SCIENCE CHINA
Technological Sciences

• Article •

November 2017 Vol.60 No.11:1716–1723
doi: 10.1007/s11431-016-9009-1

Enhanced mechanical properties and flame retardancy of short jute fiber/poly(lactic acid) composites with phosphorus-based compound

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Received October 24, 2016; accepted February 6, 2017; published online July 14, 2017

In this work, a phosphorous-based compound (DOPO-ICN) was obtained by a two-step process. 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) reacted with formaldehyde firstly, followed with reacting with 1,6-hexane diisocyanate (HDI). The chemical structure of DOPO-ICN was confirmed by Fourier transform infrared spectroscopy (FTIR) and ^{1}H NMR. The influence of DOPO-ICN on the mechanical and flammability properties of jute/PLA composites was studied. Compared to DOPO, DOPO-ICN improved the tensile, flexural and impact strength of the flame retardant jute/PLA composites. Moreover, the flammability of jute/PLA composites with different DOPO and DOPO-MA loading was investigated by thermogravimetric analysis (TGA), UL 94 test and limiting oxygen index (LOI) measurements. The results showed that DOPO-ICN imparted the flame retardancy to the jute/PLA composites.

phosphorous-based flame retardant, poly(lactic acid), jute, composite, mechanical properties, flame retardancy

Citation: Yu T, Ding D, Sheng C, et al. Enhanced mechanical properties and flame retardancy of short jute fiber/poly(lactic acid) composites with phosphorus-based compound. Sci China Tech Sci, 2017, 60: 1716–1723, doi: 10.1007/s11431-016-9009-1

1 Introduction

With the wide utilization of synthetic fiber reinforced polymer composites, the disposal of the composites is seen as an important environmental problem [1,2]. Nowadays, the materials extracted from natural resources such as biopolymers and plant fibers have shown considerable potential to replace the traditional synthetic fibers and polymers and use in automotive components and aerospace industry [3–5]. The most commonly preferred plant fiber, that is, jute fiber possesses good sound absorption, low density, no health risks and is inexpensive, commercially available in the required form and has higher strength and modulus than plastic [6,7]. Poly(lactic acid) (PLA) is a versatile thermoplastic produced from lactic acid monomer mainly deriving from the fermentation of corn, sugar beets, etc. [8,9]. Plant fiber reinforced PLA composites have been good candidates to be used in electronics, transportation or construction due to considerable mechanical properties and environmental advantages [10,11]. However, the inherent flammability and high smoke emission of plant fibers and polymers restrict their applications.

The methods to reduce the flammability of PLA have been attempted recently. The eco-friendly flame retardant including nano inorganic, phosphorus-based, intumescent flame retardants and new effective flame retardant system have been utilized as flame retardant agent for PLA [12–16]. Furthermore, several works have been reported on improving the flame retardancy of plant fiber reinforced PLA composites. Woo and Cho [17] found that the flame retardancy of kenaf/PLA composites could obtain the improvement of 66% by using aluminum trihydroxide (ATH) as flame retardant. Yang et al. [18] reported that for basalt fiber (BF)/PLA con-

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taining 19 wt% aluminum hypophosphite (AHP) and 1 wt% modified carbon nanotube (m-CNT), it achieved a V-0 classification in UL 94 testing with a high limited LOI (31%).

For plant fiber/PLA composites, the polar fibers have inherently lower compatibility with less polar PLA matrix, and the incompatibility may influence the processing and the properties of the composites. Many reports focused on using different physical and chemical methods include fiber treatments techniques such as acylation, alkali treatment, etherification, isocyanate treatment and use of coupling agents to improve interfacial bonding properties of plant fiber reinforced PLA composites [19–21]. Furthermore, the mechanical properties of the composites are usually seriously affect by the flame retardant which are due to that the compatibility between the fiber and polymer became worsen by the introduction of flame retardant. Shukor et al. [22] reported that ammonium polyphosphate (APP) was effective in improving flame retardancy properties, however, addition of APP decreased the compatibility between PLA and kenaf fiber resulting in significant reduction on the mechanical properties of PLA biocomposites. Therefore, it is a challenge to improve the flame retardancy and the mechanical properties simultaneously of the fiber/polymer composites. Phosphorus-containing monomers containing reactive function groups, such as hydroxyl and carboxyl, adhered or grafted on the fiber and molecular chain of polymer matrix which could be a good solution for solving the problem.

