

Thermal characterization of chicken feather/PLA biocomposites

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Abstract In this work, thermal properties of composites from chicken feather fiber (CFF) and polylactic acid (PLA) were investigated. CFF/PLA green composites were manufactured by extrusion and injection molding. Short and long fibers (3 and 20 mm) were used at different contents (2, 5 and 10 mass%). The effects of fiber concentration and fiber length on thermal properties of CFF/PLA composites were studied. Thermal properties of CFF/PLA composites were examined through differential scanning calorimetry (DSC), thermogravimetric analysis (TG) and dynamic mechanical analysis (DMA). From the experiments, it was found that addition of CFF was effective to improve the thermal properties of PLA. The DSC results showed that heat flow increased with the increase in CFF content on the glass transition, on the crystallization temperature and on melting temperature. The TG results revealed that addition of CFF to PLA had positive effect on the thermal stability. In addition, the results of DMA experiments showed that the $\tan \delta$ decreased with the increasing CFF content, indicating less damping and more elastic behavior in the composites. The results obtained from this study provide important information on the temperature-dependent properties of CFF/PLA and lead to new product development based on natural resources.

Keywords PLA · Chicken feather · Discontinuous reinforcement · Thermal characterization · Injection molding

Introduction

Nowadays, to overcome the dependence on petroleum-based polymers and synthetic fibers, scientists have been studying on materials made of natural fibers and biopolymers, known as “green composites” [1, 2]. Green composites have attracted enormous attention due to humankind’s health anxiety, environmental awareness and depleted petroleum resources.

PLA (polylactic acid) is one of the most popular biopolymer and is highly investigated by researchers in many green composite studies [3] because of sustainability, biodegradability and biocompatibility. However, reinforcement is needed to improve the performance and to resolve shortcomings of PLA for practical applications. Plant-based fibers such as flax [4–6], jute [7], hemp [8], bamboo [9], kenaf [10, 11] and abaca [12] in PLA-based studies have been mostly seen as reinforcements in the literature. From the experimental study in [4], it was shown that the thermal properties of the PLA are improved with an addition of flax fibers. Suardana NPG et al. [7] studied experimentally the effect of diammonium phosphate on the flammability, thermal degradation and mechanical properties of coconut fiber and jute fiber biocomposites. It was concluded that jute reduces the mass loss rate of composites. Besides, Tokoro et al. [9] showed that bamboo fibers improve thermal properties and heat resistance of composites. Although there are a wide variety of studies on the performance of plant-based fibers in PLA, a few researchers have attempted to use animal-based fibers such

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as silk and chicken feather. Mechanical and thermal behaviors of PLA-reinforced silk fibers have studied recently [13–15]. As referred to those literatures, silk fibers decrease crystallization temperature, crystallization enthalpy and $\tan \delta$, while it increases storage modulus of silk/PLA composites.

Chicken feather fibers have been studied instead of traditional reinforcement materials in recent years because they can be easily and cheaply obtained from poultry wastes and have unique advantages such as low density, biodegradability, recyclability characteristics [16]. In previous studies, chicken feather was used as reinforcement for polyethylene [17, 18], polypropylene [19–21], chitosan–starch [22] and also polylactic acid (PLA) [23, 24]. It can be concluded from this literature search that chicken feather fibers have promising advantages in modifying mechanical and especially thermal properties of PLA. It was seen that chicken feather enhances the thermal stability and storage modulus of PLA. However, there have to be more studies to understand thermal properties of CFF/PLA composites. Because materials selection decisions for components exposed to temperature and providing use of them in different technological fields require information of the thermal responses of materials, thermal properties are critical in the practical utilization of materials, especially for innovative materials. The aim of this study is to determine the effects of chicken feather length and content on thermal properties of chicken feather/polylactic acid composites and investigate how CFF–PLA interfaces affect the thermal properties through SEM images. The results obtained from this study can be useful to develop new types of composites with environmentally beneficial properties.

