



## Bio-composites of kenaf fibers in polylactide: Role of improved interfacial adhesion in the carding process

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### ABSTRACT

Bio-composites comprised of kenaf fiber reinforced polylactide are fabricated by carding followed by treatment with a 3-glycidioxypropyl trimethoxy silane and hot-pressing. The effects of the silane coupling agent on composite properties was highly beneficial leading to increased moduli and heat deflection temperatures as well as reduced water swelling. Swelling is found to increase with increasing kenaf loading levels but to decrease with increasing coupling agent concentrations. Mechanical properties of the bio-composites at temperatures above the glass transition are vastly improved in comparison to the base PLA polymer. As little as 10 wt.% kenaf fiber in PLA provides a heat distortion temperature in excess of 140 °C when combined with the coupling agent. The thermal stability towards degradation is slightly decreased relative to the base PLA and the ash content is increased. Thermal properties such as the glass transition, melting temperature, and percent crystallinity of the matrix PLA are largely unaffected by incorporation into the composites. Evidence of successful reaction of the silane with the kenaf fibers is provided by FTIR and implied by decreased swelling in water. A prototypical automotive headliner is fabricated as a means of demonstrating the viability of the present bio-composites in real manufacturing processes.

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### 1. Introduction

Advanced polymer composites containing carbon and glass fibers have been utilized extensively in the aerospace, automotive, and construction industries [1]. Since the matrices and the fiber reinforcements in these advanced composites are based on mineral resources, their long term sustainability is problematic. While recycling may be a viable strategy, the complicated mixed morphology of composite materials makes them inherently difficult to recycle. In comparison, several so called bio-composites, have been developed that offer certain environmental advantages at the end of their use cycle when composites are landfilled or incinerated [2]. For the purposes of this study, bio-composites are defined as composite materials that combine natural fibers such as sisal, jute, hemp, and kenaf with either biodegradable or non-biodegradable polymers [3]. Natural fibers have many advantages over synthetic fibers; these advantages include biodegradability, low density, high toughness, acceptable specific strength, reduced dermal and respiratory irritation, low cost, and less use on non-

renewable resources [4–6]. Often, polyolefin thermoplastics such as polypropylene and polyethylene have been used as the matrix polymer. However, combining the biofibers with a biodegradable and renewable resource based polymer offers additional sustainability benefits. In this study kenaf fibers are combined with polylactides (PLA), a biobased plastic which is commercially available. These fully renewable materials can be referred to as bio-composites due to their superior sustainability profile from the perspective of Industrial Ecology, for example, as calculated using the tools of life cycle analysis.

Several fabrication methods have been developed for manufacturing bio-composites. If particles such as cellulosic wood flour are used as the reinforcing filler, then melt mixing in a continuous extrusion process is often used. For short natural fibers of only a few millimeters in length, the same type of extrusion methodologies can be used [5–7]. Novel reactively compatibilized melt mixing processes for producing cellulosic fiber reinforced polylactides bio-composites have also been described [8]. In this method both monomer and polymer are introduced into the extruder as a means of optimizing the wetting and mixing needed to obtain good fiber dispersion. In another method of composite preparation, woven fabrics made of natural fibers such as jute, banana, hemp, and flax have been used as reinforcements. For example, Stocchi et al. fabricated a laminated composite with four layers of jute

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woven fabrics [9]. In this procedure, the jute fabrics were treated with the alkali under a biaxial tension prior to being impregnated with resin. Similarly, Jacob et al. prepared a woven fabric using sisal fiber and rubber composites [10]. In their woven fabric, the sisal yarns were used for all the warp yarns. Dhakal et al. carried out low velocity impact test of hemp fiber reinforced composites, which were prepared using unsaturated polyester resin and a needle punched non-woven mat made of hemp fibers [11]. It was demonstrated that the total energy absorbed by the hemp fiber reinforced bio-composites was comparable to the energy absorbed by the equivalent fiber volume fraction of an E-glass fiber reinforced composite made by impregnating a chopped strand glass mat with unsaturated polyester.

One potential problem in creating good bio-composites is a lack of interfacial adhesion between the natural fiber fillers and polymer matrix. The strength of the filler–matrix interface is essential for stress transfer between the two components [11]. Over the last decade, many approaches towards enhancing interfacial adhesion have been pursued including the use of coupling agents (MAPP, MAPE and silane agents [5,12]) and chemical modification (alkali [13], acid [14]). Generally improvements can be accomplished, but there must be a critical cost-benefit evaluation of using the added interfacial agents or processing steps.

