J Mater Sci (2011) 46:5761–5767
DOI 10.1007/s10853-011-5531-7

Aerosol filtration properties of PA6/PE islands-in-the-sea bicomponent spunbond web fibrillated by high-pressure water jets

Bong Yeol Yeom · Behnam Pourdeyhimi

Received: 26 February 2011 / Accepted: 4 April 2011 / Published online: 12 April 2011

Abstract The bicomponent spunbonding technology offers high productivity and provides great flexibility in the selection of polymer compositions and fiber configurations. This process was utilized to develop islands-in-the-sea (INS) fabrics consisting of 75% nylon-6 islands and 25% PE sea polymer. The number of islands was varied from 1 (sheath-core) to 108. These webs were hydroentangled at high pressure to cause fracture and fibrillation of the sea polymer and to "release" the islands to form hybrid micro and nanofiber structures. Physical properties as well as aerosol filtration characteristics of these webs were investigated to determine their feasibility for aerosol filtration. The submicron-sized dioctyl phthalate (DOP) particles were used as a challenging aerosol at face velocities in range of 3.3–11.7 cm/s. The filtration efficiency of the webs followed typical behavior for fibrous filtration media in which the most penetration particle size was in range of 0.2–0.3 micron. Surprisingly, the 1 INS web exhibited a higher quality factor compared with other INS webs due to its lower solidity. Also, it was found that corona charging improved the filtration efficiency of 1 INS web 4.3 times compared to discharged webs for 0.2 micron of DOP particles at the face velocity of 5.3 cm/s.

Introduction

Spunbonding (SB) is a manufacturing technique which offers an one-step process for producing nonwovens from the raw materials (thermoplastic polymers) as the fiber and fabric production are combined [1]. In SB, filaments are extruded from multiple banks of spinnerets. These filaments are then drawn to their final diameters (normally, about 10–20 microns) by two high-speed air-jets and laid down onto a porous substrate to form a web. The web is subsequently bonded thermally, mechanically, or chemically. A bicomponent system uses two polymer streams to form a single fiber. The arrangements of the two polymers in the cross section determine the type and the function of the resulting fibers. In the islands-in-the-sea (INS) technology, a polymer forms a matrix around fibrils (islands). The number of islands is controlled by the spinpack geometry. As we increase the number of islands, the size of the resulting fibers is reduced. This is of great interest in that aerosol filtration efficiency is influenced greatly by fiber diameter. This is a critical factor in determining the ultimate filtration efficiency and the resulting pressure drop in the filter [2].

The approach for decreasing fiber diameter is possible through a variety of strategies and processes including SB, melt blowing (MB), and electrospinning; the interest in the latter for the formation of nanofiber has gone through an explosive growth over the last couple of decades. Conventional fiber and filament spinning technologies (and the spunbond process) are limited to produce macro fibers with diameters typically larger than 10–15 microns. The MB process at reasonable throughputs is also limited to fibers larger than 500 nm and more often to produce fibers in the range of 2–5 microns at high throughputs [3, 4]. Electrospinning is the only process today that can routinely produce fibers down to 100 nm or less at throughputs several orders of magnitude lower than those of other processes [5–8]. The fibers produced by both MB and electrospinning, however, exhibit weak mechanical strength and low productivity.

B. Y. Yeom (✉) · B. Pourdeyhimi
The Nonwovens Institute, North Carolina State University, 2401 Research Drive, Raleigh, NC 27695, USA
e-mail: byeom@gw.ncsu.edu

The bicomponent spunbond process has the highest potential for producing high strength nanofibers at significant throughput [9, 10]. There are two categories of bicomponent spunbond fibers: (1) soluble, such as INS and (2) splittable, such as segmented pie, or ribbon [9]. The former generally consists of a dissolvable sacrificial polymer such as poly(lactic) acid (PLA). The polymer is removed after the web has been formed and consolidated to release the islands. The latter typically consists of two polymers with little or no affinity for one another because their diffusion coefficients are quite small and inter-diffusion of entire macromolecules across the interface is unlikely occurred [11]. The possible polymer pairs include polyethylene (PE)/nylon-6 (PA6), polyethylene terephthalate (PET)/PE, and polypropylene (PP)/PA6. These fibers can be split by high-pressure water jets (hydroentangling) that splits the fibers and also entangles the web mechanically to form a strong three-dimensional structure [3, 9, 12, 13]. This technology was first introduced commercially by Freudenberg marketed under the trade name Evolon® [1].

