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Web fabrication and characterization of unique winged shaped, area-enhanced fibers via a bicomponent spunbond process

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Abstract High surface area fibers are sought after for a variety of applications such as liquid and air filtrations, medical and biopharmaceutical applications. Typically, higher specific surface area is achieved by resorting to using smaller fibers. This paper focuses on the development of a new shaped bicomponent spunbond structure for achieving high surface area. The fibers used in the spunbond process comprise a sacrificial sheath polymer and the shaped core polymer. Nonwovens were directly produced by using these fibers in a spunbond process. The spunbond webs were mechanically bonded by high energy water-jets and subsequently, the sheath polymer was removed in a 6 wt% NaOH solution at 90 °C. The final fibers showed a unique cross-sectional shape having 32 flaps or wings held together with a reasonable backbone. We report the results for a variety of polymer combinations including PP, PET, PBT, and PLA as the core and PLA and EastONE^{TM} as the sheath. The fiber morphologies were observed by SEM and showed 7–12 micron of the minor and 12–21 microns of the major with various core polymers used. The surface area of these fibers was compared to those of other shaped fibers.

Introduction

Nonwovens are used as a platform technology for developing many functional systems. They provide the flexibility in design and process and the cost-effectiveness required for many of the high performance products in use today or sought after. High specific surface area is considered as one of the most important features of fibrous material. High surface area fibers enhance properties related to insulation, fluid retention, drapeability, and durability [1–5]. Improved performance due to increased surface area is also important to wipes, filtration, military, and medical products as well.

The surface area in conventional fabrics is typically very low, making them of little use in critical applications such as filtration and bio-separation which require significantly higher surface area. To achieve higher surface areas significantly smaller fibers are required.

The equation for calculating the specific surface area is derived from the following procedure [6]. The cross-sectional area (A) of a round fiber in cm^2 is given by,

$$A = \frac{\text{Denier}}{900000\rho} \tag{1}$$

where Denier is a linear density of fiber in g/9 × 10^5 cm and ρ is a fiber density in g/cm^3 (1.38 g/cm^3 for PET). Then, the perimeter (P) of the fiber in cm is given by,

$$P = \sqrt{4\pi A} = \sqrt{\frac{4\pi \times \text{Denier}}{900000\rho}} \tag{2}$$

Then, the specific surface area (S) in cm^2/g based on the mass for non-circular shaped fiber is given by,

$$S = \alpha\left(\frac{P}{A\rho}\right) = \alpha\sqrt{\frac{4\pi}{A\rho^2}} = \alpha\sqrt{\frac{36\pi \times 10^5}{\rho \times \text{Denier}}} \tag{3}$$

where considering the surface area variation, α, defined as $P^2/4\pi A$ is a shape factor that is equal to 1 for a circle and has a value greater than one for other shapes. This indicates that higher values than 1 would yield a higher perimeter due to the greater indentation of the cross-section of fiber. Consequently, the specific surface area would be expected

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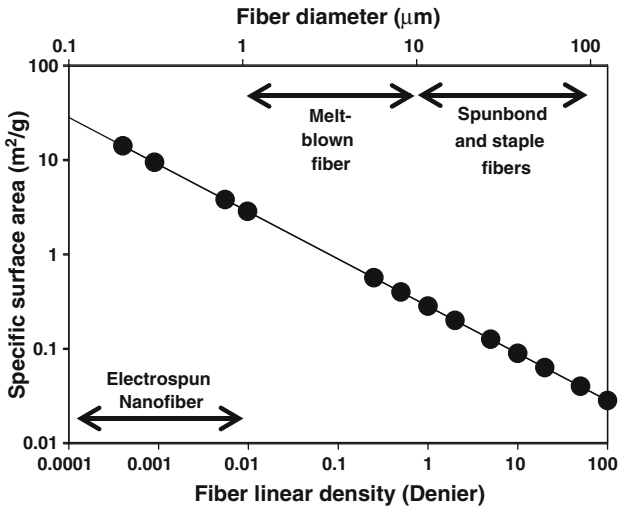

Fig. 1 Specific surface area as a function of fiber size

to increase for a given linear density. This equation is easily converted into an equivalent fiber diameter (d_f) (assuming that the fiber is round but has the same area) in micron with the following relation between the linear density and the fiber diameter.

$$d_f = 11.89\sqrt{\frac{\text{Denier}}{\rho}} \tag{4}$$

It is clear that for a given fiber, the surface area is a function of its linear density (and therefore the diameter) of the fiber. Figure 1 clearly shows this relationship for PET fibers. Other fibers would follow a similar trend but would be different in their absolute values because of the variations in density [7]. Note that a 2 denier fiber has a surface area of only 0.20 m^2/g. A 1 micron fiber will have a specific surface area of 2.9 m^2/g and a 200 nm fiber will have a specific surface area of $\sim$14 m^2/g.

