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Acetone-soluble cellulose acetate extracted from waste blended fabrics via ionic liquid catalyzed acetylation

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ABSTRACT

Isolation of cellulose from waste polyester/cotton blended fabrics (WBFs) is a bottleneck for recycling and exploiting waste textiles. The objective of this study was to provide a new environmental-friendly and efficient approach for extracting cellulose derivatives and polyester from WBFs. A Bronsted acidic ionic liquid (IL) N-methyl-imidazolium bisulfate, [Hmim]HSO_4, was used as a novel catalyst for acetylation of cellulose rather than a solvent with the aim to overcome low isolation efficiency associated with the very high viscosity and relatively high costs of ILs. The extraction yield of acetone-soluble cellulose acetate (CA) was 49.3%, which corresponded to a conversion of 84.5% of the cellulose in the original WBFs; meanwhile, 96.2% of the original poly(ethylene terephthalate) (PET) was recovered. The extracted CA was characterized by ^{1}H NMR, FTIR, XRD and TGA analysis, and the results indicated that high purity acetone-soluble CA and carbohydrate-free PET could be isolated in this manner from WBFs.

1. Introduction

It was reported that the annual production of global fiber for textiles and other applications exceeded 70.5 million tons in 2009, in which the proportion of cotton fiber and polyester fiber amounted for 40% and 45.2%, respectively (Engelhardt, 2010). Generally, used clothes and leftover materials of textile industry end in waste stations and are landfilled or incinerated (Miranda, Sosa-Blanco, Bustos-Martinez, & Vasile, 2007; Xiong, Zhang, Tian, Zhou, & Lu, 2012). These disposing methods not only create environmental pollution but also lead to great waste of the valuable resources. If the waste fabrics are in pure form of e.g. polyester or cotton fibers, different recycling or reusing techniques were developed (Wang, 2006). However, most waste textiles are generally blended fibers. Thus, separating the waste polyester/cotton blended fabrics (WBFs) into different products is attractive.

However, separating the blended fabrics into pure form can be quite challenging. Because poly(ethylene terephthalate) (PET) and cellulose in the WBFs are intricately mixed and cannot be separated mechanically. In addition, PET and cotton fibers are insoluble in most common organic liquids. Dissolving PET from the blends is not economical and environmentally friendly since PET has limited solvents which are expensive and toxic (Zou, Reddy, & Yang, 2011). In recent years, a number of methods have been developed by solubilization or degradation of cellulose to separate cellulose and PET from WBFs, but most of them had their own shortcomings. For examples, some cellulose solvents, including ionic liquids (ILs) (Hong et al., 2012; Rogers & Seddon, 2003) and N-methylmorpholine-N-oxide (NMMO) (Heinze & Liebert, 2001; Jeihanipour, Karimi, Niklasson, & Taherzadeh, 2010) have been developed to dissolve cellulose for the separation of WBFs. However, these solvent systems have many disadvantages such as low dissolution efficiency and large consumption of chemicals and energy, which limited their applications. Using NaOH (Ioelovich & Morag, 2011; Isogai & Atalla, 1998; Jeihanipour & Taherzadeh, 2009) and concentrated inorganic acids, e.g. 70–75% aqueous sulfuric acid (Negulescu, Kwon, Collier, Collier, & Pendse, 1998) and 85% phosphoric acid (Shen et al., 2013; Zhang, Cui, Lynd, & Kuang, 2006; Zhang et al., 2007) to hydrolyze cellulose have been well studied to separate WBFs. Recently, Ouchi, Toida, Kumaresan, Ando, and Kato (2010) have developed a polyester recovery technique by the strong acid (10 N H_2SO_4) degradation of cellulose and followed by a subsequent mechanical treatment. However, all these methods have the drawbacks of being ecologically unfriendly and causing considerable corrosion to the equipments. Furthermore, Vasconcelos and Cavaco-Paulo (2006) have investigated the separation of WBFs by means of the enzymatic total removal of the cellulose part of the material, but its commercialization is hindered by the harsh conditions of enzyme reaction, the prohibitive cost of the currently available enzyme preparations, and the trouble of enzyme separation (Broll et al., 1999; Hamelinck, Hooijdonk, & Faaij, 2005).