Fedorova and Pourdeyhimi [9] recently investigated the use of INS fibers in a spunbond process and demonstrated the feasibility of these structures for forming high strength fabrics. They also demonstrated that the sea polymer in INS fibers can be fibrillated to release the islands to form sub-micron fibers. Durany and Pourdeyhimi [1] reported excellent mechanical properties and relatively high water vapor transmission rate of fibrillated INS fabrics consisting of PA6/PE, PET/PA6, and PA6/PET.

Previous studies on fibrillated INS webs, however, have been focused on optimizing the mechanical and bonding strength and for forming durable nonwoven fabrics [1, 9, 12, 14, 15]. There is little research conducted to investigate the filtration performance of fibrillated INS webs. Therefore, this study is aimed at investigating the feasibility of INS webs with varying number of islands to be used as new filter media.

Experimental

Materials and INS web fabrication

Ultramid BS 700 nylon-6 (PA6) polymer (BASF) and ASPUN 6811A linear low density polyethylene (PE) polymer (Dow Chemical Co.) were used as the island and the sea polymers, respectively. Basic properties of polymers used in this study are summarized in Table 1.

The bicomponent spunbonded INS webs were produced at the Nonwoven Institute's (NWI) Partners' pilot facilities located at North Carolina State University. These webs have a polymer ratio of 25% of PE for the sea polymer and 75% of PA6 for the island with five different island counts from 1 to 108. The basis weights of unbonded INS webs were kept constant at 100 g/m^2. The spunbonded webs were fibrillated and bonded simultaneously by hydroentangling following procedures previously reported by Fedorova and Pourdeyhimi [1, 12]. The total specific hydroentangling energy was 67.5 MJ/kg. This hydroentangling energy was reported previously to be optimal for fibrillating INS fibers [1]. The SB and hydroentangling processes used in this study are fully described elsewhere [1, 9, 12].

Table 1 Properties of polymers used

Polymer	Melting temperature, T_m (°C)	Density, ρ_{fiber} (g/cm^3)	Polymer ratio (%)
PA6 (island)	220	1.14	75
PE (sea)	125	0.94	25

Web characterization and filtration property

The structure of hydroentangled INS webs was examined by using scanning electron microscope (SEM, Hitachi S-3200). According to ASTM D 5729, the thickness of the web was measured with the thickness testing gage (AMES, model BG1110-04) with a presser foot diameter of 25.4 mm at an applied force of 4.14 kPa.

The INS webs were tested under three conditions: (1) received, (2) discharged, and (3) charged. The received web refers to the webs produced and received with no treatment or pre-conditioning. The discharged web refers to the received webs after the removal (discharge) of electrostatic charge—this was to ensure that the process had not resulted in any electrostatic charging of the sample. The webs were discharged by dipping the webs in isopropyl alcohol (IPA) and allowing them to dry at room temperature for 24 h. The charged samples refer to discharged webs and subsequently being charged. The webs were electrostatically charged by a negative ion generator at 9 kV of applied voltages. The charging time and tip-to-sample distance were 10 min and 5 cm, respectively, and at 65% relative humidity.

The filtration properties of INS webs were evaluated at several face velocities ranging from 3.3 to 11.7 cm/s. DOP (dioctyl phthalate) particles were used as the challenging material generated by a collision type atomizer and evaporated through a membrane dryer and selected monodisperse particles with the size between 0.015 and 0.4 micron by a long differential mobility analyzer (DMA, TSI Inc., Model 3081). The particles were neutralized by a Kr-85 radioactive source to the Boltzmann equilibrium state and then fed into a filter holder with 100 cm^2 of effective area. Their number concentrations on the upstream side and the

downstream side of the web were measured by using two condensation particle counters (CPCs, TSI Inc., Model 3760A). The flow rate and the resistance were measured by a mass flow meter (TSI Inc., Model 4043) and an electronic manometer (TSI Inc., Model 220). Aerosol capture efficiency was calculated by following equation:

$$E = 1 - \frac{N_{\text{down}}}{N_{\text{up}}} = 1 - P \quad (1)$$

where, E(-) is a fractional efficiency for DOP particles with a specific particle size, N_{up} and N_{down} are number particle concentrations on the upstream and the downstream sides, and P(-) is a fractional penetration through the web.

Results and discussion

Filtration properties of hydroentangled INS web

Figure 1 depicts the SEM images of INS webs with various PA6 islands in PE sea. 1 INS fiber, typically called sheath–core fiber, has only one PA6 fiber covered by PE sea polymer and 37 INS fiber has 37 PA6 INS polymer. These are originally round-shaped fibers with a fiber size in the range of 15–20 microns. The islands are separated from the sea by high-pressure water jet process and form fine fibrils as they are fractured.