This study explores the use of the carding process for the production of bio-composites of kenaf fibers as a reinforcing filler in PLA. In particular, long and discontinuous natural fibers are combined with PLA fibers (mean length of 50 mm). Beyond the novel use of the carding process, the effects of a coupling agent on the composite properties was also explored and partially optimized. The mechanical and thermal properties, chemical structure and morphological properties of the bio-composites are investigated as a function of both the kenaf fiber loading level and of the silane coupling agent concentration. Strong evidence exists for the beneficial effects of adding the silane coupling agent at a level between 1 and 3 parts per hundred (pph) of the underlying bicomponent composite. In particular, significant improvements in the heat distortion temperature of the PLA may be obtained using this approach.

## 2. Experimental

### 2.1. Materials

The polymer matrix of the bio-composites discussed here is poly(lactic acid) (PLA), which was manufactured by Huvis Ltd., (South Korea). The PLA was provided in the form of fibers with average length of 52 mm, average diameter of 81  $\mu\text{m}$  and a density 1.24  $\text{g}/\text{cm}^3$ . The kenaf fibers used were bast fibers and were donated by Sutongsang (South Korea). The mechanical and physical properties of these component materials are listed in Table 1.

3-Glycidoxypropyl trimethoxy silane (GPS) from Shin-Etsu KBM 403 (Japan) was used as the silane coupling agent in the PLA/kenaf fiber bio-composites.

### 2.2. Sample preparation

The bio-composites of PLA/kenaf fiber were prepared using a carding machine (Kyowa Co. Ltd., Japan) in a process diagrammed

in Fig. 1. Carding provides a uniform blend of the two fibers, this is followed by needle punching, then pre-pressing and finally hot-pressing to form the composite material. The PLA/kenaf non-woven web produced after the carding process was pressed to reduce the thickness of the matt. Subsequently, this prepressed non-woven web was treated with the silane coupling agent in amounts of 1, 3 and 5 parts per hundred (pph) of the prepressed composite material. The silane was allowed to penetrate and pre-treated with the prepressed matt for 2 h. In the final step, the silane treated prepressed matt was hot-pressed for 5 min at 200 °C under a pressure of 0.7 MPa (70  $\text{kgf}/\text{cm}^2$ ). This process enabled melting of the PLA and good impregnation provided a well consolidated formed sheet. The compositions of the various PLA/kenaf fiber bio-composites treated with silane coupling agent investigated here are listed in Table 2.

### 2.3. Mechanical tests

Flexural strength tests were conducted according to ASTM D790-03 with a Universal Testing Machine (Zwick Co.). Following the standard, these tests were conducted at  $23 \pm 2$  °C,  $50 \pm 5\%$  RH; the thickness, width and length of flexural test specimens were 3 mm, 12 mm, and 63 mm, respectively. Flexural strength and flexural modulus were computed from the stress–strain curves. Each value reported is the average of six sample tests and error bars correspond to plus or minus one standard deviation.

### 2.4. Dimensional stability and water absorption behavior

The swelling and water absorption were conducted as ASTM D 570; swelling is measured in the direction of the thickness of the formed sheet. Samples of each biocomposite were immersed in water for 70 days. After removal from the water the samples were surface dried by patting with a towel before measuring thickness and weight. The data reported are average values obtained from five separate samples of each bio-composites; again the measurement error is reported as standard deviation of the data for each sample.

### 2.5. Dynamic mechanical analysis (DMA)

The storage modulus was measured at a frequency of 1 Hz and at a strain rate 0.05% at a heating rate of 2 °C/min over the temperature range of  $-20$  °C to 140 °C. A TA Instruments dynamic mechanical analyzer (DMA Q800, TA Instruments) employing the dual cantilever method on rectangular specimens of 60.0 mm  $\times$  12.0 mm  $\times$  3.0 mm was used.

