

An Investigation of Fiber Splitting of Bicomponent Meltblown/Microfiber Nonwovens by Water Treatment

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Received 23 September 2003; accepted 12 April 2004

DOI 10.1002/app.21036

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Meltblowing is a most versatile and cost-effective process commercially available worldwide to produce microfiber nonwovens directly from thermoplastic resins. The new bicomponent (bico) meltblown technology opens a great possibility to make even finer microfibers by subsequently fiber splitting. Water-dispersive Eastman AQ polymers were initially introduced to the meltblown process to make the mono- and bicomponent meltblown webs at Textiles and Nonwovens Development Center (TANDEC), University of Tennessee, Knoxville. The postwater treatment

was performed on the fabrics, which resulted in the dispersive part (AQ polymer) being dispersed in water and only the other part remaining in the bico web. A process–structure–property study is provided toward the research reported in this article. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 94: 1218–1226, 2004

Key words: fibers; meltblown technology; structure–property relations; dispersions; processing

INTRODUCTION

In a conventional meltblown process, a thermoplastic, fiber-forming polymer is extruded through a linear die containing closely arranged small orifices (normally, about 0.4 mm in orifice diameter, 20–30 holes/in.). The melt filaments out of the orifices are rapidly attenuated by two convergent streams of high-velocity hot air to form microfibrils (usually 1–5 μm) and blown by the air stream to a collector to form the microfiber web by self-thermal bonding. Meltblowing, or meltblown process (MB), has become an important industrial technique in nonwovens because it is versatile and cost-effective to produce fabrics of microfiber structure directly from polymers, suitable for filtration media, thermal insulators, battery separators, oil absorbents, and many laminate applications. Polypropylene (PP) is the most widely used polymer for this process. Many others, such as polyethylene (PE), polyethylene terephthalate (PET), polybutylene terephthalate (PBT), polyamide (PA), and polytrimethylene terephthalate (PTT), can also be used to produce the meltblown webs. Considerable R&D efforts have been made in recent 30 years on the process study, new resin and product development, and process improvement. Among the most advanced development is new bicomponent meltblown technology.

The term “bicomponent” usually refers to fibers that are formed by two polymers extruded separately, from at least two extruders, but spun together through the orifices. The configuration of such a bicomponent fiber may be a sheath/core arrangement wherein one polymer is surrounded by another or may be a side-by-side or other sectional configuration. In nonwovens industries, the bicomponent (bico) fibers have been obtained in recent years for the meltblown and spun-bond processes. Figure 1 is the schematic of the first Reicofil® side-by-side bicomponent meltblown line.

Water-dispersive Eastman AQ polymers were initially introduced to the meltblown process to make the mono- and bicomponent meltblown webs at Textiles and Nonwovens Development Center, University of Tennessee, Knoxville. The postwater treatment was performed on the fabrics, which resulted in the dispersive part (AQ polymer) being dispersed in water and only the other part remaining in the bico web. A process–structure–property study is provided toward the research reported in this article.

EXPERIMENTAL

Polymer resins and web specimens

Eastman AQ35 water-dispersive polymer was applied with polypropylene to produce bicomponent meltblown fabrics. Polypropylene is the most commonly used polymer for meltblown nonwoven products because it produces the best web [among the low-cost resins].⁸ The PP resins used were ExxonMobil PP 3546G (ExxonMobil, Baytown, TX) and Basell PP-PF 008 (Basell NV, Hoofddorp, The Netherlands).

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Contract grant sponsor: Reifenhauser GmbH, Troisdorf, Germany.

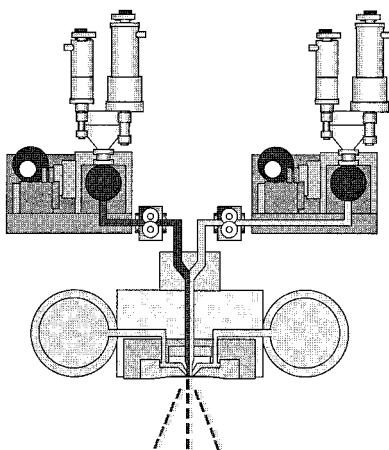


Figure 1 Schematic of the first Reicofil® side-by-side bicomponent meltblown line.

Table I lists all the web specimens used in this study, produced using Reicofil-in. 24" wide side-by-side bicomponent meltblown line.

Water treatment

Three kinds of water treatment were performed on bico meltblown fabrics of different compositions:

1. tap water (hard water)
2. soaked in distilled water for seconds
3. soaked in distilled water for hours

In the first treatment, the bicomponent specimen did not show any weight loss, indicating no AQ35 was dispersed in the tap water, because the ions such as Ca^{+2} , Mg^{+2} , Fe^{+3} , and so forth in the hard water retard the dispersion of Eastman AQ polymers; thereafter tap water treatment was not considered further in this research. In the second treatment, specimens were soaked in distilled water only for seconds. No weight loss was expected within such a short time, but the water came into contact with AQ polymer and the subsequent dispersion reaction was presumed to occur; in the third treatment, specimens were soaked in distilled water for about 20 h. Complete weight loss from AQ polymer was expected.