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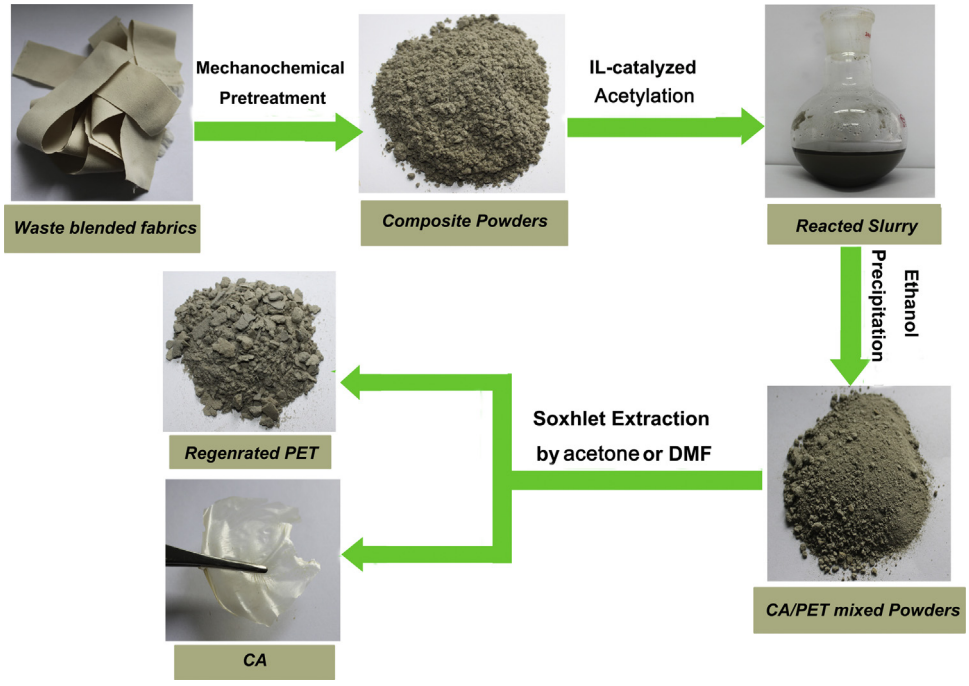

Scheme 1. The flowchart for the CA and PET recovery from the WBFs.

In this study, a new separation method using Bronsted acidic ionic liquid (N-methyl-imidazolium bisulfate) as an efficient and environmental benign catalyst rather than a solvent for the acetylation of cellulose to prepare acetone-soluble cellulose acetate (CA) so as to separate the cellulose and polyester from WBFs was developed. CA is one of the most important cellulose derivatives with a wide application in the fields of coating, film, membrane separation, textile, and cigarette industries for its low cost, toughness, gloss, high transparency and biodegradability (Aoki, Teramoto, & Nishio, 2007). Acetone-soluble CA was more industrially important due to its low toxicity and relatively cheap price of the acetone. In this process, a small amount of IL [0.1–0.6 molar equivalents of IL per molar of anhydroglucose units (AGU)] was used and it showed excellent catalytic activity for the conversion of cellulose into CA. We anticipate that this environmentally friendly and high efficient method for separation and recovery of waste blended textiles could be applied in large scale.

2. Methods

2.1. Materials

Waste polyester/cotton blended fabrics (65/35 wt%), waste PET fabrics and waste cotton fabrics were collected from tailoring workshops. All chemicals including acetone, N,N-dimethylformamide (DMF), acetic anhydride, ethyl alcohol and commercial cellulose acetate (CA) were supplied by Kelong Chemical Regent Co., Ltd. (Chengdu, China) and were used without further purification. [Hmim]HSO_4 (>99%) was purchased from Chengjie Co., Ltd. (Shanghai, China).

