

Development of needle-punched nonwovens made from waste milkweed and PET fibers

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Abstract

Despite the widespread usage and affordability of petroleum-based products, there has been tremendous effort in prioritizing and utilizing biodegradable and environmentally friendly materials. Untraditional natural fibers play a critical role in sustainability studies; however, fibers such as kapok and milkweed are quite expensive compared to other plant-based natural fibers such as cotton and flax. Therefore, it is critical to utilize these untraditional fibers in the most efficient manner that is possible. In this study, short milkweed fiber leftovers collected from the milkweed yarn spinning process were utilized as a filler material inside the needle-punched nonwoven fabrics. For this purpose, short milkweed fibers were blended with hollow polyethylene terephthalate (PET) fibers to develop nonwovens. Three different sets of weight/g fabrics were prepared for both only PET containing and short milkweed/PET blended fibers. Thickness, weight, tensile and bursting strength, thermal comfort, air permeability, and water contact angle measurements were conducted for the samples. Consequently, the thermal resistance of short milkweed/PET blended fabrics with similar weight increased by up to 34% compared to only PET-containing fabrics. Results indicate that valuable short milkweed fibers are suitable for developing nonwoven fabrics with comparable physical properties and superior thermal insulation properties.

Keywords

PET, *Asclepias syriaca*, milkweed, needle punching, sustainable nonwovens

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Introduction

The phenomenon of fast consumption directs the textile industry toward producing petroleum-based products as an economical path.¹ Although petroleum-based products are a more economical alternative than renewable natural sources, they cause irreparable damage to nature. The idea of building a sustainable and inclusive global economy has gained value in the international community, and the Green Deal Strategy has been developed by the European Union.² Therefore, there has been an increasing demand for developing natural, sustainable, and environmentally friendly

materials instead of petroleum-based products in recent years. In this regard, non-traditional natural plant-based fibers such as kapok, milkweed, poplar, nettle, etc. play a critical role.

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Milkweed seed fibers are one of the non-traditional fibers that are obtained from the seeds of *Asclepia syriaca* trees, which grow widely in the Northern Hemisphere. Since *Asclepia syriaca* plants can generally adapt to mild climates, their living spaces are quite wide. Although the length or appearance of milkweed fibers may vary slightly depending on the geography, the reported fiber lengths range from 9.5 to 43 mm in various studies.^{3–6} Fiber is mainly composed of cellulosic compounds and has approximately 40% crystallinity.⁷ These fibers have an average diameter of 20–30 μm and almost 90% of each fiber is composed of a hollow lumen. Therefore, the fiber density of milkweed fibers is around 0.97 g/cm³ while the fiber density of cotton is around 1.54 g/cm³.³ Although milkweed fibers are more rigid than cotton fibers, there are several studies reported on the spinning of milkweed fibers.^{3,8–10} Since milkweed is an untraditional fiber, it has a higher unit cost than cotton and flax.¹¹ Thus, milkweed waste obtained from the spinning process is a valuable byproduct, and it is important to utilize this leftover material.

However, these waste milkweed fibers are too short to be spun. Thus, using them as a filling material in a nonwoven product might be an alternative solution.

The type of filling material has a primary role in thermal insulation. The selected material should possess satisfactory thermal properties. The thermal properties of fiber can be influenced by its denier, fiber cross-section, porosity, etc. Gnanauthayan et al.¹² investigated the effect of physical properties on the thermal properties of non-woven fabrics made from solid and hollow polyester fibers. It was stated that thin and hollow structured fibers showed better thermal properties than thick and solid structured fibers. Needle-punched nonwovens made from solid and hollow polyesters were compared by Raesian et al.¹³ to evaluate their performance. They emphasized that hollow fibers trap air inside and have more volume and surface area, causing their thermal properties to be better than solid polyester fibers.

The needle punching process is a nonwoven production technique based on intertwining the fibers with the help of needle plates. Needle-punched nonwovens serve critical purposes in various industrial fields. For instance, it has been used as insulation materials, filtration media, cleaning materials, geotextiles, and automotive carpets.^{14–20} In addition, the needle punching process is suitable for blending long and short fibers together. For instance, short linseed fibers with an average length of 10–16 mm were blended with longer hemp fibers in a ratio of 50/50 by weight, and needle-punched nonwovens were developed for agromat application.²¹

In this study, short milkweed fiber leftovers obtained from a spinning mill were aimed to be utilized in a nonwoven fabric, rather than thrown away. For this purpose, those short milkweed fibers were blended with hollow PET fiber and turned into needle-punched nonwoven

fabrics with different thicknesses. The physical performance of the blended nonwovens was compared with nonwoven fabrics produced solely from hollow PET fibers.

