

Fiber Splitting of Bicomponent Meltblown Nonwovens by Ultrasonic Wave

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Abstract

Many technologies have been used to produce finer fibers due to their super advantages such as higher specific surface area, filtration/barrier property and absorption, as well as moderate porosity. Finer fibers are thus the great interest of many researchers in the nonwoven world, and many technologies have been used to make finer fibers. In this study, the authors addressed a novel avenue to produce finer fibers by splitting side-by-side bicomponent meltblown nonwovens composed of polyethylene terephthalate (PET) and polyamide 6 (PA6) by means of ultrasonic wave, in caustic soda and benzyl alcohol solutions respectively. The efficiency of fiber splitting was characterized in terms of dyeing ratio/percentage of the tested webs. Other properties were also examined, including fiber diameter, web weight loss, air permeability and thickness. In addition, SEM was used to observe fiber damage and fiber structure in the relevant webs.

Keywords: Bicomponent fibers, Meltblown nonwovens, Fiber splitting, PET/PA6, Benzyl alcohol, Sodium Hydroxide, Ultrasonic wave

1. Introduction

Meltblowing (MB) is a one-step process for making microfiber nonwovens directly from thermoplastic polymers with the aid of high velocity hot air to attenuate the melt filaments extruded from the orifices, originated in US Navy Laboratory in the Early 1950s. Exxon Company began to study on the MB technology in the Mid-1960s, and developed superfine fibers after five years. Subsequently, other companies produced the MB nonwovens with their own technologies, such as 3M (US), Freudenberg (Germany), NKK (Japan), etc. (Can Hong, 2004).

The nonwovens produced with MB technology are obviously featured with fine fibers compared to fibers from other technologies. The latest bicomponent MB equipment has consistently produced MB nonwoven webs containing fibers with average fiber distribution being 0.6 μ m; among which, more than 70% of the fibers have diameters less than 1 μ m, indicating that bicomponent MB technology might produce fine fibers at micro-nano scale (Xiao Jin, 2006), and thus the absorption and filtration efficiency would be improved due to the increase in specific surface area. However, it is necessary to further slenderize fine MB fibers to further improve the adsorption and filtration performance of the MB webs.

Many technologies have been employed to develop finer fibers in nonwoven field, such as direct drawing, island-in-the-sea, alkali treatment etc. For the island-in-the-sea method, bicomponent polymers are spun into island-in-the-sea bicomponent MB fibers and then dissolve the sea parts of the bicomponent fibers with appropriate solvent, resulting in finer MB fibers even at nanometer scale. Caustic soda may reduce MB fiber size to a certain degree, but the fibers are seriously damaged both in morphology and mechanical property. Drawing fibers may directly produce fine fibers, but more challenging spinning technologies will get involved, making this technology more complicated and energy-consuming, compared to the simpler post treatment technologies.

In this study, the authors would investigate the possibility of making fine MB fibers through bicomponent fiber splitting post treatments. Some researchers (Ron, 2003; Myung-Ja, 2001; Baopu Yin, 2003) applied post treatment technologies to split bicomponent MB fibers, e.g., alkali weight-reduction, hydroentanglement, heat treatment and solvent swelling methods for finer fibers with increased fiber surface area and hence the improved adsorption and filtration properties. However, such post treatments returned no significant efficiency in fiber splitting.

Based on the theory of "similarity and intermiscibility", a solute can only be dissolved or swollen in the solvent which

$$\Delta G = \Delta H_M - T \Delta S_M \tag{1}$$

Where ΔG_M – free energy of mixing, ΔH_M – heat of mixing, and ΔS_M – mixing entropy. The mixing entropy tends to be positive due to disordered alignment of the molecules during the process of mixing, and ΔH_M is also positive if the non-polar solute and solvent containing no H-bonds mix together. Therefore, the above conditions can only be satisfied when the heat of mixing is less than the product of temperature and the mixing entropy. The heat of mixing could be expressed with the following formula, if the total volume remains unchanged during mixing:

$$\Delta H_M = \varphi_1 \varphi_2 (\delta_1 - \delta_2)^2 V \tag{2}$$

Where φ_1 and φ_2 stand for the volume fractions of solvent and polymer, respectively; δ_1 and δ_2 represent the solubility parameters of solvent and polymer, respectively; and *V* is the total volume of the solution.