Materials and methods

Materials and preparation of composites

Chicken feather fibers (CFF) were used in poly (lactic acid) (PLA) to fabricate composites. Chicken feather fibers, used as reinforcement, were supplied by a local company in Manisa/Turkey. Fibers in PLA were prepared by cutting barbs manually from rachises of whole chicken feathers (See Fig. 1). They were washed with hot water for sterilization and were kept in water for 24 h. Afterward, they were kept in an oven at 333 K for 6 h in order to dehumidify. Fibers in the length of 10–30 mm had the diameter of approximately 20–40 μm . The changes in length and diameter of fibers depend on the location of the barbs along the length of the rachis (See Fig. 1). As shown in Fig. 1, cross section of the barbs has the hollow cells which were gained unique properties to the composites such as relative

density, good thermal and acoustic insulations. PLA pellets, matrix material of composites, were purchased from Resinex BMY AS (Istanbul/Turkey). Type of PLA is NatureWorks 3052D and the density of this type is 1.24 g cm^{-3} .

In order to examine the effects of amount and length of CFF, specific ratios of PLA pellets and CFF were mixed with WiseStir HT-50AX mechanical mixer and fed into ThermoFisher Scientific EuroLab 16 XL twin-screw extruder. Screw speed was 150 rpm, and barrel temperature zone profile was adjusted to 438/448/458/468/478 K. After composites with the fiber mass content of 2, 5 and 10% were manufactured, composites were cut into pieces of approximately 3 and 20 mm lengths to obtain the extruded composites reinforced with short and long fibers, respectively. Following extrusion, composite specimens of (35 × 10 × 4) mm were molded for dynamic mechanical analysis by PERMAK injection molding machine. The barrel temperatures and pressure were 427/433/427 K and 147 bar, respectively.

Characterization methods

Differential scanning calorimetry (DSC)

DSC is a thermal analysis technique used to examine the thermal transitions such as crystallization and melting. In this study, curves of heat flow versus temperature (DSC curves) were obtained with a DSC-TA Q10 instrument. The glass transition temperature where the material undergoes softening, crystallization temperature where small amount of crystallization appears and melting temperatures where the existing crystalline component is destroyed were obtained for PLA and composites. The temperature was ramped at 283 K min^{-1} from 313 to 573 K under nitrogen atmosphere. Sample mass was approximately 6 mg.

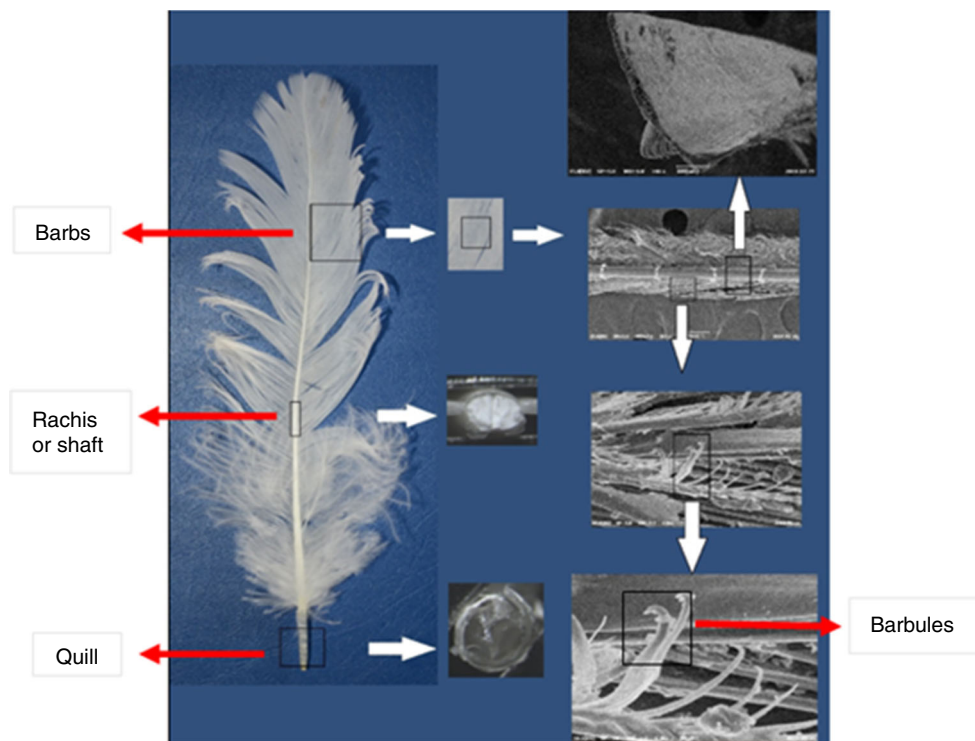
Thermogravimetric analysis (TG)