In this work, DOPO with hydroxyl group (DOPO-OH) was synthesized by the reaction between DOPO and formaldehyde, and the structure of DOPO-OH was characterized by Fourier transform infrared spectroscopy (FTIR) and ^{1}H NMR. Since isocyanates can react with hydroxyl or carboxyl groups to form the stable chemical bond [21], the isoyanate group of diisocyanate reacted with free hydroxyl group of DOPO-OH, resulting in the *in situ* formation of isocyanate group on DOPO. Free isocyanate group can then react with the hydroxyl end group of jute or PLA, and produce urethane bonds. The reaction equations are shown as Figure 1. Flame retardant jute/PLA composites were prepared by twin-screw extruder with DOPO-OH as flame retardant additive agent and 1,6-hexane diisocyanate (HDI) as compatibilizer. The mechanical properties and flame retardancy of flame retardant jute/PLA composites were investigated.

2 Experimental

2.1 Materials

The materials used were obtained from commercial supplier. PLA (NatureWorks® 4032D, M_w=140000, M_w/M_n=1.7) was purchased by NatureWorks Co. Ltd., UK. Jute fiber yarn was supplied by Shanghai Qiancong Jute fiber Co. Ltd., China. DOPO, commercial grade, was obtained from Huizhou Sunstar Technology Co. Ltd., China. 1,6-hexane diisocyanate (HDI), ethanol and formaldehyde were supplied by Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China).

2.2 Synthesis DOPO-OH

DOPO was dried to remove water at 100°C for 2 h before use, sine a certain amount of hydrated DOPO was usually found in the reagent. DOPO and ethanol were placed in a four-necked round bottom flask equipped with a stirrer. The solvent was heated to 70°C, and formaldehyde was added slowly while stirring refluxed at 70°C for 24 h. After reaction, the remained DOPO-OH was filtered and dried in vacuum at 50°C overnight.

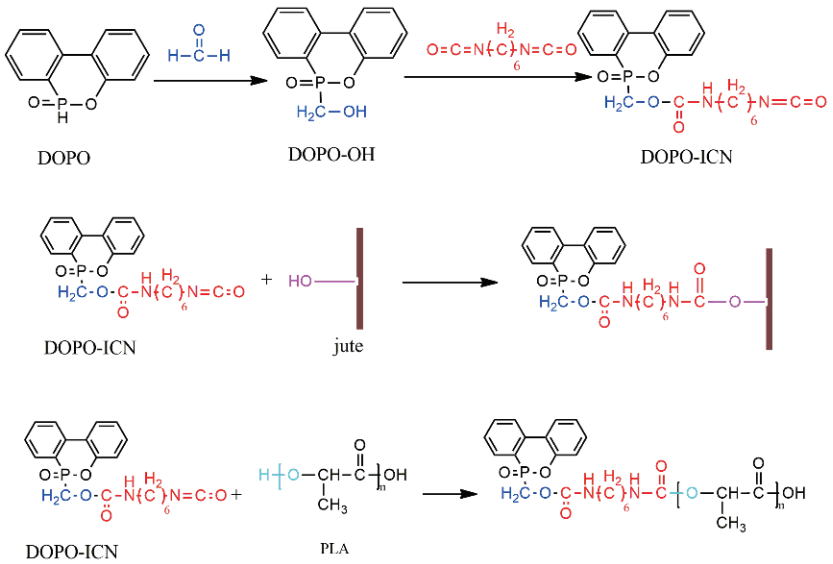

Figure 1 (Color online) Proposed chemical reaction among DOPO-OH, jute, PLA and diisocyanates.

2.3 Preparation of composites

The preparation route of flame retardant jute/PLA composites was illustrated in Figure 2. Before processing, PLA, jute yarn and DOPO-OH were dried at 60°C in vacuum for 24 h to remove the moisture content in order to avoid possible degradation. The dried PLA and DOPO-OH were firstly well mixed by a high speed mixer, then blended with jute yarn and HDI in a co-rotating twin-screw extruder (20 mm, L/D=40; Nanjing Jieya, China) at 80 r/min with the operating temperatures in the range of 155–170°C. Jute yarn was cut by the screws into about 1 mm in average length, and the diameter of jute fiber was 5–10 μm. The extrudate was cooled in a water bath and cut into granules. And then the granules were collected and dried in a vacuum oven at 60°C for 24 h before further processing. The formulations of the composites are presented in Table 1. Test specimens for the mechanical properties testing were obtained by using the injection moulding machine (Wuxi Haitian Machinery Co., Ltd., China) according to the standard. The processing temperature was varying from 170 to 190°C, and the mold temperature was from 40 to 50°C.