### 2.6. Differential scanning calorimetry (DSC)

Thermal properties were examined using a TA Instrument DSC Q 1000. About 5–7 mg of the biocomposite samples were placed in the DSC pan. A heating rate of 10 °C/min was used under a flow of nitrogen. The samples were first scanned from  $-80$  to 200 °C and then cooled to  $-80$  °C and again heated up to 200 °C in a second scan. Glass transition ( $T_g$ ) and melting temperatures ( $T_m$ ) were determined from the data of the second scan.  $T_m$  was determined from maximum region of endothermic melting peak, and  $T_g$  was a inflection temperature from baseline at the second heating scan. The degree of crystallinity ( $X_c$ ) of was calculated according following equation:

$$X_c = \frac{\Delta H_f 100}{\Delta H_f^0}$$

where  $\Delta H_f$  is the heat of fusion of the PLA and composites,  $\Delta H_f^0$  is the heat of fusion for 100% crystalline PLA ( $\Delta H_{100} = 93.7$  J/g) [15].

**Table 1**  
Physical properties of initial fiber materials at 23 °C.

Materials	Density ( $\text{g}/\text{cm}^3$ )	Mean fiber length (mm)
Kenaf fiber	1.4	$60 \pm 10$
PLA fiber	1.24	$52 \pm 5$

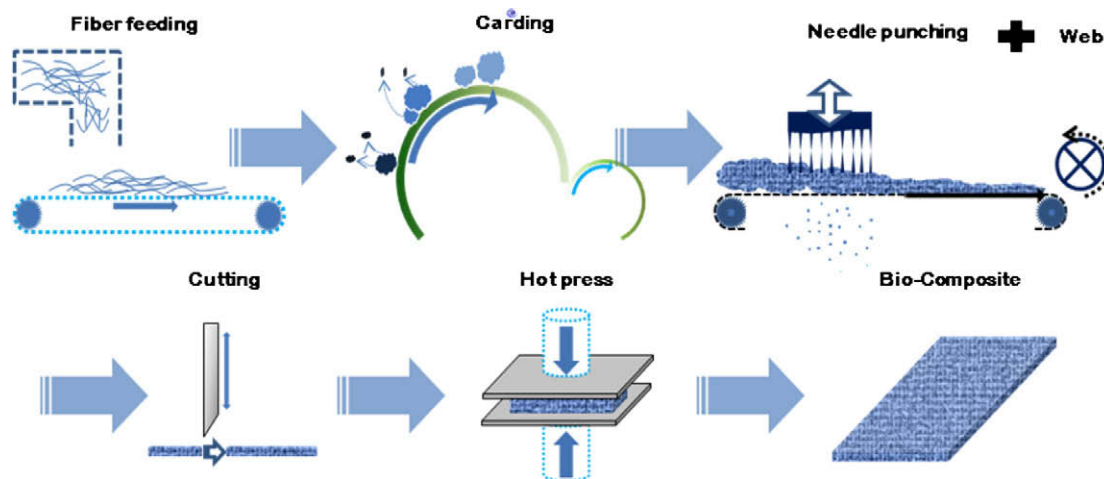


Fig. 1. Carding process for manufacturing bio-composites.

Table 2  
Composition of bio-composites.

Compositions					
PLA fiber	100	90	70	50	30
Kenaf fiber	0	10	30	50	70
GPS (pph of composite)	1, 3, 5				

2.7. Heat deflection temperature (HDT)

The heat deflection temperature (HDT) was measured by a Toyoseiki HDT tester (Japan). The dimensions of the test bars (127 mm × 13 mm × 3 mm) and the test procedure conformed with the ASTM D648-06 standard. The loading pressure was 0.455 MPa (66 psi) and samples were heated at a rate of 2 °C/min from room temperature.

2.8. Thermogravimetric analysis (TGA)

Thermal decomposition of the bio-composites was analyzed using thermogravimetric analysis (TA instrument, TGA Q500) on samples having masses from 10 to 13 mg. The heating rate was 20 °C/min over the temperature range from 30 to 600 °C; a nitrogen atmosphere was employed.

2.9. Attenuated total reflectance (FTIR-ATR) spectroscopy

Infrared spectra were obtained using a JASCO 6100 FTIR spectrophotometer. The specimens were analyzed over the range of 4000–650 cm<sup>-1</sup> with a spectrum resolution of 4 cm<sup>-1</sup>. All spectra were averaged over 30 scans.

