

Article

Influence of Spinning Temperature and Filler Content on the Properties of Melt-Spun Soy Flour/Polypropylene Fibers

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Abstract: Polypropylene (PP) fibers are heavily used in disposable nonwovens fabrics because of their desirable properties and low-cost, but they are not biodegradable. With the goal of reducing non-biodegradable plastic waste in the environment, the primary aim of this study was to produce fibers with reduced content of PP for disposable fabrics by incorporating soy flour, a bio-based renewable material. An optimum processing temperature of 190 °C was established, and thin fibers with a diameter under 60 μm were successfully melt-spun. Inclusion of compatibilized soy (SFM) at 30 wt% resulted in fibers with a tensile modulus of 674 ± 245 MPa and a yield strength of 18 ± 4 MPa. At 15 wt% SFM, fiber tensile modulus and yield strength were 914 ± 164 and 29 ± 3, respectively. Although lower than those of neat PP fibers (1224 ± 136 MPa and 37 ± 3 MPa), these SFM/PP fiber properties are suitable for nonwoven applications. Additionally, partial presence of soy particulates on fiber surface imparted enhanced water absorption and colorability properties to the fibers while imparting the fibers the feel of natural fibers. Although more difficult to produce, soy-PP fibers possessed similar properties as compared to those of than soy-PE fibers reported in earlier studies.

Keywords: polypropylene; soy flour; melt-spun fibers; hydrophilic; tensile properties

1. Introduction

In the last 60 years, world-wide plastics usage has increased from 1.5 million tons to 400 million tons [1] because plastics are low-cost materials that are suitable for a wide range of household applications due to their ease of processing and durability. A segment of these plastic products consists of nonwovens that are produced from polymeric fibers. In the USA, 51 million tons of nonwovens were produced in 2018 [2]. This large volume can be attributed to the very low cost of production of nonwoven fabrics relative to their woven counterparts. Plastics improve the quality of life by contributing comfort, convenience, and safety. The major advantage of plastics and the resulting disposables products is their affordability. Consequently, these inexpensive products have become ubiquitous. Unfortunately, the ease of disposability has also led to significant environmental pollution. It is tough to give up the utilization and production of these polyolefin polymers due to their excellent performance/cost ratio, but their massive annual production coupled with improper disposal has become a major societal problem.

The world has started to become more aware of sustainability issues and starting to follow the rule of “3Rs”, i.e., “reuse, reduce use, and recycle”. Plastic reusing and recycling are personal choices. Reusable plastics are mostly durable products, available in the market, which can be used over and over again. However, ‘reuse’ strategy (i.e., first “R”) is not applicable to sanitary items and food packaging due to the cleaning difficulties. The second “R”, recycling, can be done only when suitable facilities are

available. Also, it is costly to build recycling centers, so in the USA only about 9% of plastic waste is recycled [3] and recycled plastics lead to products that have inferior properties (compared with virgin polymers). Moreover, recycling is not feasible for disposable nonwoven fabrics/fibers such as those used for sanitary purposes. Thus, reducing synthetic polymer use, the third “R”, is an essential component of the overall solution. While eliminating the use of synthetic polymers is not an immediate solution, reducing the use of such polymers by or incorporation of bio-based fillers are potential solutions.

Soybean is an inexpensive and abundantly available agricultural material that has attracted the attention of researchers for the past several years [4–10]. Soy has been incorporated into polymer matrices primarily in its bulk form in recent studies [11–15]. The primary purposes of adding soy-based filler are to reduce the environmental impact of synthetic polymer and the cost per unit weight of the product. However, soy flour (SF) has several challenges for being utilized with polyolefins. First, it is a hydrophilic material that is not compatible with hydrophobic polymer matrices. Filler/filler interaction is favored instead of filler/matrix interaction, and this leads to agglomeration and poor dispersion of particles in polymer matrices [12,13,16]. Chemical incompatibility leads to both compounding and spinning problems and ultimately results in poor mechanical properties. Several prior studies have reported on compatibilizers such as maleic anhydride modified polymers [15,17,18] and glycerol [13,19] for better adhesion between thermoplastic polymers and soy flour to improve processibility and mechanical properties of the blends. The processability and the mechanical properties generally are improved by the existence of proper compatibilizers. In our previous paper [20], spinnability of uncompatibilized and compatibilized soy flour was reported, and the positive effect of monoglyceride on the resulting properties of SF-filled polyethylene fibers was identified.

Second, challenges are also encountered in processing due to thermal instability of soy flour at temperatures where synthetic polymers are processed. Polymer viscosity can be fairly high at a processing temperature close to polymer melting temperature. The material becomes more viscous with filler addition that can lead to localized heating due to viscous dissipation. Therefore, processing of filled systems needs more power than that of the neat polymer [21].

In our prior study [20], polyethylene (PE) was investigated due to its lower processing temperature, as compared to that of PP. A temperature as low as 140 °C could be used for soy-PE fibers, where soy degradation was minimal, but this temperature is too small for processing PP-based fibers because PP melts at 150 °C and can be spun only at temperatures at or above 160 °C (approximately 20 °C higher than melting point). However, PP is the most widely used polymer in disposable nonwoven applications because of its better mechanical properties and low cost compared to PE and other synthetic fibers [22]. However, PP has a high melting temperature of 150 °C, about 40 °C higher than that of PE. This leads to challenges for melt-mixing of soy flour in PP melt, so melt-spinnability/processing of soy/PP fibers has not been systematically investigated in prior literature studies. Therefore, the objectives of the present study were to: (i) identify melt-mixing and melt-spinning conditions for soy/PP fibers, (ii) determine the effect of soy content on the mechanical properties of soy-PP fibers and (iii) investigate the suitability of soy-PP fibers for disposable nonwovens in terms of moisture absorption and hydrophilic characteristics.

2. Experimental

2.1. Materials

Defatted soy flour, soy flour 7B, (53 wt% protein, 30 wt% carbohydrate, 7 wt% moisture, 1 wt% fat, and remainder dietary fiber) was obtained from Archer Daniels Midland (Chicago, IL, USA). A fiber grade PP, Dow 6D43 Resin, (melt flow index: 35 g/10 min at 230 °C, density: 0.9 g/cm³, Dow Chemical, Midland, MI, USA) was obtained in a pellet form. Dimodan distilled monoglyceride was used as a compatibilizer between soy flour and PP and purchased from Danisco DuPont (Century, KS, USA). Soy flour and monoglyceride were dried in a vacuum oven for 2 h at 80 °C (~100 kPa vacuum).

2.2. Spinning

Soy flour and monoglyceride were manually mixed in 4:1 ratio on a weight basis chosen according to the previous studies [20,23]. About 15 g of the mixtures were prepared by physical mixing of polypropylene pellets at 70 wt%, 85 wt%, and 95 wt% with compatibilized soy (S), and feeding to a 15 mL twin-screw extruder (MC15HT, DSM Xplore, Geleen, The Netherlands) consisting of conical screws nominally 160 mm long and having a diameter of 22 mm at the entrance that tapered down to 8 mm at the end. Because of the short length of the extruder barrel, there was not any distinction with regards to heating zone temperatures, i.e., the entire barrel remained essentially at one temperature identified as the spinning temperature. The blends and the neat PP (as a control) were compounded in the speed-controlled, co-rotating, and recirculation mode at a rotation speed of 100 rpm and mixed using a recirculation time of 2 min.