2.4 Characterization

2.4.1 FTIR

FTIR spectra were recorded on an EQUINOX 55 spectrometer using compression molded film samples at a range of 400–4000 cm^{-1}.

2.4.2 ^1H NMR

The ^{1}H-NMR spectra were determined by a Bruker AVANCE III HD 400MHz NMR. Deuterated chloroform ($CDCl_3$) was taken as solvent and tetramethylsilane (TMS) was taken as internal standard.

2.4.3 Mechanical properties

Determination of tensile and flexural properties was carried out with a computer controlled mechanical instrument (ETM-5040, Shenzhen electromechanical universal testing instrument Co. Ltd., China) according to GB 13022-91 and GB 1449-83 respectively. A crosshead speed of 2 mm/min was used. The test of Izod impact was performed by Notched Izod impact instrument (ZBC-1400-2 test machine, Shenzhen Sans Test Instruments Ltd., China) accordance to GB 1451-83. At least five specimens were used for each test.

2.4.4 Scanning electron microscopy

The fracture surfaces from the impact tests were examined by means of a VEGA TS 5136MM (TESCAN, Czech) scanning electron microscopy (SEM) with the accelerating voltage of 20 kV to observe the impact morphology. The specimens were gold coated before SEM examination.

2.4.5 Burning test

Limiting oxygen index (LOI) values were measured with an LOI instrument (HC-3 Analytical Instrument Factory, China)

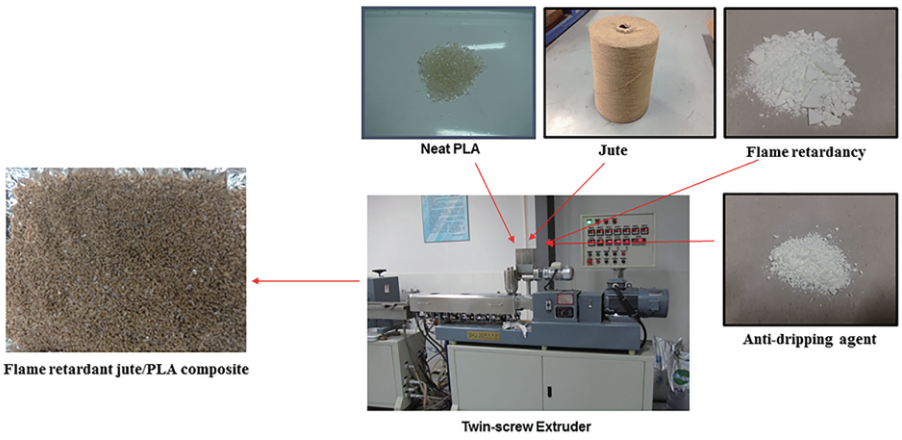

Figure 2 (Color online) Schematic illustration for the preparation of flame retardant jute/PLA composites.

Table 1 Formulations of jute/PLA composites

Samples ID	PLA (wt%)	Jute (wt%)	DOPO (wt%)	HDI (wt%)	DOPO-OH (wt%)
Jute/PLA	85	15	–	–	–
Jute/PLA/3DOPO	82	15	3	–	–
Jute/PLA/5DOPO	80	15	5	–	–
Jute/PLA/7DOPO	78	15	7	–	–
Jute/PLA/3DOPO-ICN	81	15	–	1	3
Jute/PLA/5DOPO-ICN	79	15	–	1	5
Jute/PLA/7DOPO-ICN	77	15	–	1	7

according to GB 2406-82 (China) with test specimen bars (100×6.5×3 mm). The UL-94 vertical burning test was used to test the ease of the ignition of polymeric materials using small burner. All of the bar specimens (125 mm×13 mm×3 mm) were tested by vertical burning test instrument (WC 5400) (Kunshan Wancheng Analytical Instrument Co., China) according to ASTMD 3801 UL-94 standard.