3. Results and discussion

3.1. Flexural properties

Flexural strengths of the kenaf fiber reinforced PLA bio-composites are shown in Fig. 2a. The flexural strength of the GPS treated bio-composites was significantly greater than those of the untreated bio-composites. This is presumably due to an enhanced interfacial adhesion of the PLA to the kenaf as a result of the inclusion of the silane coupling agent. For bio-composites containing 50 parts of kenaf and 50 parts of PLA, the flexural modulus increases by more than 25%. Interestingly, even the smallest concentration of the silane agent (1 pph) has nearly the same effect as the highest

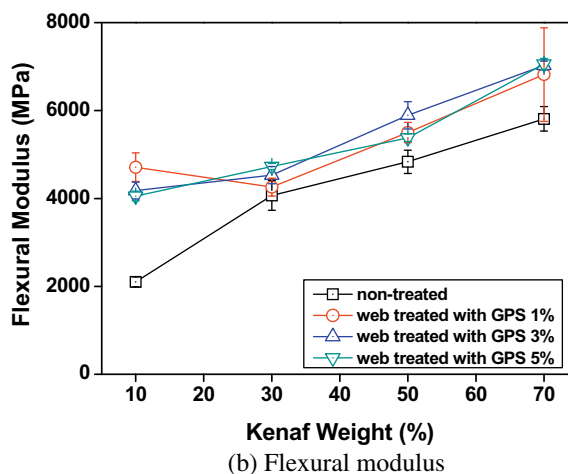
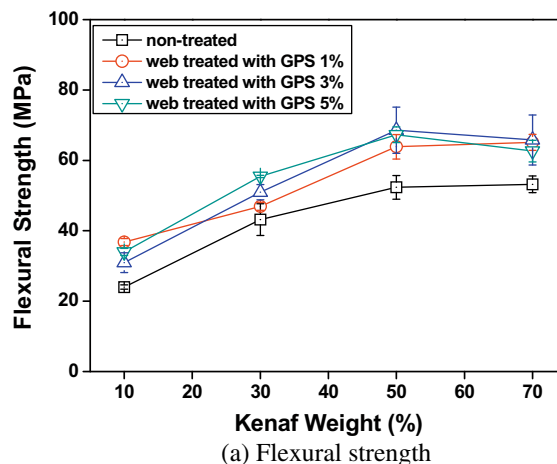


Fig. 2. Flexural modulus of the bio-composites as a function of kenaf loading and GPS concentration.

concentration (5 pph). Whereas the untreated biocomposite has a flexural modulus of 52.3 ± 3.4 MPa, all of the treated bio-composites fall within the range of 66.0 ± 2.5 MPa. These results indicate that a very low loading level of silane can provide significant benefits to the mechanical properties indicating that their use may be economically advantageous. Also noteworthy in Fig. 2 is the

decrease in flexural strength beyond 50% kenaf fiber. It is hypothesized that this may be due to a shortage of PLA to fully wet out between the kenaf fibers.

The flexural modulus were shown in Fig. 2b exhibits similar trends. Again, while the 3 pph GPS material exhibits the highest modulus, the most significant effect is between treated and untreated bio-composites – the coupling agent again shows high effectiveness at low levels of only 1 pph. The flexural modulus is increased by the adding natural fiber content up to 70% kenaf. Beyond 70% kenaf fiber content, the flexural modulus decreases.

The results for the flexural modulus and strength point out the importance of using the right amount of natural fiber as reinforcement. Using the present carding approach, it is clear that an optimal level of PLA fiber exists with respect to maximizing flexural properties and that this optimal level falls in the range of 30–70% of the binary fiber mixture being PLA; furthermore it appears the optimum is in the vicinity of 50% PLA. Importantly, the results indicate that the present silane coupling agent has very positive effects on the flexural properties of the biocomposite, even at low concentrations.

3.2. Dimensional stability and water absorption behavior

Figs. 3 and 4 show data for the thickness swelling and water absorption for the bio-composites. Swelling and water absorption of the bio-composites are seen to increase with increasing kenaf content and their values are very high compared with pure PLA. Such a finding is sensible given the hydrophobic nature of PLA and the hydrophilic nature of the kenaf fiber. Treatment of the