Thermal stability of both the pure PLA and CFF/PLA composites were observed by means of thermogravimetric analysis using PerkinElmer SII 7300TG/DTA under a nitrogen atmosphere at a flow rate of 20 mL min^{-1} . Temperature was set to start at 303 K and end at 773 K with the temperature scanning rate of 283 K min^{-1} . The samples of about 18–20 and 35–36 mg of composite and PLA were used in each test, respectively.

Dynamic mechanical analysis (DMA)

Storage modulus (E') and $\tan \delta$ curves which are used to determine stiffness and damping characteristics of

Fig. 1 A typical chicken feather fiber



PLA and CFF/PLA composites were obtained by way of dynamic mechanical analysis with DMA Q800 instrument. Beginning temperature was 308 K to be gradually increased up to 393 K with the increasing rate of 277 K min^{-1} . Analyses were made with a 3-point bending load which fluctuates as sinusoidal, and the oscillations of 1 Hz were selected. For bending test, the sample with 35 mm length, 10 mm width and 4 mm thickness was supported at both ends and force was applied in the middle.

Scanning electron microscope (SEM)

Morphology of the CFF/PLA composites was observed by a COX EM-30 scanning electron microscope at an accelerating voltage of 10 kV according to the E986 standard. The specimens were vacuum-coated with gold over a span of 2 min using argon in an ION COATER COX EM.

Results and discussion

The effects of fiber length and content on thermal properties of composites were investigated with DSC, TG and DMA tests.

Differential scanning calorimetry (DSC)

The thermal transitions generated with heat exchange were compared by using DSC results for PLA and CFF/PLA

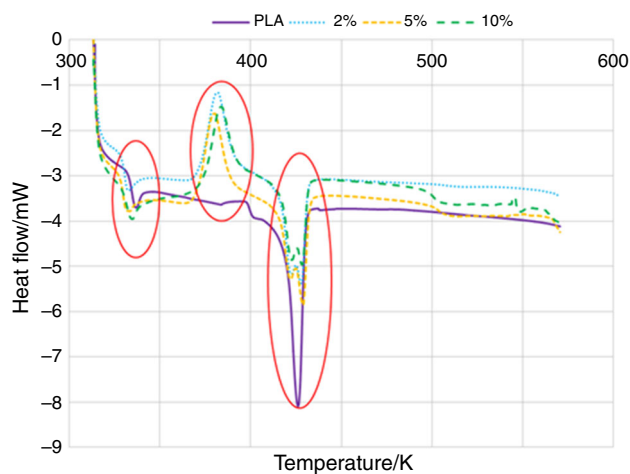


Fig. 2 DSC curves of PLA and CFF/PLA composites

composites. Figure 2 shows DSC curves (as a function of temperature) used to determine thermal properties such as glass transition (T_g), crystallization (T_c), melting temperatures (T_m), crystallization enthalpy (ΔH_c), melting enthalpy (ΔH_m) and crystallinity of CFF/PLA composites with mass content of around 2, 5 and 10%. Corresponding data are listed in Table 1. As seen from the curves, glass transition and melting peaks of PLA appear around 336 and 426 K that are consistent with the catalog values. Although the glass transition temperatures of composites are lower than the glass transition temperature of PLA with a maximum decrement of 7.3%, melting temperatures of composites

Table 1 DSC results for pure PLA and CFF/PLA composites

CFF content/mass%	T_g /K	T_c /K	ΔH_c /J/g	T_m /K	ΔH_m /J g ⁻¹	$X/\% = \frac{\Delta H_m}{\Delta H_{m0}} \times 100$
0	336.0	398.0	28.9	426.3	30.1	32.4
2	331.8	381.1	21.7	427.8	25.8	27.7
5	330.8	379.5	22.1	428.9	25.4	27.3
10	333.6	383.0	23.2	428.3	22.8	24.5