Experimental

Employed materials

Short milkweed fibers (SMF) were donated by Pangai Materials Science, Italy. At least 50 individual SMF lengths were measured under the optical microscope and the average fiber length and fiber diameter were found to be 2.13 ± 1.4 mm and 25.35 ± 3.7 μm , respectively. Hollow PET fiber used in the experiments was generously donated by SASA Polyester Sanayi A.Ş. Turkey. Hollow PET fibers have a 64 mm staple length and 6.7 denier fineness. Figure 1 displays the SEM images of a hollow PET and short milkweed fiber. Both fibers were used as they were received from the companies, without being subjected to any additional process.

Carding and needling process

About 50 g of PET fibers were manually fed into the 337A Mesdan laboratory-type carding machine to create a single PET carded web. The feeding conveyor belt of the carding machine has a width of 43 cm, while the end roller width is 48 cm and the operating speed is 10 m/min. To obtain the blended web, previously produced PET carded web was cut into two-halves and one-half placed on the conveyor belt of the carding machine. About 50 g of SMFs were evenly distributed onto this web and the other half of the PET web was placed onto the SMFs. Then, this sandwich-like assembly fed into the carding machine at once. The resulting carded web was taken from the end roller, rotated 90°, and then fed into the carding machine one more time to achieve a homogeneous distribution of the SMF inside the web.

The needling process was carried out with the Cormatex S.R.L needle punching machine. The needle punching machine has 2080 needles on the needle plates which are 104 cm width. Notched needles entered the web from the top and bottom with a 90° angle. Thus, the fibers were thoroughly intertwined, and needle-punched nonwovens were obtained. The needling machine was operated according to the parameters shown in Table 1. The needle punching stroke was fixed at 354 strokes per minute from the top and 351 strokes from the bottom per minute. The punching depth is set to 10 mm from the top and bottom stroke. To obtain multilayered needle-punched samples, a desired number of carded webs were placed on top of each other and then fed into the needle-punching machine together. The Needle-punched nonwovens used in the experiments are listed in Table 2. The pure PET samples are called PET and blended samples are called Mix respectively.

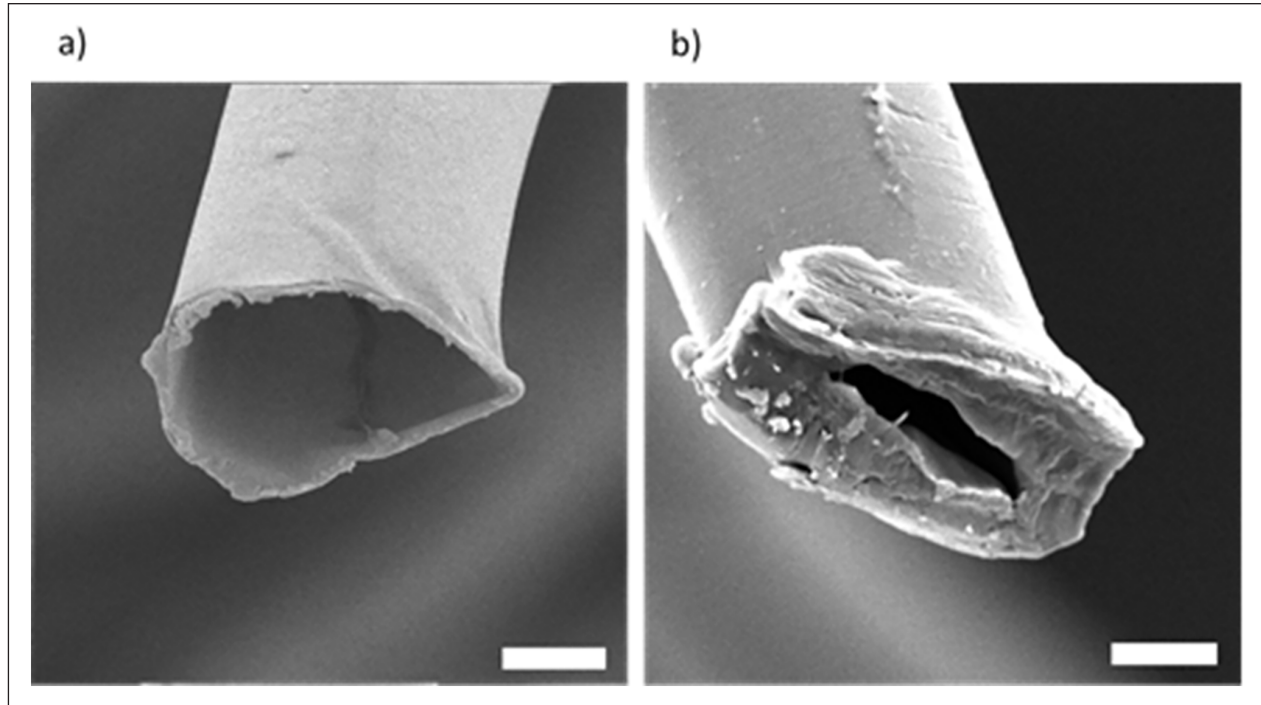


Figure 1. Cross-sectional SEM images of short milkweed fiber (a) and hollow PES fiber (b). Scale bars are 10 μm .