The blend containing 15 wt% compatibilized soy (SFM) was used to conduct viscosity testing using an ARES rheometer (TA Instruments, New Castle, DE, USA) equipped with a cone-plate fixture of 25 mm diameter and a cone angle of 0.1 rad. Shear viscosity was measured at four different temperatures: 160, 190, 220, and 250 °C. Fibers containing 15 wt% SFM (85 wt% PP) were also spun at melt temperatures of 160, 190, 220, and 250 °C. In our prior study, we had determined that soy could be incorporated into a polyethylene matrix at a temperature of only 140 °C. However, due to the higher melting point of PP, significantly higher processing temperatures had to be used, which present additional processing challenges due to degradation of soy. As shown in Figure 1, a custom-designed 3-hole spinneret, having a capillary diameter of 500 µm was attached at the end of the extruder. An air-knife was used to cool the melt right after the die exit. Fiber spinning was carried out in the force-controlled mode at a force of approximately 3000 N. Fibers were obtained using a draw-down ratio of approximately 100. SFM/PP fibers containing 70 wt% and 95 wt% PP were spun at only 190 °C. The nomenclature of the fibers is summarized in Table 1.

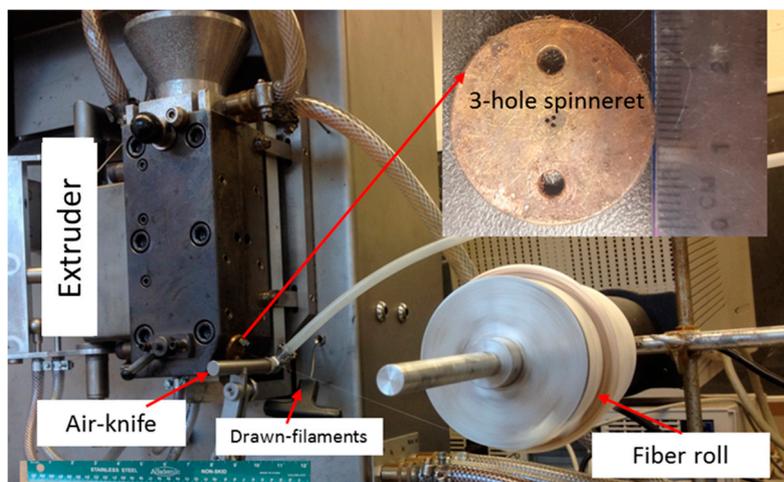


Figure 1. Spinning of SFM/PP fiber containing 5 wt% soy flour and 95 wt% polypropylene using the DSM twin-screw extruder.

Table 1. Compositions of the blends and spinning temperatures for various soy flour (SF), monoglyceride (M), and polypropylene (PP).

Fibers	SF+M (wt%)	SFM (wt%)	PP (wt%)	Spinning Temperature (°C)
SFM5P95-190	4 + 1	5	95	190
SFM15P85-190	12 + 3	15	85	190
SFM30P70-190	24 + 6	30	70	190
SFM15P85-160	12 + 3	15	85	160
SFM15P85-220	12 + 3	15	85	220
SFM15P85-250	12 + 3	15	85	250

2.3. Characterization

Thermogravimetric analysis (TGA) was conducted using a TA instrument (TGA Q5000, TA Instruments). The SFM/PP blend with a nominal sample mass of 10 mg was heated in a platinum pan under the air atmosphere at a heating rate of 10 °C/min. The temperature was increased from 25 °C to 160 °C, 190, 220 and 250 °C, as four separate experiments, and kept at those temperatures for 20 min. Differential scanning calorimetry (Perkin-Elmer Pyris 1, Waltham, MA, USA) for SFM/PP fibers was conducted to obtain glass transition, melting, crystallization, and cold crystallization temperatures. Aluminum pans were used for each sample with an average sample mass of 5 mg. The heating and cooling scan rates were 20 °C/min under a nitrogen atmosphere from 35 °C to 225 °C. The degree of crystallinity was calculated from measured cold crystallization enthalpy (ΔH_{cc}), estimated ΔH_f^0 (209 J/g), and PP concentration in SFM/PP blend (X_{pp}) using the following equation [12]:

$$w_c = (\Delta H_{cc}) \times 100 / (\Delta H_f^0 \times X_{pp}) \quad (1)$$

Scanning electron microscopy (SEM) was used to characterize the microstructure and morphology of the fibers (S-4800, Hitachi, Japan). The fibers were fractured cryogenically to obtain cross-section images and sputter-coated with platinum to avoid charging. Optical microscopy (BX60; Olympus Corp., Lake Success, NY, USA) was used to measure single fiber diameters. Average fiber diameters were calculated by using 50 measurements from each batch.

A thermo Nicolet 6700 FTIR spectrometer (Thermo Scientific, Madison, WI, USA) was used to conduct Fourier transform infrared (FTIR) spectroscopy in the attenuated total reflectance mode. Spectra for SFM30PP70-160 fibers and neat components of monoglyceride, soy flour, and polypropylene were obtained in a spectral range of 4000–400 cm^{-1} wavenumbers.

Static tensile tests were conducted following the ASTM D2256 procedure on single fibers, with an initial gage length of 2.54 cm. Mechanical testing of the fibers was performed at a cross-head speed of 0.25 cm/min with a 22 N load cell (Series 900, Applied Test Systems Inc., Butler, PA, USA). Five replicates were tested from each group.

Dynamic mechanical analysis of the fibers was conducted using a RSA3 (TA Instruments) to measure the storage modulus (E') and $\tan \delta$ as a function of frequency. The fibers were tested in the dynamic tensile mode at a dynamic strain of 1% and a frequency range of 1 to 100 rad/s. Isothermal runs were conducted at 25, 50, and 75 °C.

The contact angle between water and SFM/PP blends was measured by pressing the blends into films at 160 °C and 20 kN force (Carver hydraulic press, Wabash, IN, USA). The test was performed using a contact angle goniometer (Model DSA10, Kruss, Hamburg, Germany) using a sessile drop technique. The static contact angle measurements were obtained with distilled water at a static time of 30 s.

Fibers containing soy content of 0 (i.e., neat PP), 5, 15 and 30 wt% were dried for 4 h at 80 °C in a vacuum oven for moisture absorption studies. Approximately 1 g of fibers were exposed to moisture for 1 h. The fibers were periodically removed from the steam environment, wiped using Kim-wipes

(Irving, TX, USA), and dried with a blow-dryer for 20 s to remove the free/excess water left on the fiber surface. After weighing, the fibers were placed again in the steam environment for subsequent tests.

3. Results and Discussion

3.1. Thermal Degradation

Thermal gravimetric analysis was performed to determine the thermal stability of the SFM/PP blend, which is essential for melt-mixing and subsequent melt-spinning. Isothermal TGA scan was conducted for only 30/70 wt% SFM/PP composition because the highest soy content gave the highest resolution for weight loss during degradation. Figure 2 displays the results for 30/70 wt% SFM/PP blend for different isothermal runs with holding temperatures ranging from 160 to 250 °C. As expected, the blend displays the most stable response at the lowest temperature of 160 °C, and weight loss observed at 160 °C for a holding time of 20 min is less than 0.1%. At higher temperatures, the blend has a weight loss of about 0.2 wt% at 190 °C and 0.6 wt% at 220 °C, for a processing time of 2 min. The weight loss increases to about 0.5 wt%, 1.8 wt%, and 5 wt% at 160, 190, and 220 °C, respectively, for a longer holding time of 20 min. At 250 °C, the weight loss increases significantly from 2 wt% for 2 min holding time to about 10 wt% for 20 min holding time.

