

Poly(Lactic Acid) (PLA)-reinforced Kenaf Bast Fiber Composites: The Effect of Triacetin

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ABSTRACT: In this article, the mechanical, morphological, and dynamic-mechanical properties of the blends of PLA and kenaf bast short fiber were investigated. The composites, with different fiber loading and triacetin content, were prepared by melt blending techniques using a Brabender internal mixer at 60 rpm and 170°C for 10 min and their properties were examined. Pure PLA was used as a reference for the biocomposite samples. Triacetin was used as a plasticizer for PLA and PLA/kenaf composites in order to study the improvement in tensile properties. The tensile strength and stiffness of unplasticized biocomposite materials decreased with the addition of kenaf bast fibers but improved with the addition of triacetin. The optimum fiber loading was 30 wt% kenaf fibers in the PLA matrix with the addition of 5% triacetin. The dynamic mechanical analyses showed that triacetin improved the thermal stability of the biocomposites. The triacetin increased the storage modulus and gave a lower softening temperature for plasticized biocomposites. The micrographs of the tensile specimens and their fractured surfaces, which were examined by scanning electron microscopy, demonstrated that better adhesion between the fibers and the matrix was achieved with the addition of the plasticizer.

KEY WORDS: PLA, kenaf, triacetin, internal mixer, biocomposites, mechanical properties.

INTRODUCTION

THE DEPLETION OF petroleum resources and the increase of environmental awareness has lead to the development of composites based on natural resources. These new and advanced materials of bio-based polymeric product are derived from nature and agricultural stock. They will produce sustainable, eco-friendly, eco-efficient, and biodegradable products that can penetrate the global market, which is currently dominated by petroleum resources and their products. These biodegradable materials have wide potential in

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Figures 1–4 and 8–10 appear in color online: <http://jrp.sagepub.com>

furniture, automotives, building, household goods, and packaging industries. The solution is to combine petroleum materials with bioresource materials in order to produce useful products [1]. There are also other alternatives to produce biocomposite materials, by reinforcing the synthetic polymer with natural fiber sources as stated by Mohanty et al. [2]. This biocomposite is comparable to glass fiber reinforced composites. A variety of natural fibers, such as: kenaf, jute, sisal, wood residues, flax, ramie, and pineapple leaf have been studied using thermoplastics, to make partially biodegradable composites [2]. These composites have a wide potential in furniture, automotives, household goods, and the packaging industries [3].

Increased environmental awareness made more researchers, who were designing materials, integrate material design concepts with ultimate disposability. Efforts to replace thermoplastics with biodegradable polymers such as: poly(lactic acid) (PLA), polyhydroxybutyrate (PHB), cellulose esters, polyhydroxyalkanoates, and soy-resin produced completely biodegradable composites that are eco-friendly and coefficients.

Among the biodegradable polymers, PLA is the most promising biodegradable material because it is readily susceptible to biological attack [4]. The degradation occurs by hydrolysis due to lactic acid, which is metabolized by microorganisms, to water and carbon monoxide. By composting PLA together with other biomass waste, the biodegradation process can happen within two weeks and the materials will be fully decomposed, having disappeared within three to four weeks [5]. Since PLA is environment friendly with compostability, PLA became one of the polymers in highest demand to be used in applications that are difficult to be recycled. PLA is commonly used to replace commodity synthetic polymers that can cause deterioration of our environment, due to solid waste pollution. However, polylactic polymers are stiff and brittle materials and it is therefore necessary to use plasticizer to improve the elongation and tensile properties. PLA is highly transparent with a relatively low crystallization rate and is chosen for biaxially oriented film, thermoforms containers, and stretch blown bottles [4,5]. Many studies on the mechanical properties of natural fibers reinforced with PLA have been developed by other researchers with different types of fiber, such as: kenaf [3], flax [4], jute [6], abaca [7], hemp, [8], and cordena rayon fiber [9] as a reinforcement in order to replace synthetic fiber in polymer composites. Nishino et al. [3], performed a study on the retted long fiber as reinforcement using PLLA polymer as a matrix. They found that fiber loading, up to 70%, provided impressive mechanical properties, and it was comparable to those traditional composites. Their studies focused more on molding conditions, mechanical properties of the kenaf fiber biocomposite materials, and interfacial bonding of the biocomposites prepared. In this study, the short kenaf bast fiber, produced by the decorticator process, was selected as filler to reinforce the PLA polymer matrix. The effect of fiber loading and the amount of triacetin, as a plasticizer, on the mechanical, morphological, and thermal properties of PLA/kenaf biocomposites were investigated.