ΔH_{m0} = The melting enthalpy of the purely crystalline PLA which is 93 J/g [26]

which are around 428 K are higher than that of PLA with a maximum increment of 1.4%. Comparing the glass transition temperature of reinforced PLA composites (Table 2), sugar beet pulp reinforcement [25] and phormium tenax fibers [26] lead to a decrease in the glass transition temperature, while chopped glass, recycled newspaper [27], kenaf [28] and juvenile, mature and compression fibers [29] increase the glass transition temperature of PLA. The double melting peak in DSC curves of composites which is attributed to a lamellar rearrangement during crystallization is visible, in agreement with that observed during crystallization from the melt of PLA/CFF [24], PLA/Pinewood [30], PLA/phormium tenax fiber [26] and PLA/

oil palm empty fruit bunch fiber [31] composites. With the increasing of feather content, an increase in the values of T_g is seen, whereas there is no considerable change in T_m values [24, 29, 35]. Although the expected increase due to nucleating effect of CFF, crystallization temperatures (T_c) and crystallization enthalpy (ΔH_c) decreases with the presence of CFF from 398 to 479.5 K and from 28.91 to 21.7 J g⁻¹, respectively. This is in agreement with findings from the literature [14, 27, 32] in which the crystallization temperatures and crystallization enthalpy of the composites decrease with the addition of silk fiber, chopped glass, recycled newspaper and corn fiber into PLA. This is due to increasing of viscosity of the composites in the presence of

Table 2 Comparison of the thermal properties of PLA composites with different types of reinforcement fibers

Fiber	Fiber proportion/mass%	T_g /K	T_c /K	T_m /K	ΔH_m /J g ⁻¹	Source
Neat PLA	0	335.95	397.95	426.25	30.1	Present
	0	333.35	387.15	439.65	35.4	[14]
	0	331.85	386.05	436.55	43.2	[24]
	0	331.29	–	424.52	–	[25]
	0	333.85	405.35	426.45	–	[26]
	0	327.15	369.15	445.15	47.9	[27]
	0	331.39	389.05	425.15	–	[28]
	0	319.15	–	423.15	17.1	[29]
	0	–	381.85	442.85	37.1	[30]
	0	329.05	393.25	424.95	30.1	[35]
CFF	10	333.55	382.95	428.25	22.8	Present
	10	330.65	376.05	439.85	46.9	[24]
Silk	5	337.55	373.65	441.55	35.7	[14]
Sugar beet pulp	7	331.23	402.68	425.02	–	[25]
Phormium tenax	20	332.35	392.95	424.05	23.0	[26]
Chopped glass	30	330.15	364.15	443.15	41.7	[27]
Recycled newspaper	30	329.15	363.15	443.15	44.3	[27]
Kenaf	20	332.48	392.61	426.21	–	[28]
Juvenile wood fiber	25.6	327.15	–	424.15	21.5	[29]
Mature wood fiber	9.3	325.15	–	423.15	18.7	[29]
Compression wood fiber	18	326.15	–	423.15	20.9	[29]
Pine wood flour	20	–	367.85	441.65	32.7	[30]
Oil palm	30	329.65	392.15	413.25	–	[31]
Corn fiber	20	320.65	376.15	–	28.3	[32]
Hemp	10	329.45	392.35	425.05	28.3	[35]

CFF, which hindered the migration and diffusion of PLA molecular chains in the composite [14]. This result is not in agreement with the findings of Cheng et al. [24] who reported that the values of ΔH_c increase with the presence of CFF which plays the role of nucleating agent. In addition, melting enthalpy (ΔH_m) for PLA is around 30.1 J g^{-1} , whereas ΔH_m for composites decreases to 22.8 J g^{-1} with the addition of CFF. In other words, the degree of crystallinity defined as the fraction of the crystalline phase in the total mass decreases. The similar finding was reported by [9] when using bamboo as reinforcement in PLA. Decreased crystallinity is associated with decrease in mechanical properties such as rigidity and tensile strength. This result agrees with literature reference reported by Baba et al. [33]. A detailed comparison of the thermal properties found from DSC measurements for PLA composites with different types of reinforcement fibers is given in Table 2.