2.2. Separation of mixed fabrics

The flowchart for the CA and polyester recovery from the WBFs using IL as catalyst is showed in Scheme 1. The WBFs were first subjected to cutting and shredding processes. In order to enhance reaction accessibility of cellulose in WBFs, it was pretreated by mechanical milling according to reference (Geboers et al., 2010). The acetylation of cellulose was carried out under atmosphere pressure using an acidic IL N-methyl-imidazolium bisulfate, [Hmim]HSO_4, as the heterogeneous catalyst which could not dissolve WBFs. In a typical reaction, 4.63 g [~0.01 mol of AGU] of the pulverized WBFs powders, 20.42 g (0.2 mol) of acetic anhydride and 0.18–1.08 g (0.001–0.006 mol) of the [Hmim]HSO_4 were mixed and heated at 100 °C for 12 h. Then the reaction mixture was poured into 100 mL of ethanol. The solid, which consisted of CA and PET was filtered and washed 3 times with ethanol and then dried in a vacuum oven at 60 °C for 12 h. In order to extract acetone-soluble CA, part of the sample was refluxed for 12 h by the Soxhlet extraction method using acetone as solvent. The filtrate was then dried in a vacuum oven at 60 °C for 12 h to obtain the acetone-soluble CA product. Meanwhile, the solid part of the sample was refluxed for 12 h by the same method using dimethylformamide (DMF) as solvent. DMF-soluble CA was obtained as a result and the regenerated PET was isolated. The degrees of substitution (DS) values of the CA products were determined by ^{1}H NMR spectroscopy. The dried samples were weighted respectively to calculate the CA and polyester recovery after the isolation.

2.3. NMR

The DS values of CA were determined using ^{1}H NMR spectroscopy (Bruker AV II-600 MHz, AV II-400 MHz, at ambient temperature) after the samples were dissolved in DMSO-*d*6 containing a drop of deuterated trifluoroacetic acid, and the DS of CAs was calculated by Eq. (1) according to Goodlertt, Dougherty, and Patton (1971)

$$\mathrm{DS} = \frac{7 \times I_{\mathrm{acetyl}}}{3 \times I_{\mathrm{H,AGU}}} \quad (1)$$

where I_{acetyl} is the peak integral of methyl protons of acetyl moiety, and $I_{\mathrm{H,AGU}}$ is the peak integral of all protons of anhydroglucose unit.

Table 1
The total DS of CA and distribution of acetyl moiety among the three OH groups of AGU produced using different amounts of IL.

IL/AGU (mole rate)	DS	Distribution of substituents		
		C_6	C_2	C_3
0	0	0	0	0
0.1	2.41	0.87	0.79	0.75
0.2	1.92	0.72	0.65	0.55
0.3	2.27	0.87	0.78	0.62
0.4	2.62	0.97	0.90	0.75
0.5	2.3	0.86	0.80	0.64
0.6	2.17	0.80	0.76	0.61

2.4. FTIR analysis

Regenerated PET, pure PET, underivatized cellulose, the obtained CA and commercial CA were ground into powders, and dried in vacuum for 24 h at 60 °C. Transmission Fourier transform infrared spectroscopy (FTIR) was conducted by means of a Nicolet 560 spectrophotometer (USA), taking over 20 scans for each sample with a resolution of 2 cm^{-1}, ranging from 400 cm^{-1} to 4000 cm^{-1}. The samples were prepared by using the KBr-disk method.

2.5. XRD

X-ray diffraction (XRD) patterns were collected on a Philips Analytical X'Pert X-diffractometer (Philips Co., Netherlands), using Cu-Kα radiation (k = 0.1540 nm) atanaccel-erating voltage of 40 kV and the current of 40 mA. The data were collected from 2θ = 5–60° with a step interval of 0.03°.

2.6. Thermal property analysis

Curves of differential scanning calorimetry (DSC) analysis for the samples were recorded on a NETZSH 204 DSC differential scanning calorimeter under a flowing N_2 with sample weight 5 mg. The thermal properties of pure PET and regenerated PET were measured by heating from ambient temperature to 280 °C at a heating rate of 10 °C/min. Those of underivatized cellulose, the obtained CA and commercial CA were measured by heating from 50 °C to 250 °C at a heating rate of 10 °C/min. In order to eliminate thermal history, each sample was conducted second scan. Thermal gravimetric analyzer (TGA) under nitrogen purge (flow rate about 100 mL/min) was used on the TA-2000 analyzer (TA Instrument), the scanning rate of 10 °C/min and temperature range of 40–600 °C was used. Samples of about 5 mg were placed in an open alumina crucible. All the samples were vacuum oven dried at 60 °C for 24 h before testing.