EXPERIMENTAL

Preparation of Biocomposites

Kenaf bast (V36) was supplied by Lembaga Tembakau Malaysia (LTN), Kelantan. The kenaf were harvested and underwent a decorticator process in order to remove the bast from the stem. Then the bast fiber was dried and crushed using a fiber cutter and crusher

to reduce the size to 75–150 μm . The fibers were kept in an oven at 60°C for 24 h to minimize the moisture content. PLA (NatureWorksTM PLA 4060D) in pellet form was obtained from Natureworks[®] LLC, Minnetonka, Minnesota. The melt flow index of this PLA is 15 g/10 min (190°C/2.16 Kg). Its T_g is 52°C and its melting temperature is 135°C. The density of this PLA is 1.24 g/cm³. Triacetin, or glycerol triacetate, an oily, clear, slight odorous liquid with a boiling point at 258°C, refractive index of 1.430 at 25°C, specific gravity of 1.1596 at 20°C, was used as a plasticizer.

Prior to blending, polymer and fibers were kept in an oven at 60°C for 24 h. The PLA were premixed with 1%, 3%, 5%, 8%, 10%, 12%, and 15% triacetin and added together with several percentages of fiber content, such as 10, 20, 30, 40, and 50 wt% as tabulated in Table 1. These composites were prepared using a Brabender internal mixer at 170°C, for 10 min at 60 rpm. Neat PLA mixed with triacetin was also processed in the same way in order to obtain a reference material. The blended materials were then hot press molded to produce sheets of about 1 mm thickness. The composites were preheated for 15 min, with hot press at 150°C for 5 min and then cooled for 8 min.

Tensile Test

The tensile testing was performed according to ASTM D638, using an Instron machine (model 4201). At least seven specimens were tested for every material. The specimens were prepared by cutting them into dumbbell shapes using a hydraulic cutter machine. The width and thickness at the midpoint and at locations on either side of the midpoint of each dumbbell were measured. Specimens were then subjected to a tensile test using an Instron test machine. The machine was operating at a cross head speed of 10 mm/min. The results obtained from the computer system (using Merlin software) such as stress, strain, and elongation at break were recorded. The results were expressed as a plot of tensile strength (MPa), tensile modulus (GPa), and elongation at break (%). Seven samples were used for the tensile test and an average of seven results were taken as the resultant value.

Scanning Electron Microscope

The morphology of the tensile fractured surface of the biocomposite samples was examined under a Philips XL40 with the acceleration voltage of 30 Kv at room temperature.

Table 1. Compositions of different biocomposite materials.

PLA (wt%)	Kenaf fiber (wt%)	Triacetin (wt%)
100	—	—
70	30	—
60	40	—
100	—	5
100	—	10
100	—	15
70	30	5
60	40	5
70	30	1
60	40	1

The samples were coated with gold to avoid charging. The samples were viewed perpendicular to the fractured surface.

Dynamic Mechanical Analysis

Dynamic mechanical properties were measured in the three-point bending mode at a frequency of 1 Hz on rectangular samples of $50 \times 15 \times 1 \text{ mm}^3$. Four main formulations (PLA, PLA/kenaf, PLA/triacetine, PLA/kenaf/triacetine) were tested to study the maximum used temperature and also to observe the possible interaction between the PLA matrix and kenaf fibers. The temperature was from 25°C to 180°C with a heating rate of $1.5^\circ\text{C}/\text{min}$ under the flow of nitrogen (flow rate at $50 \text{ mL}/\text{min}$).

RESULTS AND DISCUSSION

Biocomposite materials with good strength properties can be produced when the fiber is uniformly dispersed and distributed in the matrix. Interaction and adhesion between the PLA and the bast kenaf fiber can be improved by the addition of a plasticizer, which softens the PLA matrix. Tensile test samples for PLA/kenaf biocomposites were successfully prepared by melt blending techniques and then compression molded. The surfaces of all samples were smooth and brown in color, indicating the dispersion of the bast fibers within the PLA were uniform. The effect of fiber loading and the amount of plasticizer (triacetin) on the tensile properties were determined. The dynamic mechanical properties of the composites were also studied. The morphology of the fractured surfaces from tensile testing was also examined by scanning electron microscope (SEM).

Effect of Fiber Loading on Tensile Properties

The effect of fiber loading on the tensile strength of PLA/kenaf bast fiber composites is depicted in Figure 1. In this study, the neat PLA was used as the reference sample.

