

Recycling of waste nylon 6/spandex blended fabrics by melt processing



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ARTICLE INFO

Article history:

Received 15 March 2014

Received in revised form

3 September 2014

Accepted 10 March 2015

Available online 18 March 2015

Keywords:

A. Recycling

A. Fabrics

E. Compression molding

B. Mechanical properties

ABSTRACT

A unique approach for reclaiming waste nylon 6/spandex blended fabrics was demonstrated by melt processing through mixing and molding. Spandex from the waste fabrics was removed by hydrolysis under controlled conditions. Morphological analysis showed that the fracture surface was homogeneous for samples of neat nylon 6 and treated fabrics, and voids were observed for samples from untreated fabrics. Thermal analysis indicated that the materials from waste fabrics exhibited both similar melting temperature (around 220 °C) and similar crystallization temperature (around 185 °C). Infrared spectroscopy showed nearly the same main absorption peaks of neat nylon 6 and samples from nylon 6/spandex fabrics. The viscosity of treated fabric samples was lower than that of untreated fabric composites at low and medium frequencies, as an effect of the spandex removal after treatment. The recycled plastics from treated fabrics exhibited good mechanical properties with a tensile strength of 46.6 MPa and a Young's modulus exceeding 2.4 GPa.

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1. Introduction

Remnant of cloth especially from the apparel industry is both an environmental and economical problem. An enormous amount of synthetic waste is generated, most of which is disposed in landfills [1]. Besides environmental issues with landfilling, the disposal of large amount of remnant cloth is a huge waste of resources and energy. It is a demanding issue of seeking an efficient and cost-friendly way in separating and recycling used synthetic fabrics into recycled products. The recycled synthetic fabrics especially nylon fabrics have already established great market value in many application areas such as auto parts [2], composites [3] and soil reinforcement [4]. However, recycling of blended fabrics poses a significant challenge in engineering because of the inhomogeneous nature of the materials [5]. In order for the fibers from these fabrics to be reclaimed, effective separation of synthetic fibers in a blended fabric becomes the first challenge in reclaiming and further reusing of such fabrics.

Spandex is a segment polymer containing at least 85% segmented polyurethane [6]. Spandex has superior stretch and elastic recovery ability as a result of alternating rigid and flexible segments [7–9]. Fabrics containing spandex yarns find wide applications in sports clothes, leisure clothes, hosiery, underwear, and swimwear, as well as for body-confirming garments, which assume a stable shape under loading during wearing [10–12]. Although the elastic polyurethane fibers offer reasonable elastic recovery to apparel, deficiencies in chemical resistance and temperature stability have to be managed during apparel manufacture to avoid excessive fiber property degradation and loss of elasticity [13–15].

Nylon is a generic designation for a family of synthetic polymers known as polyamides, which is made of repeating units linked by amide bonds [16]. Among many types of synthetic fabrics, nylon is one of the most widely used in various clothing products. Nylon fabric has been long heralded for its “wash and wear” quality, and has lent durability, strength and softness to a host of materials. The attractions of nylon 6 fibers are high melting point, good mechanical strength, good resistance to hydrocarbons, and the ease of fabrication and processing [17–20]. Nylon is often blended with spandex to achieve superior elastic and comfort characteristics that are also visually striking yet economically efficient [21]. Originally

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designed to be synthetic replacements for silk, nylon fabrics have a soft hand and supple nature, allowing for the manufacture of popular materials like stretchy spandex blends and breathable power mesh.

As one of existing technologies, the solvent extraction has been widely used with high recycle yield of nylon. However, the drawbacks of solvent extraction are the chemicals involved, modest temperature, pressures and time required. To recycle blended synthetic fabrics, Yin [22] et al. developed an environmentally friendly way in separating spandex components via selective degradation from Nylon/spandex blend fabrics. After heat treatment, the thermal-degraded spandex was mostly removed by ethanol washing. Following the simple separation process, the work is further extended in this study for a cost-effective and time-efficient route to produce recycled plastics by melt processing. The prewashed blended fabric was first treated in a hydrolysis reaction to degrade the spandex. Then the fabrics were directly melt-blended in a mixer. Plastic samples from waste fabrics as well as neat nylon 6 were fabricated by compression molding. Their morphological, thermal and mechanical properties were characterized. Fracture surfaces were examined by SEM. The structural and thermal properties were analyzed by FTIR-ATR and DSC. Furthermore, the tensile stress-strain behavior of the samples was investigated. The study shows that melt blending provides an effective route for recycling waste nylon/spandex blend fabrics.