There are two simple approaches to increasing fiber surface area: (1) decreasing fiber diameter and (2) modifying fiber cross-sectional shape. The first approach is possible through a variety of strategies and processes including spunbonding (SB), melt-blowning (MB), and electrospinning; the interest in the latter for the formation of nanofiber has gone through an explosive growth over the last decade. Conventional fiber and filament spinning technologies (and the spunbond process) are limited to producing macro fibers with diameters typically above 10–15 microns. The melt-blowing process at reasonable throughputs is also limited to fibers larger than 500 nm and more often to producing fibers in the range of 2–5 microns at high throughputs. Electrospinning is the only process today that can routinely produce fibers down 100 nm or less. The fibers produced by both melt-blowing and electrospinning, however, exhibit weak mechanical strength and low productivity, in particular for electrospinning [8, 9].

The bicomponent spunbond process has the highest potential for producing high strength nanofibers at significant throughput [10, 11]. Evolon® is the first revolutionary commercially available spunbond segmented pie structure wherein the fibers are split and mechanically entangled and bonded by subjecting the fibers to high-pressure water-jets [7]. The resulting fibers have an equivalent fiber diameter of about 1–2 microns. Fedorova and Pourdeyhimi [10] recently investigated the use of islands-in-the-sea (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 and form submicron fibers. Durany et al. [7] evaluated the mechanical strength and water vapor transmission rate (WVTR) of modified INS filament. Based on their estimation, high island counts above 108 are required for obtaining the fibers having 0.5 micron or less of fiber diameter. In this case, the basic assumption is that fine fibers are completely separated from one another. Most fibers on the surface can be split, but often it is not the case for the fibers in the body of the web. Thus, INS webs may have a larger effective fiber diameter than that estimated.

It is not therefore surprising to find many attempts aimed at increasing specific surface area by the use of shaped fibers that have been used for several applications such as a capillary-channeled stationary phase for liquid chromatography [8, 9], a sound insulator for acoustics [12, 13], air filtration [14], and a scaffold for tissue engineering [15]. One such fiber is the 4DG fiber developed by Eastman Chemical and Procter & Gamble and available from Fiber Innovation Technologies (FIT).

The 4DG fiber seeks to increase the depth of the grooves by providing a fiber with a specific cross-sectional geometry. The 4DG fibers are formed by the use of special spinnerets. Consequently, the process imposes several limitations on the formation of fibers such as 4DG and other fibers having a similar configuration. Many such fibers cannot be spun into fiber diameters less than about 50–60 microns; the minimum denier attainable with the 4DG fiber is approximately 6. Such fibers have a limited number of arms and grooves resulting in a relatively low surface to volume ratio. Finally, due to the size and geometry of the 4DG fiber, the arms can easily interlock (inter-digitate) during fabric formation resulting in dense and compressed materials, which diminishes their filtration and absorption properties [16].

This paper deals with the feasibility of the production of new shaped fibers with a linear density of 1–2 deniers. Their shaped cross-sections can result in significant surface area that can potentially overcome the shortcomings of the

4DG concept. Bicomponent SB technology having sheath–core fiber structure was used to spin new shaped fibers, in which the sheath part consisted of a sacrificial polymer and the core was the shape-formed polymer.

Experimental

Shaped and area-enhanced fibers were prepared through a two-step process: production of bicomponent spunbond nonwoven web hydroentangled and the removal of the sacrificial sheath component via post-treatment. Figure 2 shows the possible polymer configurations which typically results in an oval shape having a low aspect ratio of less than 1.0. The core polymer that forms a backbone and the wings is totally encapsulated and isolated by sheath polymer and is not in contact with air during spinning. The number of wings can be adjusted through the assemblage of the plates in the spin-pack in range of 12 and 64 (higher numbers are theoretically possible but more difficult to spin and control). A number of thermoplastic polymers can form the structure. Examples include polypropylene (PP), polyethylene terephthalate (PET), polybutylene terephthalate (PBT), nylon-6 (PA6), polyethylene (PE), thermoplastic urethanes (TPU), copolyester (Co-PET), biodegradable poly(lactic acid) (PLA), and liquid crystalline polymers can be used for core formation [17].

In this study, PP, PET, PBT, and PLA were used for the core component, and PLA and EastONE^{TM} were used for the sacrificial sheath. PLA is easily dissolved in NaOH solution at elevated temperature of 90 °C and EastONE^{TM}, a water-dispersible sulfopolyester developed by Eastman Chemical Company, was removed by exposure to hot deionized water. PP (Sunoco, CP360H, 34 melt flow index (MFI)), PET (Eastman, F61HV, relative viscosity: 6.1), PBT (Ticona, Celanex® 2001, 6.5 MFI), and PLA (NatureWorks, 6202D, 15–30 MFI) were used as a sheath and a core polymers