Figure 2 shows the fractional penetration of DOP particles ranging in size from 0.015 to 0.4 micron at the face velocity of 5.3 cm/s. The shape is typical of aerosol filter media and represents a combination of Brownian diffusion, interception, and inertial impaction at these particle sizes. The maximum penetration is affected by fiber diameter, solidity, and face velocity [16]. The most penetrating particle sizes (MPPS) for all samples are in range of 0.2–0.3 micron. The efficiency of 108 INS web is higher at the MPPS than other webs, and 1 INS web shows the highest penetration for all particle sizes.

Filtration performances of INS webs were measured with 0.2 micron of DOP particles at the face velocity of 5.3 cm/s and the data are summarized in Fig. 3. Compared to 1 INS fiber, the efficiencies of other INS webs show improvements, however, and the increase of number of islands only marginally improves efficiency. The efficiency increment may be from the increase in web solidity that is expected to lead to an increase in both the resistance and the efficiency [17].

The effect of the face velocity on the filtration performance is shown in Fig. 4. The efficiency of INS web decreases with increasing the face velocity. The velocity used in this study is typical of those used for media in high efficiency HAVC filter design and is based on an effective area of the filter web, not on a cross-sectional area of a filter element. Note that the efficiency of 1 INS web with the velocity decreases faster than those for other webs, in particular twice compared to 108 INS web in the velocity range used in this study. The resistance, however, shows that the resistance of all INS webs linearly increased with

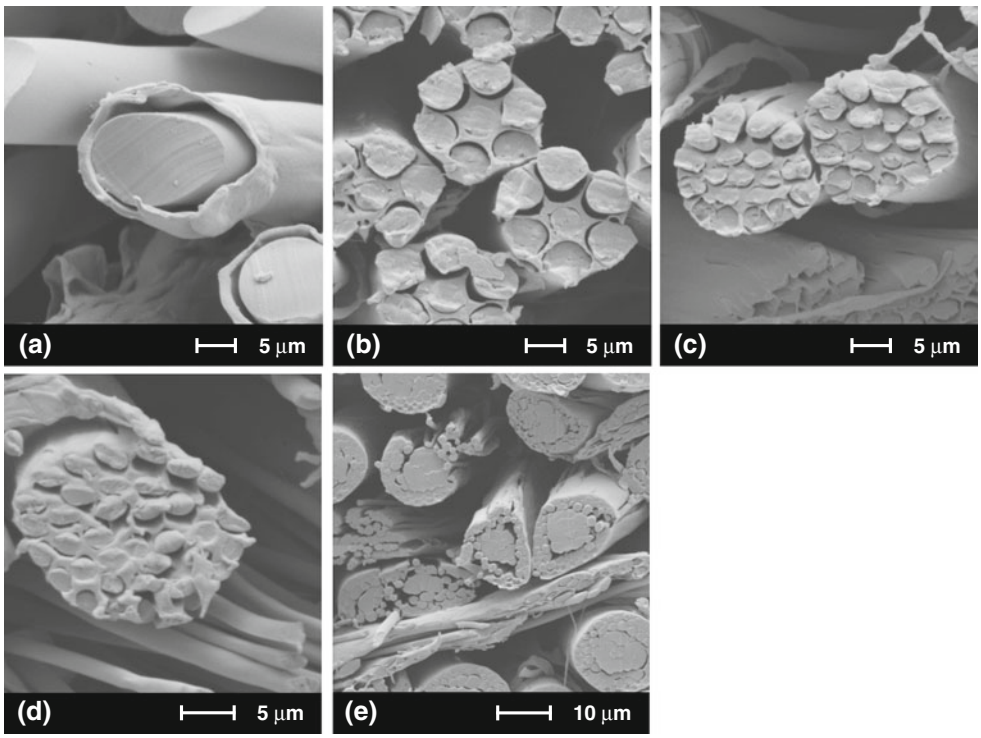

Fig. 1 SEM images of INS webs with several number of islands in the sea: **a** 1, **b** 7, **c** 19, **d** 37, and **e** 108

