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Bamboo (*Neosinocalamus affinis*)-based thin film, a novel biomass material with high performances

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ABSTRACT

Exploration of biomass based materials to replace conventional petroleum based ones has been a trend in recent decades. In this work, bamboo (*Neosinocalamus affinis*) with abundant resources was used for the first time to prepare films in the presence of cellulose. The effects of weight ratio of bamboo/cellulose on the appearances and properties of the films were investigated. It was confirmed there existed strong interactions between bamboo and cellulose, which were favorable to formation of homogeneous structure of blend films. Particularly, the presence of bamboo could improve the surface hydrophobicity, water resistance and thermal stability of blend films, and the films possessed an excellent oxygen barrier property, compared with generally used commercial packaging films. The bamboo biomass, therefore, is successfully used to create a new film material with a good application prospect in the fields of packaging, coating, and food industry.

1. Introduction

Owing to the increasing anxiety about petroleum supply and environmental threat, there has been a worldwide interest in development of biomass based materials (Zhang, Song, Wang, & Wang, 2012; Song, Tang, Wang, & Wang, 2011; Dauenhauer, Dreyer, Degenstein, & Schmidt, 2007; Huber, Iborra, & Corma, 2006). Among a large number of natural polymers, lignocellulosic is commonly regarded as one of the most promising candidates because of its advantages, such as abundant resources, low cost, nontoxicity, and biodegrability (Zhang et al., 2012; Song et al., 2011; Berndes, Hoogwijk, & van den Broek, 2003). Bamboo, a perennial woody grass belonging to the graminaceous plant composed of cellulose, hemicellulose, lignin and other small amounts of extractive minerals in an intrinsic three-dimensional cell-wall structure (Gírio et al., 2010), is widely distributed in Asia. Generally, it only takes bamboo several months to grow up, which is much shorter than wood. However, up to date, most of such biomasses have to be burned because there is no effective technology converting them into valuable materials for practical uses, which encourages us to efficiently exploit the biomass.

In the past decades, most researches on bamboo biomass were focused on its chemical modification as well as component separation (Yang, Zhong, Yuan, Peng, & Sun, 2013; Muhammad et al., 2012; Wen et al., 2011a,b; Okubo, Fujii, & Yamamoto, 2004), but how to directly employ it to fabricate materials with high-performances has still be neglected. Very recently, a bagasse-based film was developed by using DMSO/LiCl as a solvent (Chen, Zhang, Liu, Sun, & Lu, 2014), however, serious cracking and curling were hard to avoid unless performing a rigorous and complex freezing–thawing treatment, which is not easy to control and still an obstacle to real applications. It can be regarded, therefore, the film-forming property of lignocellulosic is quite poor because of the presence of lignins and/or hemicelluloses.

To solve these above problems, in the present work, we succeeded in preparing a uniform film material based on lignocellulosic biomass without defects of cracking or curling by using cellulose as a component, which has been proven to possess a good film-forming property as well as interactions with other common natural polymers previously (Takegawa, Murakami, Kaneko, & Kadokawa, 2010; Wu, Wang, Wang, Bian, & Li, 2009; Kadokawa, Murakami, Takegawa, & Kaneko, 2009). 1-Allyl-3-methylimidazolium chloride (AmimCl), a kind of ionic liquid with good solubility for lignocellulosic biomass (Zhang, Wu, Zhang, & He, 2005; Wu et al., 2004), was investigated as the solvent of bamboo and cellulose. The interactions between bamboo and cellulose were examined, and the effects of compositions of

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the blends on their structure and properties were discussed in detail.

2. Experimental

2.1. Materials

Cellulose pulp consisting of 95% cellulose (degree of polymerization, 650) was purchased from Yibin Changyi pulp Company. Bamboo (*Neosinocalamus affinis*) was obtained from Yibin (Sichuan, China). AmimCl (purity ≥99%) was purchased from Linzhou Material Science and Technology Company, China. All other reagents with analytical grade were dried at 80 °C under vacuum for 24 h before use.

2.2. Activation of bamboo

The leaves and branches of received bamboo were first removed. After that, bamboo was cut into small pieces and ground to 40 to 60 mesh size. After that, bamboo was extracted with toluene/ethanol (v/v = 2:1) for 12 h to remove wax, pigments and chlorophyll, and then dried at 80 °C under vacuum for 12 h. In the activation process, 2.5 g of dewaxed bamboo powder was immersed in 50 mL of 5% NaOH aqueous solution under constant stirring at 50 °C for 3 h, followed by filtration and washing with deionized water. The resultant bamboo powder was ground and screened to prepare particles with the size less than 200 mesh, and then dried at 80 °C under vacuum for 24 h.