2.4.6 Thermogravimetric analysis

Thermogravimetric analysis (TGA) was determined in a thermogravimetric analyzer (NETZSCH TA449C Jupiter, Germany) at a heating rate of 20°C/min from 30 to 600°C under the presence of nitrogen (80 mL/min). About 5 mg sample was used in each test.

3 Results and discussion

3.1 The characterizations of DOPO-OH

FTIR analysis of DOPO and DOPO-OH was presented in Figure 3. In the spectrum of DOPO, the characteristic absorption peak of DOPO at 2385 cm^{-1} (P-H stretching vibration), 956 and 1149 cm^{-1} (P-O-Ph vibration), 1585 and 1450 cm^{-1} (P-Ph stretching vibration), and at 1209 cm^{-1} (P=O stretching vibration) were found. Furthermore, the band at 3345 cm^{-1} was attributed to the presence of hydroxyl groups introduced by atmospheric moisture. After reaction, it was found that the absorption peak at 2385 cm^{-1} for P-H stretching vibration disappeared from the spectrum of DOPO-CH_2OH, and a new absorption peak at 2892 cm^{-1} for C-H stretching vibration appeared, which clearly indicated that DOPO-OH was achieved.

The above chemical structure was further confirmed by ^{1}H-NMR spectra, which was shown in Figure 4. The chemical shifts at 7.18–8.06 ppm can be found in both spectra which were attributed to the aromatic hydrogen. For DOPO, the chemical shift at 8.80 ppm was assigned to the hydrogen on the –O-P-H group. However, for DOPO-OH, the peak at 8.80 ppm disappeared, and the peaks at 4.30 and 2.81 ppm appeared which were attributed to the hydrogen on the CH_2 group and OH group, respectively. According to the characterizations above, DOPO-OH was successfully synthesized.

3.2 Mechanical properties

The tensile properties of jute/PLA composites with different loading of DOPO and DOPO-ICN are shown in Figure 5. From Figure 5, the untreated jute/PLA composite has a tensile strength of 47.99 MPa and a tensile modulus of 4.8 GPa. The tensile strength and tensile modulus of the jute/PLA composites showed gradual decline with the increased DOPO loading. The similar results were found by Brehme et al. [23]. The decrease in tensile strength as the flame retardant agent acted as a matrix discontinuity and stress was concentrated

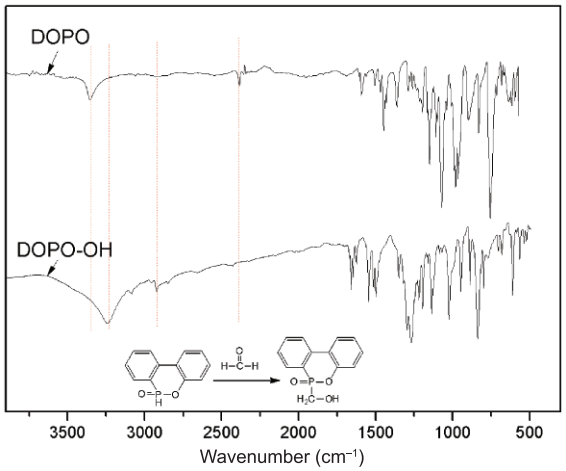

Figure 3 (Color online) FTIR spectra of DOPO and DOPO-OH.

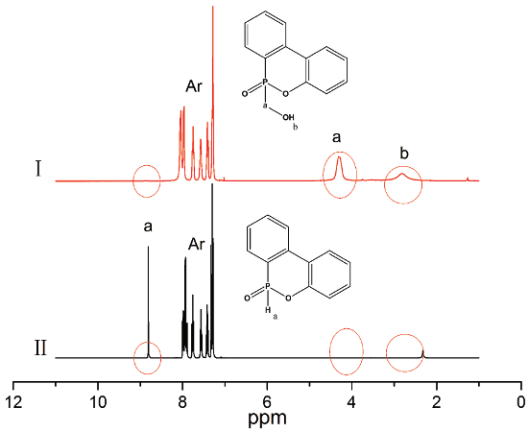

Figure 4 (Color online) ^{1}H-NMR spectra of DOPO (I) and DOPO-OH (II).

in this region, leading to premature failure. Moreover, the well-dispersed states of the flame retardant in polymer matrix and the good interfacial interaction between two components were key factors [24]. However, the tensile properties of jute/PLA composites with DOPO-ICN showed a significantly different trend. Adding 3% DOPO-ICN decreased the tensile strength and tensile modulus of jute/PLA composites. Then the tensile strength and tensile modulus increased by further adding DOPO-ICN. The increased tensile strength and Young's modulus were mainly due to the well disperse of flame retardant agent and the well interface between two components.