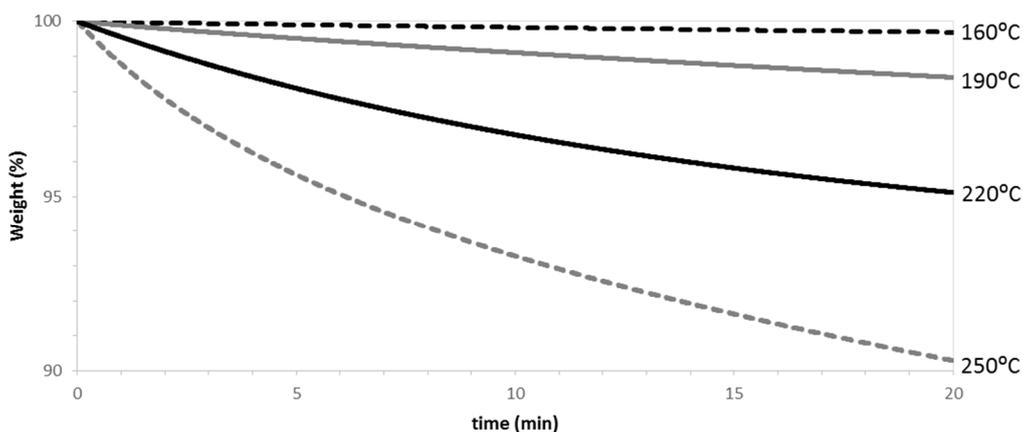


Figure 2. Isothermal TGA (at 160 °C, 190 °C, 220 °C and 250 °C for 20 min) of the blends having 30 wt% SFM and 70 wt% PP.

Above results establish that SFM/PP has good thermal stability for processing at 160 °C and 190 °C, but only moderate stability at 220 °C. At 250 °C, there is a sharp increase in the thermal degradation level. In a previous study, we have shown that monoglyceride displays a drastic weight loss around 250 °C (Guzdemir et al. [20]). Higher temperature and residence time accelerates the degradation of soy flour and also monoglyceride [20] while PP stays stable at this temperature [14].

3.2. Melting Behavior and Crystallinity

The melting characteristics of various compositions are presented in Table 2. The melting temperature peak of neat polypropylene was observed at 150 °C and its degree of crystallinity was 34%. The melting peak of the SFM/PP blends ranged from 151 °C to 146 °C, not significantly different from pure PP. A small increase in the degree of crystallinity was observed for soy-filled PP samples. These minor effects are generally consistent with those reported in prior literature studies [12,14].

Table 2. DSC results of neat PP and SFM-PP blends.

Melting			
Material	Melting Temperature T (°C)	Enthalpy of Melting ΔH_m (J/g mix)	Degree of Crystallization X_c (%)
PP	150	53.3	34.0
SFM5PP95	151	44.2	35.2
SFM15PP85	147	33	35.5
SFM30PP70	146	31	38.3

3.3. Effect of Temperature on Fiber Spinnability and Properties

Based on thermal stability analysis, fiber spinning temperatures were chosen as 160, 190, and 220 °C. The mixing time and spinning time were determined as 2 min and 20 min, respectively. Limited fibers were also spun at 250 °C to confirm the degradation observed in TGA results. For 15 wt% filler content, SFM15-PP85 fibers were successfully produced by melt-spinning. At the lowest temperature of 160 °C, the SFM/PP blend was highly viscous as compared with those at higher temperatures, and the spinnability of the blend was limited due to the fact that polymers can be drawn-down (often called melt strength) less at lower temperatures [24,25]. At 190 °C and 220 °C, the flow was smooth, and extensibility of the blend increased, so it resulted in finer fiber diameters. This observation is consistent with independent rheological measurements, shown in Figure 3. The shear viscosity of the blend at 160 °C is twice of that at 190 °C, i.e., as expected viscosity decreased as temperature increased. The viscosity behavior was fitted to a Power-law model: $\eta = K (\dot{\gamma}^{n-1})$ where η (Pa·s) is viscosity, K is consistency index (Pa·sⁿ), n is flow behavior index and $\dot{\gamma}$ is shear rate (s⁻¹). The power-law parameters, displayed in Table 3, indicate that shear-thinning is evident at all temperatures (all n values less than 1). However, an increasing value of power-law exponent at increasing temperatures indicates that the extent of shear thinning decreases with increasing temperatures because it is known that Newtonian fluids display a power-law constant $n = 1$.

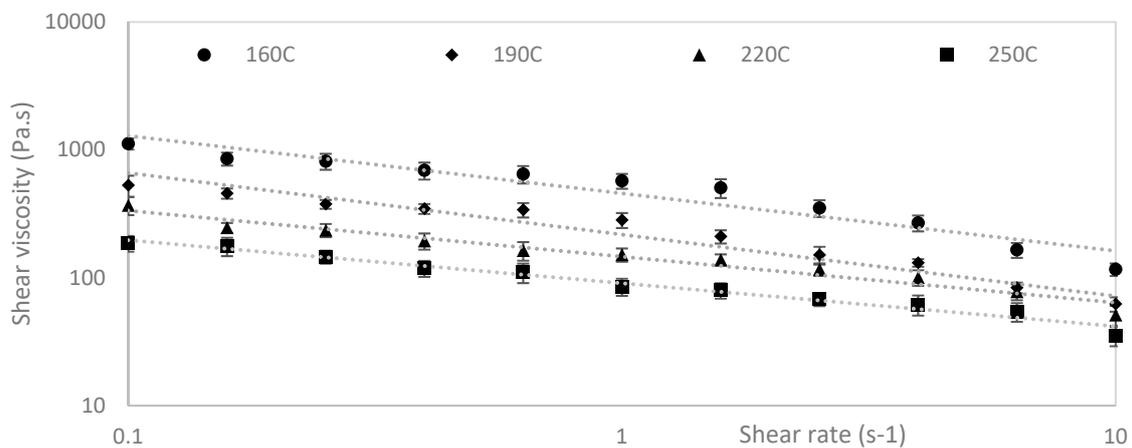


Figure 3. Shear viscosity of SFM15-PP85 blend at 160, 190, 220, and 250 °C. (Linear least square fit is shown by dotted lines for each sample).

Table 3. Power-law viscosity parameters of soy/PP blends: $\eta = K (\dot{\gamma}^{n-1})$ where η (Pa·s) is viscosity, K is consistency index (Pa·sⁿ), n is flow behavior index and $\dot{\gamma}$ is shear rate (s⁻¹).

Sample	N	K (Pa·s ⁿ)
SFM15-PP85-160	0.55	457
SFM15-PP85-190	0.52	217
SFM15-PP85-220	0.64	146
SFM15-PP85-250	0.67	91

Representative fibers produced from SFM15-PP85 are displayed in Figure 4. The top row displays photographs of fibers starting with neat PP (left, for comparison) followed by fibers spun at temperatures increasing from 160 to 250 °C. The average fiber diameters were measured at 65 ± 11 , 59 ± 4 , 50 ± 6 , 107 ± 27 μm at 160 °C, 190 °C, 220 °C, and 250 °C, respectively. This is consistent with prior literature results that confirm that at higher melt temperatures (160 to 220 °C), polymer blend has higher extensibility and easier draw-down [26]. However, at 250 °C, soy flour degradation limited the extensibility of the blend, and the final product was filaments with large diameters. Starting with neat PP (white), fiber color turned light brown at 160 and 190 °C. At 220 and 250 °C, the fibers had a dark brown color because of significant soy flour decomposition. This result is in good agreement with the ones obtained by the thermal degradation test. The color change is likely due to Maillard reaction [27,28] where the soy sugars and soy protein react leading to a decrease in the content of hydrophilic groups and improvement of some properties of soy flour like bonding strength [29]. Darker shades were produced at increasing spinning temperatures due to increasing extents of the reaction [28,30]. Also displayed in the bottom row of Figure 4 are SEM micrographs of fiber cross-sections with black arrows pointing to visible soy particles. The micrographs indicate the clear presence of soy particles at 160 and 190 °C, but a significantly rough texture is evident at the higher temperatures of 220 and 250 °C due to significant thermal degradation of soy, which led to the formation of large voids as indicated by red arrows (Figure 4h,j).