The specimens were weighed before treatment. In the second treatment (short time with distilled water), specimens were soaked in plastic containers with sufficient distilled water for no more than 10 s, and removed when the specimens looked thoroughly wet. In the third treatment (long time with distilled water), the specimens felt slippery at first with AQ polymer attached to them, but after being soaked in water about 20 h, it no longer felt slippery because AQ polymer dispersed into the water. The specimens were

then carefully removed, dried in air, and weighed again.

Physical testing

All the specimens were conditioned in standard atmosphere for testing, which is $21 \pm 1^\circ\text{C}$ ($70 \pm 2^\circ\text{F}$) and $65 \pm 2\%$ humidity.

Basis weight and thickness

Ten measurements were made on 10 random places on each specimen. The average values were reported according to INDA Standard Test Methods: IST 130.1 and IST 120.1 (ASTM D5729-97).

Air permeability

This is the rate of air flow passing perpendicularly through a known area under a prescribed air-pressure differential between the two surfaces of a material. According to ASTM D737-96, 10 specimens were taken randomly for each sampling unit. An FX 3300 air-permeability tester provided a pressure drop of 125 Pa (12.7 mm or 0.5 in. of water) across the specimen. The average values and standard deviation were reported.

Water resistance (hydrostatic pressure test)

According to IST 80.6 (98), three random specimens were tested for each material. The FX 3000 hydrostatic head tester provided a pressure gradient of 60 mbar of water/min. A supporting nylon net was attached under each specimen.

Fiber diameter

Fiber diameter was determined from images of a scanning electron microscope and an Olympus (Osaka, Japan) optical microscope, with the application of Scion software (Scion Corp., Frederick, MD). In the optical microscope measurement, a video camera was attached to the microscope, and measurement was performed on the images displayed on a computer

TABLE I
Bicomponent Samples Used for Water Treatment

Sample	Description (weight ratio, polymer)
1	Pure PP
4	Pure AQ35
7	25/75 AQ/PP
8	25/75 AQ/PP
6	50/50 AQ/PP
9	50/50 AQ/PP
10	75/25 AQ/PP

monitor. The scale setting applied in Scion software was 3.45 pixels/ μm . About 100 fiber diameter measurements were made for each sample.

Stiffness

Because of limited amount of treated samples, three or four specimens were taken only in the machine direction if no large variation was noticed. With the instruction of ASTM D5732-95, FRL cantilever bending tester was used to obtain the bending length and flexural rigidity.

Tensile strength, elongation, and tenacity

According to ASTM standard D5035-95, peak force and elongation were measured on an Instron tensile tester with a loading cell of 10-lb. capacity and a gauge length of 3 in., at a constant rate of extension. Fiber random specimens were measured for each sample.

RESULTS AND DISCUSSION

Eastman AQ polymers are thermoplastic and amorphous polyesters that disperse directly in water without the assistance of organic cosolvents, surfactants, or amines. An Eastman AQ polymer was considered "soluble" if 1 of polymer pellets was taken up in 9 gms of solvent (agitated) within 1 week at room temperature ($\sim 22^\circ\text{C}$ or 75°F). Note that the solubility may increase at higher temperature or longer time periods. Forty percent solids are near the maximum level that can be achieved for Eastman AQ 55 polymer before turning into gel. Eastman AQ 35 behaves similarly.

Dissolving a polymer is a slow process that occurs in two stages. First, solvent molecules slowly diffuse into the polymer to produce a swollen gel, and then the second stage of solution can occur if the polymer-polymer intermolecular forces can be overcome by the introduction of strong polymer-solvent interactions. Here the gel gradually disintegrates into a true solution.

Partial dispersion of AQ/PP/bicomponent nonwoven webs

To investigate the effect of time on the solubility of AQ35 bicomponent nonwoven web, first some specimens were soaked in distilled water just for seconds. Specimens after become brittle and stiff, looking more like a piece of film rather than a nonwoven web. Air permeability changed essentially on the treated web. For example, on the 75/25 AQ/PP treated fabric, air permeability decreased nearly one fourth. Within several seconds, what probably happened might be that water molecules diffused into the web and reacted with AQ polymer to produce a swollen gel left in the webs. The swollen gel

covered some open space on the web, and therefore greatly decreased the air permeability.