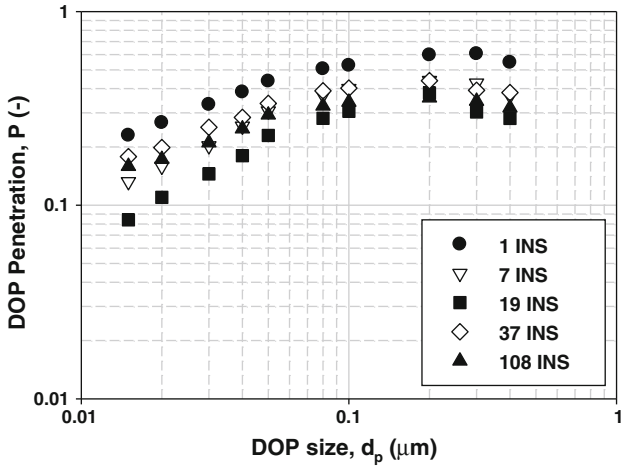

Fig. 2 Fractional penetration of DOP particles through received INS webs at the face velocity of 5.3 cm/s

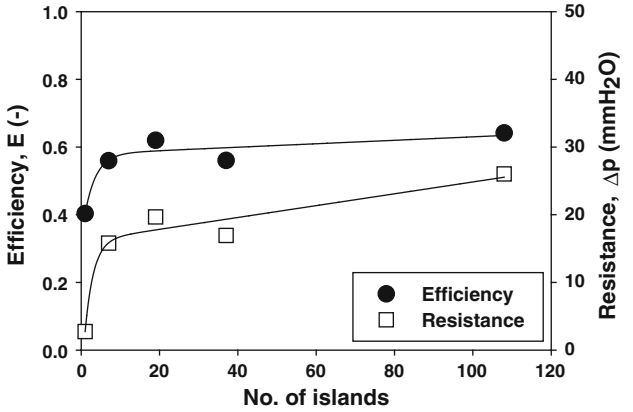

Fig. 3 Filtration performance of received INS webs with different island counts against 0.2 μm of DOP particle at 5.3 cm/s of face velocity

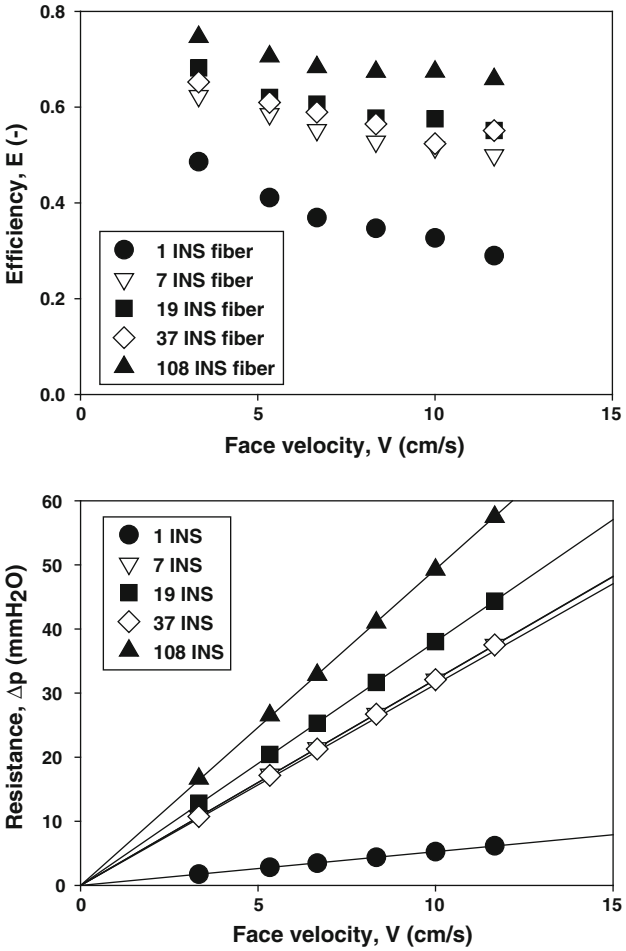

Fig. 4 Filtration performance of received INS webs as a function of face velocity with 0.2 micron DOP particle

the velocity, which means that the flow inside the web is still laminar and follows Darcy's law [17]. The 108 INS web has the steepest slope for the resistance with increasing the face velocity and INS webs. Other island counts fall between 1 INS and 108 INS.