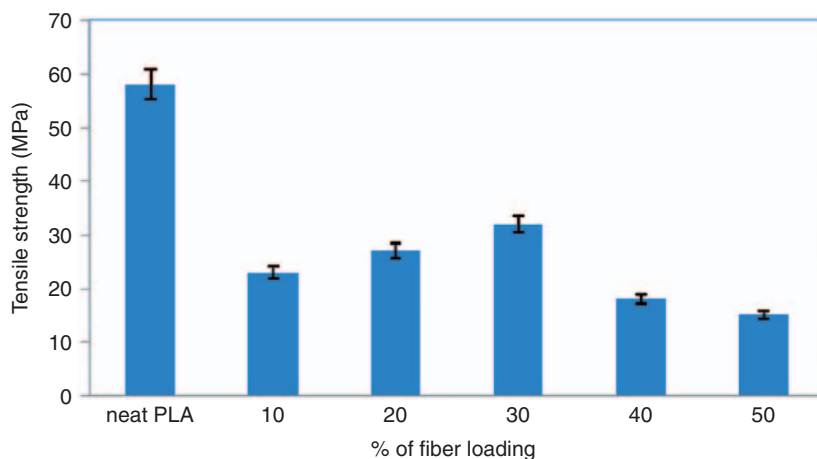


Figure 1. Effect of fiber loading on tensile strength of PLA and PLA/kenaf bio-composites.

The neat PLA present the highest tensile strength results with 58 MPa. Generally neat PLA show better tensile strength than composite PLA and these findings are in agreement with Oksman et al. [4]. As seen in Figure 1, the tensile strength increased with the increased of fiber loading but decreased thereafter. The maximum tensile strength was at 30% fiber loading and decreased as more fiber was incorporated into the matrix. The strength increased slightly from 10 to 30 wt% of fiber loading but suddenly dropped to 18 and 15 MPa with 40 and 50 wt% of fiber loading, respectively.

As per the investigation, the addition of the 10 wt% of bast kenaf fibers did not improve the tensile strength, which implies that there was poor dispersion and fiber distribution, hence, it did not improve the adhesion between the fiber and the PLA matrix. This indicates that the amount of filler is not adequate to transfer the stress efficiently. For 20 and 30 wt% of fiber loading, the fiber dispersion and distribution started to improve, so the results imply better strength compared to 10 wt% of fiber loading. The strength then dropped with 40 and 50 wt% of fiber loading because the matrix was not sufficient enough to wet out the fiber and this reduced the tensile strength of the biocomposite materials. The decrease in tensile strength may also be due to fiber–fiber interaction and agglomerates of fiber in the PLA matrix because there are no polymer chains between the fiber layers.

Other factors such as: changes of PLA crystallinity, porosity of the composites, and chemistry of the natural fibers' surfaces also contributed to the enhancement of the strength and the stiffness of the biocomposite materials [7]. The effect of fiber loading on the modulus strength of PLA and PLA/kenaf biocomposites was shown in Figure 2. The pattern of tensile modulus was similar to the tensile strength results. The results in Figure 2 shows that at 10, 20, and 30 wt% of fiber loading, the modulus of the biocomposites were increased up to 4.4 GPa; however, the tensile modulus of the PLA/kenaf biocomposites dropped to 2.1 and 1.9 GPa at 40% and 50%, respectively. Oksman et al. [4] suggested that this pattern is due to fiber orientation, since the test samples are compression molded and the fibers can possibly be oriented differently from one sample to another. Other researchers suggested that this was associated with low strength of adhesion between the fibers within the biocomposite materials [10].

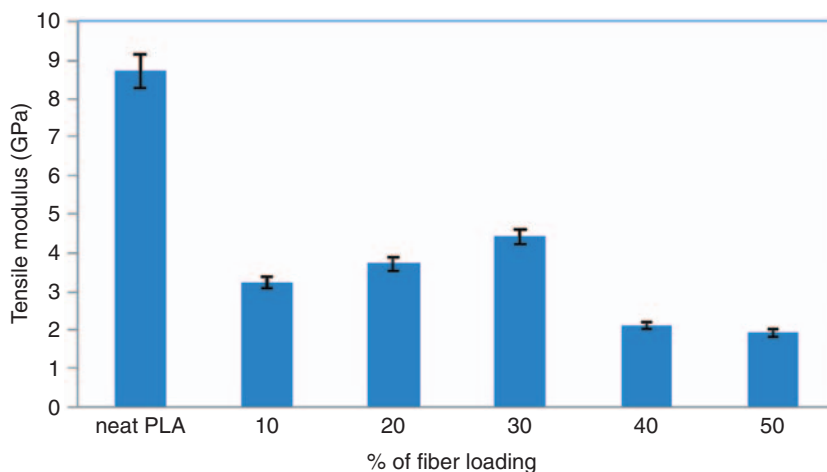


Figure 2. Effect of fiber loading on tensile modulus of PLA and PLA/kenaf bio-composites.