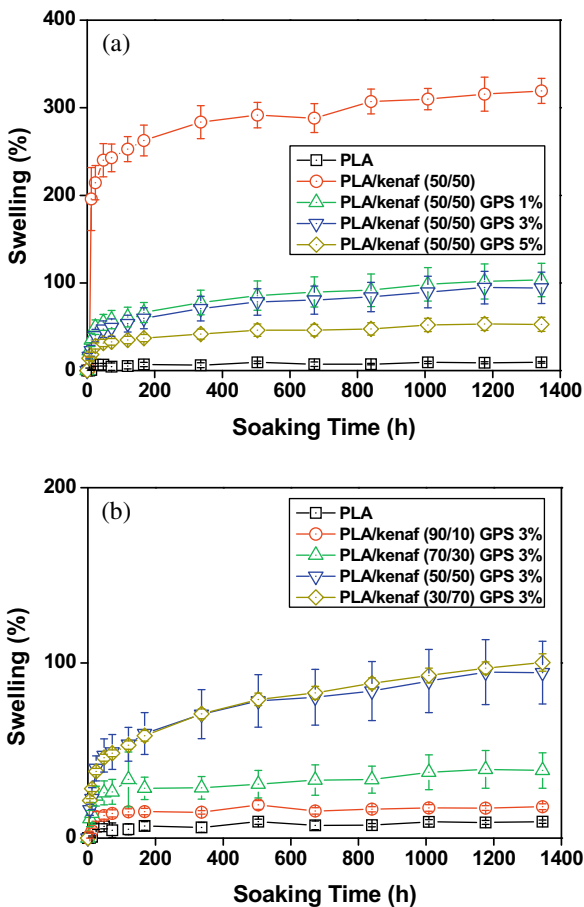


Fig. 3. Swelling of the PLA/kenaf bio-composites: (a) for different GPS concentrations and (b) for different kenaf fiber loadings at a fixed GPS concentration of 3 pph.

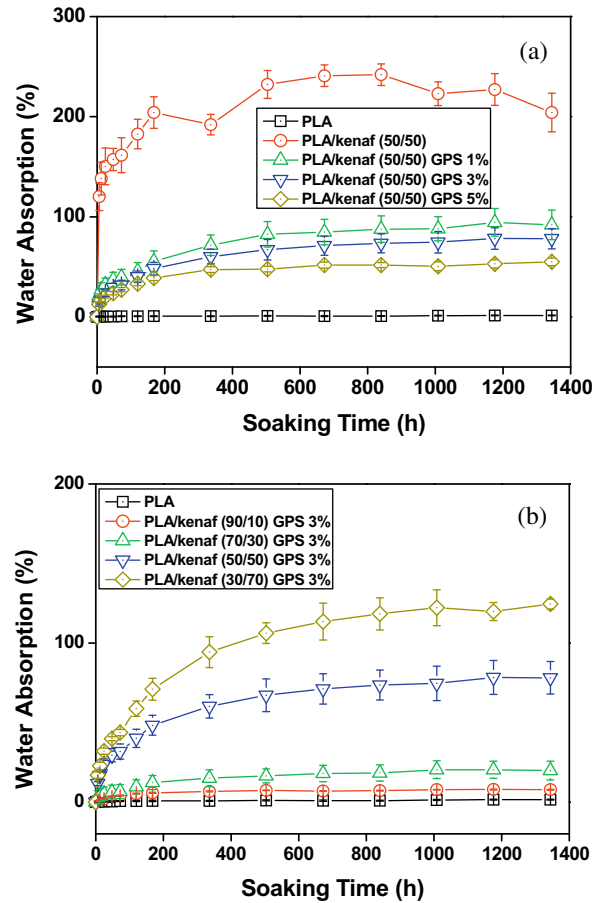


Fig. 4. Water absorption of the PLA/kenaf bio-composites: (a) for different GPS concentrations and (b) for different kenaf fiber loadings at a fixed GPS concentration of 3 pph.

50% biocomposite with the GPS coupling agent reduces swelling and water absorption compare to the non-treated biocomposite. At least two mechanisms can be considered. First, silanes are known to react with the hydroxyl groups prevalent in cellulose; accordingly when they are used the kenaf fibers are rendered less hydrophilic. In addition, better wetting out of the PLA onto the fibers means there may be less void space through which water may ingress into the samples. Such findings have been previously reported [16]. In all cases, the swelling and water absorption occurs predominantly in the first 200 h.