Another batch of specimens was soaked in distilled water for a longer time of 2.5 h within 0.2 to 0.4 wt % solution concentrations. On contact with water, AQ dispersive polymer in every bicomponent fiber first swells, and is then dispersed gradually into water. When removed from the water after 2.5 h, the specimen's surface (except #7) felt quite slippery, indicating that 2.5 h is not sufficient for swollen AQ polymer gel to disintegrate completely into water and some was still left on the specimens. For sample 7 (25/75 AQ/PP) specimens, air permeability increased by about 4% after this treatment; for samples 9 (50/50) and 10 (75/PP AQ/PP) specimens, air permeability decreased by approximately 5.5 and 87.7%, respectively. It can be readily understood because sample 7 contains less AQ polymer than samples 9 and 10, and those AQ polymers all dispersed in water within 2.5 h, leaving the web sparser, whereas for specimen samples 9 and 10, with higher AQ35 concentration, especially sample 10 with higher thickness, 2.5 h were not sufficient to disperse all the AQ35 in the water. In other words, there was AQ35 gel partly left on the webs, resulting in decreased air permeability.

Complete dispersion of AQ/PP bicomponent nonwoven webs

Most work were done on the completely dispersed AQ/PP bicomponent nonwoven webs. Specimens after 20-h water treatment are softer, lighter, and contain strong static charges.

SEM image analysis

All the images were taken with 20-kV accelerating voltage, 12-mm working distance, and $\times 500$ magnification. AQ/PP 75/25 (sample 10) fibers' shape changed most after water treatment among three composition groups (75/25, 50/50, 25/75 AQ/PP), given that the highest ratio of AQ polymer it contained was dispersed.

Figure 2 and Figure 3 show the bico web structure of 75/25 AQ/PP before and after the treatment. The fibers' shape changed from the original cylindrical shape before treatment (Fig. 2) into a prominent ribbon shape (Fig. 3). Some fibers even look transparent in the images. Besides, a "spider web," formed by some superfine fibers, can be observed in some of the images. The very thin ribbon shape of fibers is more clearly displayed from cross-sectional photos. Fibers with half side lost can be observed in the image (Fig. 4), where several fibers' ends face out of the image and show off their half-round cross section.

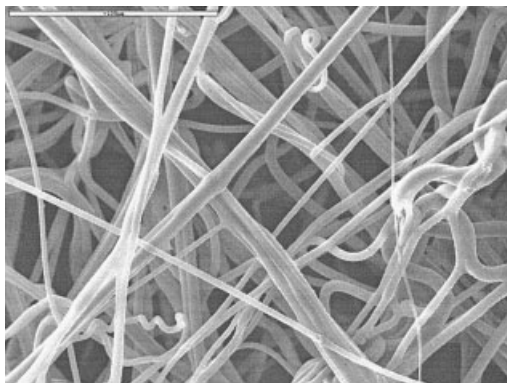


Figure 2 SEM microphotograph of sample 10, 75/25 AQ/PP meltblown nonwoven before water treatment.

Properties analysis

Basis weight

PP is hydrophobic and is not affected by water treatment. AQ dispersive polymer is dispersed in soft water under the proper conditions. It is supposed to be completely dispersed in water after the treatment. Basis weight loss [Fig. 5(A)] after water treatment is expected to agree with the bicomponent ratio. For example, 25/75 AQ/PP is expected to lose 25% weight after treatment. In the experiments, all specimens lost more weight than expected: 34.3% weight loss for sample 7 (25/75 AQ/PP), 62.6% for sample 9 (50/50 AQ/PP), and 79.6% for sample 10 (75/25 AQ/PP). One of the reasons for the extra weight loss is that some floating or infirmly bonded fibers sloughed off during the long soaking time in water.

The fragmentary fibers found later in the container are evidence for this presumption. The other possibility is that the real bicomponent ratios of the bicomponent webs are not accurately equal to the designed ratios because of all the possible problems during production. This phenomenon may imply an efficient method to check the real bicomponent ratios for the

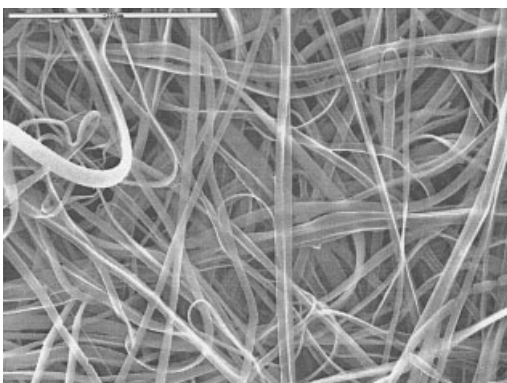


Figure 3 SEM microphotograph of sample 10, 75/25 AQ/PP meltblown nonwoven after water treatment.

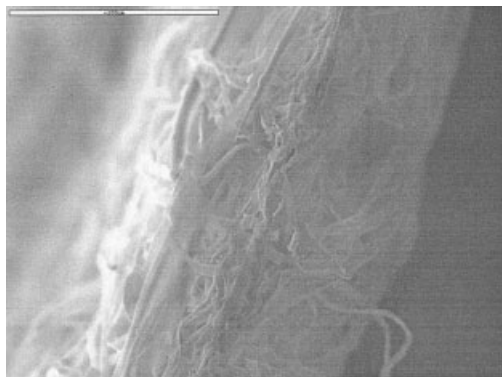


Figure 4 Cross-section SEM microphotograph of sample 10, 75/25 AQ/PP meltblown nonwoven after water treatment.

bicomponent nonwoven webs, and further examine production parameter designs and equipment working conditions.