Combining Equations (1) and (2) we can find that spontaneous dissolving readily occur when a smaller ΔH_M value is available, i.e., when the values of δ_1 and δ_2 are close to each other. Generally, dissolving or swelling of the solute will occur when the difference between δ_1 and δ_2 is less than 1.7~2.0, which is shown in Equation 3 as below:

$$\left|\delta_1 - \delta_2\right| < 1.7 \sim 2.0\tag{3}$$

In this study, PET/PA6 melt blown nonwoven web samples were treated with alkali and benzyl alcohol solutions respectively, with the aid of ultrasonic wave, to produce finer fibers through fiber splitting technology. The solubility parameters of PET and PA6 are $10.7 \text{cal}^{1/2}/\text{cm}^{3/2}$ and $13.0 \text{cal}^{1/2}/\text{cm}^{3/2}$ respectively, and that of the splitting reagent, i.e., benzyl alcohol, is $11.6 \text{cal}^{1/2}/\text{cm}^{3/2}$.

2. Experimental

2.1 Web sample description

The side-by-side (S/S) MB nonwoven sample composed of 50PET/50PA6 was produced from Textile and Nonwoven Development Center (TANDEC), The University of Tennessee, Knoxville, USA.

2.2 Post-treatment

The specimens were treated in benzyl alcohol and in NaOH solutions respectively, at the concentration of 5g/L, liquor ratio of 1/100, temperature of 60°C, for 45 minutes, with the aid of ultrasonic wave at the powers of 0, 100, 120, 150, 180, 210 and 240W, respectively. Five specimens (20cm by 20cm for each) were used in each experiment.

2.3 Characterization

Along with the controls, the treated web samples were tested for air permeability (ISO9237-1995), thickness (ISO9073-2-1989), dyeing rate, weight loss, and fiber diameter.

2.3.1 Fiber diameter and web morphology

The optical microscope system and image analyzing software were utilized to capture the images of fibers and measure fiber diameter. The averages of 200 readings of fiber diameters for each specimen were recorded. The coefficients of variance in fiber diameter were in the range of 20% to 32%. The web morphologies were examined with a KYKY-2800 scanning electron microscope (SEM).

2.3.2 Degree of fiber splitting

The degree of fibers splitting was characterized in terms of dyeing rate. Firstly, specimens were treated with the penetrating agent of JFC at 1g/L, 40°C for 5 minutes, and then, the weakly acidic red dye, GRS was added to the treatment solution at 0.5% owf and 100/1 liquor ratio. The dye absorption remained for 45 minutes at 40°C. After dye absorption, the treated web samples were thoroughly rinsed with 50°C water and then let dry at room temperature. The maximum absorption wavelength of GRS was detected on the visible spectrophotometer VIS-723, and the absorption wavelength. The dyeing bath before and after dyeing, A_0 and A_i , were measured respectively at the maximum absorption wavelength. The dyeing rate can be calculated as:

$$S = (1 - A_i / A_0) \times 100 \,(\%)$$

Where A_0 and Ai stand for the absorbencies of the dyeing bath before and after dyeing, and S represents dyeing rate. Specimens were conditioned for 24 hrs at standard temperature and humidity, before being measured on JA5003 electronic balance (accuracy: 0.001g). Weight loss was characterized based on the equation below:

$$W = (W_0 - W_i) / W_0 \times 100 \,(\%)$$

Where W_0 and W_i are the weight values of the samples before and after dyeing, and W is weight loss.

3. Result and Discussion

3.1 Change in web morphology

The SEM photos of the web samples before and after post treatments were shown in Figure 1.

It was observed after comparing the photos in Figure 1, that the web structure showed no obvious change when specimens were treated in NaOH solution at low concentration of 5g/L, while fiber diameter decreased mainly due to the effect of fiber splitting and hydrolysis of ester group in PET and amide group in PA6. This effect became extremely significant, especially when ultrasonic wave was incorporated.

Benzyl alcohol is one of the swelling agents of PA6, whose molecules may pass amorphous region, occupying the space among chain segments of PA6.

It has been reported based on SAXS observations that the swelling agent only swelled amorphous region inside the fiber along fiber axis (Zeyong Zheng, 1989), whereas the molecules in crystalline region across the fiber axis still exhibited a dense arrangement, indicating no swelling effect from the swelling agent along this direction, which resulted in significantly anisotropic swelling effect from benzyl alcohol on PA6 fiber swelling (J. Baldrian, 1991). The bicomponent would become separated when the stress at the interface of PET and PA6 was generated and became large enough; the addition of ultrasonic wave would enhance the bicomponent splitting, and hence shorten the time for this process.