3. Results and discussion

3.1. CA and polyester recovery

The DS values of CA produced using different amounts of IL at the reaction temperature of 100 °C and the acetic anhydride/AGU mole ratio of 20 is shown in Table 1. Without IL, the DS of CA is very low. It was difficult to isolate cellulose and PET from WBFs because most of the cellulose was not reacted. It can be seen that the DS values of CA first increased significantly with only a small amount of IL (0.1 molar equivalents of IL per molar of AGU) and obtained CA with a DS of 2.41. Further increase the amounts of IL could not resulted in the rise of DS values apparently.

In this work, we used acetone and DMF as solvent by the Soxhlet extraction method to separate PET and the obtained CA. The DS values have great influence on the solubility of CA. It is well known that all the CA products are readily soluble in DMF, while CA only with the average DS ranging from 2.0 to 2.7 are soluble in acetone (Wu et al., 2004). As plotted in Fig. 1(a) and (b), 0.1–0.6 molar equivalents of IL per molar of AGU for acetylation cellulose is proper to achieve acetone-soluble CA which is important in industry. The CA product with the DS values of 2.62 (0.4 molar equivalents of IL per molar of AGU) is the most acetone-soluble and almost completed soluble in acetone. Meanwhile, the yield of acetone-soluble and DMF-soluble CA are 49.3% and 51.0%, and the corresponding conversion are 84.5% and 87.40%, respectively. The yield and regeneration rate of PET with various IL content are shown in Fig. 1(c). The recovery of PET from WBFs in our experimental work is more than 90%, indicating that PET can be effectively separated from the WBFs after the acetylation of cellulose using IL as catalyst.

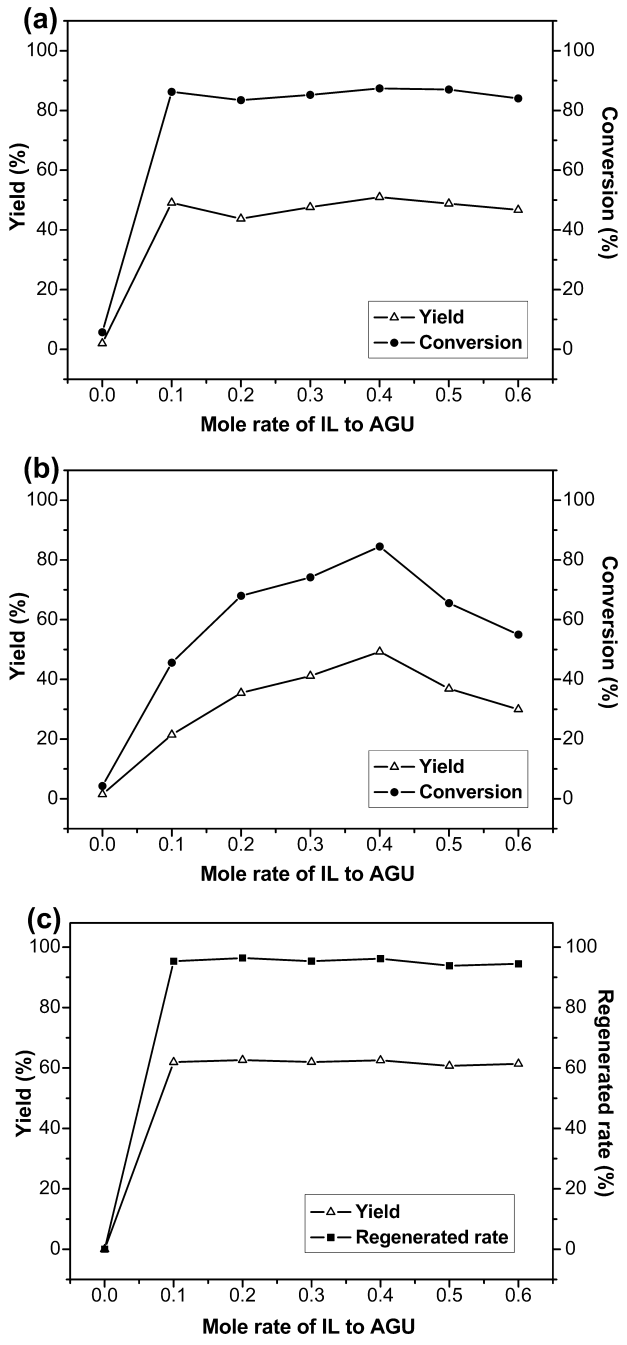

Fig. 1. (a) The yield and conversion of DMF-soluble CA, (b) the yield and conversion of acetone-soluble CA, (c) the yield and regenerated rate of PET using different amounts of IL.