Thermogravimetric analysis (TG)

Thermal stability which has critical role in final performance of composites was evaluated by thermogravimetric analysis. The thermogravimetric (TG) and derivative thermogravimetric (DTG) based on the TG data curves are used to determine mass loss and any change in the rate of mass loss of materials, respectively. The TG and DTG curves as a function of temperature of pure PLA, CFF and composites with various fiber contents are shown in Figs. 3–4, respectively. In addition, TG-related data of these materials are listed in Table 3. From the table, the temperature range of thermal degradation (T_{range}) and loss of mass in these ranges, the temperature at which rate of degradation was highest (T_{peak}), the maximum mass loss rate at T_{peak} (R_{peak}) and residual mass at 723 K can be seen.

TG curves contain horizontal and curved portion indicating constant mass and the rate of mass loss, respectively. As shown in Fig. 3, four zones are seen during the thermal process for chicken feather fiber. The initial zone from 303 to 373 K corresponds to removal of moisture with a total

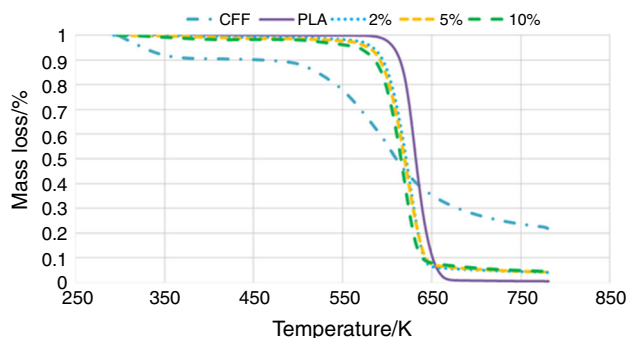


Fig. 3 TG curves of CFF, PLA and CFF/PLA composites

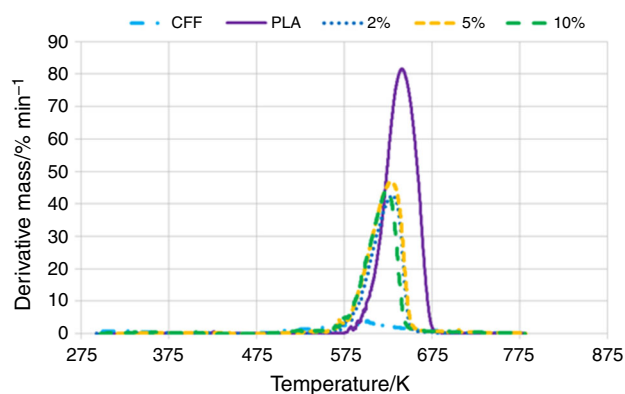


Fig. 4 DTG curves for CFF, PLA and CFF/PLA composites

mass loss of about 10%. This initial zone is followed by the second zone from 373 to 498 K without thermal degradation. At third zone (503–683 K), chicken feather is thermally degraded (mass loss around of 70%). This zone which shows rapid thermal degradation can be followed from DTG curve in Fig. 4. The DTG curve of CFF shows an initial peak associated with loss of moisture at 323 K. After this small peak, the curve shows single peak of maximum mass change at 601 K. After 723 K, chicken feather is degraded and mass percentage gradually decreases (mass loss up to 78%).

TG of PLA shows one step of degradation represented by a single peak. Thermal decomposition of PLA starts at about 583 K and completes above 683 K with a total degradation of 99.4%. The DTG curve of PLA shows one peak at 640 K. In other word, the maximum change in the rate of mass loss is observed at this temperature. The sample mass remains almost constant after 683 K. The decomposition range of PLA is smaller than that of CFF. In addition, while CFF leaves residue of 25.63%, a small amount of residue remains for PLA at the same temperature.