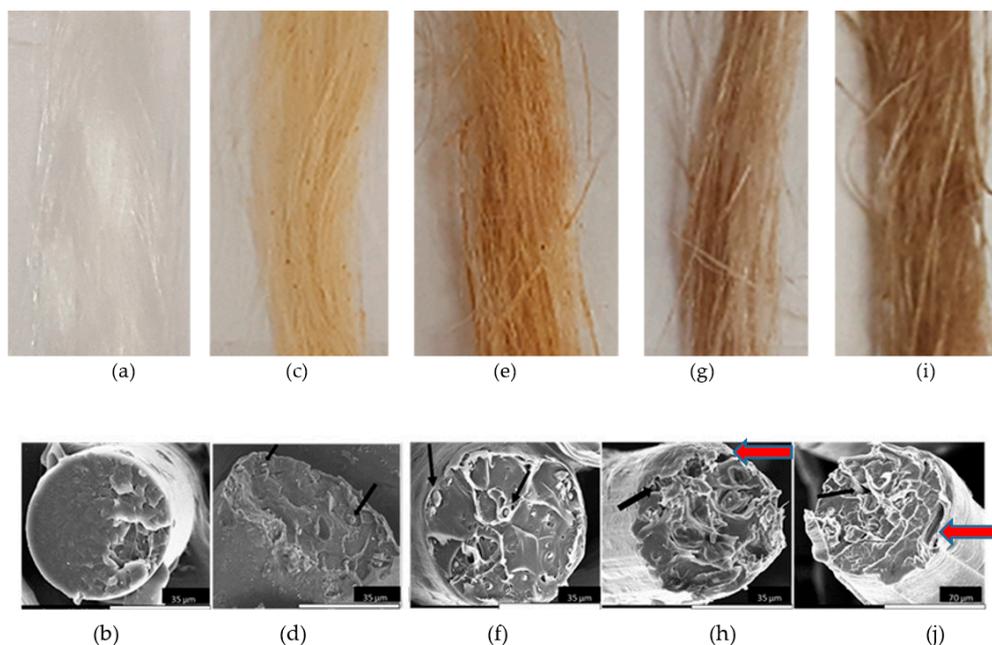


Figure 4. Representative photographs and cross-sectional SEM images, respectively of (a,b) neat PP fibers (for comparison); and soy filled PP fibers (SFM15-PP85) spun at various temperatures: (c,d) 160 °C, (e,f) 190 °C, (g,h) 220 °C and (i,j) 250 °C. Black arrows represent soy particulates, whereas red arrows indicate voids.

Representative tensile stress-strain curves for SFP15-PP85 fibers spun at four different temperatures are reported in Figure 5, and various material properties are summarized in Table 4. As the spinning temperature increased from 160 to 220 °C, the yield stress decreased from 35 ± 6 MPa to 19 ± 4 MPa, which is an acceptable value for nonwoven fabrics. It is noted that when fibers are converted into fabrics, yield strength is the more relevant material property as compared with ultimate strength because fibers start to undergo plastic deformation at yield stress, so that is the upper limit of how fast the fibers can be pulled during fabric formation. At 250 °C, all fibers properties were reduced due to significant degradation of soy flour. In comparison, neat PP displayed a tensile modulus, yield strength, tensile strength, and strain-to-failure values of 1224 ± 136 MPa, 37 ± 3 MPa, 104 ± 10 MPa, and $260 \pm$

35%, respectively. Although lower than those of neat PP fibers, SFM/PP fibers processed at 160 and 190 °C had similar tensile properties, but fiber spinning was easier at 190 °C. Properties deteriorated significantly at 220 and 250 °C. The drastic reduction of yield strength and strain-to-failure (over 50% reduction) for fibers spun at 250 °C is consistent with the presence of voids, as observed in Figure 4j. Therefore, for further studies, 190 °C was chosen for subsequent mixing/spinning trials.

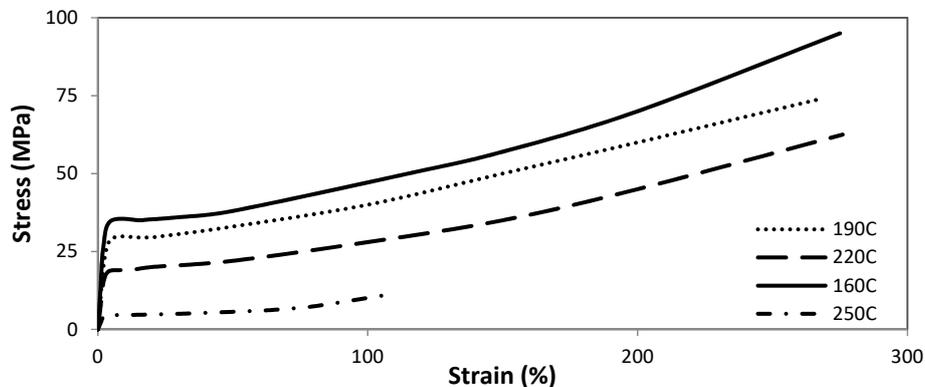


Figure 5. Stress-strain curves of SFM15PP85 fibers spun at 160, 190, 220, and 250 °C.

Table 4. Summary of static mechanical properties of SFM15-PP85 soy flour-filled PP fibers.

	Processing Temperature (°C)	Tensile Modulus (MPa)	Yield Stress (MPa)	Yield Strain (%)	Failure Strength (MPa)	Strain-To-Failure (%)
1	160	1313 ± 419	35 ± 6	4.8 ± 0.4	95 ± 30	275 ± 42
2	190	914 ± 164	29 ± 3	5.0 ± 1.0	74 ± 7	268 ± 57
3	220	743 ± 217	19 ± 4	3.8 ± 0.7	63 ± 9	276 ± 41
4	250	207 ± 108	4.5 ± 2	2.6 ± 0.9	11 ± 5	106 ± 48

3.4. Effect of Filler Composition on Mechanical Properties

Next, fibers filled with 5, 15, 30 wt% SFM were spun at 190 °C. SEM micrographs of the lateral (left) and cross-sectional (right) surfaces of neat PP and soy-PP fibers containing 5, 15 and 30 wt% SFM are displayed in Figure 6a–d.

The nominal circular cross-sectional shape of neat PP fibers was retained in soy-PP fibers, although the presence of soy particles imparted more texture. As the soy content increased, the lateral surfaces clearly display increasing texture, i.e., soy particles protruding from the fiber surface. This is a consequence of soy not being miscible in PP, which results in only partial dispersion of soy particles within the PP matrix. Contrary to the thinking that rough surface is undesirable, this rough surface is actually desirable because it provides such blend fibers with a tactile feel similar to natural fibers such as cotton, which have a crenulated and irregular cross-section (i.e., not smooth and circular that pure PP has). In prior studies, we have established that polyolefin bi-component fibers with an outer sheath containing an immiscible blend, with the minor component protruding out of the surface, provide a desirable tactile feel [31].

Representative tensile stress-strain characteristics of various fibers are displayed in Figure 7 and the material properties are summarized in Table 5. The yield strain and strain-to-failure of SFM/PP fibers are not statistically different from those of neat PP fibers and are not affected by the soy composition because the continuous PP phase dominates the strain behavior of the composite.