2.3. Preparation of blend films

The cellulose solution was prepared by dissolving 2.5 g of dried cellulose in 47.5 g AmimCl at 80 °C, and the dissolving procedure of bamboo in AmimCl was the same with cellulose except that the heating temperature was 90 °C. The solutions obtained above were then mixed under different weight ratios of bamboo versus cellulose. The mixture solutions were vigorously stirred at 90 °C for 1 h, and then degassed and casted onto a glass plate with a thickness of about 0.5 mm. The glass plate covered with sample solution was coagulated in distilled water to prepare transparent gelatinous bamboo/cellulose films, which were washed with distilled water for several times to completely remove Cl^- ions. As a control, pure cellulose film was also prepared by the same method. All the sample films were finally dried and stored before tests, whose thickness was about 0.10 mm.

2.4. Characterization

The Fourier transform infrared (FTIR) spectroscopy was used to investigate the chemical structure of the films and the interactions between bamboo and cellulose. Samples were ground into powder and dried under vacuum for 24 h before the measurement, and the FTIR spectra was conducted in the range from 4000 to 500 cm^{-1} by using a Nicolet 6700 spectrometer (USA). The transparency of the blend films was determined by a UV–vis spectrometer (Varian Cary50, USA) within the wavelength from 300 to 800 nm. X-ray diffraction patterns were recorded by an X-ray diffractometer (Philips X'Pert X-ray diffractometer) with Cu $K\alpha$ radiation at 40 kV and 30 mA. The morphologies of the films were evaluated by scanning electron microscopy (SEM, Philips XL-3, FEI Company, USA). All the testing samples were fixed on stubs and coated with gold prior to measurement. In order to investigate the cross-section morphology of films, the samples were cryofractured in liquid nitrogen before the fixation. Thermal gravimetric analysis (TGA) was carried out under a nitrogen atmosphere on a SDT Q600 apparatus (TA Co., USA) using a heating rate of 10 °C min^{-1} from 50 to 500 °C. Mechanical properties were investigated using a universal testing machine (CMT6503, Shenzhen SANS Test Machine Co., Ltd.) with a tensile rate of 10 mm min^{-1} at room temperature according to the standard of ASTM D638. The testing samples were shaped with a dumbbell-shaped cutter, whose thickness and width were 0.10 and 4.00 mm, respectively, and five parallel measurements were conducted for each sample. Contact angles measurements were conducted on a contact angle analyzer (Model DSA-100 TC40-MK1, Germany). The surface of the samples was washed with ethanol and dried prior to test, and then the samples were horizontally fixed on a movable stage. The water droplet with the volume of 3 μL was deposited on the sample surface by a micro-syringe and a CCD video camera was used to record the shape of droplet. The experimental results were the average values of five measurements performed for each sample. Oxygen permeability measurements were performed by using a gas permeation instrument (VAC-V1, Labthink Instrument Co, China) according to the standard of GB/T1038-2000. All samples were cut into circular discs with a diameter of 50 mm and a thickness of 1 mm, and each measurement was continued until a stable oxygen permeability rate was reached.

3. Results and discussion

In order to prepare bamboo/cellulose blend films, the two components were both dissolved in AmimCl. As detected by an optical microscope with a hot stage, the dissolution property of cellulose in AmimCl was stronger than bamboo, which was due to the presence of lignin in bamboo (Kilpeläinen et al., 2007). Therefore, the dissolution of cellulose and bamboo was performed at different temperatures, i.e., 80 °C for cellulose and 90 °C for bamboo. Owing to the low content of cellulose as well as rigidity of lignin, the obtained film prepared from pure bamboo solution was too brittle to be peeled off from the mold, and thus the maximum content of bamboo in the blends was 80%.

