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Recyclability of cellulose acetate butyrate (CAB) matrix for controllable and productive fabrication of thermoplastic nanofibers

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ABSTRACT

A high-throughput, controllable and environmentally benign fabrication process of thermoplastic nanofibers with a biobased material, cellulose acetate butyrate (CAB) as the matrix was recently reported. To prove the recyclability of the CAB, a method was proposed to theoretically demonstrate the ability of the recycled polymer for repeated melt extrusions based on a comparison of thermal degradation and flow activation energy of the recycled polymers. The flow activation energy was derived from rheological behaviors of the original and recycled CAB at different temperatures. The thermogravimetric analysis (TGA) was utilized to characterize the thermal degradation behaviors of the polymer. The change in the number average molecular weight of the recycled CAB was studied by measuring the intrinsic viscosity and the Mark-Houwink equation. To display the practical recyclability, the recycled CAB was successfully employed to serve as the matrix for the preparation of iPP (polypropylene), PTT (poly trimethylene terephthalate) and PE-co-GMA (poly (ethylene-co-glycidyl methacrylate)) nanofibers. The proposed quantitative method could be extended to evaluate the recyclability of the other thermoplastics.

1. Introduction

Cellulose esters are prepared from the esterification of renewable and biodegradable cellulose which is abundant in agricultural wastes such as straws and residues or so called biomasses. These derivatized cellulose polymers have attracted considerable research interests as promising bio-based material candidates to unhook widespread dependence on petroleum based materials and reduce the growing environmental pollution resulted from the increasing amount of non-biodegradable wastes (Dale, 2003; Mohanty, Mirsa, & Drzal, 2002). Bio-based cellulose esters are thermoplastics and are typically immiscible with most petroleum based materials due to their structure differences. Moreover, by varying the substitution of the cellulose ester they can be either melt processed or readily dissolved into acetone, and then made into different products (Buchanan, Buchanan, Debenham, Gatenholm, et al., 2003; Muller & Leuschke, 1996). With above advantages, we have recently developed a novel technique to mass produce thermoplastic nanofibers with the thermoplastic, cellulose acetate butyrate (CAB), as a sacrificial matrix through a conventional melt blending and extrusion process. The thermoplastics which can be made into nanofibers with this process cover a wide range, including polyesters, polyolefins, functional copolymers and polyurethanes (Wang & Sun, 2007; Wang, Sun, & Chiou, 2007, 2008a,b; Wang, Sun, Chiou, & Hinestroza, 2007). In this process, CAB was melt blended with immiscible thermoplastics and extruded into *in situ* nanofibrillar blends under certain conditions. When the CAB/thermoplastic nanofibrillar blends were immersed into the acetone solvent, CAB matrix was rapidly dissolved, the thermoplastic nanofibers in continuous forms were obtained. The dissolved CAB can be precipitated into solid form with addition of water, and then filtered and recycled. The recycled CAB could be used for repeated thermoplastic nanofiber productions from the viewpoints of environmental friendliness and economic concerns. The recyclability of the extruded CAB is therefore worth of thorough investigating.

Thermal degradation of the CAB matrix often occurs during the melt blending and extrusion process due to its own relatively poor thermal stability and some interactions between CAB matrix and the thermoplastics which may accelerate the degradation of the CAB matrix. The degree of thermal degradation plays an important role in determining the reusability of the CAB matrix. However, it is very difficult to estimate the recyclability of the CAB only from the thermal degradation parameters by themselves. The overall evaluation of the molecular weight, rheology and thermal degradation properties of the recy-

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cled CAB is essential in investigating the recyclability of the CAB matrix.

In the present study, a quantitative method of evaluating the recyclability of the CAB is proposed. This method is based on a comparison of the thermal degradation activation energy and the flow activation energy calculated from the rheological properties of the CAB. The higher thermal degradation activation energy indicates before the serious thermal degradation happened the CAB already could become fluid and be able to flow. On the contrary, the higher thermal flow activation energy shows before the CAB became melt it already thermally degraded, therefore losing the recyclability. The output of this method may be extended to evaluate the recyclability of other polymer systems.