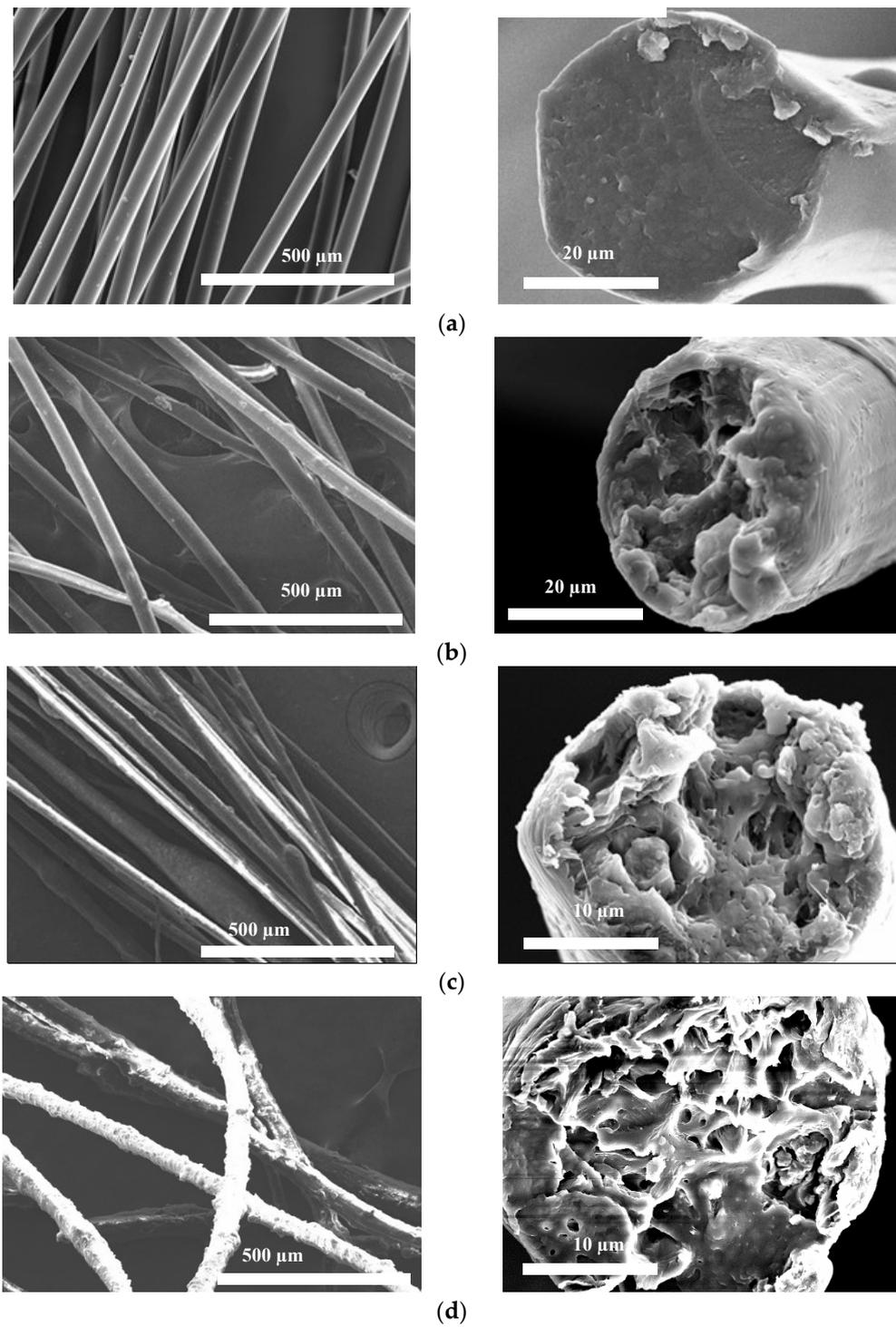


Figure 6. Scanning electron micrographs of lateral (left) and cross-sectional (right) surfaces of soy-PP fibers spun at 190 °C and containing various soy contents: (a) neat PP, (b) SFM5PP95 (c) SFM15PP85, and (d) SFM30PP70.

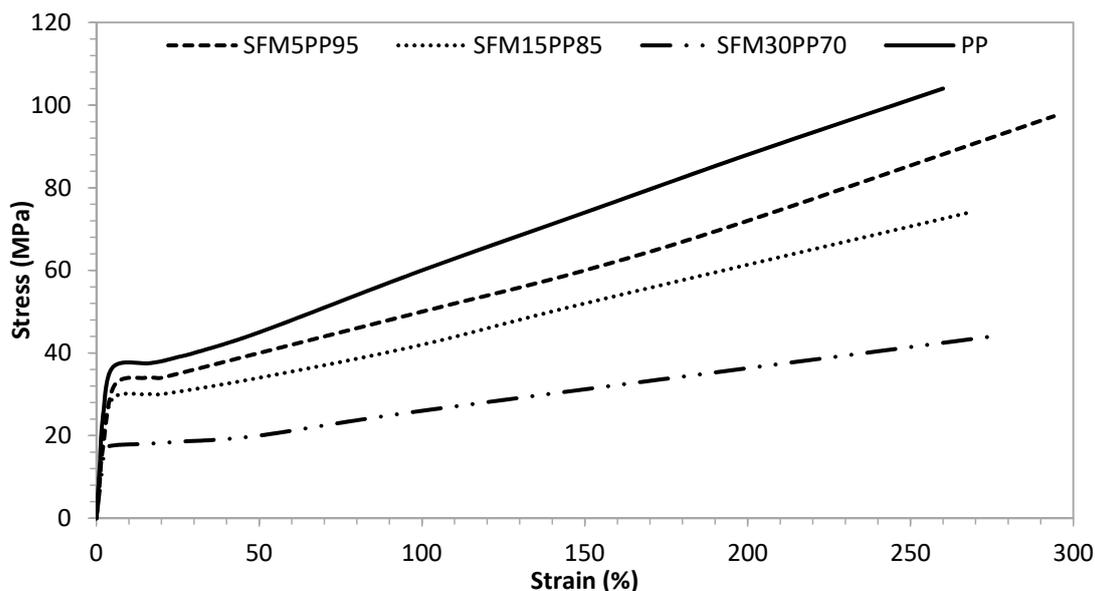


Figure 7. Stress-strain curves of SFM/PP fibers spun at 190 °C with SFM contents of 5, 15, and 30 wt%.

Table 5. Static mechanical properties of the fibers spun at 190 °C and containing 5, 15, and 30 wt% SFM. Also shown is the response of neat PP fibers.