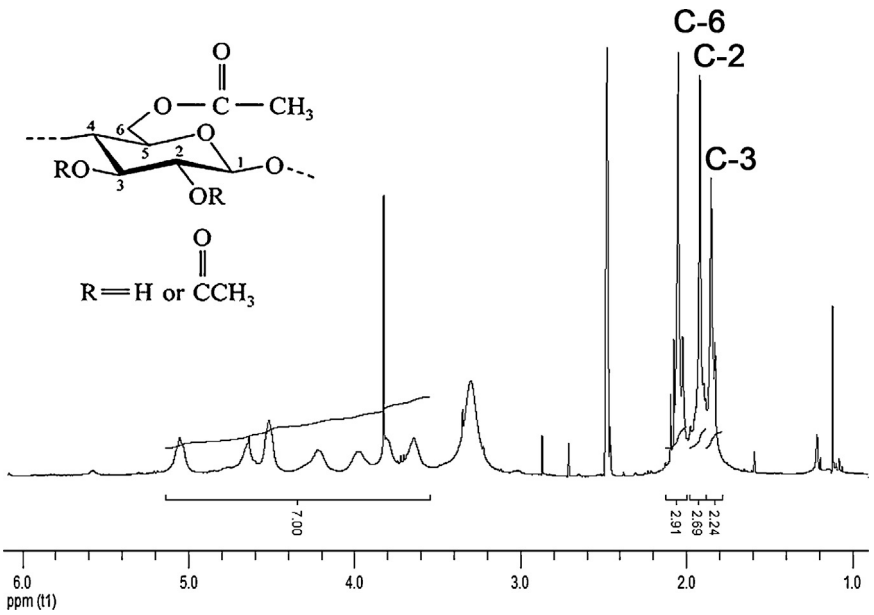

Fig. 2. Typical ^{1}H NMR spectrum of the acetone-soluble CA (DS = 2.62, 0.4 molar equivalents of IL per molar of AGU).

3.2. NMR

^{1}H NMR analysis was used to determine not only the DS of CA but also the distribution of acetyl moieties among the three OH groups of the AGU, which have significant influence on the dissolution behavior of CA. A typical ^{1}H NMR spectrum of the CA sample with DS of 2.62 (0.4 molar equivalents of IL per molar of AGU) is listed in Fig. 2. The acetyl moieties among the three OH groups are calculated from the integration of carbonyl carbon area of the ^{1}H NMR spectrum, and the results are presented in Table 1. It can be seen that the three hydroxyl groups at C_2, C_3, and C_6 position exhibit different reaction activities, and the order of reactivity is C_6-OH > C_2-OH > C_3-OH. This result is similar to the acetylation of cellulose in LiCl/1, 3-dimethyl-2-imidazolidinone (DMI) (Takaragi, Minoda, Miyamoto, Liu, & Zhang, 1999) and LiCl/DMAc solutions (Marson & EI Seoud, 1999). However, the result is quite different from that of commercial CA sample whose substituent distribution is C_3-OH > C_2-OH > C_6-OH (Buchanan, Edgar, & Wilson, 1991). This phenomenon is considered to be the major reason of the difference in solubility for CA samples synthesized from different paths. For the obtained CA sample, the acetylation of the OH group at C_6 position is expected to be preferred because hydroxyl group of this position is the least sterically hindered one of the AGU. For instance, the DS of sample shown in Fig. 2 is 2.62 and their partial DS on C_6-OH position is 0.97, which is considered to be almost completely substituted.

3.3. FTIR

The FTIR spectra of underivatized cellulose, the obtained CA and commercial CA sample are given in Fig. 3(a). Compared with the spectrum of underivatized cellulose, the spectra of the obtained CA provide an obvious evidence of acetylation by showing the presence of two important ester bonds at 1752 cm^{-1} assigned to C═O ester stretching (Biswas et al., 2008) and 1235 cm^{-1} band assigned to –CO– stretching of acetyl group (Chen, Zou, Ni, & Feng, 2003). A corresponding decrease is also seen in the OH stretch regions at 3412 cm^{-1}. Intensity of the peak at 1714 cm^{-1} (Shen, McKellop, & Salovey, 1996) corresponds to C═O in the carbonyl group is not presented in CA, indicating that PET was removed from the obtained CA products. On the other hand, without of appearance of new peaks, the spectrum of the obtained CA is quite similar to that of commercial CA. These results further confirm the purity of the obtained CA sample.