2. Experiments

2.1. Materials

Cellulose acetate butyrate (CAB; butyryl content 35–39%) was purchased from the Acros Chemical (Pittsburg, PA). Isotactic polypropylene (iPP) and Poly (trimethylene terephthalate) (PTT) were kindly supplied by ExxonMobile Co., and Shell Chemicals, respectively. Low density polyethylene, (LDPE, MFI: 25 g/10 min, 190 °C/2.16 kg), Polyethylene-co-glycidyl methacrylate (PE-co-GMA, GMA 8 wt%) and poly(vinyl alcohol-co-ethylene) (PVA-co-PE, ethylene 27 mol%) were purchased from Aldrich Chemical (Milwaukee, WI). Acetone solvent was purchased from VWR Scientific. (West Chester, PA).

2.2. Preparation

The mixtures of CAB/iPP, CAB/LDPE, CAB/PTT, CAB/PE-co-GMA and CAB/PVA-co-PE were gravimetrically fed into a Leistritz co-rotating twin-screw (18 mm) extruder (Model MIC 18/GL 30D, Nurnberg, Germany) at the blend ratio of 80/20. The feeding rate was 12 g/min and the screw speed was 100 rpm. Barrel temperature profiles were 150 °C, 180 °C, 200 °C, 220 °C, 235 °C and 240 °C. The blends were extruded through a 2 strand (2 mm in diameter) rod die. The extrudates were hot-drawn at the die exit by a take-up device keeping a drawn ratio of 25 (the area of cross section of the die to that of the extrudates) and air cooled to room temperature. The original CAB was extruded with the same procedure as a control, labeled as the extruded CAB.

The extruded CAB and CAB/thermoplastics blends were immersed into acetone solvent at room temperature for 2 h. The dissolved CAB in the acetone solvent was then precipitated with water. Then recycled CAB in the wet state was placed into an oven for drying until a constant weight was reached. The recycled CAB in solid form was stored in a desiccator for future testing.

2.3. Characterization

The intrinsic viscosities ([η]) of original, recycled CAB from extruded CAB and from CAB/thermoplastics were measured using a Ubbelohde rheometer with acetone as a solvent at 25 °C. The rheological properties of original CAB, and recycled CAB were measured by a Capillary Rheometer LCR 8052 (Kayness, Inc., PA 19543), using a capillary round die with a L/D ratio of 30 and an entrance angle of 120°. Barrel temperature was set at 230 °C, 240 °C and 250 °C.

The thermal degradation analysis of the original CAB and recycled CAB were performed with a thermogravimetric analyzer (TGA-50, Shimadzu, Columbia, USA) at constant heating rates of 2 °C/min, 5 °C/min, 10 °C/min and 20 °C/min under a steady nitrogen flow with a flow rate of 50 ml/min from ambient to 500 °C, respectively. The recycled CAB was used as the matrix to prepare the thermoplastic nanofibers. The extrudates of CAB and thermoplastic blends from twin-screw extruder were immersed in acetone at room temperature for 15 min to remove the CAB matrix from the blends. The morphology of obtained polymer nanofibers was characterized using a Philips XL30 Scanning Electron Microscope (SEM).

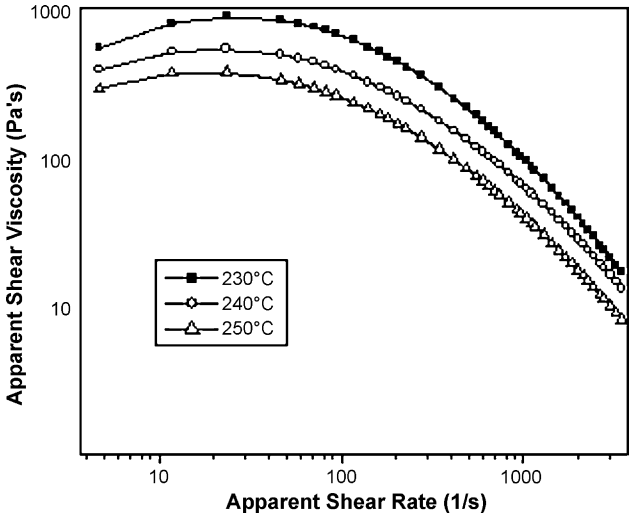

Fig. 1. Apparent shear viscosity as a function of the apparent shear rate at 230, 240 and 250 °C for original CAB.