2. Experimental

2.1. Materials

Nylon 6/spandex blended fabrics (223.8 g/m², knitted, brown) obtained from Aquafil USA, Inc., were used in this experiment. Dimethylformamide (DMF) and ethanol of analytical reagent grade were supplied by Sigma–Aldrich Co. LLC. Virgin nylon 6 was obtained from ICI plastics in the form of pellets. The nylon 6 pellets were dried in oven at 80 °C for 24 h before use.

2.2. Removal of spandex from nylon 6/spandex blended fabric

Solvent extraction: Pure nylon 6 fabrics were obtained by washing waste nylon 6/spandex in the DMF solvent (solvent ratio 50:1 in mass) at 70 °C for 4 h. The sample was washed with deionized water and dried at 50 °C for 24 h before use. According to the results observed by Yin [22] et al., spandex was completely dissolved in DMF.

Heat treatment: The blended fabrics were washed with deionized water, and then dried in vacuum at 60 °C for 24 h. A customized stainless steel reaction chamber was heated to 220 °C, and the dried fabrics were placed in the reaction chamber with the valve closed and heated for 2 h. After heating, the treated fabrics were transferred and washed by ethanol at 60 °C for 30 min with a magnetic stirring apparatus. Finally the treated fabrics were washed 3 times with deionized water. The waste fabrics before and after heat treatments were noted as untreated and treated fabrics, respectively.

2.3. Melt mixing

The untreated polyamide/spandex blended fabrics, treated fabrics and virgin nylon 6 pellets each were separately melt-processed in a Brabender Intelli-Torque Rheometer. The respective materials were melted and homogenized by the torque rheometer. The rheometer head was mounted with Sigma blades capable of tumbling and distributive mixing. A compounding temperature of

240 °C and a mixing speed of 45 rpm were used for the mixing process. The shear rate at this mixing speed with a rotor diameter of 35.5 mm was estimated to be 47.5 s⁻¹. To minimize the hydrolysis of nylon 6 at the high compounding temperature, the fabrics and the nylon 6 pellets were all dried in an oven at 90 °C for 12 h and melt-processed immediately under nitrogen protection. The achievement of homogeneous material inside the mixing chamber was established by the development of a constant mixing torque. For all materials processed, a homogeneous melt was obtained after approximately 15 min when the torque reached a stable value. The melt was immediately scraped out of the chamber and quenched in tap water. The cooled material was dried in an oven at 90 °C for 4 h and stored in a desiccator before the next step of experiment. The material from virgin nylon 6 pellets was named neat nylon 6 as a reference for future discussions.

2.4. Compression molding

A hot press was used to prepare sample sheets to be used for mechanical testing and other characterization. Each mixture, enclosed in a spacer frame between two steel plates, was placed between the upper and lower platens of the hot press and pressed at a temperature of 240 °C with a ram force of approximately 5.0 GPa for 15 min. The time taken to fully close the platens was approximately 30 s. The molded sheets were dried at 60 °C for 24 h and stored in a desiccator before characterization. The thickness of sheets was approximately 2 mm.

2.5. Characterization

SEM was used to observe the appearance and morphology of the molded samples. Fractured samples after tensile testing were dried with liquid carbon dioxide, sputtered with gold–palladium, and analyzed under an SEM (ISI model DS130).

A Thermo Nicolet Nexus FTIR-ATR spectrophotometer (Thermo Electron Co., MA, USA) equipped with an OMNI-Sampler was used to examine the chemical structure of the molded samples.

A differential scanning calorimeter (Q200, TA Instruments) was employed for thermal analysis of the original fabrics, the treated fabrics and the virgin nylon 6 polymer. All samples were heated from 40 °C to 240 °C at a rate of 10 °C/min, held for 10 min at 240 °C, and then cooled back to 40 °C at a rate of 10 °C/min. The holding stage was considered necessary to erase possible effects of thermal history of the sample on the subsequent melt crystallization.