3.2 Change in fiber diameter

Figure 2 showed a consistent tendency that the fiber diameter decreased with the power of applied ultrasonic wave during the three post treatments, suggesting that ultrasonic wave, as an auxiliary means, could indeed assist fiber splitting in PET/PA6 side-by-side bicomponent MB fibers. However, it was found that ultrasonic treatment alone only resulted in fiber splitting to a limited extent; it only could help the splitting agent enter into the interface between PA6 and PET. In bicomponent fiber splitting, swelling/splitting agent played a key role, while ultrasonic wave only assisted the diffusion of swelling agent in the bico fibers. When the bico fibers were treated with NaOH solution, with the aid of ultrasonic vibration, NaOH solution might enter into the interface of the bicomponent in PET/PA6 fiber, resulting in the different shrinkage ratios of PET and PA6 components in the bico fibers(less shrinkage from PET; more from PA6), and thus expanding the crack at the interface (Bin Yang, 2002); meanwhile, ultrasonic application further enhanced the crack, and eventually leading to the fiber splitting; therefore, fiber size decreased significantly. On the other hand, at 60°C, the ultrasonic vibration enhanced the hydrolysis effect from NaOH on the ester bonds in PET, and the whole fiber became thinner, as a result. Therefore, finer fibers were the result from both the foregoing action processes. As for benzyl alcohol, it served as a swelling agent of PA6, favoring the splitting of PA6 and PET with no caustic effect on the fiber; ultrasonic treatment would play the same role in fiber splitting, as addressed before.

3.3 Change in web adsorption

The separation of the two components in bico fibers would definitely cause the increase in specific surface area of the fibers and hence the adsorption property of the webs, which could be characterized with the degree of adsorption to certain dye, in terms of Dyeing ratio(percentage). Therefore, the values of dyeing ratio would be an index indicating the degree of fiber splitting in bico MB webs in this study. The weak acidic red dye GRS, which only could be absorbed by PA6, was selected to dye PET/PA6 bico MB webs, and 40 °C was chosen as the initial dyeing temperature, to lower the possibility of the dye to penetrate inside the bulk fibers and thus minimize the experimental error. The results from the dyeing experiments were shown in Figure 3. It was observed from the dyeing results that the dyeing percentage (%) of all the web samples treated with ultrasonic wave, NaOH/ultrasonic wave and benzyl alcohol/ultrasonic wave increased with the increasing ultrasonic power, which indicated the increase of specific surface area of the split fibers. This suggested that the dyeing experimental results were consistent with those from fiber diameter measurement tests and hence it could be concluded that fiber splitting in bico MB webs resulted in finer fibers and thus increased surface adsorption ability.

3.4 Change in web weight loss

The results from weight loss measurement tests were summarized in Figure 4. As can be seen in Figure 4, the webs after three different treatments (i.e., ultrasonic, NaOH/ultrasonic and benzyl alcohol/ultrasonic) lost their weights and the weight loss increased with increasing ultrasonic power. This phenomenon could be explained by the characteristics of MB nonwoven webs. As we know, MB webs were produced in one-step process, during which the fibers were formed by the attenuation from the hot air, and the semi-molten state fibers were deposited on the web screen to form MB webs immediately by self-adhesion. Therefore, the bonding/adhesion between MB fibers was not strong enough to survive the ultrasonic vibration treatment, and thus the MB fibers might be pulled out of the MB web, leading to the weight loss of the treated MB webs. Increasing ultrasonic power would provide more vibration energy to detach the weakly bonded fibers from the MB webs, and even split the poorly adhered bicomponent in the MB fibers, resulting in increasing

weight loss of the treated web samples.

The web samples treated with alkali solution/ultrasonic wave showed much higher weight loss compared with the web samples treated with ultrasonic wave alone or benzyl alcohol/ultrasonic wave, indicating the possibility that alkali solution hydrolyzed and thus damaged the fibers in the nonwoven webs.

Small fibers were found to be left in the residue of benzyl alcohol solution. However, no short fibers were found in the residue of NaOH solution, possibly because these fibers were hydrolyzed in the alkali solution with the help of ultrasonic wave at 60° C.

Furthermore, the weight loss curve of the web samples treated with ultrasonic wave showed similar tendency to that with benzyl alcohol/ultrasonic wave treatment; and thus, it was suggested, on the other hand, that benzyl alcohol treatment brought no damage to the fibers.

3.5 Change in web thickness

Figure 5 shows an increasing web thickness with increasing ultrasonic power, which was because that ultrasonic vibration energy could impart fibers in the webs with sufficient dynamic energy for fibers to detach from the webs and then further entangled one another, which finally caused the web shrinkage to occur. Also, post treatment of the nonwoven webs in 60° C aqueous solution would cause the relaxation of the processing stress created during bico MB web production, and finally result in web shrinkage and thickness increase. More powerful ultrasonic wave would provide the fibers with more mobility to move around and get entangled together.