Fibers	Tensile Modulus (MPa)	Yield Strength (MPa)	Yield Strain (%)	Tensile Strength (MPa)	Strain to Failure (%)
Neat PP	1224 ± 136	37 ± 3	5.0 ± 1.3	104 ± 10	260 ± 35
SFM5PP95-190	843 ± 140	32 ± 4	5.5 ± 0.5	97 ± 16	294 ± 46
SFM15PP85-190	914 ± 164	29 ± 3	5.0 ± 1.0	74 ± 7	268 ± 57
SFM30PP70-190	674 ± 245	18 ± 4	6.1 ± 2.6	44 ± 11	275 ± 42

The yield strength of soy/PP fibers decreased to 32 MPa, 29 MPa, and 18 MPa for 5, 15, and 30% SFM/PP fibers, respectively, as compared with neat PP yield strength of 37 MPa. A similar decreasing trend was observed for tensile strength; values of 97, 74, and 44 MPa were measured for fibers containing 5, 15, and 30% soy, respectively. The reduction of strength with increasing soy content is consistent with prior literature results of Sailaja et al. [13] for bulk soy/PP composites. Although it was more difficult to spin soy-PP fibers relative to their PE counterparts (due to the higher temperatures for PP where soy degrades), these tensile properties are similar (or slightly better) to those for soy-PE fibers reported in our earlier study [20]. For instance, the yield strength and modulus of soy-PP fibers at 30 wt% SFM was 18 and 674 MPa, respectively, when the values were slightly lower at 15 and 615 MPa for PE-based fibers (also at 30 wt% soy). However, due to the slightly higher degradation of soy at the higher spinning temperature of 190 °C for PP-based fibers, as compared with only 140 °C for PE-based fibers, the ratio of properties for the blend fibers relative to their neat polymer counterparts was slightly lower at 0.5 for PP-based as compared to 0.6 for PE-based fibers.

It is noted that while clays and hard inclusions lead to an increase in composite modulus [32–35], inclusion of soy flour led to a reduction in tensile modulus for 15 and 30 wt% composites, as compared to that of neat PP fibers. The decrease in tensile modulus is explained by the lower stiffness of SFM. The reduced stiffness provides a softer feel to the composite fibers as compared to that of neat PP fibers. These results indicate that fibers containing up to 30 wt% SFM are potential as fibers for disposable nonwoven fabrics.

Figure 8a,b displays the dynamic storage moduli and $\tan \delta$ for various fibers. The storage moduli were measured in the range of 1047–1570 MPa, 988–1270 MPa, 664–1130 MPa, and 450–706 MPa for neat PP, SFM5PP95, SFM15-PP85, and SFM30-PP70 fibers, respectively. The storage moduli decreased with increasing SFM content, consistent with the trend discussed earlier for static moduli. The storage

moduli and $\tan \delta$ for SFM15PP85 fibers at three different temperatures are presented in Figure 8c,d. As expected, fibers showed a decrease in the storage moduli with increasing temperature. Also, a slight increase in moduli is observed with increasing frequency due to a relatively greater elastic response typically observed for all viscoelastic materials. Also, as expected, $\tan \delta$ increased with increasing temperature consistent with the fact that polymeric materials display greater viscous (as compared with elastic) response at elevated temperatures.

To investigate the nature of interactions among the three components in SFM-PP blend, FTIR analysis was conducted. Spectra for neat monoglyceride, soy, and PP, and SFM-PP are displayed in Figure 9. Monoglyceride has two strong peaks at 3000–2850 cm^{-1} due to C-H stretching and one peak at 1715 cm^{-1} due to the carbonyl (C = O) [36]. For the protein component of soy, the amide I bond (C = O) stretching in the protein secondary structure, appears at 1630 cm^{-1} , whereas soy carbohydrate bands are located between 1200 and 1000 cm^{-1} due to C-O, C-C, and C-O-H stretching/bending [20]. For PP, the main absorption bands are at 2951, 2839, 1450, and 1375 cm^{-1} attributable to alkyl groups (C-H stretching) [37]. SFM-PP fiber spectrum consists of a combination of these aforementioned peaks, with broad band at 3300 and 3307 cm^{-1} attributable to O-H from soy protein and monoglyceride, and N-H from soy protein [20,38,39]. Finally, a numerically superposed spectrum was created by combining the spectra of neat components (soy/monoglyceride/PP). When the actual spectrum of SFM-PP fibers is compared with the numerically superposed spectra, it is found that there is not any new peak that is formed during melt processing of SFM-PP fibers. This indicates that the interaction between soy, monoglyceride, and PP is primarily physical in nature. This is consistent with the absence of any additional phase in the SEM micrographs.

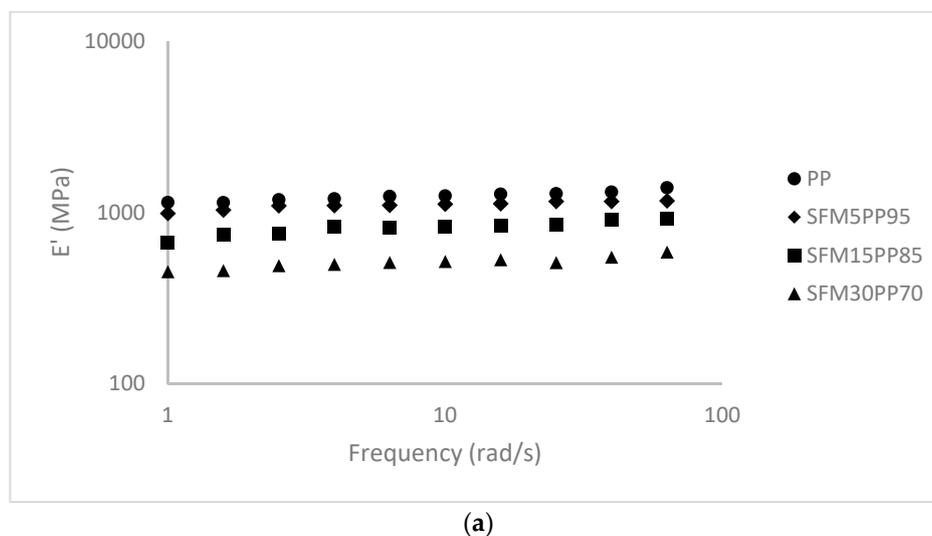
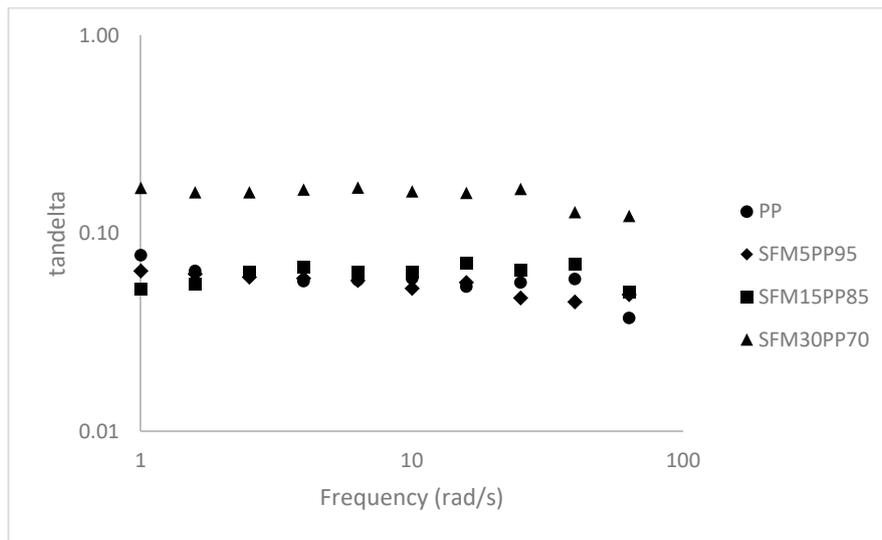
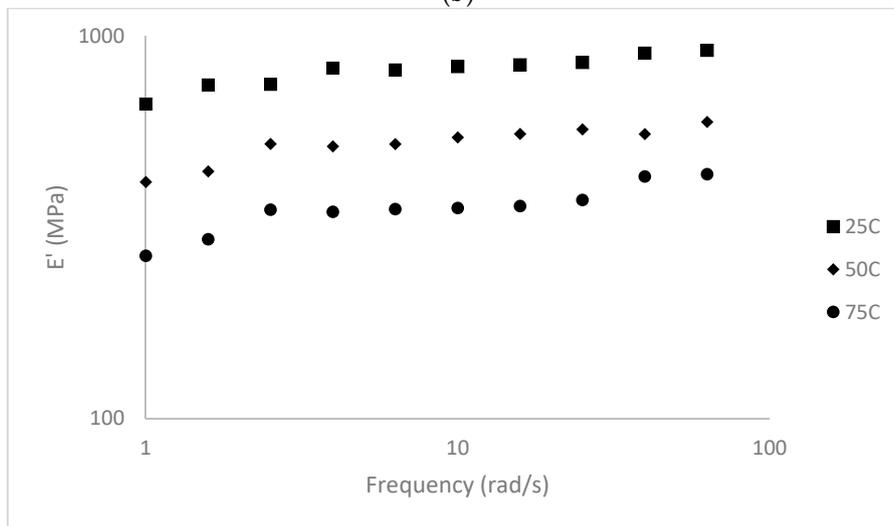