Effect of Triacetin on PLA and PLA/Kenaf Biocomposites

The mechanical properties of PLA and PLA/kenaf biocomposites were also studied with the addition of the plasticizer. Since PLA is brittle in nature, triacetin was used as a plasticizer to plasticize the neat PLA and PLA/kenaf biocomposite materials. This modification was done in order to enhance the strength properties of the PLA/kenaf biocomposites. Plasticizers are small molecules added to soften the polymer by lowering its glass transition temperature or reducing its crystallinity or melting temperature [9].

The amounts of triacetin used were 1%, 3%, 5%, 8%, 10%, 12%, and 15%. Based on other researchers' findings [9,11], about 12–15% of triacetin was needed to improve the properties of PLA. In this research, the optimum triacetin content used was 5%. The strength deteriorates after addition of more than 5%. Below 5% the effect is not significant and these observations agreed with another researcher's [4].

The effect of triacetin on PLA and PLA/kenaf biocomposites on the tensile strength and modulus was revealed in Figures 3 and 4, respectively. Figure 3 shows that the tensile strength of 30:70 kenaf/PLA composites decreased with the increase of triacetin content. This result indicates that triacetin improved the adhesion between the fiber and the matrix. From the graph, the tensile strength for unplasticized PLA is 58 MPa. Then, as 5% triacetin was incorporated into the PLA, the tensile strength of the matrix seemed to decrease to 46 MPa and continued to drop to 40 MPa and 36 MPa as the percentage of triacetin was increased to 10% and 15%. However, the tensile strength for biocomposite samples did improve with the addition of the triacetin content. As shown in the graph, the unplasticized biocomposites have low tensile strength, with only 32 MPa. The triacetin did affect the tensile strength of the biocomposite materials and improved the strength to 44 MPa with 5% triacetin. Unfortunately, the tensile strength started to reduce as the percentage of triacetin was increased, up to 10% and 15%, with 38 and 23 MPa, respectively. According to Espert and Drzal [12], as the plasticizer content increased up to 10%, the tensile strength of PLA dropped to 50 MPa. Oksman et al. also found the same results for PLA/flax fiber composites. The results reveal that the PLA strength decreased from 50 to 37.2 MPa when the content of plasticizer increased up to 15%. However, at 5% triacetin

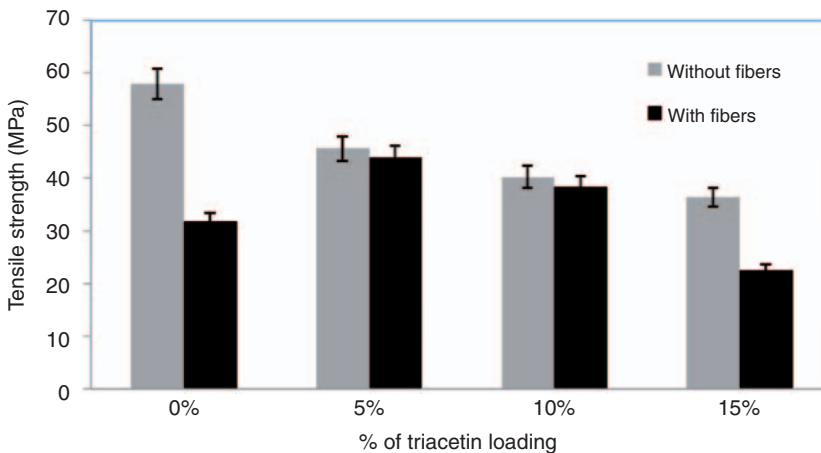


Figure 3. The effect of triacetin on tensile strength of 30:70 bast kenaf fibers/PLA composites.

content, the PLA/flax composites show good tensile strength with 41.7 MPa. Figure 4 shows similar trends with tensile strength where the addition of triacetin decreased the stiffness of the PLA matrix and 5% triacetin content contributed to a higher modulus for biocomposite samples. This indicates that 5% triacetin is the optimum amount to improve the biocomposite materials where more stress is required in order to produce a given amount of strain. This result also reveals that 5% triacetin resists deformation from happening. However, the tensile modulus decreased significantly with the addition of 10% and 15% triacetin as shown in Figure 4. Yang et al. [13] also reveal the same finding, as addition of higher plasticizer percentage will affect the glass transitions' temperature of the materials, thus decreasing the T_g , and generally reducing the tensile modulus and tensile strength of the amorphous polymer.