Table 1. Needle punching machine parameters (m/min) employed to produce waste milkweed/PET containing nonwovens.

Feeding top	Strokes top	Delivery top	Feeding bottom	Strokes bottom	Delivery bottom
0.97	354	1.07	1.03	351	1.11

Table 2. Milkweed fiber composition of the webs.

Sample	Milkweed fiber (%)
Mix1	17.42 ± 0.73
Mix2	17.66 ± 0.80
Mix3	18.95 ± 0.66

Thickness measurements. Thickness values of the pure hollow PET needle-punched nonwovens and the mixed samples are measured using a James H. Heal cloth thickness tester, with a probe diameter of 1.12 cm, according to ISO 9073-2. Each sample was measured 5 times and average values were reported.

Scanning Electron Microscope analysis. Images of PET fibers, milkweed fibers, and needle-punched nonwovens were captured by using a TESCAN VEGA3 Scanning Electron Microscope. Before the SEM analysis, each sample was coated with Au/Pd using a Quorum Sputter Coater instrument under vacuum for 120 s. The acceleration voltage was 10 kV, and the working distance was between 8.34

and 13.72 mm. To investigate the cross-sections of the fibers, samples were cut using a razor blade and placed on the appropriate sample holder.

Determining the composition of the webs

Quantitative chemical analysis was conducted under BS EN ISO 1833-11:2017 standard to determine the mass percentage of short milkweed and hollow PET fibers in blended fabrics. First, the mixed fabrics are dried in the oven and the dry fabric mass is measured. About 200 ml of 50 wt% sulfuric acid (H_2SO_4) per gram of fabric is added to the fabrics in the glass beaker. The bottle is shaken periodically for 1 h at $(40 \pm 5)^\circ\text{C}$ then filtered through filter paper. The residues that cannot pass through the filter paper are washed, dried, and reweighed. This process was repeated two times and the mean mass of short milkweed fibers was calculated. In this way, quantitative chemical analysis of fabrics was investigated.

Characterization of mechanical properties. To analyze the mechanical properties of the nonwoven samples, the James

H. Heal, Titan 2 was used and tests were conducted according to the ISO 9073-3 standard. Each specimen was cut into $16\text{ cm} \times 2\text{ cm}$ rectangle shapes and then placed in the tensile testing instrument. The initial gage distance was set to 10 cm, and the upper gage's separation speed was set to 100 mm/min. About 120 N load cell was employed for all experiments. At least five samples were tested for each set of samples. Breaking Stress (MPa) values were calculated using the original cross-sectional area of each specimen.

Bursting test. To analyze the burst strengths of samples, Dachang QC 115D was employed under the TS EN ISO 13938-2 standard. The samples were kept in standard conditions for at least 24 h before performing the test. The pneumatic diaphragm operating speed is adjusted to be $20 \pm 5\text{ s}$ to detonate the sample. Pressure is applied to the sample until the fabric bursts.

Wear and abrasion analysis. According to the TSE EN ISO 12947-2 standard, the abrasion test is based on the principle of abrasion of the standard fabric with a test sample. A woven wool felt fabric with a mass of $750 \pm 50\text{ g/m}^2$ and a thickness of $2.5 \pm 0.5\text{ mm}$ is used as a standard fabric according to TSE EN ISO 12947-1 standard. The test specimen is mounted on the test piece holder with a foam backing. Before starting the experiment, the samples were conditioned under standard atmospheric conditions. The abrasion test is carried out at certain intervals according to the friction number at which the sample ruptures.

Water contact angle analysis. Water contact angle analysis was conducted using the ThetaLite Optical Tensiometer TL 101 to measure the water contact angle of the nonwoven fabric. The tests were performed according to the BS EN 828:2013 standard, and each sample was tested from at least three different locations.

Air permeability analysis. The air permeability of the samples was assessed using a PROWHITE tester (document number 1) under the EN standard for plate number 1. The test was conducted using a 20 cm^2 test head and a prescribed air pressure of 200 Pa for 1 min. To ensure accuracy, at least five specimens were tested for each group of fabrics.