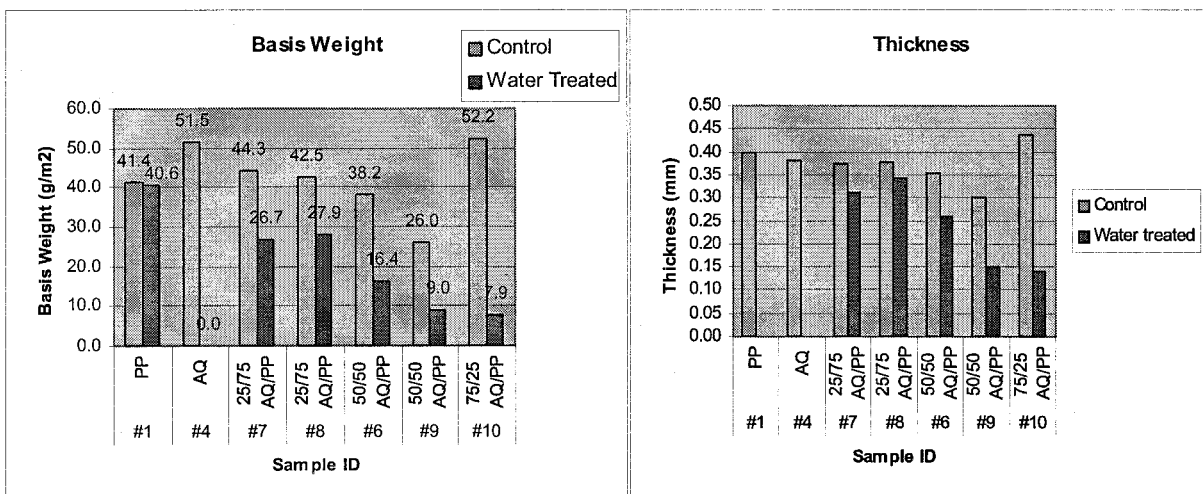
The other possible reason is the dispersion of the hydrophobic polypropylene, not just the AQ polymer, because Eastman AQ polymers have the capacity to disperse many water-insoluble, hydrophobic materials in water, although a later experiment proved this was not the case. Pure PP web did not lose any weight even after a week's soaking in AQ dispersion solution.

Thickness, flexural rigidity, and bulk density

All specimens decreased sharply in thickness and flexural rigidity (stiffness), as shown in Figure 5(B) and Figure 5(H), especially #10 with 75% AQ decreased most, 67% thinner in thickness and 98% in stiffness. Specimens became obviously lighter and softer. The bulk density (basis weight divided by thickness) is showed the same decreasing trend.

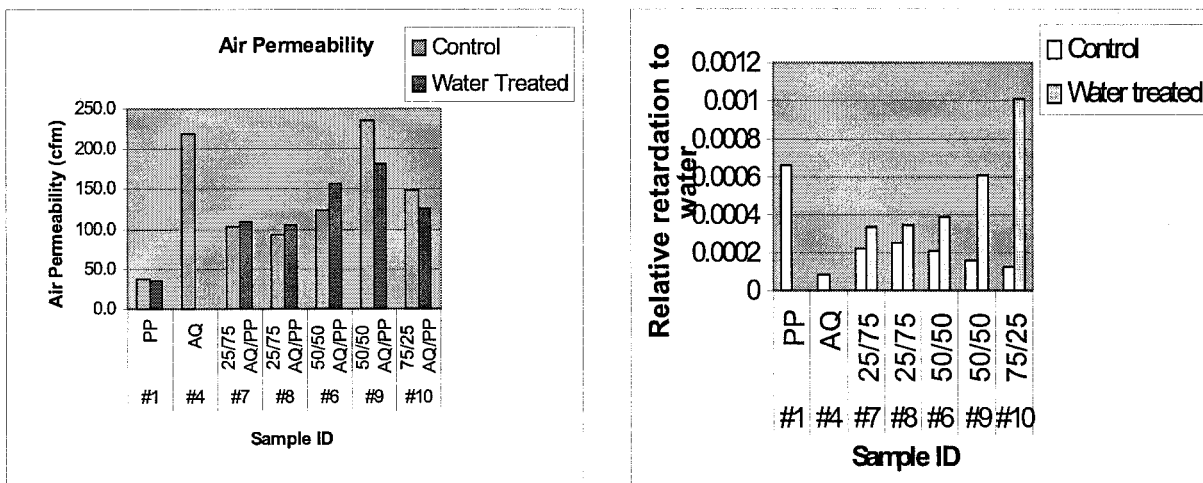
Hydrostatic head, relative hydrostatic head

The hydrostatic pressure test measures the resistance of nonwoven fabrics to the penetration of water under low hydrostatic pressure. All specimens experienced a decline in hydrostatic pressure to some extent [Fig. 5(E)]: 25% AQ polymer specimens samples 7 and 8 decreased less than samples 9 and 10 with 50 and 75% AQ polymer component separately. Conversely, when converted to relative hydrostatic head pressure (divided by corresponding basis weight), the hydrohead values increased dramatically [Fig. 5(F)], which indicates that based on the same basis weight, the resistance to water penetration increased after water treatment on these samples. Possible reasons are attributed to increased specific surface area.