3.1. FTIR and SEM analyses

Compatibility of different components is a generally important issue need to be considered for blends. To confirm the structure of bamboo and cellulose as well as their interactions, FTIR measurement was carried out for cellulose, bamboo, and bamboo/cellulose blend films. For both cellulose and bamboo, the characteristic peaks of C—H and C—O—C stretching were detected at 2900 and 1120 cm^{-1} (see Fig. 1). Compared with cellulose, the peak at 1508 cm^{-1} was only found for bamboo, which originated from the aromatic skeletal vibration of lignin (Lopez et al., 2000). In addition, it was worth noting that O—H stretching peaks of cellulose and bamboo were observed at 3425 and 3422 cm^{-1}, respectively, however, the peak was broaden and shifted to 3415 cm^{-1} for bamboo/cellulose, which suggested new intermolecular hydrogen bonds were formed between the two components (Kubo & Kadla, 2005; Liang, Zhang, & Xu, 2007). The tentative mechanism regarding the interactions between lignin and cellulose molecules is presented in Fig. 1. A further investigation was conducted on the RC and bamboo/cellulose films by SEM analysis. As shown in Fig. 2, both the surface and cross-section of bamboo/cellulose film showed a uniform morphology, which was similar with RC film, indicating a good compatibility between bamboo and cellulose.

3.2. XRD analysis

As a further confirmation of the interaction between bamboo and cellulose, XRD analysis was employed to investigate the crystallization of bamboo, cellulose, and bamboo/cellulose blend film. As shown in Fig. 3, the typical diffraction peaks were detected at 16.3 and 22.6° for bamboo and cellulose, indicating the existence

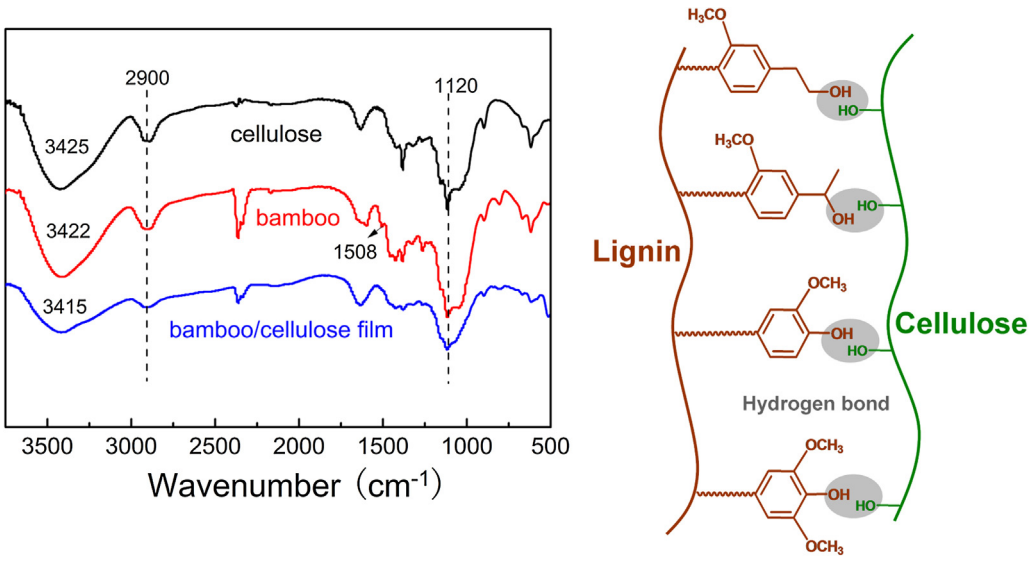

Fig. 1. FT-IR spectra of cellulose, bamboo, and bamboo/cellulose (4/6) blend films, and a given tentative mechanism regarding the interactions between lignin and cellulose.

of cellulose I (Zavrel, Bross, Funke, Büchs, & Spiess, 2009). Once dissolved in AmimCl and coagulated with water, the resultant regenerated bamboo (RB) exhibited a similar diffraction pattern with original bamboo, while the diffraction pattern of regenerated cellulose (RC) changed obviously, showing a broad peak at 20.6° attributing to cellulose II. As reported previously, a transformation from cellulose I to cellulose II was brought during the regeneration process (Raymond, Kvick, & Chanzy, 1995). Therefore, it can be regarded the well kept crystallization of RB was mainly resulted from the high content of lignin. However, the diffraction pattern of blend film was nearly the same with RC even when bamboo content was as high as 40%, suggesting the crystallization transfor-