Flexural strength and flexural modulus of jute/PLA composites with different loading of DOPO and DOPO-ICN are shown in Figure 6. It was found that flexural strength and flexural modulus of jute/PLA composites decreased with the increased DOPO loading. However, the increase in flexural strength and almost no significant reduction in flexural modulus were observed for the composites with the increased DOPO-ICN loading. These reasons were consistent with the tensile properties described previously.

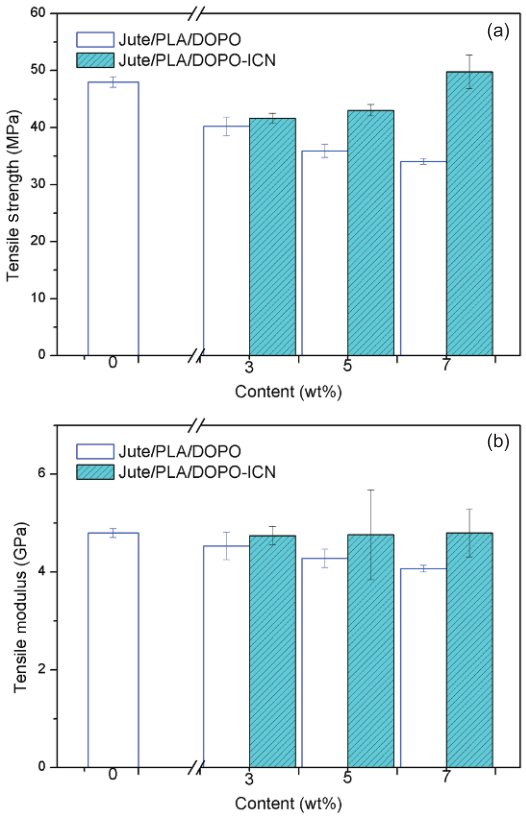

Figure 5 (Color online) Tensile properties of flame retardant jute/PLA composites. (a) Tensile strength; (b) tensile modulus.

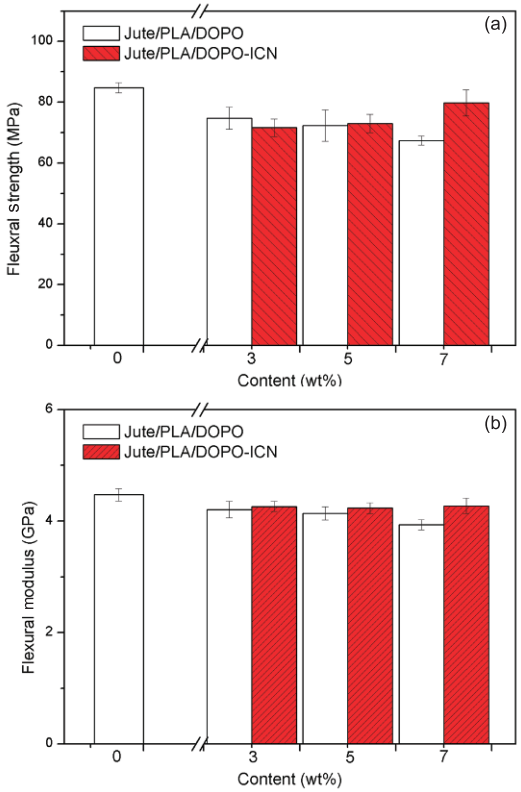

Figure 6 (Color online) Flexural properties of flame retardant jute/PLA composites. (a) Flexural strength; (b) flexural modulus.

Figure 7 shows impact strength of jute/PLA composites with different loading of DOPO and DOPO-ICN. From Figure 7, it can be found that the impact strength of the composites decreased with the increased DOPO loading, but increase with the DOPO-ICN loading. It was due to that DOPO after modification provided effective resistance to crack propagation during impact test.