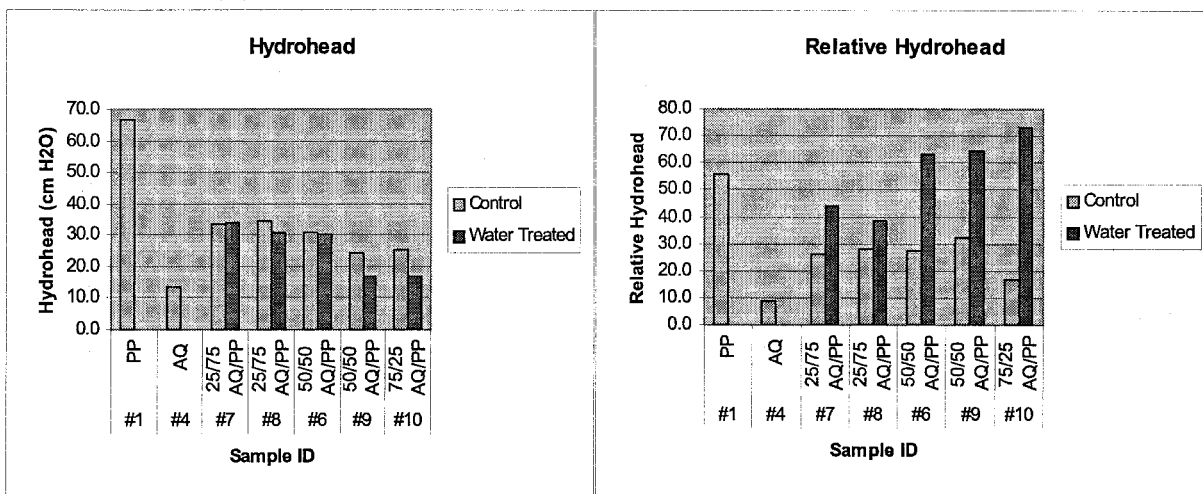
(A) Basis Weight

(B) Thickness



(C) Air Permeability

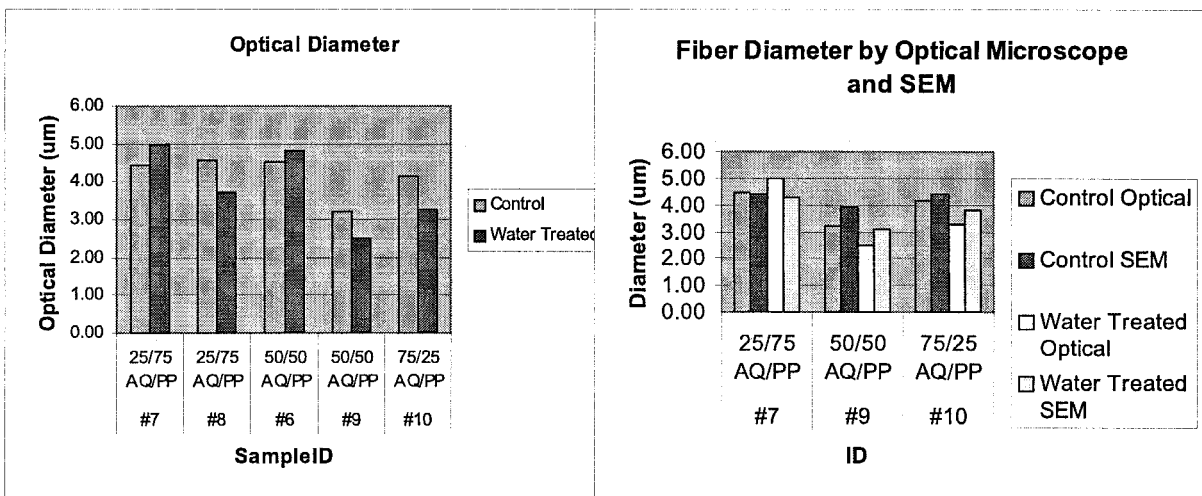
(D) Retardation to Air



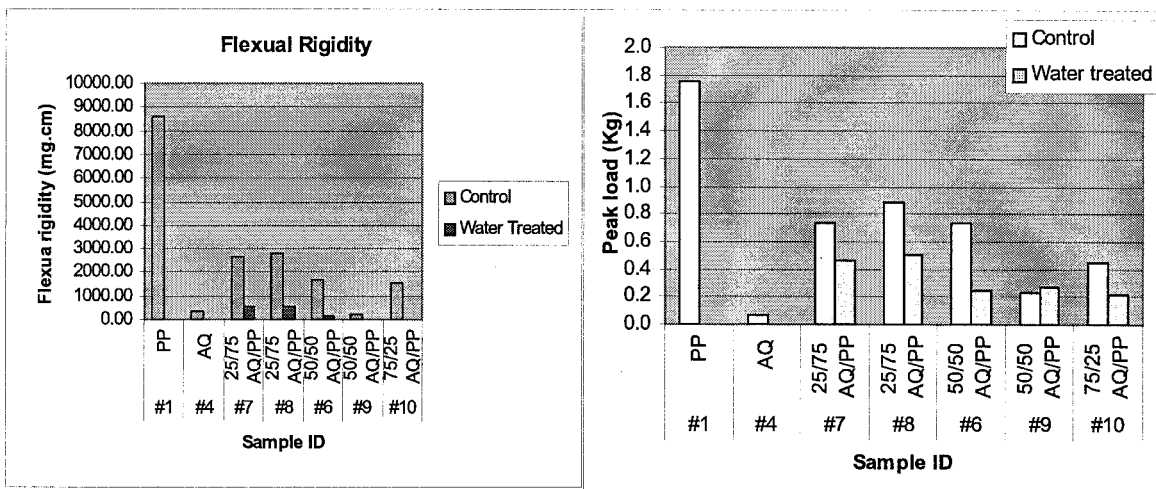
(E) Hydrostatic Head (Water Resistance)

(F) Relative Hydrostatic Head

Figure 5 Physical testing on AQ/PP bicomponent nonwoven webs.

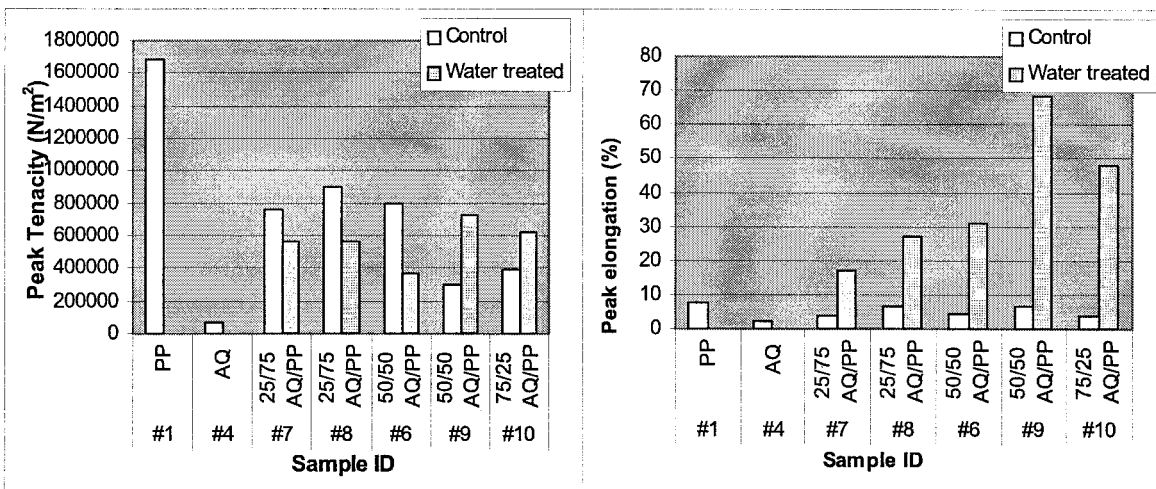


(G) Fiber Diameter by Optical Microscope (G-1) Fiber Diameter by SEM and Optical Microscope



(H) Flexural Rigidity

(I) Peak Load



(J) Peak Tenacity

(K) Peak Elongation

Figure 5 (Continued from the previous page)

Tensile testing

Generally the meltblown nonwoven web is considered to be soft and bouncy, and a method that does not require a direct measurement of web thickness to obtain tenacity is suggested, to avoid the difficulty of measuring the actual web thickness or maintaining the same thickness under different conditions. Choi's application was on pure polypropylene webs, where PP's density was applied directly in the calculation. For bicomponent webs, the overall polymer density still can be obtained, according to each polymer's component ratio in the bicomponent fibers, with knowledge of each polymer's density. However, the density accuracy is not known for certain. Two problems need to be considered at the same time: one is that the real component ratio may not be the same as the labeled ratio; the other is that there are some big bundles and bonding points contained in the web, whose polymer composition may be different from the overall ratio. As a result, another simple method based on original definition for tenacity is suggested here, which should be closer to the true value.

Tenacity (N/m²) =

$$\frac{\text{Load(kg)} \times 9.0 \text{ m/s}^2}{\text{Width of specimen (m)} \times \text{thickness of specimen (m)}}$$

From Figure 5(I), peak loads all decreased except for sample 9 is unexpected increase, which might be induced by the nonuniformity of the web. Peak tenacity decreased for 25% AQ, but increased for sample 10 with 75% AQ [Fig. 5(J)]. Two 50% AQ specimen samples 6 and 9, behaved oppositely: the former decreased and the latter increased. The trend of elongation change is certainly interesting: all increased to some extent [Fig. 5(K)]. Water molecules' movement appeared to enhance the web extension.

