Effect of sea component dissolution on fibrous structure of islands-in-the-sea spunbond nonwovens

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Abstract. This work presents the preliminary results of our efforts that focused on the development of lightweight and more fibrous nonwoven. For this objective, nonwoven webs that contain bicomponent filaments with island-in-the-sea cross section was produced by spunbonding, which involves extruding of sea and island polymer melts through dies, cooling, and attenuating the bicomponent filaments by high velocity air streams. Nylon-6 and Polyethylene were chosen as the island and sea polymers, respectively. Bonding process was applied to web first to keep structural integrity after removing the sea polymer. The web was hydroentangled with high speed water jets prior to the dissolving process to obtain fiber entanglement. Xylene, which is one of the few chemical that can dissolve Polyethylene, was used for the dissolution of the sea component from the fibrous structure of the spunbond nonwoven. Removal of the sea polymer from spunbond nonwovens that contain bicomponent filaments with islands-in-the-sea cross section was achieved by the developed dissolution process. Weight, thickness, and area of the nonwoven samples changed after the dissolution. After removing the sea polymer, spunbond nonwoven contains only thin island fibers and also gets lighter. Lightweight and more fibrous nonwovens can be obtained with the method given in this study.

1. Introduction
Nonwovens are complex fibrous and porous structures with very low solidity. They are inherently lightweight due to pores filled with air. The pores (voids) are also in the form of a bundle of capillaries. Their thin fibrous structure is useful especially for sound absorption applications since frictional resistance of fibers dissipates propagating sound wave energy [1].

Spunbonding process with bicomponent filament technology coupled with a mechanical fiber-splitting process allows production of submicro- and microfiber based nonwovens. Segmented-pie or islands-in-the-sea cross sections are often chosen to obtain fiber diameters ranging from 0.1 to 5 µm [2]. Producing thinner fibers with islands-in-the-sea cross section is more feasible since the number of islands in a circular bicomponent filament is limitless theoretically and the sea component that surrounds island fibers makes the whole bicomponent filament more spinnable. Island fibers of the resulting nonwoven can be split by mechanical forces. For instance, high pressure hydroentangling water jets can be used for this purpose [3]. Another method that can release island fibers from bicomponent filament is dissolution of the sea component. After removing the sea polymer from the bicomponent filament by dissolving, nonwoven only contains thin island fibers and also gets lighter.

It is known that lightweight materials are advantageous in many applications especially in automotive industry due to fuel consumption and CO₂ emission concerns. For this target, in this work,
we tried to dissolve sea polymer from spunbond nonwovens that contain bicomponent filaments with islands-in-the-sea cross section to obtain submicro- and microfibrous lighter nonwovens.

2. Materials and Methods

Web that contain bicomponent filaments with island-in-the-sea cross section was produced by spunbonding, which involves extruding of sea and island polymer melts through dies, cooling, and attenuating the bicomponent filaments by high velocity air streams. Nylon-6 (PA6) and Polyethylene (PE) were chosen as the island and sea polymers, respectively. Filaments in the web have a weight ratio of 75% for the island polymer and 25% for the sea polymer. Spunbonded web with islands count of 108 was produced at the Nonwovens Institute’s Partners’ Pilot facilities located at North Carolina State University. In Figure 1, scanning electron micrograph (SEM) of the fiber cross-sections of the spunbonded web is given. Sea polymer (PE) surrounds thin circular fibers (island fibers) in a bicomponent filament (Figure 1).

Bonding process should be applied to web first to keep structural integrity after removing the sea polymer [2]. The web was hydroentangled with high speed water jets prior to the dissolving process to obtain fiber entanglement and consolidation. 22,484 kJ/kg energy was transferred to the spunbonded web with single pass.

Xylene, which is one of the few chemical that can dissolve PE, was used for the dissolution of the sea component from the fibrous structure of the spunbond nonwoven. It should be noted that PA-6 (island polymer) provides chemical resistance to xylene.