3.3 Morphologies

The SEM morphologies of the fracture surfaces of jute/PLA composites with 7 wt% DOPO and DOPO-ICN from impact tests were shown in Figure 8. From Figure 8(a), the compatibility of the jute and the PLA matrix with adding DOPO, as evidenced by the presence of the voids and exposed fibers on the fracture surface. Furthermore, the compatibility between the jute and PLA matrix improved when the DOPO-ICN added, as seen in Figure 8(b). The gaps between the fiber and the matrix became small, which was evidence that DOPO-ICN had played the role of the compatibilizer between the fiber and the polymer matrix, leading to the better adhesion. The improved interface bonding strength between jute and PLA could be attributed to the formation of urethane bonds between the hydroxyl groups of jute or PLA and the terminal isocyanate groups of DOPO. The results were consistent with the mechanical properties.

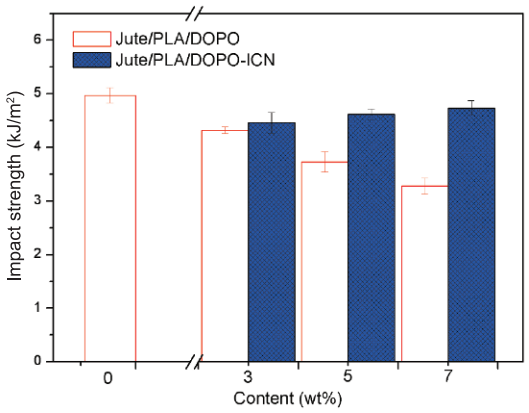

Figure 7 (Color online) Notched Izod impact strength of flame retardant jute/PLA composites.

3.4 Flammability

The flammability of jute/PLA composites without and with DOPO and DOPO-ICN was evaluated by LOI measurements and the UL 94 tests, and the results are shown in Table 2. Jute/PLA composite without flame retardant was highly flammable and completely consumed with serious dripping. The UL 94 vertical test of the composite classed no rating, and LOI value was only 21.6%. When 3 wt% DOPO and DOPO-ICN were added, the composites passed the UL-94

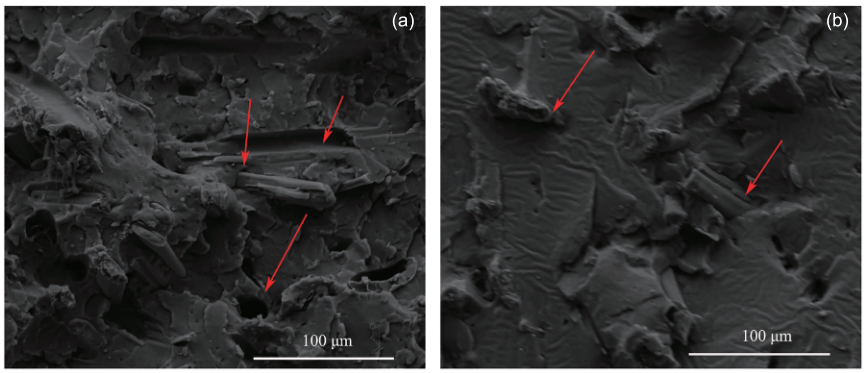

Figure 8 (Color online) SEM micrographs of fracture surface of notched Izod impact specimen. (a) Jute/PLA/7DOPO; (b) jute/PLA/7DOPO-ICN.

Table 2 Combustion parameters obtained from LOI and UL-94 test[a)]

Sample ID	LOI (%)	Dripping	UL-94
Jute/PLA	21.6	Y	NC
Jute/PLA/3DOPO	25.2	Y	V-1
Jute/PLA/5DOPO	27.3	Y	V-0
Jute/PLA/7DOPO	28.1	Y	V-0
Jute/PLA/3DOPO-ICN	25.5	Y	V-2
Jute/PLA/5DOPO-ICN	27.0	Y	V-0
Jute/PLA/7DOPO-ICN	28.3	Y	V-0

a) Fire not extinguished after ignition; NC, not classified by UL-94 rating.

V-1 rating and V-2 rating in the vertical burning test, and the LOI values were increased to 25.2% and 25.5%, respectively. With the further increase of the loading content of flame retardant, the LOI values continued to increase. The vertical burning test results showed that all of the jute/PLA composites with DOPO and DOPO-ICN could reach UL-94 V-0 rating when the loading content of flame retardant was more than 5 wt%, although little dripping could be found. Furthermore, the LOI value of the composite with 7 wt% DOPO-ICN was higher than that of the composite with 7 wt% DOPO, which indicated that DOPO-ICN could enhance the mechanical properties and the flame retardancy of jute/PLA composites simultaneously.