3.3. Dynamic mechanical analysis (DMA)

DMA is a powerful test method for investigating the viscoelastic behavior of the bio-composites. The storage modulus of PLA/kenaf bio-composites is shown in Fig. 5. The  $E'$  values of PLA and the bio-composites is decreasing with increasing temperature because of increased chain mobility of the matrix polymer, the drop in modulus is precipitous near the glass transition of the PLA (around 60 °C)[17]. As shown in Fig. 5a, the  $E'$  values of the bio-composites are significantly higher than pure PLA over the whole temperature range. Treatment with GPS further enhances the storage modulus values compare to the untreated bio-composites. However from Fig. 5b, the storage modulus of the bioscomposite having 70 wt.% kenaf is lower than other bio-composites; again this is attributable to not having sufficient PLA present to fully wet out the fibers thereby creating voids which restricts the stress transfer at the interface [18]. And also, the drop point in  $E'$  is slightly shifted to



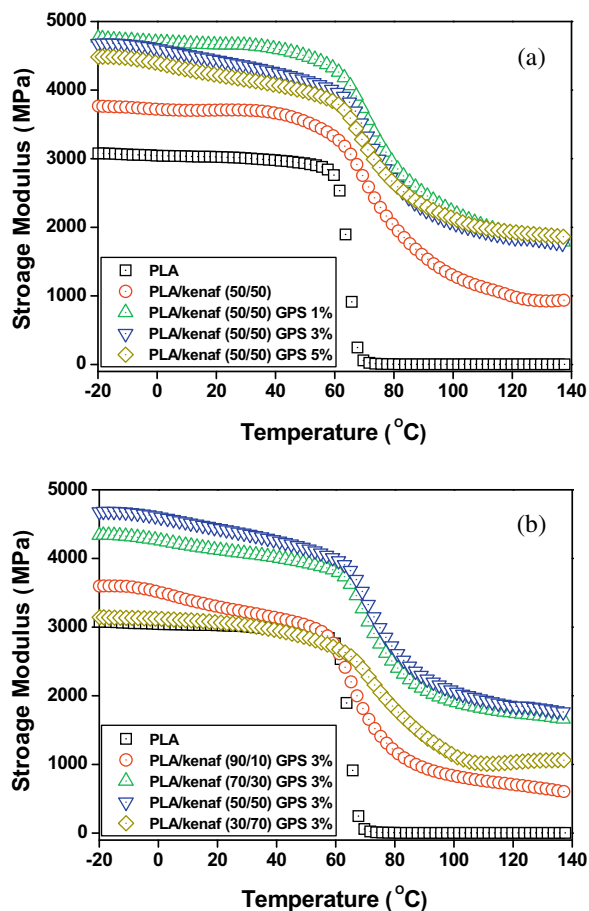


Fig. 5. Storage modulus versus temperature of the PLA/kenaf bio-composites: (a) for different GPS concentrations in a 50/50 mixture and (b) for different kenaf fiber loadings at a fixed GPS concentration of 3 pph.

higher temperatures and broadened upon incorporation of the kenaf fibers. The value of the temperature at drop point coincides with the glass transition temperature ( $T_g$ ). As seen in Fig. 5a, the  $T_g$  of bio-composites with treated GPS is not strongly affected; the majority of any effects associated with chain confinement occur as a result of adding the kenaf fiber [19]. From the DMA results, it can be concluded that the kenaf fibers affect the viscoelastic properties to a much greater extent than the silane coupling agent treatment.

### 3.4. Differential scanning calorimetry (DSC)

Thermal properties including glass transition temperature ( $T_g$ ), melting temperature ( $T_m$ ) and heat of fusion ( $\Delta H_f$ ) can be directly measured in DSC. The results for all of the investigated bio-composites are summarized in Table 3. The melting temperatures,  $T_m$ , do not exhibit sharp differences. However, GPS treated bio-composites are just slightly increased (+4 °C) compared to untreated bio-composites. The  $T_g$ ,  $T_m$ , and of bio-composites are not fully affect by the GPS treatment. But, Table 3 reveals  $\Delta H_f$  and degree of crystallinity ( $X_c$ ) is a bit improved, about 4 J/g and 4%, respectively. Untreated PLA/kenaf (50/50) biocomposite contain 20.5% crystallinity. The percent crystallinity increases in the bio-composites as GPS is added with 1, 3, and 5 pph resulting in 24.3%, 24.6% and 24.9%, respectively. Again as with other properties, the major change takes place upon incorporation of any GPS and significant increases are not seen as higher concentrations are used. This increase in crystallinity can be explained by the kenaf fiber serving

Table 3  
Thermal properties of kenaf-filled PLA bio-composites.

	$T_g$ (°C)	$T_m$ (°C)	$\Delta H_f$ (J/g)	$X_c$
PLA	60.0	160.3	–	–
PLA: Kenaf (5:5)	56.9	164.2	19.2	20.5
PLA: Kenaf (5:5) GPS 1%	58.7	168.6	22.8	24.3
PLA: Kenaf (5:5) GPS 3%	59.3	168.1	23.1	24.6
PLA: Kenaf (5:5) GPS 5%	59.1	167.3	23.3	24.9

to promote heterogeneous crystallization – effectively serving as a nucleating agent.