3. Results and discussion

3.1. Melt flow behaviors

To investigate the melt flow behaviors of the CAB, the plots of the apparent shear viscosity as a function of the apparent shear rate at 230 °C, 240 °C and 250 °C for original and recycled CAB are shown in Figs. 1 and 2, respectively. The recycled CAB was prepared by dissolving the extruded CAB in acetone, then precipitating from mixing its acetone solution with water and drying. It can be found that the apparent shear viscosity decreased with increasing the apparent shear rate for both original and recycled CAB, indicating that the recycling process did not change the pseudoplastic fluid feature of the recycled CAB. The slightly higher apparent shear viscosity of the recycled CAB than the that of

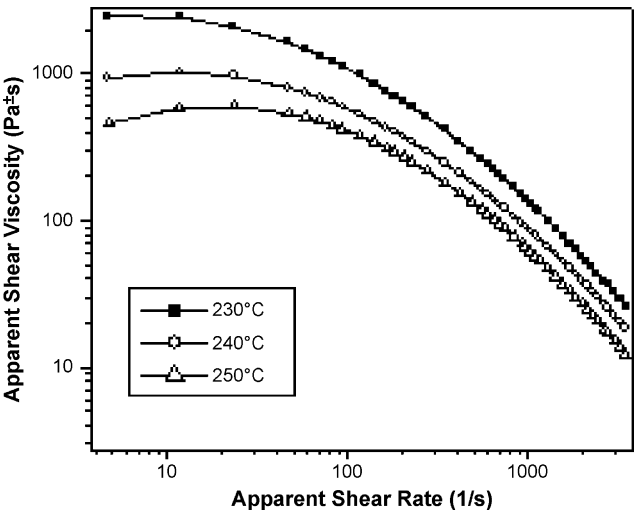

Fig. 2. Apparent shear viscosity as a function of the apparent shear rate at 230, 240 and 250 °C for recycled CAB.

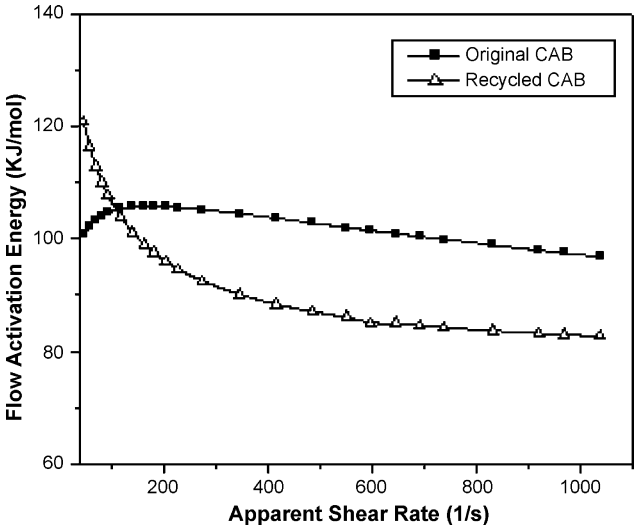

Fig. 3. Flow activation energy as a function of the apparent shear rate for original and recycled CAB.