Dynamic rheological properties were measured on a parallel-plate rotational rheometer (AR2000ex, TA Instruments). The plate diameter and the gap between the plates were 25 mm and 1 mm, respectively. The strain applied was 1%. The sample sheets were melted and equilibrated at 240 °C. The initial gap was set to a value equivalent to the final gap plus 5 mm. After the excessive sample squeezed out was carefully trimmed off, the upper plate was moved to the final gap size. To remove existing crystallization and residual stress, the melted sheets were held for about 10 min at the heating temperature and then cooled at a rate of 10 °C/min for temperature-sweep rheological measurements. The sheets were also cooled to the predetermined temperature at a rate of 10 °C/min for time-sweep rheological measurements.

Tensile tests were carried out on a tensile test machine (Instron Universal Testing Machine 5166 Series, Instron Corp., MA) at room temperature with a crosshead speed of 5 mm/min. All sample sheets were cut into dog-bone shaped testing specimens using a cutting die according to DIN-53504. The specimen has a gauge length of 50 mm and a width of 10 mm. Three specimens were tested for each sample.

3. Results and discussion

3.1. SEM observation

Fig. 1 shows SEM micrographs of fracture surfaces of different materials prepared by melt mixing and compression molding. It can be observed that the surfaces of samples from neat nylon 6 and the treated fabric were relatively smooth. However, the sample from the untreated fabric contained a number of sizable voids. In fact, the fracture surface of untreated fabrics was sticky, and some fluid-like droplets were visually observable before drying. As supported by the results from FTIR-ATR (to be presented later), spandex fibers in the untreated fabrics was largely degraded during melt mixing, and the voids observed in SEM may therefore be attributed to the degraded spandex. These voids can act as defects and cause premature failure of the molded sample. Much fewer defects were observed in molded sheets from virgin nylon 6 and treated fabrics. Additionally, all SEM micrographs showed no fibrous structures, indicating that all fabric samples were melted completely during melt mixing.

3.2. DSC investigation

To eliminate the effects of structure and molecular weight on the thermal and rheological behavior of nylon 6, the samples in DSC and rheology tests are all from the waste fabrics. DSC was used to determine the melting temperature (T_m) and crystallization temperature (T_c) of different materials involved in this study.

As shown in Fig. 2, the melting peaks of samples from waste fabrics were very close, at around 220 °C. A noticeable minor peak several degrees below the melting temperature were also observed for all three samples, which might be originated from a solid-to-solid transition between different crystalline structures of nylon 6. This is supported by the fact that the peak below 220 °C is clearly shown and well separated from the melting point in the nylon 6 fabrics. The additional spandex content to the nylon 6 matrix would diminish the boundary between the two peaks, due to its lack of structural regularity as a non-crystalline elastomer and typically shows broad peaks during thermal transitions. The much better separation of peaks for the treated fabric as compared to the untreated one indicated that the treatment have substantially reduced the content of spandex in the fabric. Spandex is not a thermoplastic material and it degrades before it melts. Thermal gravitational analysis (TGA) showed that spandex fibers were thermally stable as far as decomposition was concerned till about 180 °C. On the other hand, the residual of spandex have slight contribution to the increase of melting temperature of nylon 6 (Fig. 3).

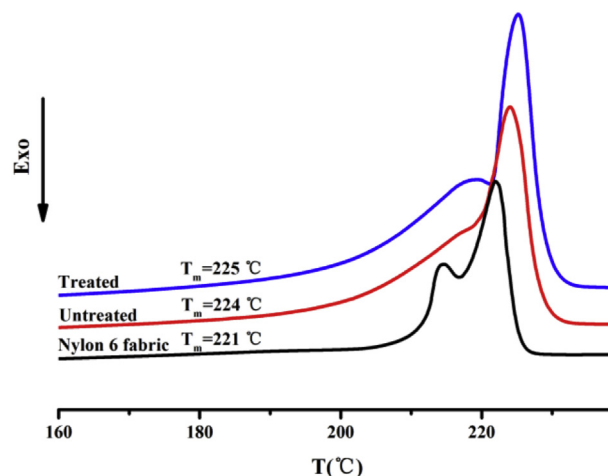


Fig. 2. DSC curves obtained on heating samples from waste fabrics.