Fig. 3(b) shows the FTIR spectra of regenerated PET and pure PET. In this figure, the peaks at the wave numbers of 1714, 1250, 1120, 1046 and 726 cm^{-1} are specific for PET (Awaja & Pavel, 2005). It can be seen that the regenerated PET is quite similar to the pure PET and no new peaks appear in the regenerated sample, indicating no chemical reaction occurred during this regeneration processes of PET and CA is also not present in the regenerated PET.

3.4. XRD

The X-ray diffraction (XRD) patterns of underivatized cellulose, the obtained CA and commercial CA are shown in Fig. 4(a), which allow us to analyze changes in the crystallinity of the samples after the reaction. The underivatized cellulose exhibits main crystal planes of the crystalline cellulose I structure with the main characteristic peaks localized at 14.5°, 16.0°, 22.5°, and 33.5°, which are assigned to the diffraction planes of 1 0 1, 1 0 1, 0 0 2, and 0 4 0, respectively (Matsumura, Sugiyama, & Glasser, 2000). The XRD pattern of the obtained CA shows a decrease and broadening in the intensities of the peaks at around 14.5°, 16.0°, and 22.5°, while the intensity of the corresponding peak of 33.5° is disappeared. This reduction in crystallinity compared with the underivatized cellulose could be ascribed to the substitution of the hydroxyl groups by acetyl groups. The larger size groups may result in the broken of inter- and intra-molecular hydrogen bonds of cellulose. Some new diffraction peaks ranging from 5.0° to 12.0° are observed, which are cited as the principal characteristic of semicrystalline acetylated derivative cellulose (Barud et al., 2008; Filho et al., 2000) and indicate the generation of disorder when the cellulose was acetylated. Similar results were reported by Li et al. (2009). On the other hand, the XRD patterns of the obtained CA and commercial CA are similar. The whole diagram of commercial CA is diffuse, indicating it has a relatively poorer local order than that of the obtained CA. This result might be associated with the fact that commonly used cellulose diacetate esters were obtained by hydrolyzing fully substituted CA and the hydrolyzing process might result in the decrease of its crystallinity.

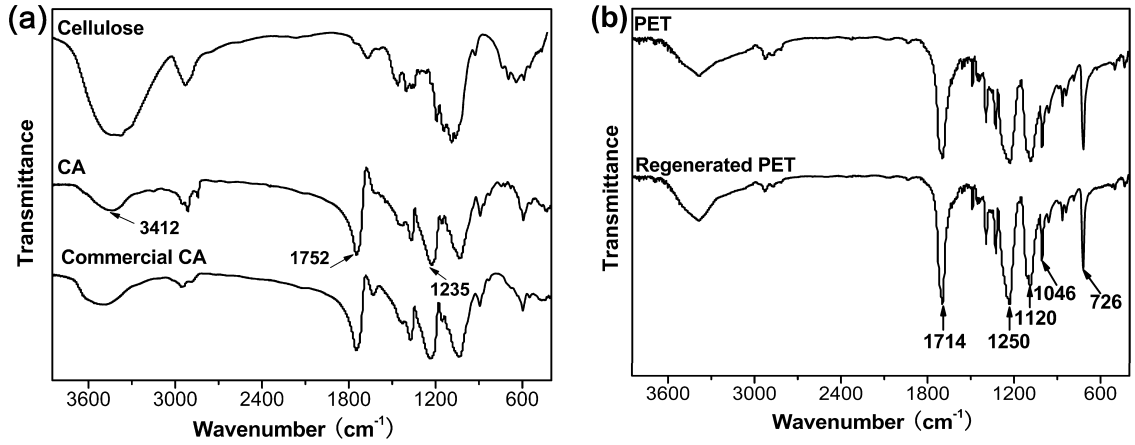

Fig. 3. FTIR spectra of (a) underivatized cellulose, the obtained CA and commercial CA, (b) regenerated PET and pure PET.