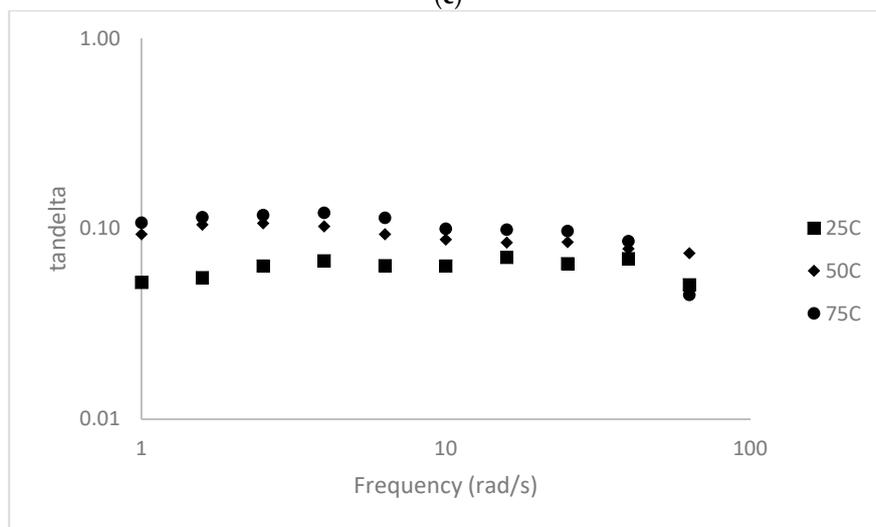
Figure 8. Cont.



(b)



(c)



(d)

Figure 8. Dynamic storage modulus and $\tan \delta$ at 1% strain as a function of frequency: (a,b) for fibers containing various SFM contents, and (c,d) for SFM15PP85 fibers at various temperatures.

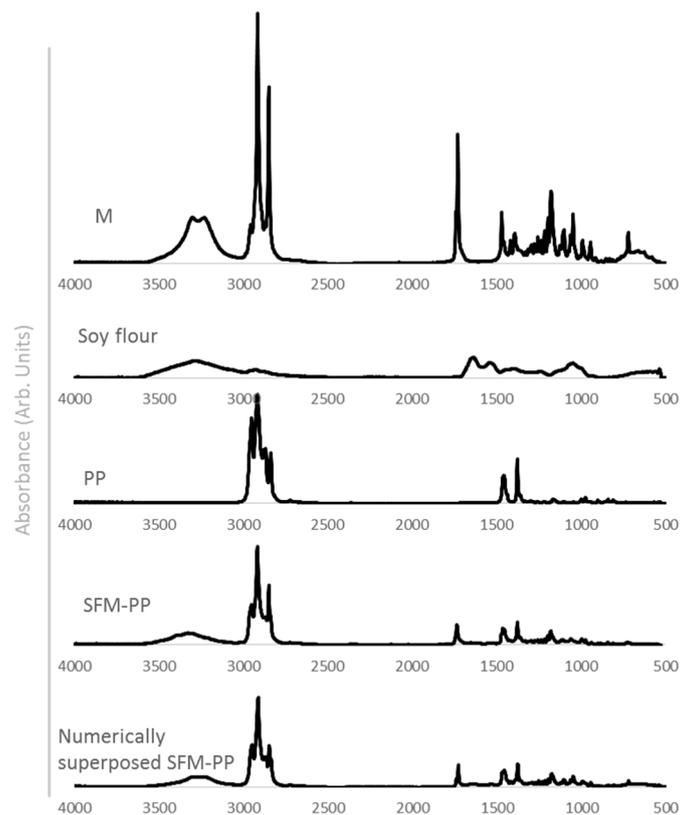
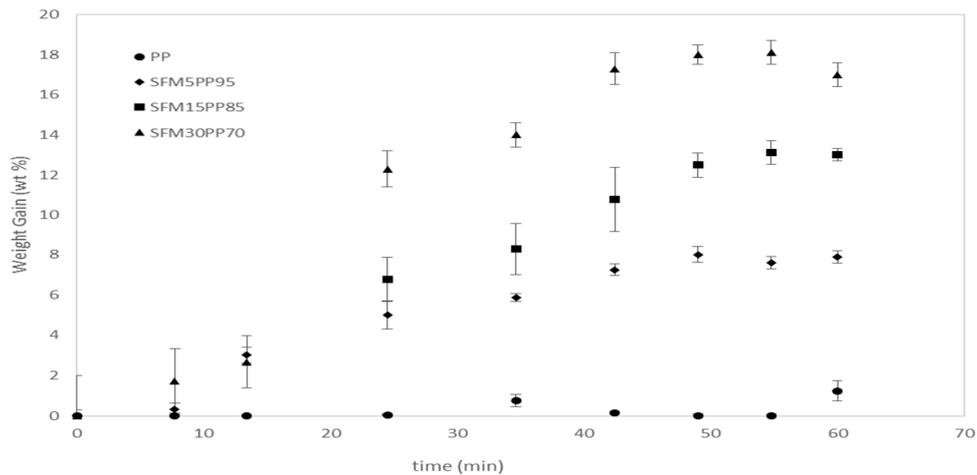


Figure 9. FTIR spectra of neat monoglyceride, neat soy flour, neat polypropylene, SFM30-PP70-160 fiber, and a superposition of all three neat component spectra.

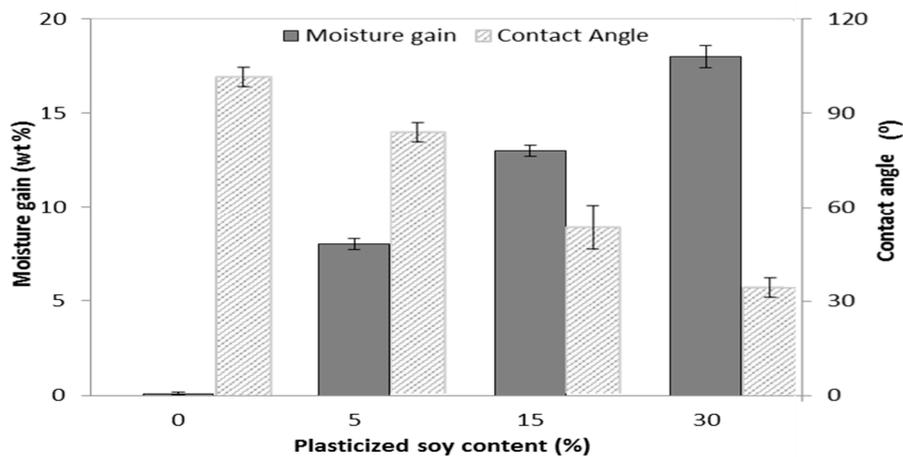
3.5. Hydrophilicity/Coloring

For use in disposable nonwoven fabrics, other properties of fibers are also important and were investigated next. Some disposable nonwoven fabrics come in contact with water and body fluids, e.g., sanitary pads, diapers, and band-aids. Therefore, contact angles with water were measured for the three different soy contents of 5, 15, and 30 wt%. These measurements were performed on films obtained from pressing appropriate extrudates into films. The contact angles were measured at $101 \pm 3^\circ$, $83 \pm 3^\circ$, $53 \pm 7^\circ$ and $34 \pm 5^\circ$, respectively, for the blends having soy contents of 0 (PP as control), 5, 15, and 30 wt%. Contact angles larger than 90° indicate a hydrophobic surface. The lower angles measured for SFM/PP surface revealed that presence of soy particles on the surface improved the hydrophilicity of the composite.