Figure 5 shows the effect of the amount of triacetin on the elongation at break for PLA and PLA/kenaf composites. Among biocomposite samples, the elongation at break of

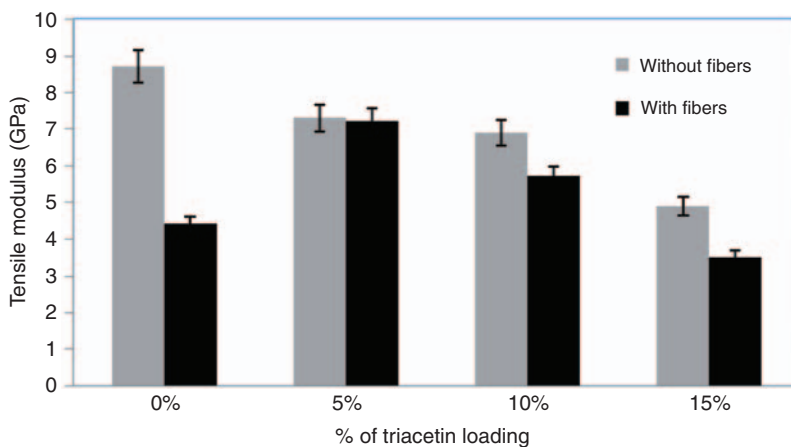


Figure 4. The effect of triacetin on tensile modulus of 30:70 bast kenaf fibers/PLA composites.

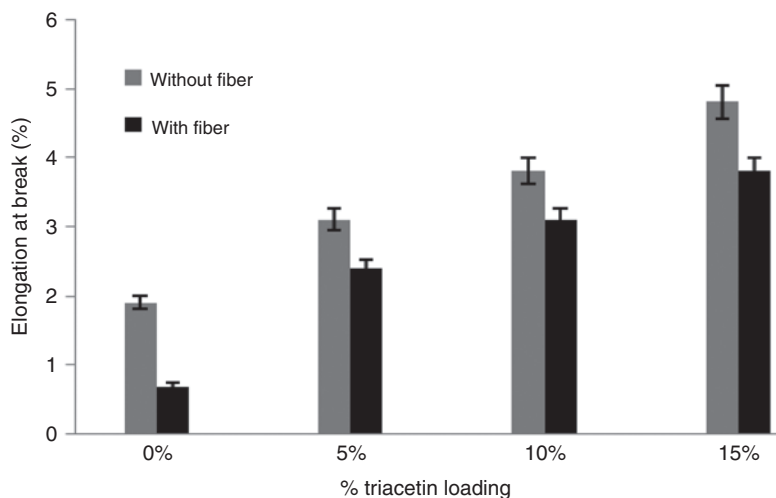


Figure 5. The effect of triacetin on elongation at break of 30:70 bast kenaf fibers/PLA composites.

10% and 15% triacetin is slightly higher than 5% triacetin samples. This is due to the lower strength and stiffness, which is attributed to weak adhesion between fiber and polymer matrix. Researchers noted that [8], as the toughness of the biocomposite is reduced, lower elongation at break results. Higher toughness can be obtained from weak interfacial adhesion as shown by 15% triacetin in biocomposite samples in Figure 5. Weak interfacial bonding resulted in an energy adsorption mechanism such as bond breakage at the fiber/matrix interface and pulled out fiber created better toughness in the biocomposite materials. As reported in references [14,15], it was demonstrated that the tensile properties of biocomposite materials, with the addition of a suitable percentage of plasticizer, could enhance the segmental mobility of the PLA chain and thus increase the ability of the amorphous PLA, until plastic deformation occurred, decreasing the yield stress and increasing the elongation at break of the PLA.

SEM Analysis

Figure 6 shows the fracture surface of the PLA/kenaf composites with 30 wt% of fiber content. It is possible to see from the microscopy picture that there are many fibers pulled out. Besides, the fiber surfaces are shown clean which indicated poor adhesion between the fibers and the PLA matrix. The micrographs reveal that the fibers are oriented in the form of single fibers, hence implying that the fibers have been dispersed during the blending process. The 30 wt% fiber seems considerably well dispersed in the PLA matrix. As reported by other researchers [16], good dispersion of single fibers and good fiber orientation shall contribute to produce good mechanical properties and thus result in good tensile strength for 30 wt% fiber loading for this biocomposite material.