The filtration performance shown in Fig. 5 was measured with the web having 1–4 layers at 5.3 cm/s of face velocity. The size of DOP particle used was 0.2 micron, i.e., the MPPS for most of INS webs. The slopes of each line, a ratio of particle penetration to resistance, indicate the quality factor (QF), also called the filtration index, expressed by Eq. 2.

$$\mathrm{QF} = -\frac{\ln(1-E)}{\Delta p} \tag{2}$$

We used the layered webs from 1–4 layers to obtain the QF from the slope since QF is not a function of the thickness of a filter media having a same fiber and web properties. This factor is useful for evaluating filter effectiveness based on energy saving [12] and the fitted line with greater downward slope indicates a higher performance [18]. In the case of the samples used in this study, as shown in the Fig. 5, the 1 INS fabrics has the highest QF and the 108 INS fabrics show the lowest value. Even though the efficiency of 108 INS web is higher than those of other island counts, the web has a lower QF due to its higher resistance. The higher efficiency is due to a larger proportion of smaller fibers and the higher solidity of the samples with high island counts. The QF eventually decreases with higher solidity in the vicinity of MPPS [17, 19]. This generally agrees well with our experimental data.

Electrostatic properties of hydroentangled INS web

The filtration efficiencies of 1 INS and 108 INS webs are shown in Fig. 6, in which the webs have three different conditions of received, discharged, and corona charged. The MPPS for 1 INS web was 0.2 micron, not depending

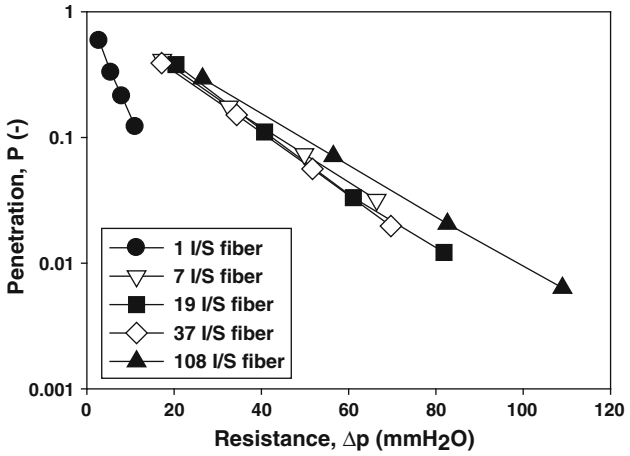

Fig. 5 Penetration curve through multi-layered received INS webs with 0.2 micron DOP particle at 5.3 cm/s face velocity

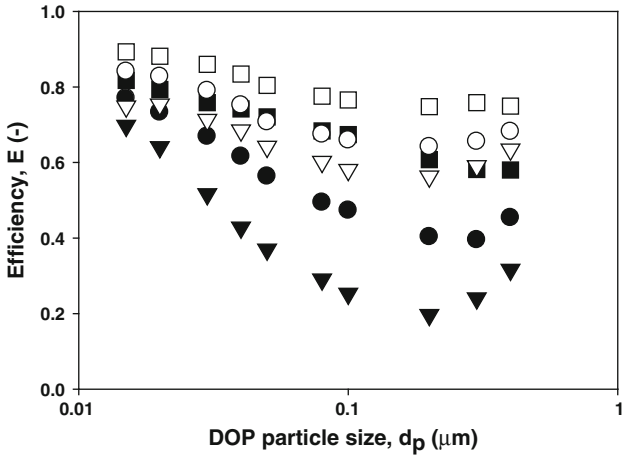

Fig. 6 Filtration efficiencies of 1 INS (*filled symbols*) and 108 INS (*unfilled symbols*) webs with different conditions at 5.3 cm/s of face velocity: *circle* received, *reverse triangle* discharged, and *square* charged

on the sample condition. Its filtration efficiency was 40.3 and 19.6% for received and discharged web with 2.8 mm H_2O of the resistance, respectively. The efficiency of 1 INS web discharged by IPA solution was significantly decreased for all DOP particle size, particularly in the vicinity of MPPS. And, 108 INS web exhibited the slight difference of the efficiency with the conditions, which were 64.1 and 56.3% for received and discharged web with 27.0 mm H_2O of the resistance, respectively. The efficiency difference between received and discharged conditions indicates the effect of electrostatic charges that make the efficiency increase without sacrificing the resistance. The formation of electrically charged polymer has been studied to make an electret, which is a quasi-permanent electrically charged material and is widely used in the aerosol filtration due to its enhanced filtration properties [20–22]. The water jet spraying on the nonwoven fibrous web is a modified method being used to improve the efficiency of aerosol filter media in which several charging enhancing additives are also used to maximize not only the initial charging capacity, but the charging stability. This charging technology is affected by a zeta potential of polymer, and electrical conductivity of water, melt additives used in or on the fiber, and water spraying conditions [22]. The hydroentangling process, the high-pressure water spraying for both fibrillating fibers and bonding the web, is very similar to this charging technique and improves the electrostatic charging property of the filter media, even the INS web used in this study did not include any melt additives and therefore, the hydroentangling conditions were not optimized for the electrostatic charging of filter media. It is, however, clearly shown that PE/PA6 INS webs are electrostatically charged by the hydroentangling process which can be used for developing the electret filter media and forming the composite web simultaneously.