Thermal conductivity and resistance analysis. The Alambeta testing instrument (Sensora, Czech Republic) was used to measure the thermal conductivity and thermal resistance of the fabrics according to the ISO 11092 standard. Each sample was tested from at least three different locations.

Results and discussions

In this study, 100% PET and 50/50 wt % PET/short milkweed webs were produced. SEM images of the needle-punched PET and Mix samples are shown in Figure 2(a)

and (b), respectively. As seen in both figures, longer PET fibers are intertwined as a result of the needle-punching process. Since SMFs are thinner and shorter than PET fibers, needle-punched blended samples can accommodate SMFs. This can be seen in Figure 2(b) and also in the porosity analysis results. The SEM image of the mixed sample has a large number of SMFs and less space between the fibers. While a PET sample has 36.3% porosity, this value almost drops to half for the mixed sample (See Table 3). As indicated by the lower porosity percentages of the mixed sample, SMFs are filling the interspaces between the PET fibers, and their presence also slightly hinders the entanglement of PET fibers during the needle punching process. This is concluded from the weight and thickness results listed in Table 4. Samples with similar layer numbers and weight/g should have similar thickness values after the needle punching process. However, mixed samples are thicker compared to their PET counterparts. Less intertwinement of PET fibers in the mixed samples not only increases the sample thickness but also results in a decrease in the mechanical strength of the mixed nonwoven samples.

The percentage of short milkweed fiber by mass of the hollow PET/short milkweed blended fabrics is given in Table 2. To create a carded web 50 g each of hollow PET and short milkweed fibers are used. Since the short milkweed fiber leftovers from the milkweed yarn spinning process are employed, the fiber length is very short ($2.13 \pm 1.4\text{ mm}$). During the carding process, short milkweed fibers flew around. Therefore, when the quantitative chemical analysis was performed on the blended fabrics, the milkweed fiber ratio for Mix1, Mix2, and Mix3 was found to be 17.42 ± 0.73 , 17.66 ± 0.80 , and 18.95 ± 0.66 , respectively.

As seen in Table 5, feeding a higher number of carded PET webs to the needle-punching machine results in more durable nonwovens due to the stronger intertwining of the fibers. Among the PET samples, increasing the layer number from single layer to triple layers increases the stress at break values from 0.5 to 2.76 MPa. To visualize the effect of adding SMFs into the PET webs, stress-strain curves are plotted for the same layer numbered groups separately in Figure 3. In all layer numbers, the mixed samples have nearly 50% lower stress at break values compared to their PET counterparts. However, their breaking strain values are comparable mainly due to the breaking mechanism of the needle-punched samples.

ANOVA results indicate that there is no statistically significant difference ($p > 0.05$) among the values in terms of breaking strain values of all six sample groups. During the mechanical tests, needle-punched samples initially exhibit an elastic elongation where the non-intertwined PET fibers align with the extension direction. Then, intertwined PET fibers and nots created by the intertwined fibers deform and break, yielding the full breakage of the sample. Since