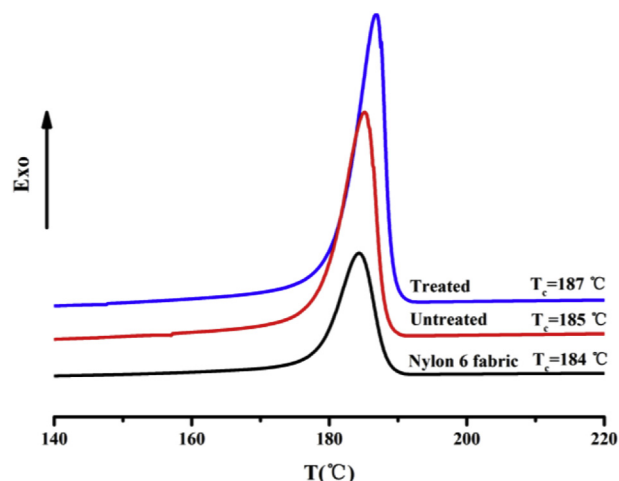


Fig. 3. DSC cooling curves of samples from waste fabrics.

On cooling, pure nylon 6 fabrics exhibited an exothermic T_c dip at 184 °C, whereas T_c 's of samples from treated and untreated waste fabrics were significantly higher, at 187 °C and 185 °C, respectively. As a result of spandex residue, the crystallization temperature was relatively higher than that of pure nylon 6 fabrics. Moreover, the DSC results of samples based on waste fabric showed

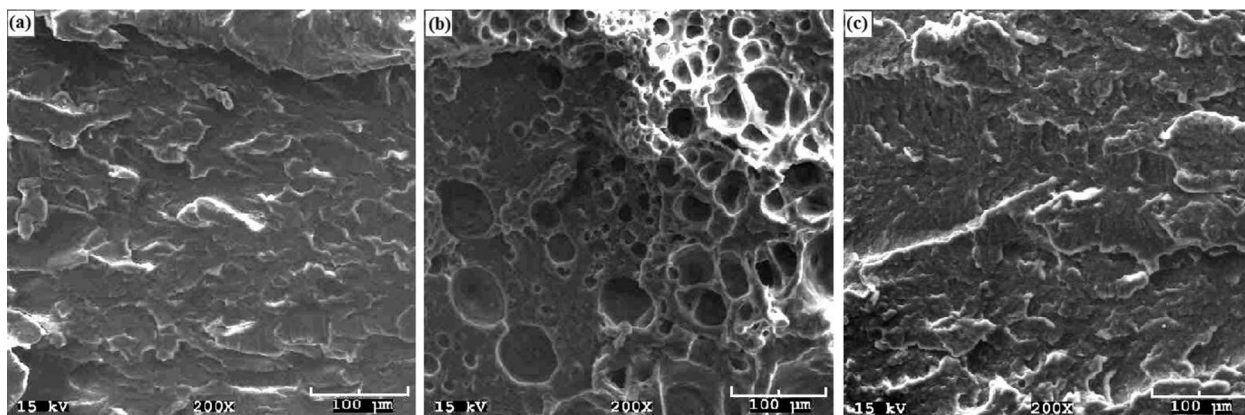


Fig. 1. SEM micrographs of fracture surfaces of different materials (a) neat nylon 6; (b) untreated fabrics and (c) treated fabrics prepared by melt mixing and compression molding.

narrower crystallization temperature ranges in contrast to the wider temperature range for neat nylon 6. This much narrower crystallization range may be attributed to the existence of spandex residuals (degraded or not degraded), which may result in increased crystallization rate. To some extent, nylon 6 and spandex share some common properties as a result of the amide group present in their molecular chains.

3.3. FTIR-ATR analysis

Spectroscopic methods have an important role in characterization of polymers [23–25]. The use of methods such as FTIR-ATR for surface characterization is very effective in investigating molecular structure and organization of polymers.

From Fig. 4, the main absorption peaks of the samples obtained from waste fabrics were nearly the same as those of neat nylon 6, and there were no new peaks or fading peaks in the whole spectra. From TGA, the degradation temperature of spandex was determined to be 177 °C. Above the degradation temperature, degradation would occur, and the long molecular chain would break and the amide group be damaged. However, because there was spandex residue in the sample and there were also amide groups in nylon, the absorption peak of —CON— was still present. After the spandex residue was washed with ethanol, the absorption peaks at 1538 cm^{-1} and 1634 cm^{-1} were weakened in intensity. It was probably due to the damages to the amino group in spandex molecules as they were degraded into oligomers. Overall the FTIR-ATR absorption peak only showed slight difference between treated and untreated fabrics.