Fiber diameter

Fiber diameter images were obtained by means of both optical microscopy (OM) and scanning electron microscopy (SEM). It can be seen in Figure 5(G) and (G-1), that most fibers tended to decrease in diameter after water treatment, as expected. However, two specimens' fiber diameters "increased" unexpectedly. They are sample 7 and sample 6 with 25 and 50% AQ, respectively. Theoretically, it is not likely to happen because of the prominent weight loss of the specimen. It more likely resulted from measurement error. The polypropylene component of sample 7 and sample 6 specimens is from Exxon Company with a melting flow rate (MFR) of 1200 g/10 min, whereas for other specimens, PP is from Basell Company with a MFR of 800 g/10 min, which is closer to the MFR of Eastman

AQ35 polymer. Besides, samples 7 and 6 are quite nonuniform, having stripes along the machine direction and large thickness variation across, the machine direction. One conjecture about OM measurement is that the water-treated specimen measured happened to be picked up from sample area with higher thickness or area mainly distributed with coarser fibers. In the first case, the transmission of light is less than that of the thinner specimen area, and fewer fibers could be observed except some larger size fibers.

With SEM images, about 150 fiber diameter measurements on four or five images were made for each specimen. In Figure 5(G-1), all three compositions specimens decreased in fiber diameter, including sample 7; the same sample showed the opposite result with OM measurements, which accents the necessity of applying two methods on the fiber diameter measurements.

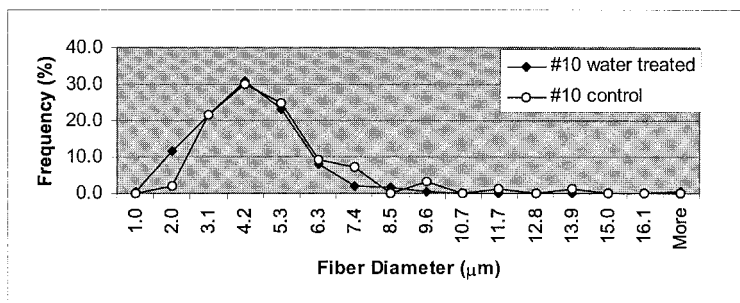
During diameter measurements, particularly large fibers and bundles of fibers were frequently or more easily observed, which would raise the average value to some extent. To understand the diameter distribution in the web, and to compare OM and SEM measurements, the fiber diameter distribution for sample 10 is plotted in Figure 6, where fiber diameter is plotted against frequency percentage. It clearly shows the wide distribution range of meltblown fiber diameter. Even with one side of the fiber washed off, a few fibers of diameter > 10 μm still exist. A similar analysis was also performed for the other specimens and it was found that OM and SEM image measurements were statistically consistent to show the trend of diameter change.

Figures 7 and 8 show DSC scans for the film remaining after water treatment. The two scans look different, which indicates the thermal property is not uniform in the film. Their shapes are closer to the AQ pure nonwoven web thermograph than to the AQ pellets thermograph. Besides, no typical thermal characteristics were observed in the thermograph just like AQ resins; therefore, basically what was washed off in the water is still AQ polymer with some changes, which suggests the possibility of recycling the AQ polymer.

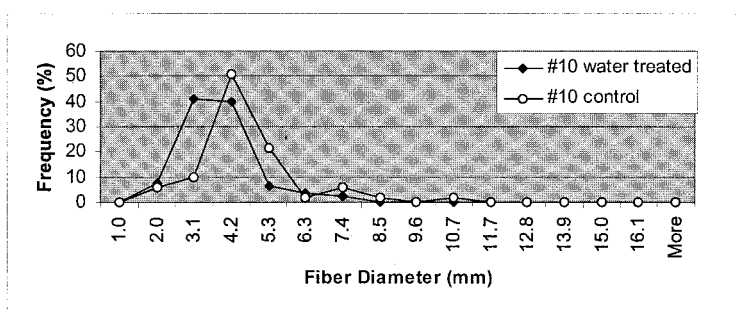
CONCLUSIONS

Water-dispersive Eastman AQ polymers were initially introduced to the meltblown process to make the mono- and bicomponent meltblown webs at Textiles and Nonwovens Development Center, University of Tennessee, Knoxville. The postwater treatment was performed on the fabrics, which resulted in the dispersive part (AQ polymer) being dispersed in distilled water and only the other part remaining in the bico web.

The weight loss was dramatic, proportional to the sample's bicomponent ratio, which suggests an effective method to verify the bicomponent ratio. The treat-



(A) Images Taken by Scanning Electron Microscope



(B) Images Taken by Optical Microscope

Figure 6 Fiber diameter distribution of sample 10 (75/25 AQ/PP) meltblown webs before and after water treatment, determined from images.

ment tends to decrease thickness, fiber diameter, stiffness, and tenacity of the bicomponent webs, whereas relative hydrostatic pressure, resistance to air penetra-

tion, and elongation increased. The webs after treatments exhibited excellent softness and had a silklike hand.