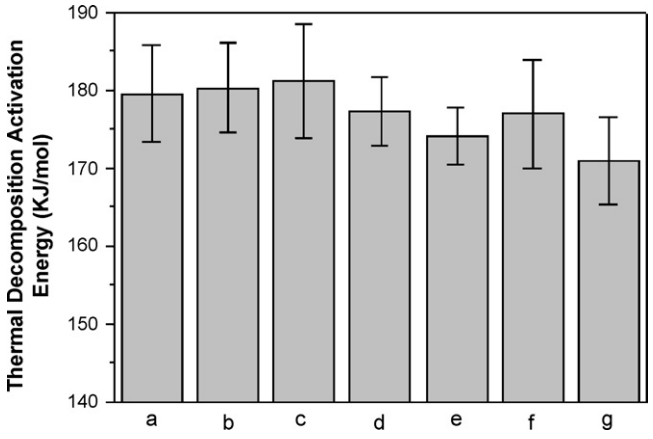

Fig. 4. Thermal degradation activation energy of (a) original CAB, (b) recycled CAB and from blends of CAB and (c)LDPE, (d) iPP, (e) PE-co-GMA, (f) PVA-co-PE and (g) PTT (Unit: kJ/mol).

the original one was observed, which might be attributed to the facts that more butyryl substitution groups were released from CAB molecules than the acetyl groups during the melt extrusion and the intermolecular interactions between CAB molecules in the molten state were enhanced (Chen, Zhong, & Gu, 2010; Muller & Leuschke, 1996). The mathematical relationship between the apparent shear viscosity and the temperature could be expressed with the Arrhenius–Frenkel Eyring equation as follows (Munari, Pezzin, & Pilati, 1990; Nair, Oommen, & Thomas, 2002).

$$\eta_r = B \exp\left(\frac{E_r}{RT}\right)$$

where η_r melt viscosity at a given shear rate, E_r is the melt flow activation energy, B is a constant, T is absolute temperature and R is the gas constant.

The values of $\ln \eta_r$ were plotted against $1/T$, and then E_r could be determined from the slope of the plot at a given apparent shear rate. Fig. 3 presents the calculated flow activation energy E_r for original and recycled CAB at apparent shear rates ranging from 46.8 to 1037 s^{-1}. The melt flow activation energy of the recycled CAB is in the range of the 83–106 kJ/mol as the apparent shear rate is above 100 s^{-1}, lower than that of the original CAB. Compared with the original CAB, the decreasing tendency of the melt flow activation energy of the recycled CAB is sharper with increasing the apparent shear rate. The result suggests that the molecular weight of recycled CAB has dropped and its molecular weight distribution became broader. The flow activation energy could be considered as an indicator to evaluate the capability of the CAB melt to flow at certain temperature. The higher the E_r, the worse the flow ability is.

3.2. Thermal degradation behavior

Thermal degradation of cellulose acetate butyrate readily occurred with debutyrylation and deacetylation at the beginning of melting, followed by the break of the cellulose skeleton (Huang & Li, 1998; Jain, Lal, & Bhatnagar, 1985). Therefore, the thermal stability of CAB plays an important role in determining its reusability as a sacrificial matrix in the production of thermoplastic nanofibers. Thermal degradation activation energy is accepted as one of the key kinetic parameters to evaluate the thermal stability. The thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of original CAB, recycled CAB from extruded CAB as well as the recycled CAB from melt extruded blends with LDPE, iPP, PE-co-GMA, PVA-co-PE and PTT were performed at constant heating rates of 2, 5, 10 and 20 °C/min in N_2 atmosphere, respectively. The Flynn-Wall-Ozawa method is utilized to calculate the thermal degradation activation energy (Liu, Yu, Sun, Zhang, & He, 2003).

$$\ln \beta = -0.4567\left(\frac{E_a}{RT}\right) + \ln\left(\frac{AE_a}{R}\right) - \ln F(\alpha) - 2.315$$

β is the constant heating rate dT/dt, E_a is the thermal degradation activation energy, T is absolute temperature, α is the conversion (weight of materials volatilized/initial weight of materials) and R is the gas constant. Thus, E_a could be obtained from the slope of the plot of $\ln \beta$ against $1/T$.