All samples revealed strong O—H absorption peaks at 3075 cm^{-1} and 3295 cm^{-1} . The strong O—H absorption bands may be related to moisture absorption on the sensitized nylon 6 surfaces after the specimens were exposed to the atmosphere. The moisture absorption can lead to the formation of a boundary lubrication film of water on the surface of the sample.

In the wave number range of 2800–3000 cm^{-1} the band corresponding to valence vibrations of methyl and methylene groups, with two peaks at 2860 cm^{-1} and 2930 cm^{-1} , was found. In this region there are peaks corresponding to antisymmetric C—H stretching vibration of methyl and methylene groups and also the bands attributed to symmetric C—H stretching vibration of above mentioned groups [26,27]. The broad absorption band between 1000 cm^{-1} and 1200 cm^{-1} corresponding to the —C—O—C— ether

group was also observed for the samples from waste fabrics. However, there was no peak around 1100 cm^{-1} for neat nylon 6, and therefore it should be attributed to the existence of spandex and its residual.

3.4. Rheological behavior

The rheological behavior of polymer blends depends not only on the composition but also to some extent on the morphology. The complex viscosities at 240 °C for materials from waste fabrics as a function of frequency were presented in Fig. 5.

In the entire range of frequencies, the complex viscosities of all samples decreased with the increase of frequency, indicating a non-Newtonian shear thinning behavior and pseudo-plastic characteristics of materials. No obvious influence of the spandex was detected at low shear rates. However, as the frequencies climbed up, the spandex content in the fabrics began to play a role in elevated viscosities, which may be originated from the inclusion of spandex residuals. The viscosity of treated fabric samples was lower than that of untreated fabric composite at low and medium frequencies, as an effect of the spandex removal after treatment.

The difference in complex viscosity is primarily caused by changes in the storage modulus G' , as can be seen in Fig. 6A. The dynamic storage modulus is related to the elastic storage energy while the dynamic loss modulus as shown in Fig. 6B is related to the energy dissipation. The pure nylon 6 fabrics exhibited homopolymer-like terminal behavior at low frequencies. Both the storage modulus and loss modulus of materials from waste fabrics were increased relative to the pure nylon 6 fabrics, particularly at low frequencies. Compared to the case for pure nylon 6 fabrics, the decrease in the G' and G'' for materials from waste fabrics could be explained by the incorporation of spandex residuals. Furthermore, removing spandex had insignificant effect on the rheological behavior of waste fabrics.

3.5. Mechanical property

Fig. 7 shows the stress strain behavior of neat nylon 6 and the materials from the treated and untreated fabrics. The strength and elongation at break of the recycled materials were significantly lower compared with neat nylon 6. Besides, neat nylon 6 and the materials from the treated and untreated fabrics exhibited a Young's modulus at around 2.7 GPa, 1.9 GPa and 2.4 GPa, respectively. The Young's modulus also decreased to some extent. It could

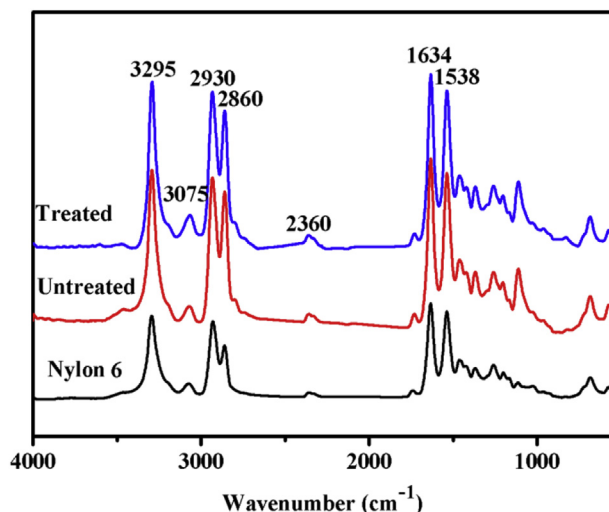


Fig. 4. FTIR-ATR spectra of samples from neat nylon 6 and waste fabrics.