Dissolution process developed for removing the sea polymer is carried out in the following sequence: Flat bottom flask with 250 ml xylene is placed on hot plate at 100°C surface temperature. Head/joint of the flask is sealed with PTFE joint sleeve to establish leak-proof connection. Then flask is fitted to reflux system. Reflux system allows liquid circulation at outer wall of gas-outlet spiral to drop temperature of the vapour. Sufficient temperature drop leads condensation. Xylene in the flask is heated up to its boiling point (137°C). After removing the flask, nonwoven specimen is put into flask with hot xylene, and then flask is fitted to reflux system again. Heat evaporates xylene during dissolution but xylene mass remains the same because liquid drops come back to flask by condensation. After a specific time, nonwoven sample is taken out from the xylene and then put into hot pure water to clean remaining xylene and sea polymer from fabric surface. Sample is also cleaned in beaker with cold pure water. Finally, nonwoven sample is placed in hot oven at temperature of 60°C for 15 minutes for drying.
Dissolved polyethylene content precipitates after cooling down xylene solution. It can be seen in white color in solution (Figure 2).

Quantity of the dissolved polymer can be calculated by measuring weights of the samples before and after the dissolution process. It is expected to get 25% lighter nonwoven after removal of the sea polymer.

Thickness and area of the samples were also measured before and after sea polymer removal. All the samples were conditioned at 65 ± 2% relative humidity and temperature of 21 ± 1°C before each measurement.

3. Results and discussion
Weights of the 108 islands nonwoven samples changed after the dissolution process. The results are given in Table 1. Most of the sea polymer (PE) (average 22.75 %) was removed from the fibrous structure of the samples according to weight loss results in Table 1. Note that 25 % weight decrease of the nonwoven sample corresponds to full sea polymer dissolution.

| Sample no | Weight [g] | Weight loss [%] |
|-----------|------------|-----------------|
|           | Before dissolution | After dissolution |
| 1         | 0.552      | 0.437           | 20.83          |
| 2         | 0.561      | 0.430           | 23.35          |
| 3         | 0.542      | 0.415           | 23.43          |
| 4         | 0.539      | 0.413           | 23.38          |

Thickness values of the nonwoven samples also changed after the dissolution process. The results are given in Table 2. Thickness decrease can be explained by the movement of the island fibers. It seems that island fibers come closer due to sea polymer removal. Areal shrinkage after the dissolution corroborates this result (Table 3).

| Sample no | Thickness [mm] | Thickness decrease [%] |
|-----------|----------------|------------------------|
|           | Before dissolution | After dissolution |
| 1         | 0.49            | 0.44                   | 10.20         |
| 2         | 0.49            | 0.42                   | 14.29         |
| 3         | 0.50            | 0.46                   | 8.00          |
| 4         | 0.47            | 0.43                   | 8.51          |
Area of the 108 islands nonwoven samples decreased after the sea polymer removal (Table 3). Decrease is under 5%.

| Sample no | Area [cm$^2$] Before dissolution | Area [cm$^2$] After dissolution | Areal decrease [%] |
|-----------|---------------------------------|---------------------------------|--------------------|
| 1         | 49                              | 47.6                            | 2.86               |
| 2         | 49                              | 46.9                            | 4.29               |
| 3         | 49                              | 47.6                            | 2.86               |
| 4         | 49                              | 46.9                            | 4.29               |

Samples shrank three-dimensionally according to the results in Table 2 and 3. This structural change affects solid volume fraction of the samples.

4. Conclusion
Removal of the sea polymer from spunbond nonwovens that contain bicomponent filaments with islands-in-the-sea cross section is achieved by the dissolution process. Submicro- and micro-fibrous lightweight nonwovens can be obtained with spunbond web production and following chemical sea polymer removal method.

More porous fiber network can be expected after the dissolution process. However, nonwoven shrinkage from all dimensions shows us island fibers in adjacent bicomponent filaments move and come closer during the sea polymer removal.

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