The thermal degradation activation energies of original and recycled CAB are summarized in Fig. 4. The original, recycled CAB from extruded CAB and from CAB/HDPE blends have the highest thermal degradation activation energy of about 180 kJ/mol, followed by the CAB recycled from CAB/iPP and CAB/PVA-co-PE, about 177 kJ/mol. For the CAB recycled from blends of CAB with PE-co-GMA and PTT, the thermal degradation activation energy E_a decreased to 174.12 and 170.84 kJ/mol, respectively. The decreased thermal stability may be attributed to the possibly accelerated release of the acetyl and butyl groups from the side chain of the cellulose acetate butyrate by the esters of PE-co-GMA and PTT (Chen et al., 2010).

To compare the thermal degradation difference under air and N_2, respectively, the TG and DTG curves for original CAB, recycled CAB from melt extruded blends of CAB with iPP, PE-co-GMA, and PTT at constant heating rates of 2, 5, 10 and 20 °C/min in air were carried out, and the thermal degradation activation energy E_a were obtained with the same method, as listed in the Table 1. For original CAB, there is no significant difference in the thermal degradation activation energy under air and N_2, respectively. However, the E_a decreased by 35.13 and 56.79 kJ/mol to 135.71 and 117.33 kJ/mol, for CAB recycled from the blends with PTT and PE-co-GMA in air because the oxygen accelerated the thermal degradation of the CAB. Figs. 5 and 6 display the TG and DTG curves of the original CAB and recycled CAB from blends with iPP, PE-co-GMA, and PTT at a constant heating rate of 10 °C/min in N_2 and air, respectively. The thermal degradation kinetic parameters are listed in Table 1. Both the onset thermal degradation temperature T_{onset} and the temperature at the maximum degradation rate T_{dm} significantly decreased in the oxidative atmosphere for original and all recycled CAB, compared with them under the inert N_2 atmosphere. The CAB recycled from CAB/PTT blends have the lowest thermal degradation temperatures in air, followed by the PE-co-GMA, iPP and the original CAB, consistent with the tendency observed for the thermal degra-

Table 1
Thermal degradation kinetic parameters of the original and recycled CAB.

Sample	In air			In nitrogen		
	E_a (kJ/mol)	T_{dm} (°C)	T_{onset} (°C)	E_a (kJ/mol)	T_{dm} (°C)	T_{onset} (°C)
Original CAB	180.26 ± 5.80	367.76	339.02	179.48 ± 6.17	380.49	345.58
Recycled CAB from						
CAB/iPP	157.36 ± 28.68	365.60	336.99	177.24 ± 4.40	377.88	344.06
CAB/PTT	135.71 ± 6.65	361.88	329.64	170.84 ± 5.65	377.59	340.78
CAB/PE-co-GMA	117.33 ± 16.42	363.70	331.92	174.12 ± 3.60	377.79	342.30

The T_{dm} and T_{onset} are obtained from the DTG curves at a constant heating rate of 10 °C/min.

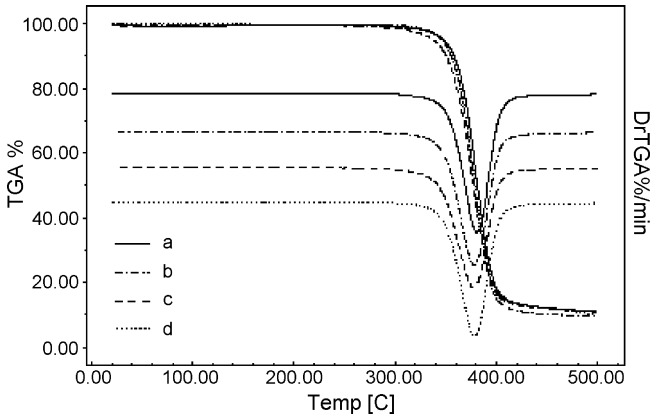

Fig. 5. TGA and DTG curves of (a) original CAB, recycled CAB (b) from CAB/iPP blends, (c) from CAB/PTT blends and (d) from CAB/PE-co-GMA blends in nitrogen atmosphere at a heating ramp of 10 °C/min.

dation activation energy and the thermal degradation behaviors under N_2.