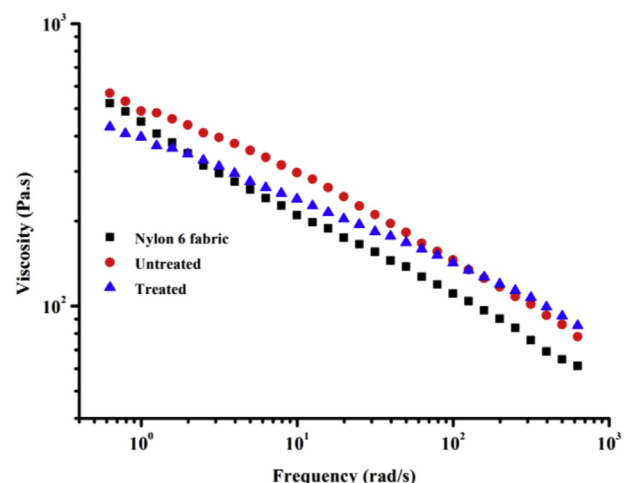


Fig. 5. Complex viscosities at 240 °C of materials from waste fabrics.

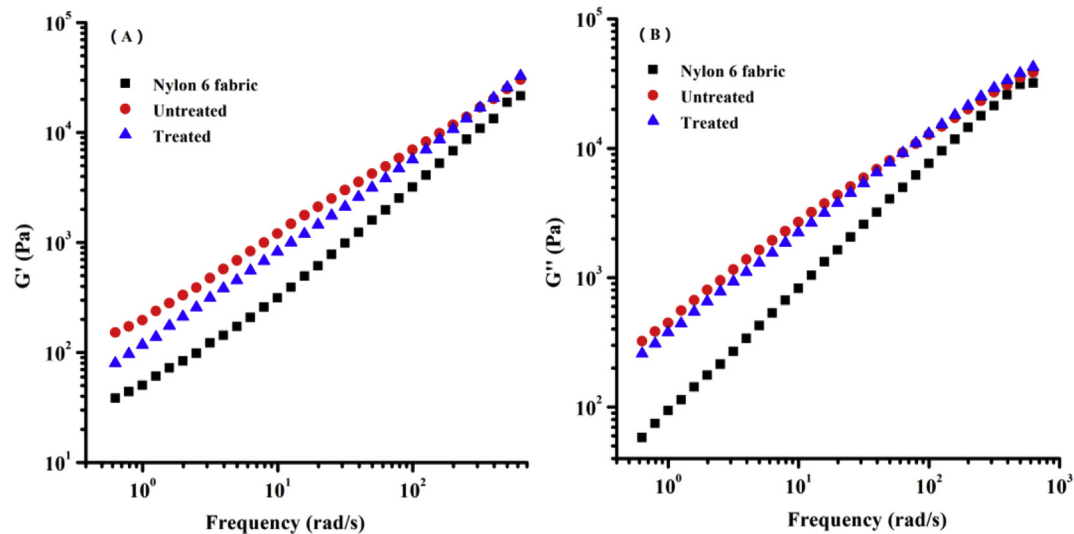


Fig. 6. (A) Storage modulus G' at 240 °C of materials from waste fabrics; (B) Loss modulus G'' at 240 °C of materials from waste fabrics.

be attributed to the presence of spandex residuals and voids in the recycle samples, leading to stress concentrations and therefore a brittle and weak material. The results nonetheless showed that waste fabrics can be recycled without losing elastic modulus significantly. The strength and elongation are still reasonable to meet requirements for many applications.

In comparison, the sample from the untreated fabric showed lower mechanical properties than the sample from the treated fabric. The sticky feel of the untreated sample indicated the existence of spandex-derived residuals in the molded sample which affected the mechanical properties. Such degraded residuals were in fact observed in SEM, showed as voids in the SEM images after washing. During the heat treatment of the waste fabric, the spandex macromolecule degraded into some short-chain spandex residues, so that they were easily washed away from the nylon fabric by alcohol. After removal of the spandex component, the treated fabric had better homogeneity in material composition, resulting in a higher strength and elongation at rupture. Removing the spandex was beneficial to the improvement on the mechanical properties of recycled materials from blended fabrics.