Figure 6 also shows the efficiency of INS webs electrostatically charged by the negative corona discharger. 1 INS and 108 INS webs showed 60.7 and 74.8% charged filtration efficiency, respectively. The efficiency of corona charged web improved significantly comparing to the discharged and received webs.

Web properties of hydroentangled INS web

A characteristic of hydroentangled webs is the presence of jet streaks corresponding to high and low densities in the form of peaks and valleys running the length of the web. The period of the streaks corresponds to the spacing of the nozzles used in the jet strips employed during hydroentangling [23]. Figure 7 shows the cross-sectional SEM images of the fibrillated INS web. These images clearly show the jet streaks where the fibers are clearly more highly fibrillated and entangled as compared with other parts [1]. The structure is composed of fine fibrils of irregularly shaped PE and separated PA6 island fibers completely or partially from PE sea polymer. SEM observations indicate that even at the high pressures utilized, un-fibrillated fibers still remain throughout the structure.

Table 2 summarizes the basis weight and thickness for all samples, where σ is a standard deviation. Note that the sheath–core (1 island) web is not as highly consolidated although the basis weight is lower. This is expected because the fiber diameter is larger in these structures; consequently, these require higher hydroentangling energy to reach the same level of consolidation. These web properties are used for calculating the solidity of the webs.

As may be evident from the SEM images, the fiber diameter distribution is rather broad for these webs because

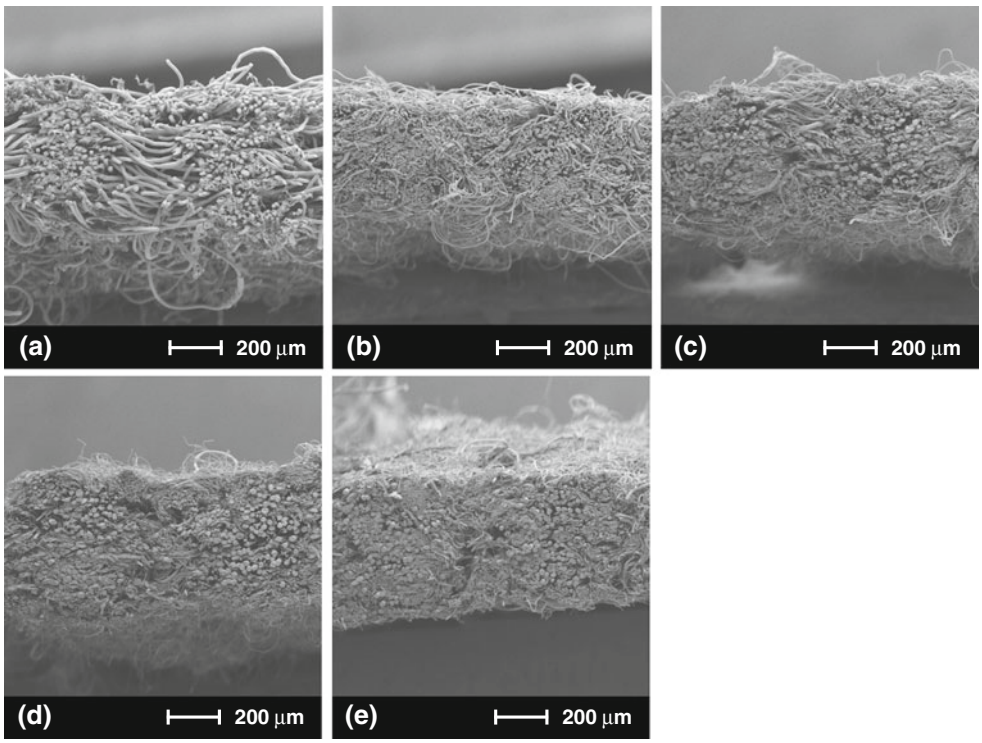

Fig. 7 SEM images of fractured INS webs with several number of islands in the sea: **a** 1, **b** 7, **c** 19, **d** 37, and **e** 108