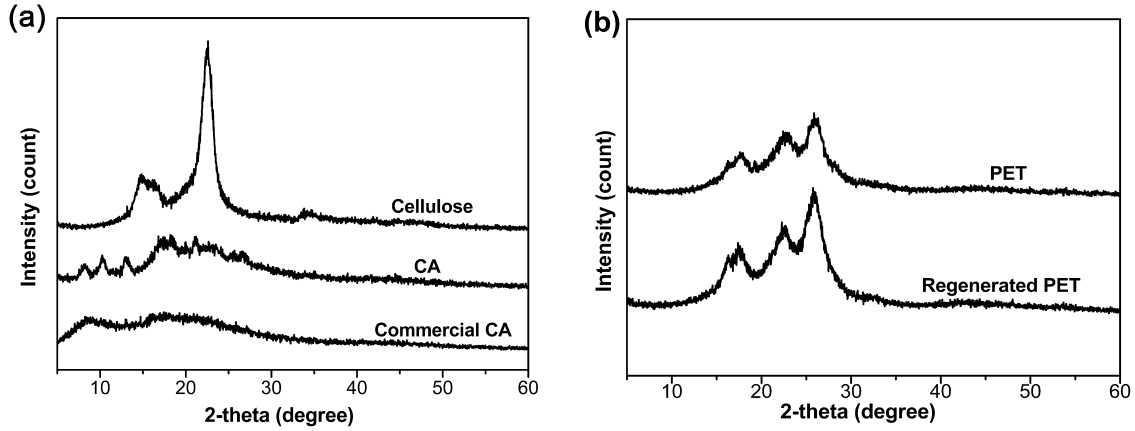

Fig. 4. XRD patterns of (a) underivatized cellulose, the obtained CA and commercial CA, (b) regenerated PET and pure PET.

Fig. 4(b) illustrates the XRD of the regenerated PET and pure PET. It is clearly seen that there is no significant change in shape and position of the diffraction peaks. Both of them show three peaks at 2θ of 17.5°, 22.6° and 25.6° (Murthy, Correale, & Minor, 1991), correspond to the 100, −1l0 and 010 spacings, respectively.

3.5. Thermal property analysis

Differential scanning calorimetry (DSC) curves obtained from underivatized cellulose, the obtained CA and commercial CA are shown in Fig. 5(a). In conventional DSC, a reversing event like the glass transition may be hidden by a nonreversing event, such as enthalpic relaxation (Cao et al., 2007). In our experiments, the diagram of underivatized cellulose does not display the glass transition temperature (T_g) and molten peak until it decomposed at standard heating scan (Fig. 5(a)). However, the DSC diagram of the obtained CA exhibits a T_g at 187 °C, which is similar to that of the commercial CA (190 °C). It should be noted that the diagram of commercial CA shows a more obvious T_g compared with that of the obtained CA. This phenomenon may be due to the fact that commercial CA

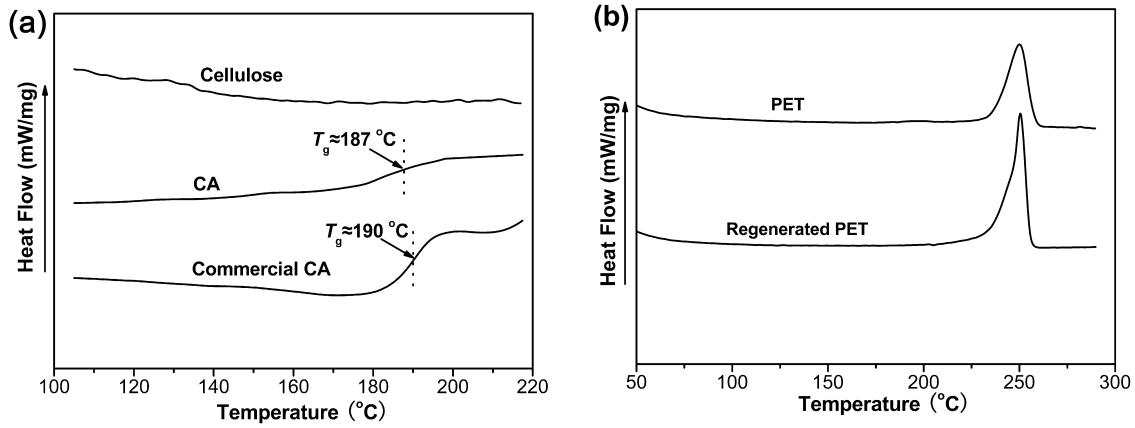

Fig. 5. DSC curves of (a) underivatized cellulose, the obtained CA and commercial CA, (b) regenerated PET and pure PET.