3.3. Number average molecular weight of CAB

The thermal degradation of the CAB typically involves the break of the cellulose skeleton after releasing acetic and butyric acid, causing the decrease in the molecular weight and the widened molecular weight distribution. Intrinsic viscosity measurement is often conducted to characterize the number average molecular weight of a polymer. Tamblyn et al. established the quantitative relations between the intrinsic viscosity and the number average molecular weight for the CAB which have been moderately degraded by heat, mechanical working by fractionation theory. As the CAB is dissolved in acetone at 25 °C the equation could be expressed as follows (Tamblyn, Morey, & Wagner, 1945):

$$[\eta] = 18.5 \times 10^{-5} M_n^{0.83}$$

$[\eta]$ is the intrinsic viscosity, M_n is the number average molecular weight.

The intrinsic viscosities of the original CAB, recycled CAB from the blends with LDPE, iPP, PVA-co-PE, PE-co-GMA and PTT were measured and converted to the number average molecular weight M_n, as shown in Fig. 7. It indicated that the recycling of the original CAB led to the decrease in the M_n from 6.87×10^5 to 6.24×10^5, while the M_n of the CAB recycled from the blends with LDPE and iPP had M_n of 6.82×10^5 to 6.20×10^5, respectively. On the contrary, the significant decrease in the M_n of the CAB recycled from the blend with PTT was observed, reaching 4.92×10^5, which could be assigned to the following explanation. During the melt extrusion, the CAB thermally hydrolyzed and produced butyric acid, acetic acid as well as the hydroxyl groups. The hydroxyl groups and adsorbed water moisture facilitated the breakage of the ester bonds in the PTT, creating the carboxylic acid and more hydroxyl groups which in turn accelerated the deacetylation and debutylation of the CAB. As a result, more acid gas generated broke the bonds between the two glucose units or rings of the cellulose chain, lowering the molecular weight of the recycled CAB. The CAB from the blends with PVA-co-PE and PE-co-GMA showed the values of M_n of 5.83×10^5 and 5.45×10^5, respectively. The tendency was in good agreement with the results of the thermal degradation activation energy.

3.4. Overall evaluation of the recyclability of CAB

Based on the comparison of the thermal degradation activation energy and the flow activation energy of the recycled CAB, a method could be used to evaluate the theoretical possibility of the repeated use of CAB in the melt process. The thermal degra-

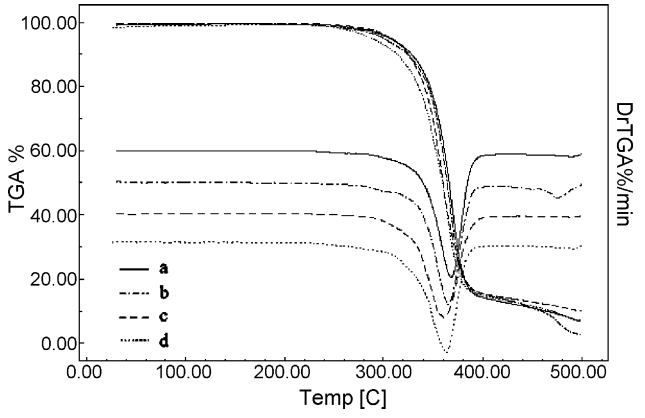

Fig. 6. TGA and DTG curves of (a) original CAB, recycled CAB (b) from CAB/iPP blends, (c) from CAB/PTT blends and (d) from CAB/PE-co-GMA blends in air atmosphere at a heating rate of 10 °C/min.