### 3.5. Heat deflection temperature (HDT)

The HDT or softening point of polymer-based materials is an important factor for designing industrial products [20]. Fig. 6 shows the HDT value for the various bio-composites. The HDT value of pure PLA is 57 °C. The HDT of PLA/kenaf fiber bio-composites was increasing with increasing kenaf loading. Such a result is sensible given that the HDT value of the kenaf reinforcement is greater than that of the PLA matrix [20]. The HDT values of GPS treated bio-composites were a little higher than values of untreated bio-composites. This is attributable to the improved interfacial adhesion and higher crystallinity of the bio-composites. The HDT values of bio-composites steeply increases with increasing kenaf fiber loading; already at only 10% kenaf the HDT is much greater than that of pure PLA. In addition, it is again observed that the addition of only small amounts of the coupling agent has very beneficial effects. Finally it should be remarked that the HDT more or less plateaus near 160 °C for high values of the kenaf loading, accordingly this must be considered in deciding on ultimate applications for this class of bio-composites.

### 3.6. Thermogravimetric analysis (TGA)

The TGA data for the various biocomposite formulations is shown in Fig. 7. In all of the TGA curves, two main degradation regions can be observed. One is reportedly due to the thermal degradation of cellulose, hemicelluloses, and lignin in the kenaf fiber [17,21], and the other higher temperature one is attributed to depolymerization of the PLA [22]. The onset of thermal decomposition of the bio-composites is slightly lower than pure PLA indicating that introduction of the fibers reduces the thermal stability. However, the thermal stability of the silane treated bio-composites is slightly higher than the untreated ones. Finally, Fig. 7 shows that

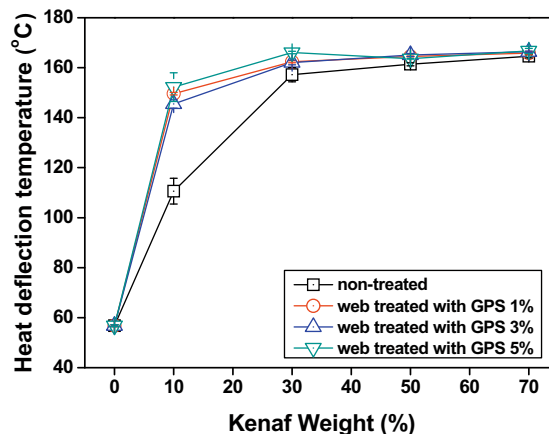


Fig. 6. Heat deflection temperature (HDT) of the PLA/kenaf bio-composites.

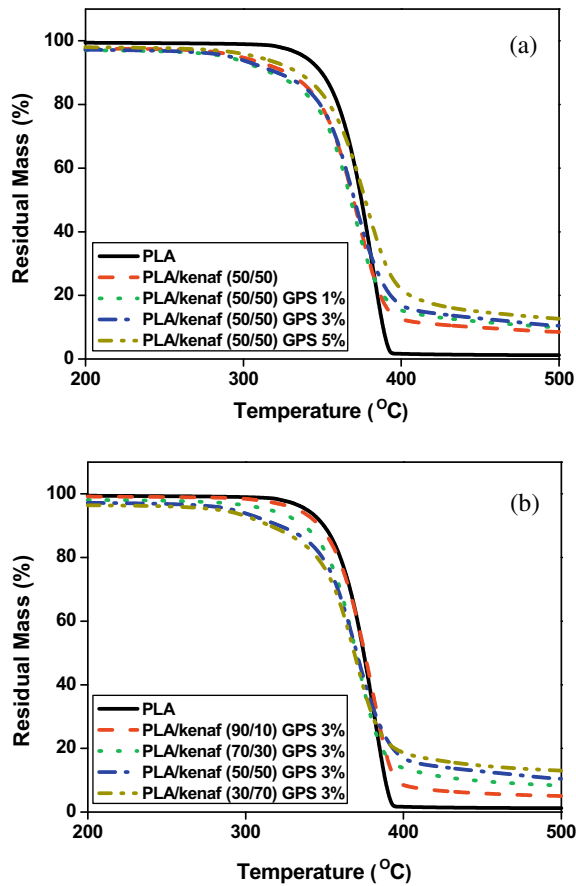


Fig. 7. TGA curves of PLA/kenaf bio-composites: (a) with different GPS treated concentration and (b) with different kenaf fiber contents treated with GPS 3%.

the residual mass of ash content of the bio-composites increased when 5 pph of GPS was used. In summary, the kenaf fibers decrease the thermal stability but increase the residual ash content.