Recycled nylon material can be blended with virgin nylon 6 to improve its mechanical properties. Recycled nylon fabrics and

virgin nylon pellets were melt mixed at 240 °C, and then pressed by compression molding at 240 °C for 15 min. Fig. 8 showed typical stress-strain curves of the materials. Uniaxial tensile tests were taken three times at the same strain rate. The error for the strain to failure point is ± 0.3 , but the fluctuations in the results should not affect the overall conclusions made here. In general, the tensile strength of the nylon blend decreased with the increase of the recycled content. Also shown in Fig. 8 is that the maximum tensile strain was achieved at a fabric fraction of $W_f = 40\%$. Further increase of the waste fabric content would reduce the tensile strength and strain of the blends. The decrease in mechanical properties after the peak at 40% was probably caused by the increasing occurrence and size of fabric clumps as fabric weight fraction increased [28]. However, the strain for sample with $W_f = 60\%$ was significantly larger than the strain of samples from 100% waste fabrics. Waste fabrics and nylon 6 pellets have good compatibility, allowing the two to be blended at a wide range of ratios to meet the demand of various applications.

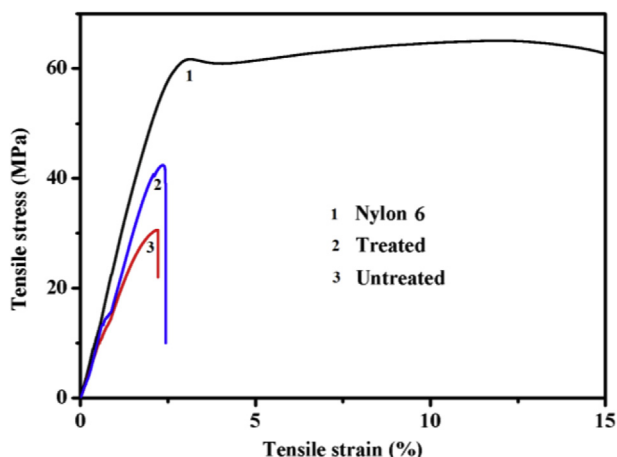


Fig. 7. Tensile strain-stress curves of neat nylon 6 and materials from waste fabrics.

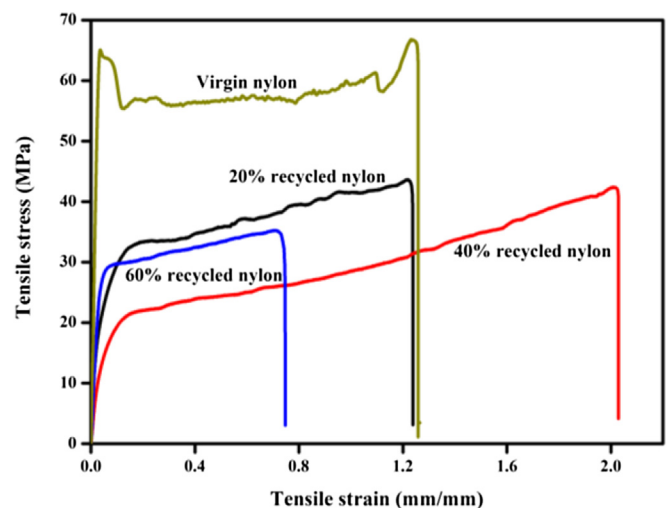


Fig. 8. Tensile stress-strain curves of blends of recycled nylon fabrics with virgin nylon 6.

4. Conclusion

Converting blended fabrics into useful products reduces the need for waste disposal and conserves valuable resources. A simple approach developed in this study converts nylon 6/spandex blend fabrics into recycled plastics suitable for various applications. The process involves heat treatment, washing and melt mixing. The fracture surface of samples from treated fabrics was homogeneous without fibrous residue. Recycled plastic samples from treated and untreated fabrics exhibited slightly higher crystallization temperature, compared with that of pure nylon 6 fabrics. FTIR-ATR revealed no significant difference between treated and untreated fabrics, and there was no new peak or fading peak in the whole spectrum. The complex viscosities decreased with the increase of frequency, indicating a non-Newtonian behavior and pseudo-plastic characteristics of materials from waste fabrics. The strength and elongation at break of the recycled materials were significantly lower compared with those of neat nylon 6. Compounding of neat nylon 6 pellets with the recycled plastics produced a blend material with good strength and failure strain properties. This research thus demonstrates that direct melt mixing can be an effective approach for recycling nylon/spandex blend fabrics.

Acknowledgments

The authors wish to thank Aquafil USA for their support of this research effort, and also thank National Natural Science Foundation of China (21174055), Graduate Students Innovation Project of Jiangsu Province in China (CXZZ1207246), the Fundamental Research Funds for the Central Universities (JUDCF10038) and the International Joint Research Laboratory for Advanced Functional Textile Materials of Jiangnan University.

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