CFF/PLA composites have one-step decomposition pattern which is similar to that of PLA (Fig. 3). The smooth TG curves correspond to the homogenous loss mass behavior which indicates homogenous structure of composites. Moisture trapped in composites is evaporated between 303 and 553 K. Through this range, the mass losses are less than 5%. Beyond 553 K, composites begin to degrade. Composites drastically degrade with prominent mass loss of about 90% at temperatures of 553 and 658 K. After 723 K, mass loss remains almost constant. Mass loss of composites does not reach up to 100% due to existence of chicken feather in composites. The residual mass changes between 4.7 and 5.4% due to content of CFF in composites. Although not very significant, chicken feather content affects thermal degradation of composites in all stages. During elimination of moisture, the mass

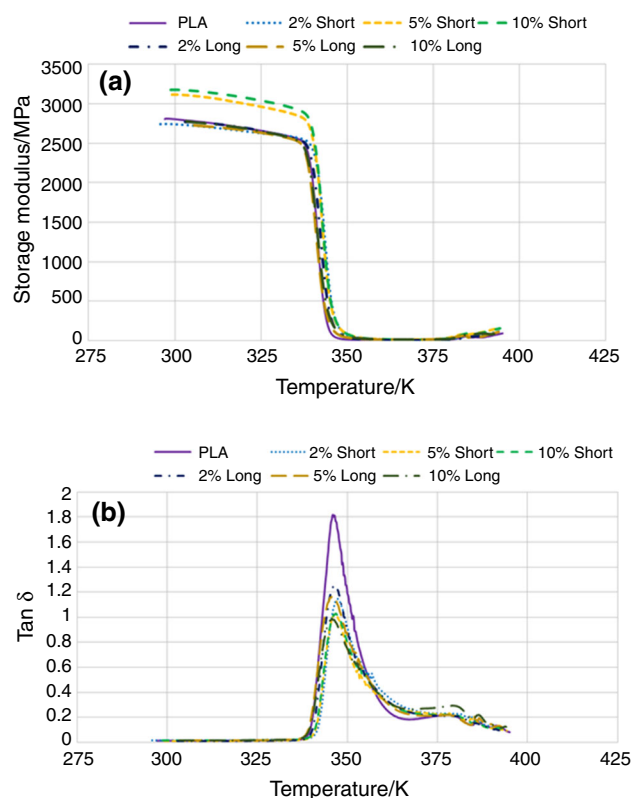
Table 3 TG and DTG results for CFF, pure PLA and CFF/PLA composites

Samples	T_{range}/K TG	Loss of mass/% TG	T_{peak}/K DTG	$R_{\text{peak}}/\% \text{ min}^{-1}$ DTG	Residual mass at 723 K/%
PLA	583–683	98.4	640	81.5	0.7
CFF	498–723	63.2	601	3.9	25.6
%2 CFF/PLA	564–652	91.5	629	42.5	4.7
%5 CFF/PLA	557–654	90.5	628	46.7	4.8
%10 CFF/PLA	555–649	87.8	624	43.9	5.4

percentage of composites decreases with the increasing chicken feather content in composites. However, onset degradation temperature of the composites is shifted to lower temperature than that of PLA (from 583 to 555 K) but to higher temperature than that of CFF (from 498 to 555 K). This information is important to identify the processing limits and operating temperatures of composites. In case of decomposition, residual mass increases with the increasing chicken feather content. Decomposition of CFF/PLA composites occurs with lower peak indicating slower decomposition rate compared with PLA (See Fig. 4). Decomposition peaks (the fastest mass change) of CFF/PLA composites having 2, 5 and 10% of CFF are at 629, 628 and 624 K, respectively. Addition of 10% CFF decreases the thermal decomposition peak of PLA from 640 to 624 K. This decrement in the thermal decomposition peaks is in agreement with findings of Porras et al. [34], Masirek et al. [35], Goriparthi et al. [36] and Rosa et al. [26] that the *Manicaria Saccifera* palm, hemp fibers, jute fibers and phormium tenax fibers were used as reinforcement in the PLA, respectively. As can be seen, composites possess lower degradation temperature at maximum mass loss than PLA due to the degradation of CFF at lower temperature. However, residual mass of composites increases as the amount of CFF increases in the temperature range above 653 K. The higher residual mass indicates enhanced thermal stability of composites in comparison with PLA.