Table 2 Basis weight and thickness of hydroentangled INS webs

Number of islands	Basis weight, W (g/m^2)	Thickness, L (mm)
1	106.3 ($\sigma = 1.5$)	0.62 ($\sigma = 0.02$)
7	125.7 ($\sigma = 1.4$)	0.49 ($\sigma = 0.02$)
19	136.3 ($\sigma = 1.3$)	0.50 ($\sigma = 0.02$)
37	126.1 ($\sigma = 1.0$)	0.49 ($\sigma = 0.03$)
108	127.4 ($\sigma = 1.8$)	0.49 ($\sigma = 0.02$)

of incomplete fibrillation. Thus, the true fiber diameter is difficult to establish. In this case, an useful and practical method to define the fiber diameter is an effective fiber diameter, $d_{\text{f,effective}}$, which has been widely used to predict a collection efficiency of challenging particles on the fiber [2]. This value is calculated from testing fluid properties and web properties and is valid in range of 0.006–0.3 of solidity. Effective fiber diameter is defined as:

$$d_{\text{f,eff}} = \left(\frac{\mu V L}{\Delta p}\left(64\alpha^{1.5}\left(1 + 56\alpha^3\right)\right)\right)^{0.5} \tag{3}$$

where, μ (Pa s) is the fluid viscosity, V (cm/s) is the face velocity, L (cm) is the fabric thickness, Δp (mm H_2O) is the resistance across the filter media, and α(-) is the solidity (or packing density) defined as the following expression, which means how much filled the fibers in the fabric.

$$\alpha = \frac{\rho_{\text{web}}}{\rho_{\text{fiber}}} = \frac{W/L}{\rho_{\text{fiber}}} \tag{4}$$

where, ρ_{web} (g/cm^3) and ρ_{fiber} (g/cm^3) are the bulk web density and the fiber density, respectively.

Figure 8 shows the solidity and effective fiber diameter for all samples. The solidity of INS webs increased to 0.25 from 0.16 of 1 INS web and the effective fiber diameter for the 108 INS web decreased to 5 micron from 10 micron for the 1 INS web. Note that the island counts greater than 1 show similar properties and are not affected significantly by island count. Ideally, the 108 INS web should have a fiber diameter approximately 0.5 micron for a 75/25 island to sea ratio. This indicates that the fibrillation is not complete [1, 9].

For observing the effect of the number of passes through the hydroentangling unit on the filtration performance, INS webs were hydroentangled with 3 and 6 passes, which are equivalent to 67.5 and 135.0 MJ/kg of water jet energy, respectively. Their filtration efficiency and resistance data are shown in Fig. 9. The capture efficiency and the resistance of INS web hydroentangled with 6 passes increased for all webs used. Note that the resistance increases linearly with the number of islands, which indicates that at the same hydroentangling energy INS webs with a higher number of islands are consolidated more easily than those with fewer number of islands.

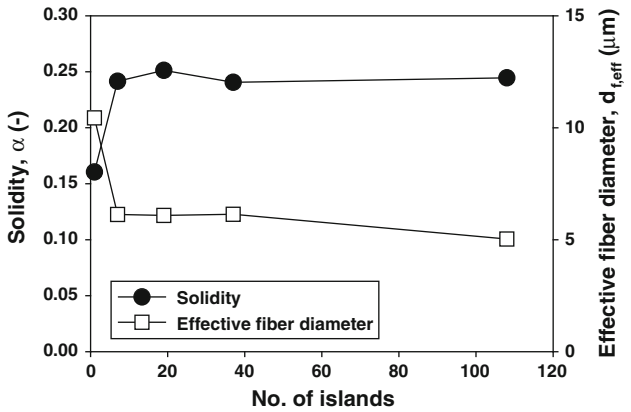

Fig. 8 Solidity and effective fiber diameter of received INS webs with different number of islands