Figure 9 shows jute/PLA composite with 7 wt% DOPO and DOPO-ICN after vertical burning test. The residue char as a protective shield and thermal barrier could be observed on the surfaces of the composites with DOPO and DOPO-ICN, respectively. Compared to the composites with DOPO, the dripping of the composites with DOPO-ICN were lightened, which was due to the char. The fact revealed that DOPO-ICN showed the outstanding performance in promoting the yield of char. The mechanism of DOPO-ICN in reducing the flammability of the composites was probably attributed to that the better interface between flame retardant agent and fiber or matrix created a barrier which could slow down the heat and mass transfer between gas and condensed phases, and protect the inner materials.

Figure 9 (Color online) Digital photos of the specimens after vertical burning test. (a) Jute/PLA/7DOPO; (b) jute/PLA/7DOPO-ICN.

3.5 Thermal properties

Figure 10 shows the TGA curves of jute/PLA composites without and with 7 wt% DOPO and DOPO-ICN under nitrogen. The relative thermal stability of the samples was evaluated by the temperature of the 5%, 50% and maximum weight loss (T_5, T_{50} and T_{max}) and the percent char yield at 600°C, and the data are listed in Table 3. T_5, T_{50} and T_{max} of jute/PLA composite appeared at 315, 354 and 381°C, respectively. Compared to jute/PLA, T_5 of the composites decreased with the loading of DOPO due to the early degradation of the flame retardant. The residue char of jute/PLA with DOPO was higher than that of jute/PLA, which revealed that DOPO present a effective char forming ability. Furthermore, compared to the composites with DOPO, the composites showed

Table 3 TGA characterization of the samples

Samples	T_5(°C)	T_{50}(°C)	T_{max}(°C)	Residue (600℃) (%)
Jute/PLA	315	354	381	1.41
Jute//PLA/7DOPO	287	358	396	5.58
Jute/PLA/7DOPO-ICN	315	367	392	5.42

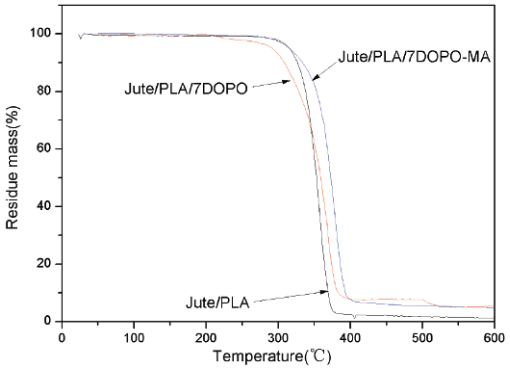

Figure 10 (Color online) TGA curves of jute/PLA, jute/PLA/7DOPO, and jute/PLA/7DOPO-ICN.

higher T_5 with the loading of DOPO-ICN, which was attributed to the relatively high temperature of the degradation of DOPO-ICN. Simultaneously, compared to DOPO, the residual weight was improved by the addition of DOPO-ICN. The TGA results revealed that DOPO-ICN could enhance flame retardancy by increasing the char yield.

4 Conclusions

The DOPO-based compound was synthesized in this study. When jute/PLA composite incorporated only 5 wt% DOPO-ICN, the LOI value and UL-94 of the rating can reach 27% and V-0 level. The flame retardant mechanism of DOPO-ICN was mainly related to that the addition of DOPO-ICN led to the formation of the char layer to protect the inner composites from further burning. The mechanical properties results showed that with the incorporation of DOPO-ICN, the improvement in tensile, flexural and impact strength was observed compared to those of DOPO. The face was ascribed to the DOPO-based compound provided the active sites linked between DOPO and PLA or jute, which ensured the good interface. The approach described herein could provide a promising solution in the development of an efficient flame retardant which can enhance the mechanical properties and the flame retardancy of jute/PLA composites simultaneously.

This work was supported by the Ministry of Industry and Information Technology of China (Grant No. MJ-2015-H-G-103), the National Natural Science Foundation of China (Grant Nos. 51103108, 11172212) and the Fundamental Research Funds for the Central Universities.

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