Dynamic mechanical analysis (DMA)

Storage modulus (E') and tan delta ($\tan \delta$) curves of PLA and CFF/PLA composites were evaluated by DMA. In addition, the effects of the fiber length and content on the thermal properties of composites were determined. Storage modulus related to elastic response of materials is a measurement of stored energy during the deformation, while $\tan \delta$ providing information on the relationship between the elastic and inelastic components is a measurement of energy dissipation. Figure 5 shows the variations of storage modulus and $\tan \delta$ as a function of temperature for PLA and CFF/PLA composites containing 2, 5 and 10% of CFF

**Fig. 5** a Storage modulus and b Tan delta curves of PLA and CFF/PLA composites

(short and long). The corresponding data are also listed in Table 4.

Figure 5 shows that thermal properties of PLA are improved with adding CFF. As shown in the storage modulus curves, it is obvious that the storage modulus decreases with the increasing temperature, as expected (See Fig. 5a). In particular, the stiffness drops rapidly between 336 and 346 K temperatures which are the glass transition temperatures as it is seen in the results of DSC. At this temperature range, the mobility of the polymer chains increases significantly and stiffness gradually diminishes to zero. The temperature of PLA where storage modulus sharply decreases is about 336 K. When CFF are added into PLA, the temperature slightly shifts to 339 K. Over the whole range of testing temperature, the addition of CFF

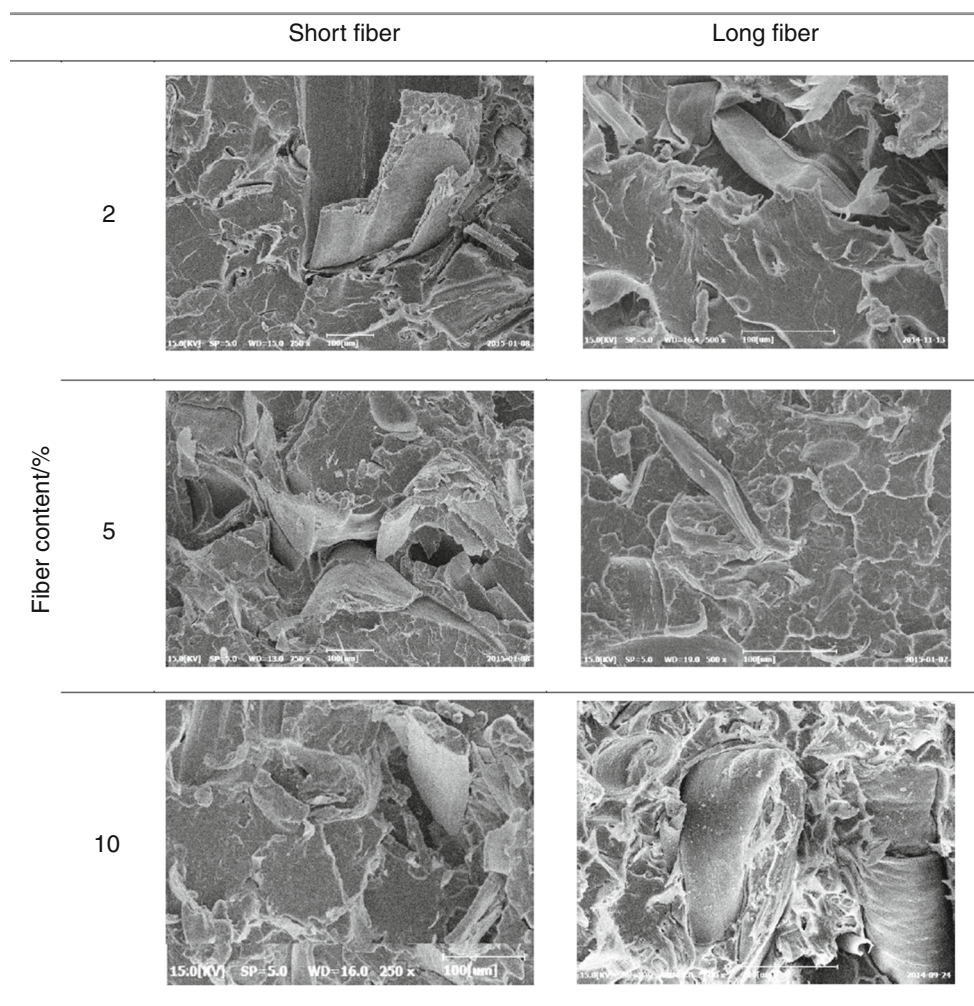
Table 4 DMA results for pure PLA and CFF/PLA composites