### 3.7. Attenuated total reflectance (FTIR-ATR) spectroscopy

GPS treated and untreated bio-composites were examined by the FT-IR spectra. From FT-IR results, bio-composites treated with GPS showed two absorption peaks at 704 and 762  $\text{cm}^{-1}$ . The presence of the absorption band at 704  $\text{cm}^{-1}$  corresponds to the  $-\text{Si}-\text{O}-\text{Si}-$  symmetric stretching band [23,24]. The peak at 762  $\text{cm}^{-1}$  was assigned to symmetric  $-\text{Si}-\text{C}-$  stretching of the silane agent [23,24]. The reaction between hydroxyl groups on the surface of kenaf with the silane coupling agent can enhance interfacial adhesion. When successful, this reaction renders the kenaf surface more hydrophobic thereby increasing the wetting of the surrounding PLA matrix which results in improved interfacial properties.

### 3.8. Industrial application of bio-composites

Over the past several years, many reports in the field of bio-composites filled with natural fibers such as jute, flax, hemp, kenaf and others have been reported. Many researchers are trying to make building interior, electronics, automotive, and other products. To date, few manufactured goods are available in the marketplace. Here we report on a prototype automotive interior part utilizing the formulated bio-composites. An interior headliner prototype for an automobile is shown in the photograph of Fig. 8; headliners are the interior ceiling in automobiles. The headliner prototype is made of PLA/kenaf 50 wt.% without GPS. Table 4



(a)



(b)

Fig. 8. Prototypical automotive headliner made from a 50/50 PLA/kenaf fiber biocomposite from the: (a) back side and (b) front side.

shows that the test results on the headliner utilizing standard test methods from the automotive industry. However, not all needed qualifying tests for the application have been conducted. Nonetheless, it can be confirmed that the tensile strength, elongation and flexural strength requirements are satisfied. Clearly these results are encouraging enough to warrant further development of these biocomposite materials and the manufacturing process based on carding.

## 4. Conclusions

As climate change and resource depletion enter into the broader societal consciousness, there will be an increasing demand for sustainable products based on renewable resources. Presently, the PLA resin cost make the bio-composites detailed here difficult to produce economical when compared to polyolefin matrix resins such as PP, LDPE and HPDE. However, the price volatility of petroleum

**Table 4**  
Mechanical properties of headliner made of bio-composites.

Test	Unit	Demand <sup>a</sup>	Result		
Tensile strength	Vertical	kgf/5 cm	Above 95	97.51	Satisfied
	Horizontal		Above 115	131.91	Satisfied
Elongation	Vertical	%	Above 3	6.75	Satisfied
	Horizontal			4.97	Satisfied
Flexural strength	Vertical	kgf/5 cm	Above 1.6	1.36	Non-satisfied
	Horizontal		Above 2.8	1.7	Non-satisfied
Dimension stability	Vertical	%	±1.0	0.26	Satisfied
	Horizontal			0.23	Satisfied

<sup>a</sup> The standard of automotive industry field.

based resins causes has led to time periods when the bioplastic composites are price competitive and in the near future, fully biodegradable bio-composites are bound to appear in many more commercial products. The replacement of glass fiber or carbon fiber filled composites presents a large potential opportunity for composite developments which will help in protecting an increasingly threatened global environment.

Here it is demonstrated that long kenaf fibers derived from the bast part of the plant may be used to successfully reinforce PLA; these novel and useful bio-composites are made using a combination of carding and punching processes followed by hot press compression molding. The effect of silane coupling agent on the mechanical properties of the kenaf fiber reinforced PLA bio-composites is shown to be highly beneficial. Even a small amount of 1 pph silane coupling dramatically increases the mechanical properties, improves the heat distortion temperature and decreases the moisture sensitivity of the composites. Roughly an optimal formulation comprised of 50% kenaf and 50% PLA fibers to which 3 parts of GPS coupling agent is added represents an optimal formulation which may prove useful in a variety of applications. It is demonstrated that such materials can be successfully utilized to manufacture automotive headliners.

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