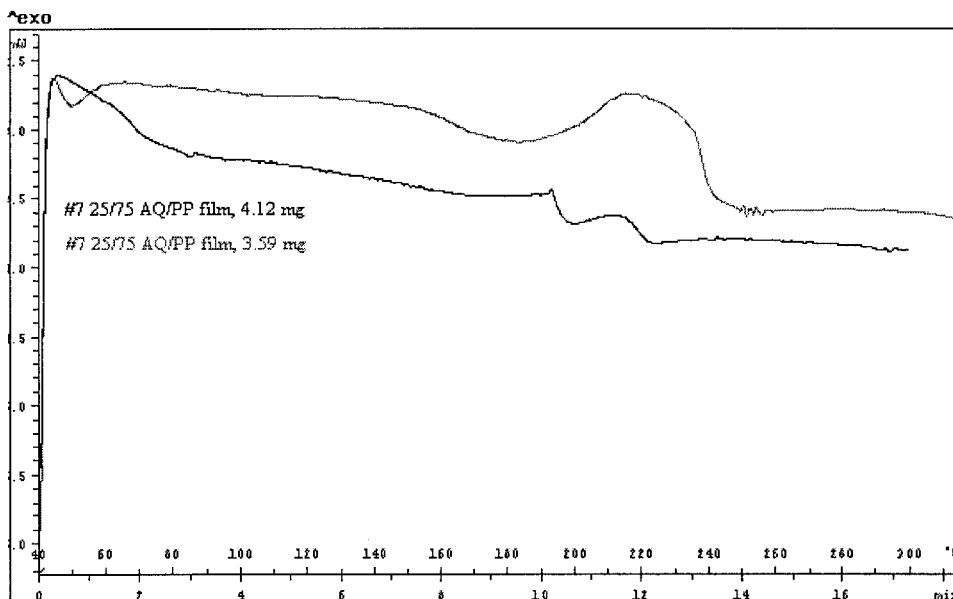


Figure 7 Two DSC scans of film extracted from sample 7 (25/75 AQ35S/PP) water treatment solution by natural evaporation.

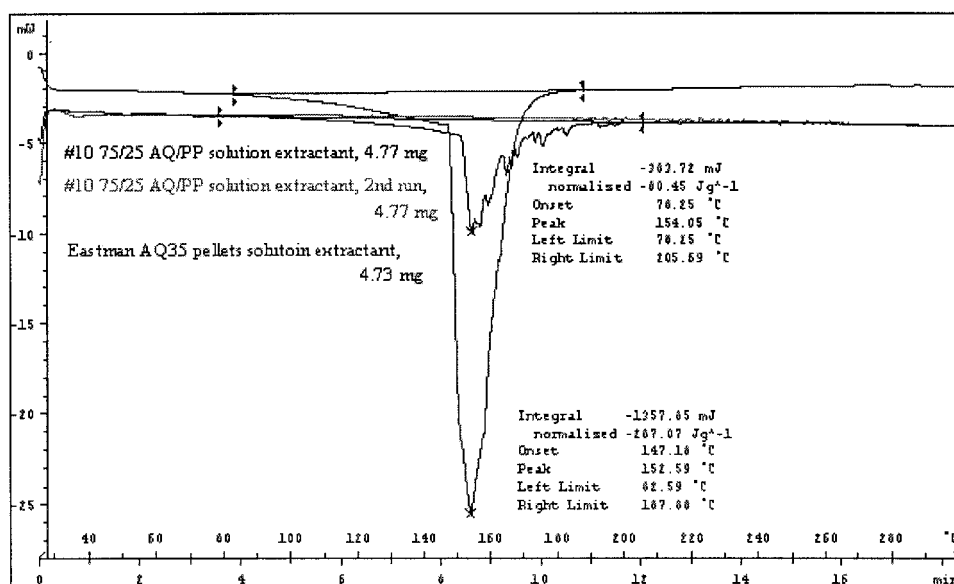


Figure 8 DSC scans of solution extractants from sample 10 (75/25 AQ35/PP) nonwoven web and Eastman AQ35S pellets by heating solution.

The fiber morphology was changed from cylinder to ribbon shape. The diameter measurement of such non-round cross-sectional fibers by OM and SEM methods is directly affected by fiber alignment in the web and orientation upon testing. The trend of the change after treatment, however, is still reliable, based on the large number of diameter measurements by optical microscopy and SEM.

DSC analysis was performed to study the thermal properties of Eastman AQ dispersive polymer and corresponding nonwovens. The similarity between AQ resin and polymer extracted from dispersion those of the solution was found, which indicates the possibility of recycling the AQ polymer after dispersion into water.

The authors express their thanks to Reifenhäuser GmbH at Troisdorf, Germany for financial support on this project and Eastman Chemicals for providing AQ polymers.

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