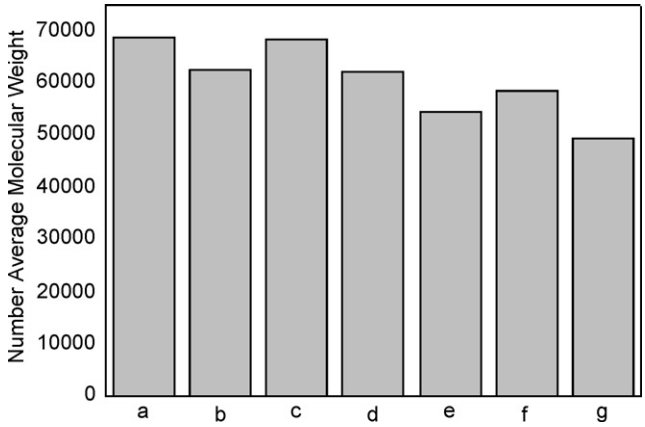

Fig. 7. Number average molecular weight M_n of (a) original CAB, (b) recycled CAB and from blends of CAB and (c) LDPE, (d) iPP, (e) PE-co-GMA, (f) PVA-co-PE and (g) PTT.

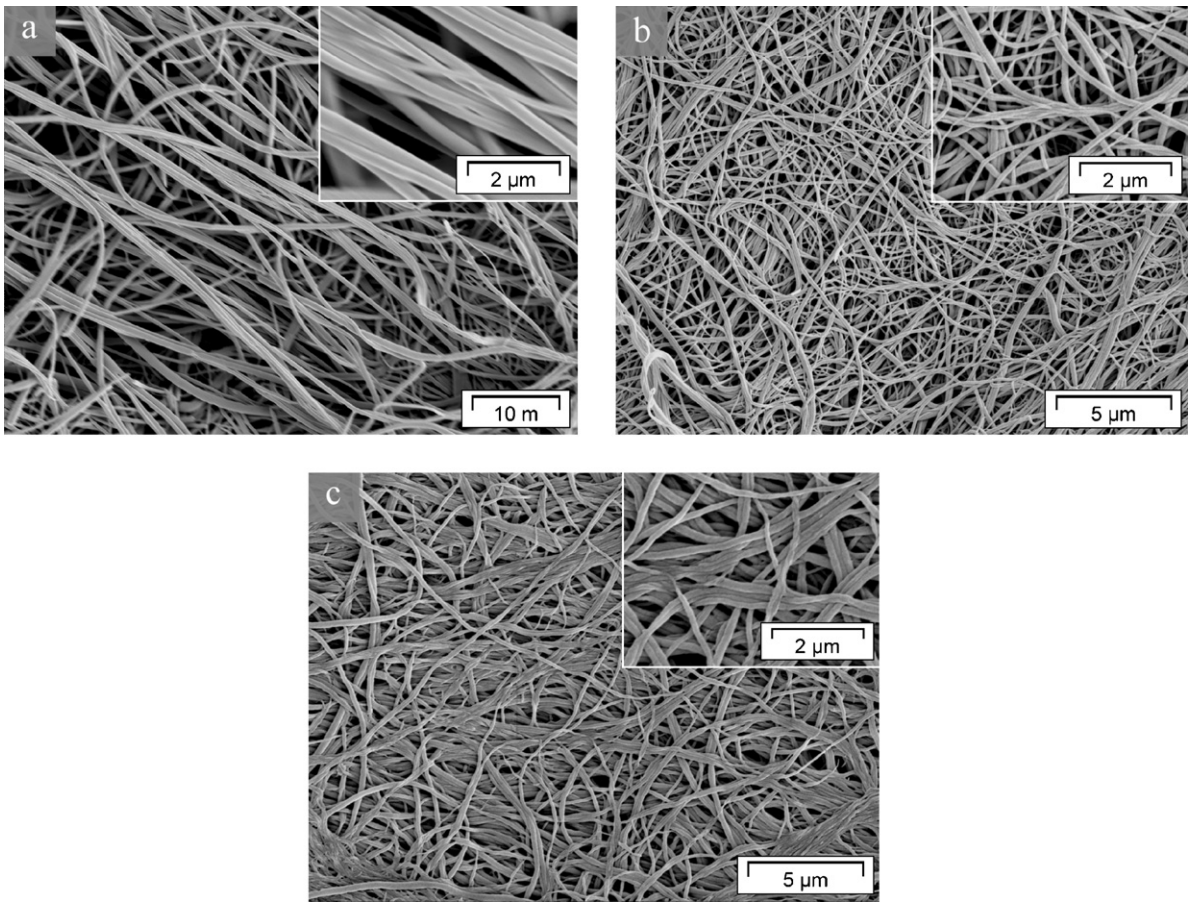

Fig. 8. SEM images of (a) iPP, (b) PTT and (c) PE-co-GMA nanofibers prepared from the recycled CAB blends.