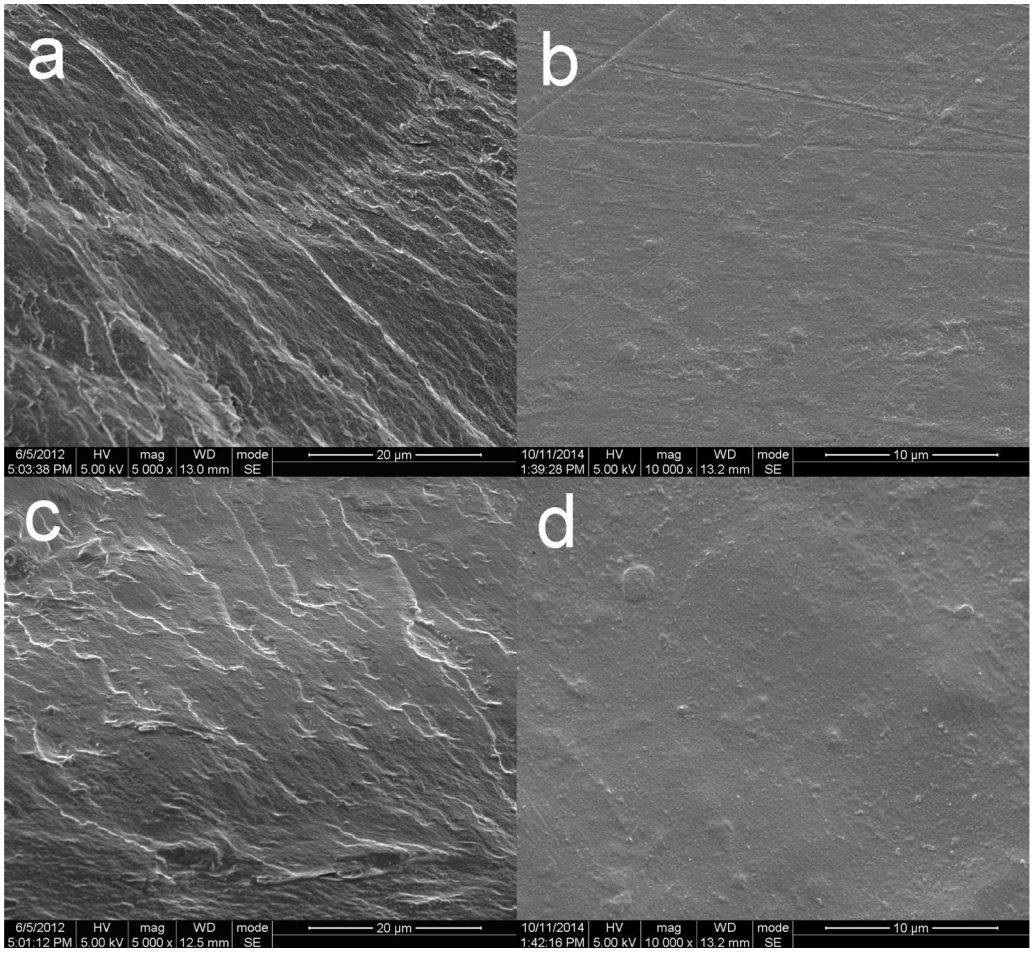

Fig. 2. SEM images of the cross-section and surface of (a and b) RC and (c and d) bamboo/cellulose (4/6) films, respectively.

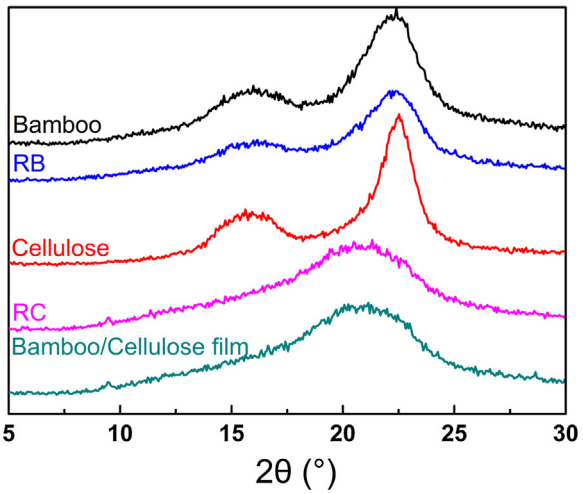

Fig. 3. XRD patterns of original bamboo and cellulose, regenerated bamboo (RB) and cellulose (RC), and bamboo/cellulose (4/6) film.

mation of cellulose in bamboo may be induced by the interaction between bamboo and cellulose.

