when comparing the two frequencies 1Hz and 20Hz. This means

that frequency has an effect on the composites' storage modulus. More specifically, according to the theory of viscoelastic materials described in the literature, at low frequencies these materials tend to behave like a viscous fluid or a soft solid, thus decreasing their storage modulus E'. At high frequencies, on the other hand, the stress is more rapid and the molecular chains have not had time to rearrange themselves, so they behave like rigid materials.

The loss modulus characterizes the viscous character of materials. For both frequencies, and between 0 and 120°C, the loss modulus decreases with increasing temperature, but increases with increasing fiber content compared to virgin LDPE. This temperature-dependent reduction in modulus is confirmed by the work of Ndlovu and al. [28], Mijiyawa and al [29], Lai and al. [30], [31].

In addition, the damping rate at all frequencies from 1Hz to 20Hz increases with increasing temperature. However, this rate decreases as the fiber fraction increases. This behavior can be explained by the non-mobility of LDPE molecules. These results are in line with those found and explained by authors Mijiyawa and al. [29], who used HDPE with birch and aspen fibers; and Ndlovu and al. [28], who used LDPE with wood flour. The transition from the rigid to the ductile and rubbery state of materials is theoretically marked by the glass transition temperature of the amorphous phase of semi-crystalline materials. In fact, a small, faintly detectable peak was observed between 0°C and 5°C, which would probably be the end of a phenomenon that would have begun below 0°C if the analysis range had been extended to negative temperatures. Referring to the work of Ndlovu et al, we note that this phenomenon appears at -5°C, and the authors attribute this phenome to β-relaxation, i.e., the glass transition of LDPE. It was also found that, in both frequency cases (1Hz and 20Hz), there was a clearly expressed peak around 120°C, while at the same time, the composites showed no peak at this temperature. In other words, the reinforcements used in this study contributed to shifting the transition phenomenon towards higher temperature values, as the energy required to initiate chain mobility is high. This observation has also been made in the literature, precisely by the authors [32][33]. A low rate of energy dissipation by the materials is due to the high immobility of the molecular edifices in the amorphous phase of the composites. Since composites are reinforced with treated or untreated fibers, which contribute to the consolidation of macromolecular chains, the result is an entanglement of bonds that prevents free movement of the molecules. In effect, this increases the glass transition.

Conservation energy proportionality factor

Figure 7 shows the evolution of the temperature dependence of the storage energy according to the frequencies studied. There is a slight difference between the values at 1Hz and 20Hz. Although this difference is not significant, it does not rule out the frequency dependence of storage energy on deformation. Considering the range 0°C to 40°C, the proportionality factor for PEBD15ENT, PEBD15ET and PEBD composites falls more rapidly at 1Hz than at 20Hz. Between 40°C and 80°C, the same behavior is observed, and, from 80°C to 120°C the same behavior appears, but this time, at 1Hz, the values are closer to zero than in the case of 20 Hz. An increase in temperature above 120°C would favour a drop to zero, which would first be noticed in the 1Hz case. This means that at higher temperatures, the composites and LDPE matrix will become more viscous, will not store energy and consequently lose their rigid character, becoming more ductile or rubbery. This information provided by the storage energy proportionality factor is important for predicting the viscoelastic behavior of materials, as well as explaining their stiffness, ductility and durability. The work of Ropers and al. [26] confirms this.
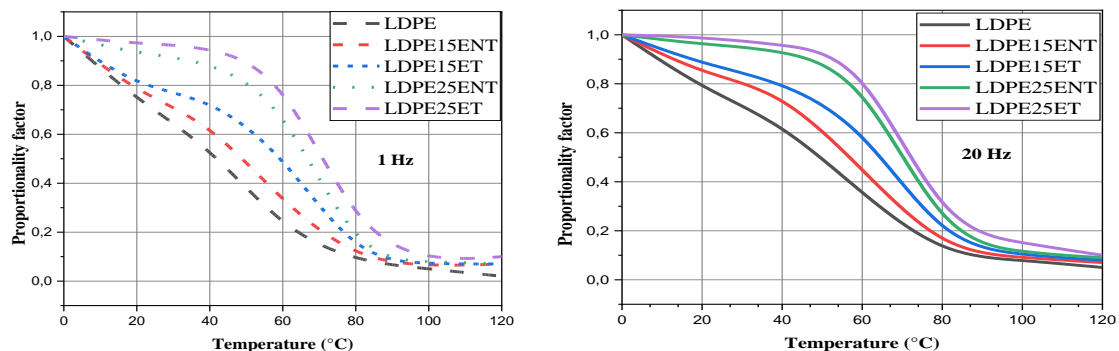

Figure 7: Storage module proportionality factor at 1 Hz and 20 Hz

Moreover, this proportionality factor confirms that the fiber content has a considerable influence on the conservation energy of the materials studied. For both frequencies, composites filled with 25% fiber have a greater proportionality factor plateau than composites filled with 15% fiber. This proves that the fibers have played their reinforcing role, consolidating and strengthening the amorphous zones that are the seats of deformation in semi-crystalline materials.

A variation is also noted in relation to fiber treatment. Composites with treated fibers were found to have a better proportionality factor than those whose fibers were not treated with NaOH. The aim of the treatment was to delignify the fiber to obtain a more crystalline cellulosic fiber and also to reduce the roughness of the fibers, giving them the power to adhere better to the matrix via the MAPE coupling agent. Referring to figure 4, the presence of lignin in the fiber shows that most of the lignin has not been eliminated, or that it is rather the roughness that has been more favorable during treatment. This is an advantage for good bonding between the composite's constituent elements, and consequently shifts the ductile factor towards the rigid one. In fact, fiber content outweighs processing when it comes to improving the dynamic mechanical properties of our composite materials.

4. Conclusion

Thermoplastic matrix composites reinforced with plant fibers are of great importance. However, knowledge of the thermal and mechanical properties of the components is essential to define the field of application of the composite product. Among these searches for information for the choice of materials are the storage energies E' of loss E', depreciation, the proportionality factor Pi which is the ratio of the storage energies E' (Ti) to temperature Ti by the storage energies E'(Tbase) a base temperature studied. This factor allows to predict forms of stiffness or ductility while ensuring a durability of the materials thus studied. The results presented in this work and those we have not presented because they are not subject of this manuscript will allow to manufacture a useful eco-material that is ecological pavé which can be used to replace conventional made based on cement, sand and gravel, whose associated costs are exorbitant and whose recyclability is expensive, which is not the case for eco-pavers.

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