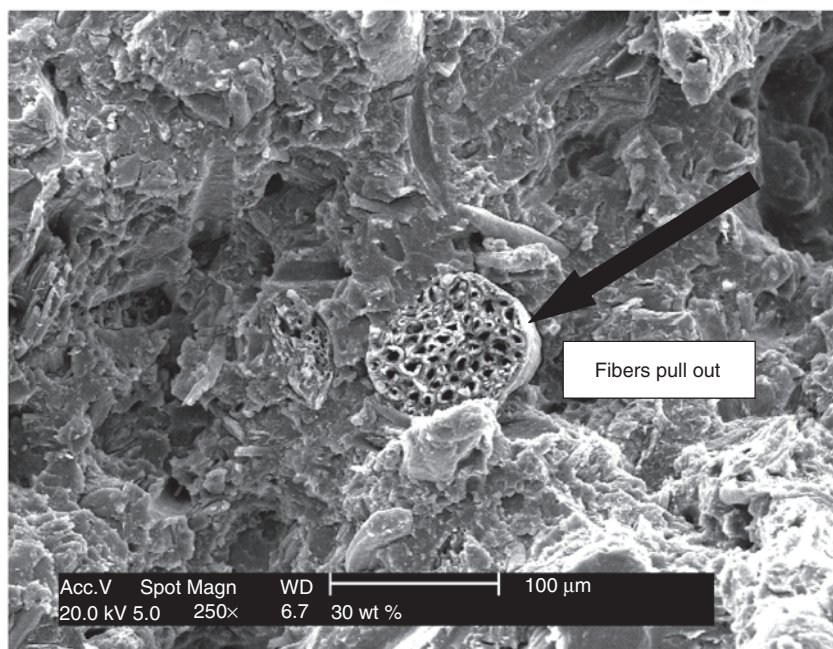


Figure 6. Fractured surfaces of unplasticized PLA/kenaf composites at 30 wt% fiber content.

Figure 7 shows the fracture surfaces of plasticized PLA/kenaf composites at 5% triacetin with 30 wt% fiber content. As reported [16] an increase of the plasticizer content resulted in a large amount of plastically deformed polymer on fracture surfaces. Ghazanfari et al. [10], found that the blends with PLA/PEG 5% have some voids, whereas increasing the percentage of the plasticizer used, up to 12.5%, resulted in the broadening of voids, thus reducing the strength and the stiffness of the matrix. However, in this research, the fibers seem to fill the void, which is caused by the addition of 5% plasticizer; and it did significantly alter the mechanical properties of the materials. However, as the percentage of the plasticizer is increased up to 15%, the voids become wide, and the 30 wt% fiber was not sufficient for filling the voids, thus resulting in lower strength and stiffness of the composite materials.

In plasticized PLA/kenaf biocomposite samples, crazes were evidenced by the SEM, as similarly described by Drzal et al. [16]. A low crystallinity level and a fine spherulitic structure, like in the materials studied, were advantages for development of crazes in semi-crystalline polymers.

Dynamic Mechanical Analysis (DMA) Properties

The temperature curves of the storage modulus, loss modulus, and loss factor (tan delta) for neat PLA, PLA/kenaf, and plasticized PLA/kenaf at 30 wt% fiber content were presented in Figures 8–10, respectively. The results show that the plasticized composite materials have a higher storage modulus than unplasticized composites and neat PLA. This is due to the plasticizing effects of the triacetin content [17]. As seen in Figure 8, the composite samples present a longer plateau effect on the storage modulus when compared to neat PLA. The softening temperature for PLA is 52°C and this temperature increased to