Figure 10 displays the moisture absorbed after 1 h exposure time for SFM/PP fibers at different compositions, with neat PP fibers included as a control. One hour exposure time was chosen to mimic how such fibers might behave when used in disposable sanitary nonwovens. It is evident that neat PP fibers (i.e., without soy flour) have the lowest moisture absorption capacity with no measurable uptake (~ 0 wt%), consistent with the hydrophobic nature of PP. At 5 wt% soy content, the moisture gain by fibers was measured at 8 wt%. This is an indicator that even a small soy content can impart some hydrophilic character to PP-based fibers. At 15 wt% SFM content, the fiber weight gain was 13 wt%, increasing to 18 wt% for composite fibers containing 30 wt% soy. This hydrophilic fiber property is desirable in many nonwoven fabrics that come into contact with human skin, such as in disposable/sanitary applications. Further, this moisture gain is similar to that observed for soy-PE fibers (about 20 wt%) reported in our earlier study [20]. This is consistent with the fact that the soy particles present on the surface are primarily responsible for moisture gain as PE and PP matrices by themselves are both highly hydrophobic.



(a)



(b)

Figure 10. (a) Moisture absorption of SFM/PP fibers over time and (b) moisture absorption and contact angles of SFM/PP blends having 5 wt% soy (SFM5PP95), 15 wt% SFM and 70 wt% PP (SFM15PP85), 30 wt% SFM, and 70 wt% PP (SFM30PP70). Also shown are data for PP (control) fibers (0 wt%). The absorption test was done for 1 h.

To determine the extent of deterioration of fiber properties, tensile testing was also conducted on fibers exposed to moisture. Also, displayed in Table 6, the fibers preserved almost 90% of their tensile properties. Therefore, the fibers are suitable for applications where the fabrics may be exposed to moisture.

Table 6. Mechanical properties of SFM15PP85-190 fibers after moisture-contact. Pure PP fibers were used as control samples.

Fibers	Tensile Modulus (MPa)	Yield Stress (MPa)	Yield Strain (%)	Failure Strength (MPa)	Strain to Failure (%)
PP	1210 ± 122	36 ± 3	5.1 ± 1.1	100 ± 12	262 ± 31
PP washed	1214 ± 83	43 ± 6	7.4 ± 1.0	90 ± 25	215 ± 47
SFM15PP85-190	914 ± 164	27 ± 3	5.0 ± 1.0	74 ± 7	268 ± 57
SFM15PP85-190-washed	780 ± 96	28 ± 5	5.4 ± 1.2	58 ± 9	247 ± 61

The other desired property for any textile product is its colorability. Figure 11 displays neat and filled PP fibers before and after coloring in a water-soluble red food dye (McCormick™, Baltimore, MD, USA). While control sample (neat PP fibers) could not absorb any color after immersion in colored

water for 10 min, SFM/PP fibers turned light pink on the matrix; the red dots on the fiber represent soy agglomerates. This observation is consistent with the studies show that neat PP is hard to dye with organic colors by classical methods because of its non-polar (purely aliphatic) structure as well as high crystallinity [40,41]. Increased hydrophilicity provides ease of coloring to the fibers. In addition, even without any coloring, SFM/PP fibers have a tan color that resembles some natural fibers like flax that have the potential for disposable nonwovens.

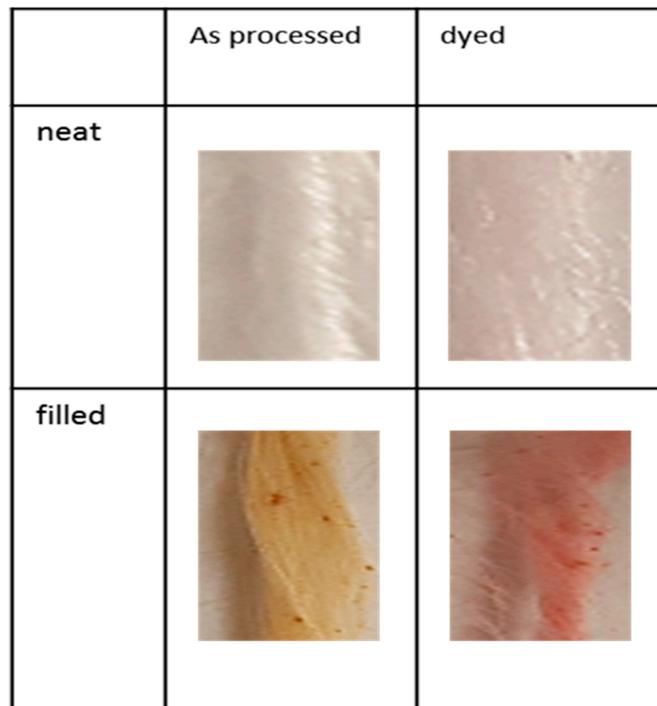


Figure 11. Images of neat PP and SFM15PP85-190 fibers before and after immersion in red dye.

4. Conclusions

With an important goal of reducing the content of synthetic/non-biodegradable polymers in disposable nonwoven fibers, soy flour was successfully incorporated into PP fibers using a scalable melt-spinning route. SFM/PP fibers, having 15 wt% soy, spun at 190 °C showed 25% less tensile modulus and tensile strength than those of neat PP fibers. As the spinning temperature increased from 160 °C to 220 °C, yield stress decreased monotonically from 35 ± 6 MPa to 19 ± 4 MPa due to the increased thermal degradation of soy flour. Tensile modulus and strengths were adversely influenced by increasing soy content, but the strain-to-failure was not significantly affected as it was dominated by the matrix response. The inclusion of 30 wt% soy resulted in fibers with a tensile modulus of 674 ± 245 MPa and a tensile strength of 44 ± 11 MPa, as compared to neat PP fibers with a tensile modulus of 1224 ± 136 MPa and a tensile strength of 104 ± 10 MPa. Thus, a spinning temperature of 190 °C and a soy content of 15 wt% provided ease of processability combined with adequate retention of tensile properties of SFM/PP fibers for potential use in disposable nonwoven fabrics. Increasing soy content led to the fibers with improved hydrophilic characteristics relative to the neat PP fibers, which are highly hydrophobic. Moisture absorption studies revealed that the composite fibers containing 30 wt% SFM gained 18 wt% moisture. Also, the presence of soy agglomerates on the surface provided the composite fibers a natural fiber-like feel and ease of colorability. While the soy-PP fibers were more difficult to produce than soy-PE fibers, overall soy-PP fibers possessed similar properties as their PE-counterparts. However, because PP fibers are heavily used in disposable nonwovens, even partial substitution of PP with soy represents a step in the right direction of reducing plastic waste in the environment.

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