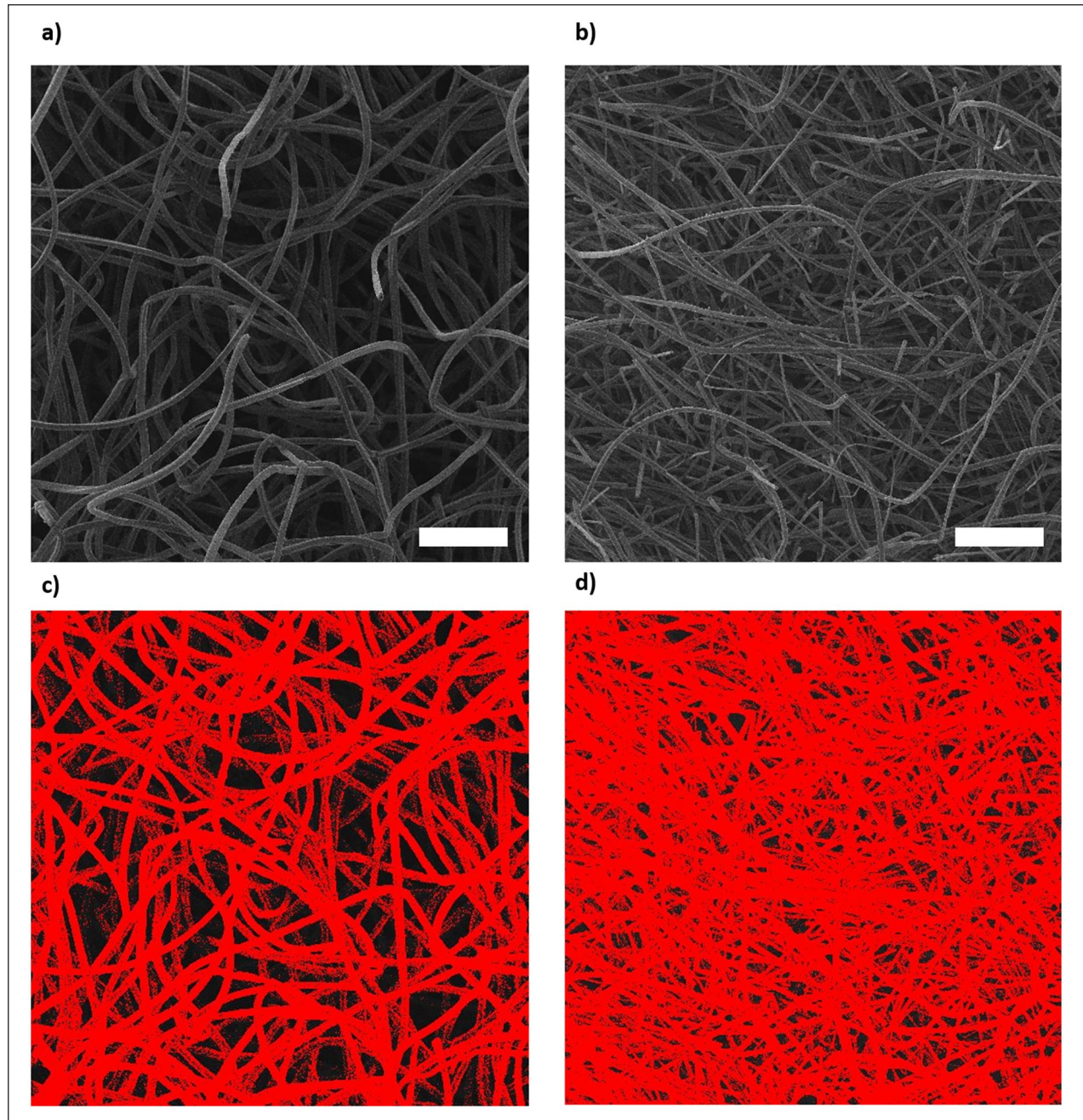


Figure 2. SEM images of PET (a) and mix (b) nonwoven samples. Scale bars are 500 μm . Fiber fraction and porosity analysis of PET and Mix samples were conducted in c and d, respectively.

Table 3. Fiber fraction and porosity values obtained from representative images of PET1 and Mix3.

Sample	Fiber fraction (%)	Porosity (%)
PET	63.7	36.3
Mix	83.0	17.0

very short milkweed fibers do not play a significant role in this breaking fashion of the nonwoven samples, all samples exhibit a similar elongation range.

Similar mechanical performance was observed when the bursting test was conducted on each sample. As the number of webs increases in the nonwoven fabric, both bursting strength and height increase as seen in Table 6. Mixed samples have lower bursting strength and height compared to their pure PET counterparts. However, differences in their bursting strength are not as low as their breaking strength values. This is due to the difference in the tensile and bursting tests working principle.

Martindale resistance to wear test method was applied to analyze the abrasion performance of the needle-punched

nonwoven samples. Among all samples, the single layer PET/SMFs sample exhibited the worst abrasion resistance. The Mix1 sample was heavily worn at 3000 cycles and the test was discontinued. All other samples survived until 4500 cycles and they have resulted in similar nep formation at 4500 cycles as seen in Figure 4.

As discussed in the literature, milkweed fibers have a waxy surface which makes these natural fibers highly

water-repellent. High water contact angles of hollow PET fibers have also been reported and results are shown in Figure 5 as well.²² As a result, both PET and mixed non-wovens exhibited high hydrophobicity. There is no statistically significant difference among the values in the PET and Mix samples according to ANOVA ($p > 0.05$). The contact angle of the 100% PET sample was measured as $143^\circ \pm 9.8^\circ$. Increasing the layer number has not affected

Table 4. Weight and thickness values of pure PET and mixed samples. Numbers in the sample names indicate the layer numbers of the web feed into the needle-punching machine.

Sample	Thickness (mm)	Weight (g/m ²)
PET1	4.15 ± 0.21	125
PET2	4.35 ± 0.19	179.1
PET3	6.35 ± 0.15	367.3
Mix1	4.12 ± 0.16	113
Mix2	5.81 ± 0.13	182
Mix3	6.92 ± 0.11	335

Table 5. Breaking strength and breaking strain values of PET and Mix samples.