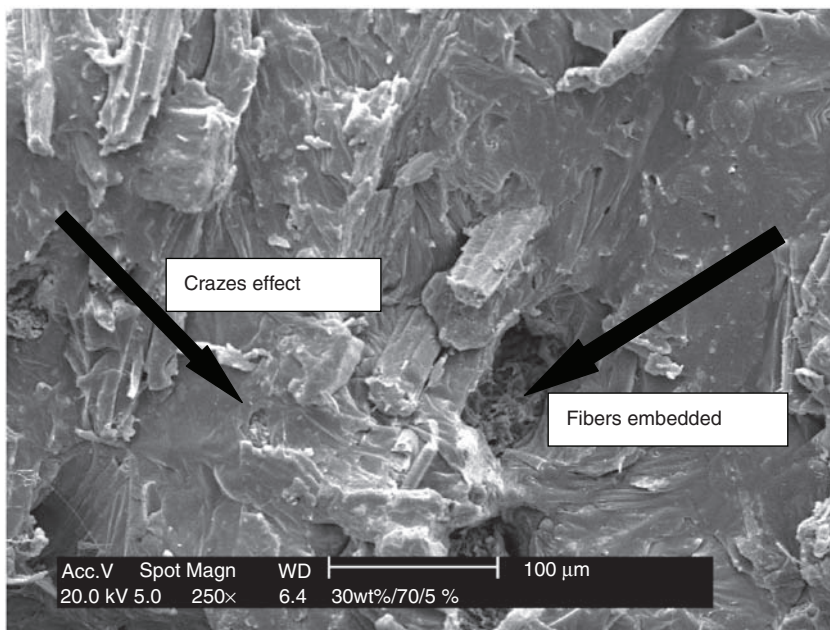


Figure 7. Fracture surfaces of plasticized PLA/kenaf composites at 5% triacetin and 30 wt% fiber content.

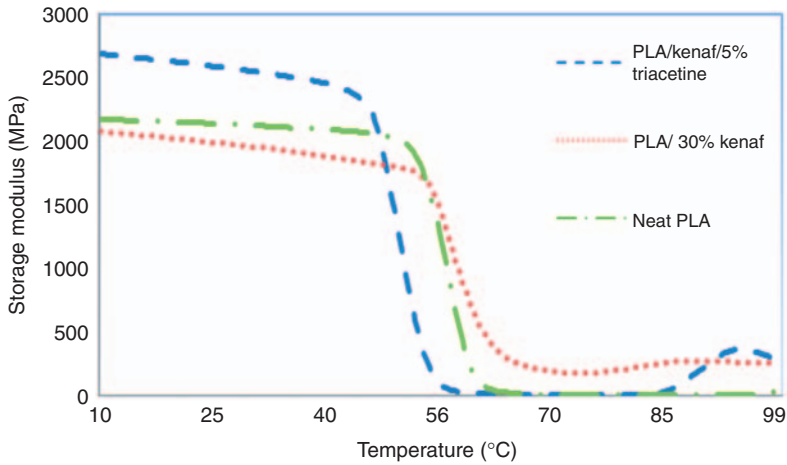


Figure 8. Temperature dependence on storage modulus of neat PLA, PLA/kenaf, and plasticized PLA/kenaf at 30 wt% fiber content.

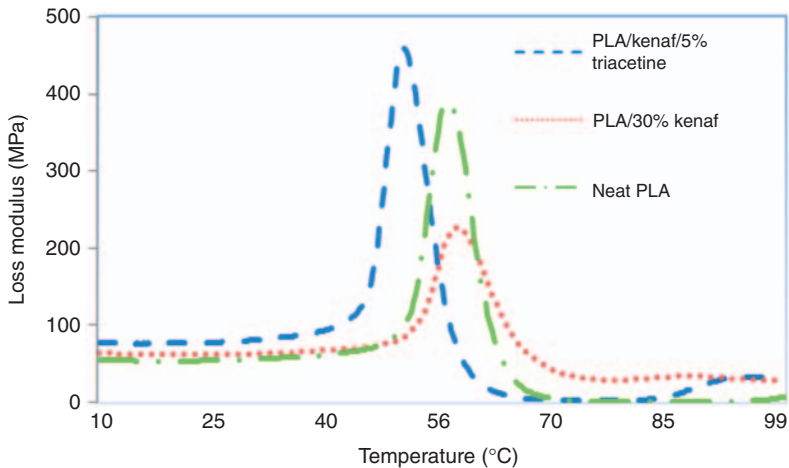


Figure 9. Temperature dependence on loss modulus of neat PLA, PLA/kenaf, and plasticized PLA/kenaf at 30 wt% fiber content.

56°C for unplasticized composites, which indicate an increase in thermal stability of the composite materials, which will be further increased as the composites crystallize [18,19]. Thus, the addition of fibers and crystallization will increase the softening temperature of the composite materials. However, the softening temperature for plasticized composite materials decreased to 46°C because of plasticizing effects.

As seen in Figure 9, the loss modulus curve is presented for neat PLA, unplasticized, and plasticized PLA composites. The glass transition temperatures (T_g), which are derived from the loss modulus curves, are included in Table 2. Based on Huda et al. [19] the T_g is usually interpreted as the peak from the loss modulus curves obtained by dynamic mechanical testing. As shown in Table 2, the T_g for neat PLA, unplasticized, and plasticized composites with 5% triacetin content shifted to lower temperatures, from 67°C for neat PLA to 58°C and 52°C, respectively.