dation and flow activation energy represent the energy barriers required to surpass and trigger the degradation and flow. The larger thermal degradation activation energy suggested that when the energy adsorbed by the CAB is above the flow activation energy but below the thermal degradation activation energy, prior to the occurrence of the thermal degradation, solid CAB could become flow-able melt, which presents the theoretical possibility of CAB for melt processing and recycling under certain conditions. The flow activation energy study indicated that at the typical apparent shear rate of 115 s^{-1} for the recycled CAB, the flow activation energy is 104 kJ/mol. The number could be lower as the molecular weigh of the recycled CAB further decreased. The thermal degradation activation energies under N_2 are all above 170 kJ/mol for recycled CAB and the CAB recycled from blends with iPP, PVA-co-PE, PTT and PE-co-GMA. Even in the oxidative atmosphere causing the significant decrease in the thermal degradation activation, E_a is still above 117 kJ/mol. The comparison clearly provides the theoretical evidence to prove the recyclability of the CAB for repeated melt extrusion. Furthermore, the results showed that the decrease in number average molecular weight of the CAB recycled from the CAB/PTT blend was less than 30% even undergoing the most severe thermal degradation.

3.5. Thermoplastic nanofibers preparation from recycled CAB

To practically demonstrate the ability of the recycled CAB serving as the sacrificial matrix in making the thermoplastic nanofibers, the CAB recycled from the blend with PTT were melt blended and extruded with the iPP, PTT and PE-co-GMA at the CAB/thermoplastic blend ratio of 80/20. The extruded composite fibers were soaked into the acetone at room temperature to remove the CAB matrix. The morphology of the iPP, PE-co-GMA and PTT nanofibers prepared were characterized with SEM, as shown in Fig. 8. Although some changes in the rheological behavior and the molecular weight of CAB occurred after recycling, recycled CAB was still able to mass produce the versatile thermoplastic nanofibers in proper sizes.

4. Conclusions

A theoretical method was proposed to evaluate the recyclability of the CAB recycled from the melt extruded blends of the CAB with iPP, PTT, PVA-co-PE, PE-co-GMA based on a quantitative comparison of thermal degradation and flow activation energy of CAB. The flow activation energy of the recycled CAB was lower than that of the original CAB due to the release of the acetic and butyric acid, followed by the chain breakage. The flow activation energy of the recycled CAB was 104 kJ/mol at the apparent shear rate of 115 s^{-1} which is a typical melt processing shear rate. The CAB recycled from the blends with PTT had the lowest onset and maximum thermal degradation temperatures, as well as the thermal degradation activation energy of 170.84 kJ/mol under N_2, followed by the PE-co-GMA, PVA-co-PE, iPP and LDPE. In air, the thermal degradation became worse for the recycled CAB. The thermal degradation activation energy of CAB recycled from the blends of PTT and PE-co-GMA decreased to 135.71 and 117.33 kJ/mol, respectively. The higher value of the thermal degradation activation energy of the recycled CAB indicated the good recyclability of the CAB. The number average molecular weight of the recycled CAB from the measurement of the intrinsic viscosity decreased by 1%, 9.8%, 21%,

15% and 28.5% for the CAB recycled from the blend with LDPE, iPP, PE-co-GMA, PVA-co-PE and PTT, respectively. The recycled CAB as a matrix was able to prepare iPP, PTT and PR-co-GMA nanofibers by melt extrusion. This method could be used to guide the evaluation of the recyclability of other thermoplastics.

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