Furthermore, benzyl alcohol molecules might enter into the interface between PET and PA6 and then split the two components, leading to increased specific volume per unit mass of the treated webs, and the increased porosity in the webs, which actually increased the bulkiness of the treated webs, in terms of thickness, as shown in Figure 5. In the case of caustic soda, although the alkali solution also could cause bico fiber to split and thus lead to the increase in the web thickness, it would additionally hydrolyze the fibers in the treated webs and thus attenuate the web thickness eventually. With the aid from the auxiliary ultrasonic wave, the attenuating effect from the alkali hydrolysis of the fibers became dominant and thus the NaOH/ultrasonic wave treated bico MB webs showed less thickness than the ones treated with ultrasonic wave alone, which could be easily seen in Figure 5.

3.6 Changes in air permeability

Figure 6 shows relations between air permeability and the ultrasonic power used. Air permeability decreased, as a whole, with the increasing ultrasonic wave power used, which was consistent with the results from the change in thickness (Figure 5); usually air permeability of nonwoven webs exhibit decreasing trend with increasing web thickness.

For the ultrasonic wave treatment alone, 100 Watt ultrasonic wave power was possibly not sufficient to split large amount of bico MB fibers, only caused fibers to entangle one another, and the relaxation of the processing stress existed in the fibers, the ultrasonic wave treated webs only shrank, as a result, generating no additional pores in the treated web samples, which led to the web samples with the least air permeability, compared to other samples treated with the other methods.

But in the existence of ultrasonic wave, both the webs treated with benzyl alcohol and alkali solutions showed higher air permeability than that treated with ultrasonic wave alone, indicating that benzyl alcohol treatment and alkali treatment could both split bico MB fibers in the webs and thus increased the porosity of the webs, with NaOH treated web samples showing higher air permeability due to the extra fiber hydrolysis effect from the alkali solution.

The air permeability of the web treated with ultrasonic wave alone increased initially and then decreased with the increasing power of ultrasonic wave; this was possibly because at lower ultrasonic wave energies, fibers were split and interstice of fibers was increased, that resulted in the air permeability increased. When the power of ultrasonic wave was increased continuously, the decrease of the air permeability was caused by fibers entangling severely.

When the power of ultrasonic wave was increased all the way up to 250 Watt, the web samples treated with ultrasonic wave alone showed significantly higher air permeability than those treated with the other two methods. This was possibly because that swelling agents brought about fibers entangling and nonwoven shrinkage, especially samples treated at high power, and that the phenomenon became more serious.

4. Conclusion

The ultrasonic technology was successfully applied to fiber splitting for side-by-side PET/PA6 bicomponent MB webs, and had a remarkable effect. The fiber diameter was reduced from $3.62\mu m$ to $2.11\mu m$, when NaOH, the concentration of 5g/L, and ultrasonic wave, whose power was only 240w, were utilized. The weight loss was only 8.34% by the way, while 50% in tradition (J. Baldrian, 1991) .SEM photograph shows damage of fibers was less, and main loss was attributable to fibers which were reinforced in webs. The porosity of treated specimens was reduced 7.59% than control,

which was449.5m/s.

Benzyl alcohol was selected as separation reagent to swell PET and PA6 on the basis of similarity and intermiscibility, because solubility parameters are close. Dyeing rate of webs treated reached 50.47%, while control 19.00%. It was proved that benzyl alcohol was useful to fiber splitting of PET/PA6. At the same time, damage of webs was less by benzyl alcohol. Finer fibers whose specific surface area was greater, could absorb more particles. And benzyl alcohol is recycled and utilized repeatedly, and it is consistent with Green environmental protection and Energy Saving.

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(c)





(d)



Figure 1. SEM photographs (a) control (b) ultrasonic treatment(c) NaOH treatment (d) NaOH and ultrasonic treatment (power 240W)(e) benzyl alcohol treatment (f) benzyl alcohol and ultrasonic treatment(power 240W)



Figure 2. The effect from ultrasonic power on fiber diameter during ultrasonic treatment, NaOH/ultrasonic treatment and benzyl alcohol/ultrasonic treatment



Figure 3. Dyeing rate of webs at various powers



Figure 4. Weight loss of webs at various powers



Figure 5. Thickness of treated webs at various powers



Figure 6. Air permeability of webs at various powers