3.3. DSC analysis

Furthermore, DSC measurement provided an additional proof for the interaction between bamboo and cellulose. As seen in Fig. 4, glass-transition and melting behaviors were not detected for cellulose because of the rigidity of molecules as well as their close intermolecular packing through hydrogen bonds, while the glass transition temperature of lignin was generally found at about 120 °C (Mousavioun, Halley, & Doherty, 2013). However, interestingly, the cellulose/bamboo films displayed an obvious endothermic peak at about 200 °C, which can be attributed to the cleavage of hydrogen bonds between cellulose and bamboo (Shi et al., 2011). As reported previously, furthermore, aliphatic hydroxyl groups can form stronger hydrogen bonds than phenolic hydroxyl groups, and the dimeric biphenyl-type structure existed in lignin can further form significantly strong intermolecular hydrogen bonds (Kubo & Kadla, 2005), and thus the newly formed hydrogen bonds between cellulose and lignin are weaker, resulting in an easy breakage at lower temperature.

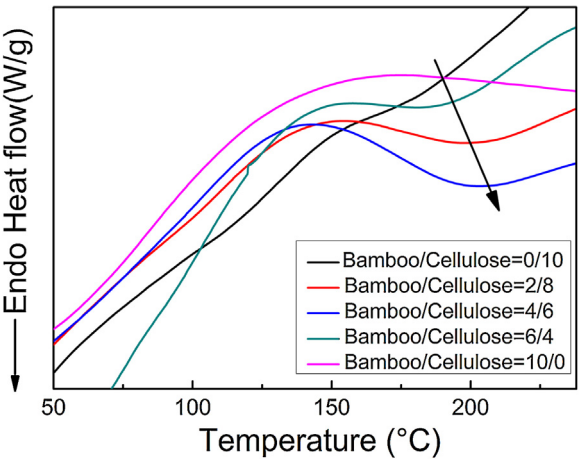

Fig. 4. DSC thermograms of bamboo, cellulose, and bamboo/cellulose blend films.

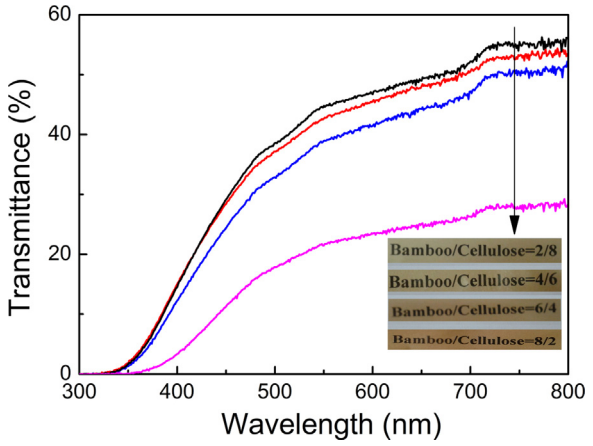

Fig. 5. Optical transmittance curves of bamboo/cellulose films. Inserted were the photos of the films.

3.4. UV–vis analysis

Generally, optical transmittance is a quite important characteristic to evaluate the transparency of films as well as a signal for the compatibility between different components (Xie, Song, Zhang, Wang, & Wang, 2014). As illustrated in Fig. 5, the marks underneath the bamboo/cellulose films can be clearly seen, implying the films possess a good transparency property. In addition, the transmittance of the blend films was dependent on their components. A higher content of bamboo in the blends resulted in a decrease in the transmittance. In particular when the bamboo content was 80%, the transmittance of obtained film was remarkably decreased lower than 30%, which suggested excessively high amount of bamboo was not beneficial for the preparation of blend film with satisfied transparency.

3.5. Mechanical property

To understand whether the mechanical property of the blends was affected by the content of bamboo or not, the tensile strength (TS) and elongation at break (ε_b) were evaluated for bamboo/cellulose with various compositions. As shown in Table 1, the blend film with weight ratio of 2/8 possessed a high TS and a high Young's modulus of 121.0 MPa and 5.1 GPa, respectively. An increase in bamboo content resulted in a gradual decrease in TS but did not exhibit an obvious influence on ε_b. When the bamboo amount was 80%, TS and Young's modulus of the blend were decreased to 67.3 MPa and 3.3 GPa, which can be attributed to the low mechanical property of bamboo (Fort et al., 2007).