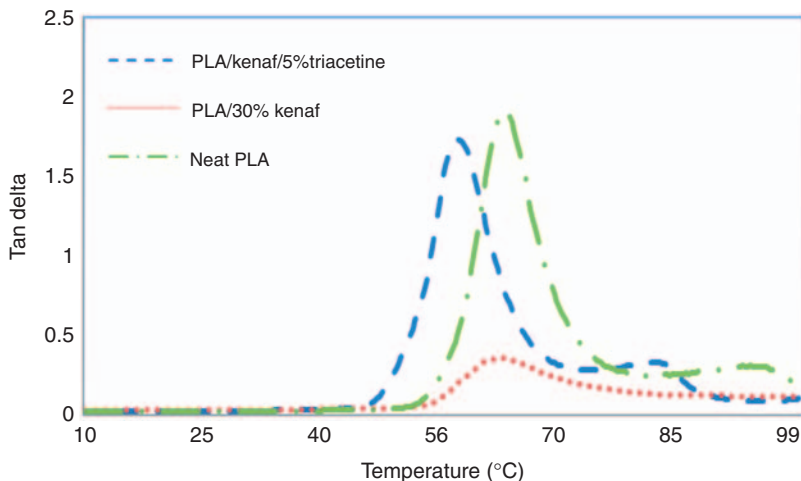


Figure 10. Temperature dependence on tan delta of neat PLA, PLA/kenaf, and plasticized PLA/kenaf at 30 wt% fiber content.

Table 2. Thermomechanical properties of the PLA, unplasticized PLA composites, and plasticized PLA composites.

Materials	T_g (°C) (from loss modulus curves)	Storage modulus (GPa) at 25°C	Storage modulus (GPa) at 40°C	Storage modulus (GPa) at 60°C
Neat PLA	67	2.14	2.11	0.25
PLA/kenaf	58	2.00	1.92	0.50
PLA/kenaf/5% triacetine	52	2.61	2.50	0.13

Martin et al. [22], found that a lowering of the modulus value was associated with the T_g of plasticized PLA, which decreased almost linearly with increasing plasticizer concentration, which ranged from 67°C for pure PLA to 54°C and 46°C for PEG concentrations of 10% and 20%, respectively. Moreover, the tan delta peak decreased accordingly [20].

This phenomenon has also been observed by Huda et al. [21]. They suggested that this is because of the degree of crystallinity, where amorphous PLA show a quite sharp and intense peak since there are no restrictions to chain motion, whereas the crystallinity of the plasticized PLA hinder the chain mobility, resulting in the reduction of the sharpness of the tan delta peak. These dynamic mechanical analyses' results represent higher thermal stability for plasticized biocomposite materials as compared to neat PLA matrix.

Shown in Figure 10 is the loss factor, which is the tan delta of the neat PLA, unplasticized, and plasticized biocomposite materials as a function of temperature, where the ratio of the storage modulus and loss modulus produce the tangent of the phase angle delta, which is the measure of the energy dissipation [22]. The tangent delta is the transition region measuring the imperfections in the elasticity of the polymer, with the possibility of additional loss that occurred at the fiber–matrix interfaces [22,23]. As referred to in Figure 10, the fibers' contribution to the damping is extremely low as compared to the plasticized PLA and plasticized PLA composites.

This situation showed that the combination of the kenaf fibers within the unplasticized PLA composites, would be mainly caused by the molecular motion of the PLA and their interaction at the fiber–matrix interface [24].

CONCLUSIONS

This study demonstrated that plasticized biocomposites with good mechanical and thermomechanical properties could be developed using kenaf bast fiber as a reinforcer, triacetin as the plasticizer, and PLA as a matrix. The results of the study revealed that triacetin improves the compatibility between the PLA matrix and kenaf bast fibers. The tensile strength properties of the plasticized biocomposite materials were significantly higher than those of unplasticized biocomposite materials.

It is believed that the plasticizer improved the interfacial interaction, thus resulting in good strength and stiffness of the biocomposite materials. The SEM micrographs of the unplasticized and plasticized biocomposites clearly indicated the extent of the fiber–matrix interface adhesion. The dynamic mechanical testing was carried out on the neat PLA matrix and showed plasticized biocomposites have a higher storage modulus, which corresponds to a higher tensile modulus. The addition of the plasticizer also gives a lower softening temperature than unplasticized and neat PLA matrix. Addition of 5% triacetin improves the thermal stability of the biocomposite materials.

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