CFF Content/mass%	E' at 313 K/MPa	Onset temperature of the E' drop/K	Peak $\tan \delta$	Temperature at peak $\tan \delta$ /K
0	2739	338.9	1.81	346.02
2-SF	2690	340.5	1.14	347.40
2-LF	2734	339.5	1.25	346.36
5-SF	3055	339.5	1.02	346.53
5-LF	2695	337.9	1.16	345.85
10-SF	3125	339.6	1.03	346.94
10-LF	2726	338.3	0.98	345.82

SF short fiber, LF long fiber

also increases the storage modulus with the highest value for PLA/10% short CFF. For example, at 313 K, the storage modulus of PLA is 2739 MPa, while the same for PLA/10% short CFF is 3125 MPa. In other words, storage modulus of composites including 10% short CFF content improves 14% in comparison with PLA. This can be attributed that chicken feather fibers embedded in

composites reduce the strain on loading. Increasing fiber content also reduces mobility and deformation of material for high concentration; therefore, the stiffness increases. Unexpectedly, in the case of adding long CFF into PLA, there are no significant changes at temperatures and corresponding storage modulus in comparison with that of PLA. In addition, a slight change in storage modulus for

**Fig. 6** SEM images of CFF/PLA composites

composite with 2% short CFF reinforcement is obtained with respect to the PLA. These results may originate from the manufacturing process where the long fibers are chopped and degraded in extruder and molding. Further, these results can be a consequence of inadequate wetting of fibers by PLA and curling of fibers due to the increasing long CFF loading. From the SEM images for composites having the different lengths of CFF for the same fiber content in Fig. 6, curling of long fibers and gap between matrix and fibers is clearly seen.

In Fig. 5b, $\tan \delta$ curves are shown; when CFF were added into PLA, the thermal transition peaks are shifted to higher temperature, although not very significant. However, $\tan \delta$ values decrease as adding of CFF. $\tan \delta$ known as damping occurs in the glass transition region where molecules start moving. As a result of restricting the movement of molecules due to the existence of CFF, damping decreases. The decreasing $\tan \delta$ indicates that composites have more energy storage potential rather than dissipating it. The energy dissipation decreases 43% at 348 K for the composites having 10% long CFF. Further, increasing of the fiber content in the composites also decreases the $\tan \delta$ from 1.25 to 0.98 (as shown in Fig. 5b). This case shows that the composites with the high CFF content have better energy absorption characteristics than the others with low CFF content.

Conclusions

In this study, PLA composites reinforced with chicken feather fiber were prepared with a twin-screw extruder and an injection molding machine. DSC, TG and DMA tests were carried out to investigate the role of fiber content and fiber length on thermal properties and to evaluate the potential of CFF/PLA green composites. From the present experimental study, the following conclusions can be made.

1. DSC test results show thermal transitions of composites are affected by increasing chicken feather content. The addition of CFF as a reinforcement in PLA decreases glass transition temperature, whereas glass transition temperature increases with the increase in CFF content. Besides, the melting temperature of composites increases slightly (approximately 1.4%), while the crystallinity of PLA in composites decreases with the increase in CFF due to increasing of viscosity of composites.
2. From the TG results, mass loss decreases when the fiber content increases in case of degradation. Although increasing CFF content decreases the temperature at which rate of degradation is highest, it causes increasing of residual mass which indicates enhanced thermal stability.
3. DMA test results reveal that the storage modulus (stiffness) of composites is improved and the damping (energy dissipation) is lowered with addition of CFF in PLA. This change, associated with decreasing mobility of polymer chain, shows that CFF/PLA composites have more elastic behavior and less damping. However, the effects of long fibers on storage modulus are not apparent when compared with short fibers.

The results in this study show that chicken feather fiber is a promising material to improve thermal properties of biopolymers, like PLA. However, further investigations are needed to find optimum fiber content and fiber length, to improve fiber-matrix adhesion and fiber dispersion and also to implement different manufacturing process to avoid fiber degradation.

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