3.6. Water resistance

The water resistance of the blend films was investigated by means of water contact angle analysis and moisture absorption. The effect of bamboo content on the surface hydrophilicity/hydrophobicity of the films is shown in Fig. 6. It can be seen that the RC film had a low contact angle (70°) due to its hydrophilicity,

Table 1
Mechanical properties of bamboo/cellulose blend films.

Weight Ratio of Bamboo/Cellulose	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
8/2	67.3 ± 3.7	3.3 ± 0.2	3.4 ± 0.1
6/4	109.8 ± 4.0	4.3 ± 0.2	9.6 ± 0.6
4/6	111.7 ± 1.0	4.2 ± 0.1	6.7 ± 1.4
2/8	121.0 ± 8.6	5.1 ± 0.3	5.4 ± 0.3

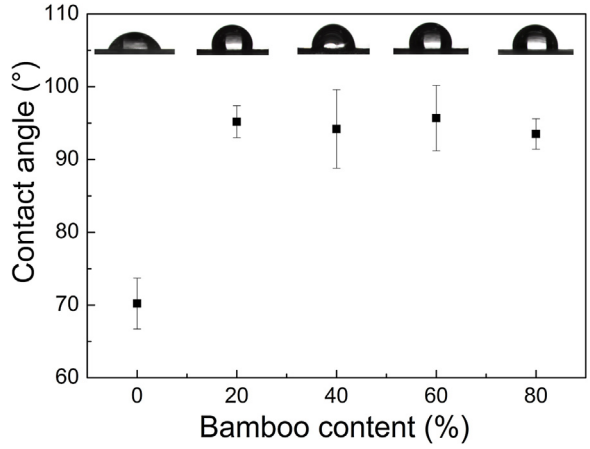

Fig. 6. The effect of bamboo content on the water contact angles of the bamboo/cellulose films.

which was in good agreement with previous report (Cao, Deng, & Zhang, 2006). With the incorporation of bamboo, the contact angle of the blends was increased to 95° and showed nearly no dependence on the amount of bamboo. A further investigation of moisture absorption was conducted to understand the water resistance of the blend films under simulated application conditions. Fig. 7 illustrates the equilibrium moisture absorption of the blend films at different relative humidities. At low relative humidities of 50% and 75%, there were no obvious differences among pure cellulose and bamboo/cellulose films, however, the moisture absorption of blend films was lower than the cellulose film at relative humidity of 100%. The results demonstrated the presence of bamboo was favorable to the improvement of water resistance for resultant films, which can be explained by the reason that lignin, a major ingredient composed of methoxylated phenylpropanoid units in bamboo, held stronger hydrophobicity than cellulose.

3.7. Oxygen barrier property

Good oxygen barrier property is urgently required for packaging materials (Aulin, Gällstedt, & Lindström, 2010). According to previous reports (Krochta & De Mulder Johnston, 1997), the oxygen permeability of packaging materials with a good barrier property should be less than 10 mL μm m^{-2} day^{-1} kPa^{-1}. Table 2 lists the oxygen permeability of bamboo/cellulose films as well as two generally used commercial packaging films, high density polyethylene

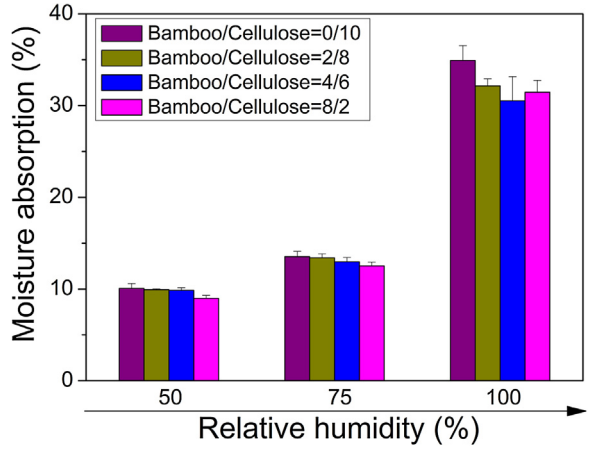

Fig. 7. Equilibrium moisture absorption of cellulose and bamboo/cellulose blend films at different relative humidities.

Table 2
The O_2 permeability of bamboo/cellulose blend films and commercial HDPE and EVOH films (Lei et al., 2013).