Sample	Breaking strength (MPa)	Breaking strain (%)
PET1	0.50 ± 0.07	56.09 ± 3.14
PET2	1.58 ± 0.27	44.13 ± 3.58
PET3	2.76 ± 0.41	42.48 ± 0.78
Mix1	0.21 ± 0.04	48.07 ± 5.34
Mix2	0.72 ± 0.18	54.78 ± 1.64
Mix3	1.50 ± 0.1	52.28 ± 1.63

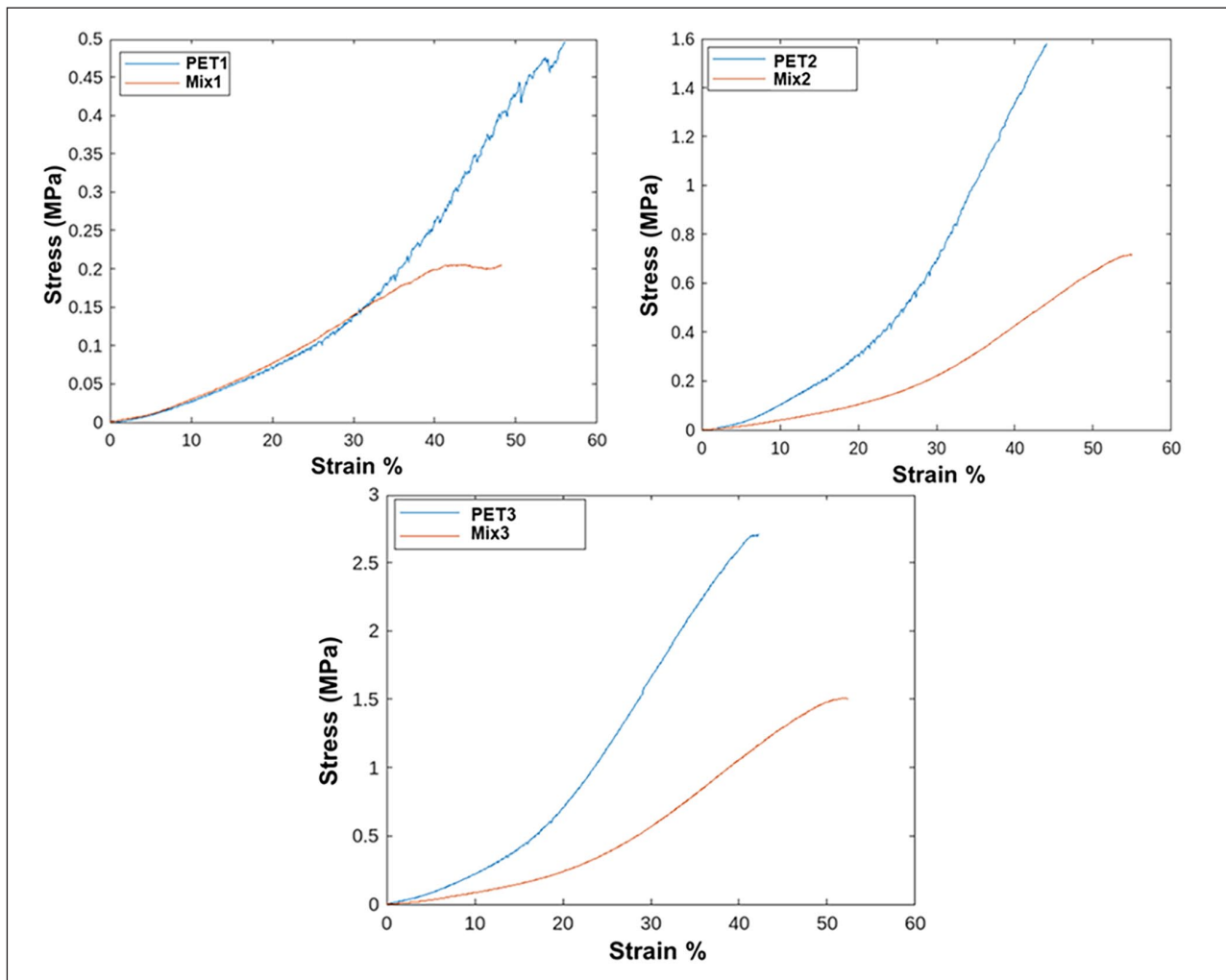


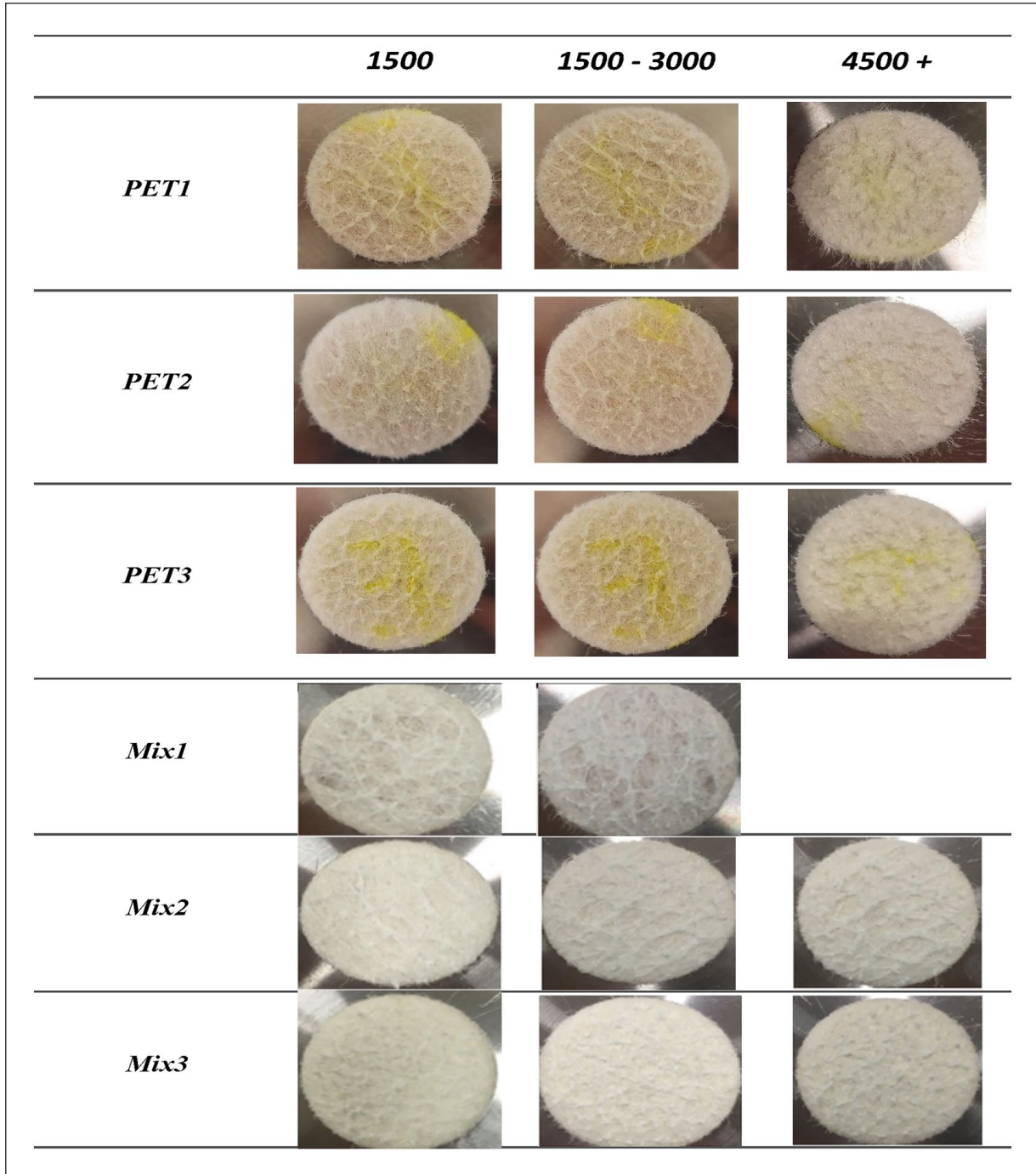
Figure 3. Comparison of the strength of PET and mixed fabrics with the same number of layers.

Table 6. Bursting strength values of PET and Mix samples.

Sample	Burst pressure (kg/cm ²)	Burst height (mm)
PET1	10.15 ± 0.73	14.4 ± 0.4
PET2	11.28 ± 0.85	17 ± 1
PET3	26.18 ± 2.06	19.53 ± 0.58
Mix1	8.73 ± 0.39	10.5 ± 0.7
Mix2	11.75 ± 1.46	13 ± 0.26
Mix3	18.55 ± 3.77	17.67 ± 2.08

the water contact angle of the mixed samples. Mix1, 2, and 3 samples' water contact angles were measured as 150.7 ± 7.4 , 146.7 ± 4 , and 148.6 ± 6.2 respectively.