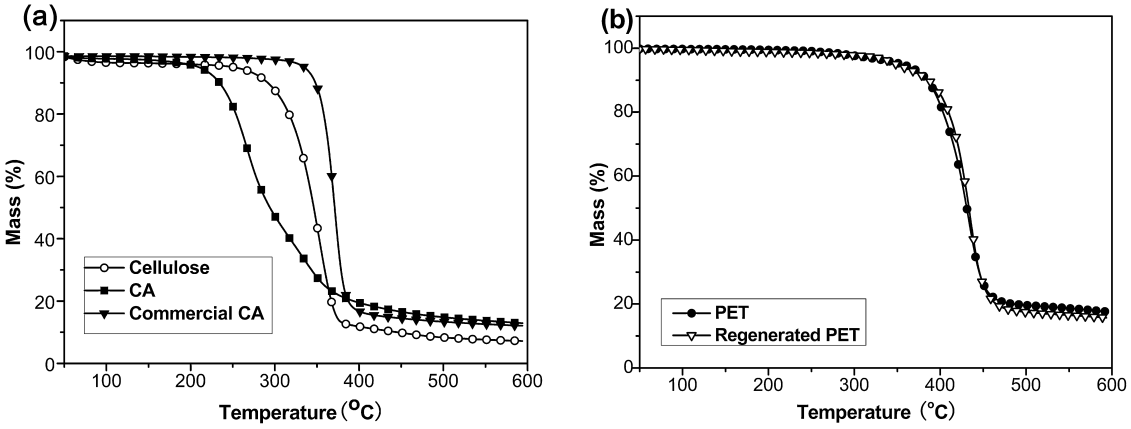

Fig. 6. TG curves of (a) underivatized cellulose, the obtained CA and commercial CA, (b) regenerated PET and pure PET.

were obtained by partial hydrolyzing fully substituted CA, which significantly broke the inter- and intra-molecular hydrogen bonds of cellulose. As a result, the movement of molecular chains and chain segments can be relatively free. Fig. 5(b) compares thermal properties of the regenerated PET and pure PET. The regenerated PET and pure PET have the same melting temperature (T_m) of 250 °C. The thermal behavior of the regenerated PET is identical to that of pure PET. DSC result also shows that the regenerated PET and the obtained CA are pure.

Fig. 6 shows the TGA curves of underivatized cellulose, the obtained CA and commercial CA (Fig. 6(a)) as well as the regenerated PET and pure PET (Fig. 6(b)) at a heating rate of 10 °C/min. The TGA curves of underivatized cellulose, the obtained CA and commercial CA exhibit a main stage of decomposition which starts around 251 °C, 210 °C and 334 °C and shows maximum decomposition at around 352.7 °C, 267 °C and 371 °C, respectively. The TGA diagram of the obtained CA indicates that both the onset temperature and the temperature at maximum decomposition rate of CA are lower than those of the underivatized cellulose, which is in accordance with the literature (Cao et al., 2007). This result indicates that acetylation reduces the thermal stability of cellulose. On the other hand, the thermal stability of commercial CA is much better than that of underivatized cellulose and the obtained CA. It could be resulted from different raw materials used (Loo, Hashim, & Leh, 2012; Zugenmaier, 2004) since the commercial CA products were usually derived from wood and bamboo pulp.

The TGA results obtained from the regenerated PET and pure PET are shown in Fig. 6(b). Both of them show a single degradation step, which starts from 350 °C with the major degradation peak at 435 °C. These results clearly illustrate that the thermal stability and maximum decomposition temperature of the regenerated PET is the same with those of pure PET.

4. Conclusion

We have demonstrated a facile approach to extract acetone-soluble CA from WBFs by using Bronsted acidic IL as a novel catalyst for the acetylation of cellulose. The highest yield of acetone-soluble CA was 49.3%, which corresponded to the conversion of 84.5% of cellulose in the WBFs; meanwhile, more than 96% of PET was recovered. This method provides an environmentally friendly and efficient process for the separation of WBFs, which is indispensable for the recycling of the blended fabrics.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.carbpol.2013.05.089.

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