Sample	O_2 permeability (mL μm m^{-2} day^{-1} kPa^{-1})
Bamboo/cellulose = 0/10	0.17
Bamboo/cellulose = 4/6	0.28
Bamboo/cellulose = 6/4	3.1
Bamboo/cellulose = 8/2	42.0
HDPE	306
EVOH	3.3

(HDPE) and ethylene vinyl alcohol copolymer (EVOH) (Lei, Du, Li, Li, & Guo, 2013), of which the oxygen permeability was determined to be 306 and 3.3 mL μm m^{-2} day^{-1} kPa^{-1}, respectively. Compared with that, the oxygen permeability of cellulose film was as low as 0.17 mL μm m^{-2} day^{-1} kPa^{-1}, which can be attributed to the abundance of hydrogen bonding among the cellulose chains as well as the small free volume (Miller & Krochta, 1997). Even though an increase in the bamboo amount induced a decrease in oxygen barrier property of blend films, their oxygen permeability was still lower than EVOH that was generally regarded as a material with an excellent oxygen barrier property. It was worth noting the oxygen barrier property could not be well maintained when the bamboo content was 80%. Under the consideration of water resistance, mechanical and oxygen barrier properties, therefore, the optimum contents of bamboo in the blend films were 40% or 60%.

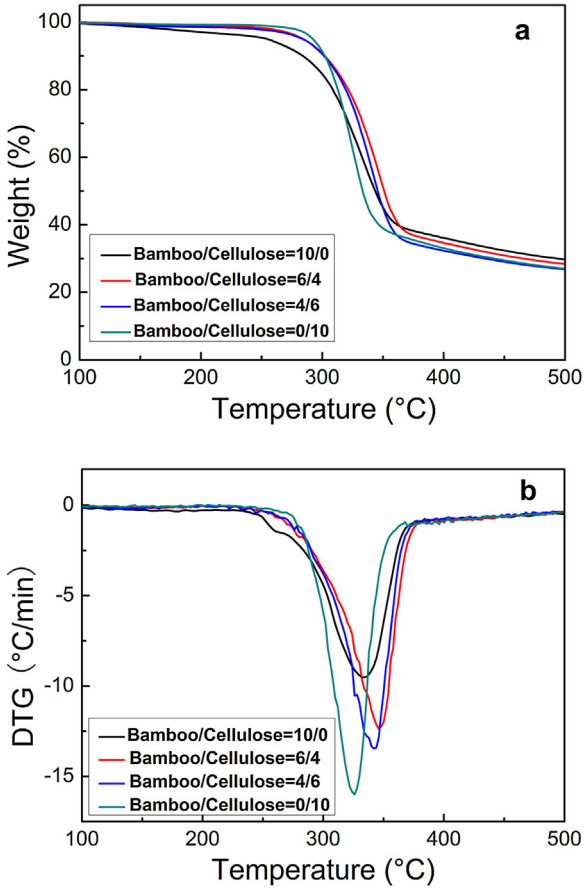

Fig. 8. TGA (a) and DTG (b) curves of bamboo, cellulose, and bamboo/cellulose blend films.

3.8. TGA analysis

In addition, thermal stability is also important for packaging materials. Fig. 8 illustrates the TGA and DTG curves of cellulose, bamboo, and bamboo/cellulose blend films. Regardless of the tiny weight loss at about 100 °C caused by the moisture evaporation, the initial degradation temperatures were determined to be 250 °C and 270 °C for bamboo and cellulose, respectively, while their maximum degradation temperatures were 325 and 313 °C. In particular, initial degradation temperatures of the blend films were close to cellulose and higher than bamboo. Moreover, their maximum degradation temperatures were higher than both bamboo and cellulose. It suggested that the resultant films possessed a good thermal stability owing to the existing interactions between bamboo and cellulose (Zhu Ryberg, Edlund, & Albertsson, 2011).

4. Conclusions

Homogeneous bamboo-based films without cracking and curling defects were prepared successfully for the firstly by a facile coagulating method. For the purpose of improving the film-forming property of bamboo, cellulose was used as its component which possessed good compatibility and strong interactions with bamboo confirmed by FTIR, XRD, DSC, SEM, and TGA measurements. The resultant films showed good surface hydrophobicity, water resistance, and thermal stability. In particular, a superior oxygen barrier property was achieved for blend films when bamboo content was 40% or 60%, suggesting an effective creation of converting an abundant bamboo biomass to valuable film materials with high performances that held a great potential application in food and packaging industry.

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