Sample thickness plays a critical role in the air permeability of the samples. As shown in Figure 6, increasing the layer number decreases the air permeability of both PET and Mix samples. However, the presence of the SMFs determines the final air permeability because mixed samples have lower air permeability even when they are

**Figure 4.** Abrasion performance of nonwovens.

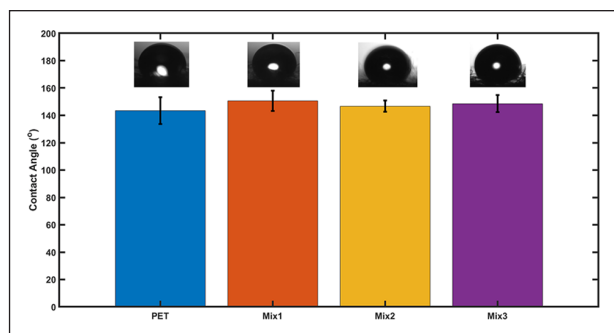


Figure 5. Water contact angle values and representative goniometer images of the samples.

thinner than PET samples. For instance, Mix1 is thinner than PET2, and Mix2 is thinner than PET3. Yet Mix1 and Mix2 have lower air permeability than PET2 and PET3, respectively. When a *t*-test was conducted on the air permeability performance of PET2 and Mix1 samples, which showed similar results in Figure 6, no statistically significant difference was found between them. Similarly, according to the *t*-test on PET3 and Mix2, there is no statistically significant difference in the air permeability performance between samples. Since SMFs are filling the gaps between the PET fibers and resulting in lower porosity, airflow through the nonwoven samples decreases.

It is well known in the literature that the air in the hollow fiber structures reduces the thermal conductivity and the sample thickness has an effect on the thermal properties.^{23–25} Thermal resistance and conductivity values are listed in Table 7. When compared with the same number of layer PET and Mix samples, it is clear that the presence of SMFs increases the thermal resistance and decreases the thermal conductivity. The huge hollow lumen of the milkweed fibers allows blended nonwovens to provide better insulation properties compared to hollow PET nonwovens. When ANOVA (Tukey's Post Hoc Test with 95% confidence interval) was performed to analyze the thermal resistance performances of the samples, it was determined that there was a statistical difference between all PET and Mix samples when they had the same layer numbers, except for PET2 and Mix1.

Conclusion

In summary, 100wt% PET and 50/50wt% PET/SMFs blended needle-punched nonwovens were produced. The addition of SMFs decreases the porosity and air permeability of the PET blended nonwovens. To test the mechanical properties of the samples, tensile and bursting tests were conducted. The results showed that mixed samples have lower tensile strength, bursting strength, and height compared to their pure PET counterparts. Although mixed samples have lower strength, their strain values were

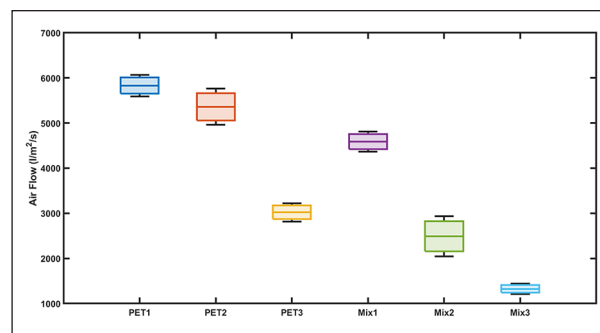


Figure 6. Air permeability performances of PET and Mix samples.

Table 7. Thermal resistance and thermal conductivity values of the test specimens.

Sample	Thermal resistance (mK/W/m ²)	Thermal conductivity (mW/m K)
PET1	98.10 ± 0.41	42.27 ± 0.12
PET2	102.17 ± 0.58	42.63 ± 0.21
PET3	135.17 ± 0.60	46.93 ± 0.31
Mix1	101.77 ± 0.62	40.47 ± 0.05
Mix2	137.53 ± 0.76	42.23 ± 0.09
Mix3	157.13 ± 0.61	44.03 ± 0.25

comparable. Both blended and unblended samples exhibit high water contact angles. The presence of the SMFs increases the sample thickness and thermal resistance while decreasing the thermal conductivity. While the PET 3 sample showed 135.17 ± 0.60 mK/W/m² thermal resistance, the Mix3 sample showed 157.13 ± 0.61 mK/W/m² thermal resistance. The thermal conductivity values of mixed fabrics give similar and better results compared to commonly used thermal insulation materials. Therefore, this study indicates that extremely short milkweed fiber leftovers from the milkweed yarn spinning process can be utilized as a filler material in needle-punched nonwovens to obtain better thermal insulation.

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