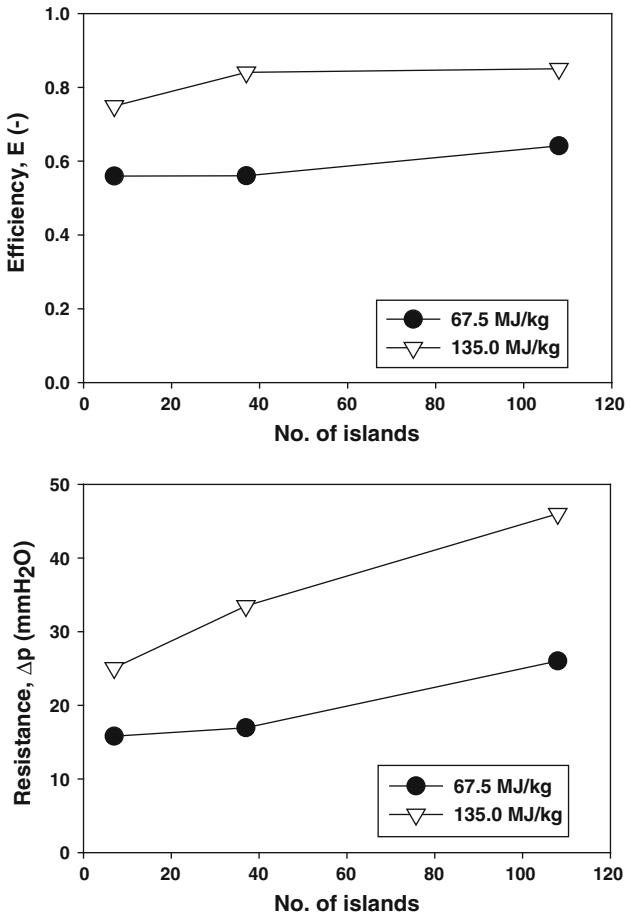

Fig. 9 Effect of number of hydroentangling pass on filtration performance with 0.2 micron DOP particle at the face velocity of 5.3 cm/s

Conclusion

The PA6/PE INS webs with several numbers of PA6 islands were fabricated by the bicomponent SB technology and fibrillated to form fine fibers by high-pressure water jet spraying. The number of island in the sea had little effect on the increment of web solidity after one island. The most penetration particle size for all INS webs used in this study was about 0.2 micron, but was slightly shifted to larger particle size after corona charging. We found that the hydroentangling process improves the aerosol filter efficiency by increasing the electrostatic charging on the PA6/PE INS web. And, larger number of islands is more consolidated at the same hydroentangling energy level. Our results show the feasibility of INS web to be used for new aerosol filter media with corona charging depending on the applications.

References

1. Durany A, Anantharamaiah N, Pourdeyhimi B (2009) J Mater Sci 44:5926. doi:10.1007/s10853-009-3841-9
2. Hinds WC (1982) Aerosol technology: properties, behaviors, and measurement of airborne particles. Wiley, New York
3. Zhang D, Sun C, Song H (2004) J Appl Polym Sci 94:1218
4. Zhao R, Wadsworth LC (2003) Polym Eng Sci 43:463
5. Chronakis IS (2005) J Mater Proc Technol 167:283
6. Huang ZM, Zhang YZ, Kotaki M, Ramakrishna S (2003) Compos Sci Technol 63:2223
7. Gupta P, Wilkes GL (2003) Polymer 44:6353
8. Subbiah T, Bhat GS, Tock RW, Parameswaran S, Ramkumar S (2005) J Appl Polym Sci 96:557
9. Fedorova N, Pourdeyhimi B (2007) J Appl Polym Sci 104:3434
10. Fedorova N (2006) Dissertation, North Carolina State University
11. Sun C, Zhang D, Liu Y, Xiao R (2004) J Appl Polym Sci 93:2090
12. Anantharamaiah N, Verenich S, Pourdeyhimi B (2008) J Eng Fibers Fabr 3:1
13. Ghassemieh E, Acar M, Versteeg HK (2001) Compos Sci Technol 61:1681
14. Fedorova N, Verenich S, Pourdeyhimi B (2007) J Eng Fibers Fabr 2(1):38
15. Pourdeyhimi B, Fedorova N, Sharp SR (2006) US Patent 0223405 A1
16. Brown RC (1993) Air filtration: an integrated approach to the theory and applications of fibrous filters. Pergamon Press, Oxford
17. Wang J, Kim SC, Pui DYH (2008) J Aerosol Sci 39:323
18. Barrett LW, Rousseau AD (1998) Am Ind Hyg Assoc J 59:532
19. Kalayci V, Ouyang M, Graham K (2006) Filtration 6:286
20. Chudleigh PW (1976) J Appl Phys 47:4475
21. McCarty LS, Whitesides GM (2008) Ang Chem Int Ed 47:2188
22. Sebastian JM, Jones ME, Li FB, Chou S, Schultz NE, Keough JL, Shah RR, Japuntich DA (2009) US Patent 0293279 A1
23. Shim E, Pourdeyhimi B (2005) Textile Res J 75:569