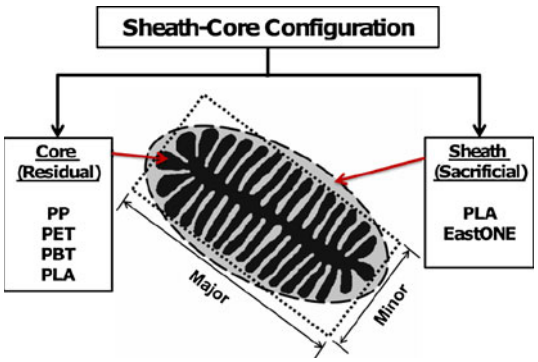

Fig. 2 Possible polymer configurations of shaped, area-enhanced fiber

without further treatment. Bicomponent fabrics were produced at the Nonwovens Cooperative Research Center (NCRC) Partners' pilot facilities located at North Carolina State University. The take-up velocity and polymer throughputs were 2000 m/min and 0.30–0.35 g/hole/min, respectively. Bicomponent fibers are typically formed by co-extruding two polymer streams that form a single fiber once they reach the spinneret. The bicomponent character of the fiber is determined by the manner in which the two polymer streams are metered and channeled in the spinpack. The winged fiber is formed by channeling the wing segments appropriately as in the case of segmented fibers except that one polymer stream is directed to form a sheath around the wing segments. The spinpack was designed specially by Hills Inc. for this study.

And then, these spunbond webs were entangled by high energy water-jet three times on the Fleissner hydroentangling unit at the pilot facilities of NCRC at a speed of 10 m/min by using 5 manifolds with water-jet pressures ranging from 50 to 220 bar. In pre-experiments, we established the level of energy required to consolidate the web sufficiently and to ensure that the web would withstand the washing step. Subsequently, the sheath polymer was removed in a wash bath equipment consisting of a constant temperature bath and a glass tube in which NaOH solution of 6 wt% was stored at 90 °C. The weight reduction, ΔW (%), was calculated by the following equation:

$$\Delta W = \frac{W_0 - W}{W_0} \times 100(\%) \tag{5}$$

where, W_0 and W are the initial sample weight and weight at any washing time, t, after drying for 12 h at 70 °C.

The cross-sectional shapes of fibers were examined with scanning electron microscope (SEM, Hitachi S-3200). The perimeter was determined experimentally by using image analysis techniques. Fiber cross-sectional shape was characterized by aspect ratio and the specific surface area. We can define a bounding rectangle which is the smallest rectangle covering the object with the same orientation. Aspect ratio (AR) is defined as a ratio of the minor and the major of the rectangle that are shown in Fig. 2.

$$\text{AR} = \text{Minor}/\text{Major} \tag{6}$$

The specific surface area (A_s) was determined as described above.

Results and discussion

Figure 3 shows the surface (a) and the cross-section (b) of PLA/PBT winged fibers after etching to distinguish the two components. These fibers comprise an internal PBT core

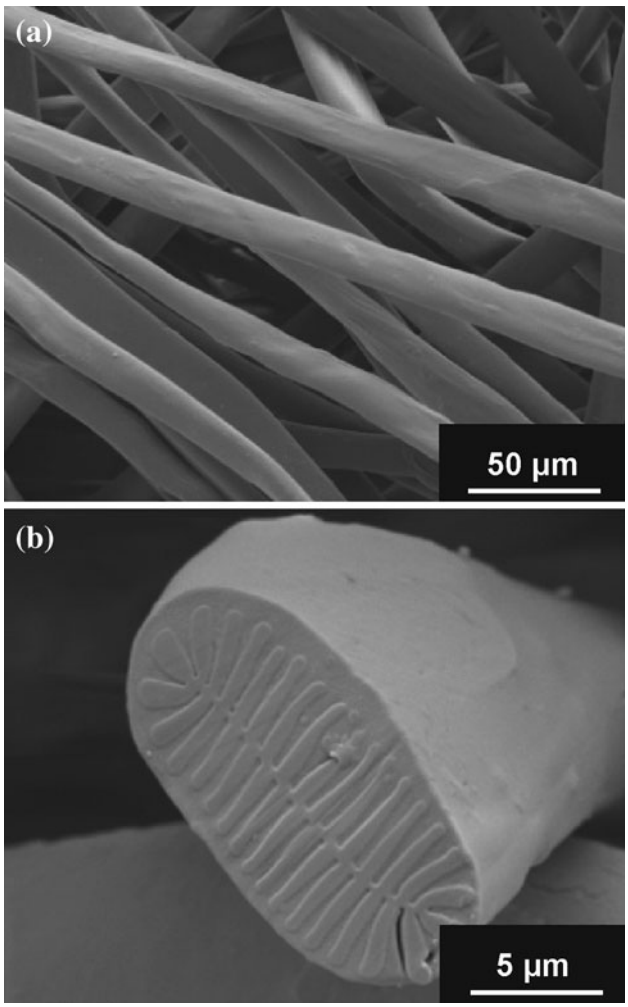

Fig. 3 SEM images of spunbond PLA/PBT winged fiber with a ratio of 60 to 40: **a** surface and **b** cross-section of fiber

polymer and external PLA sheath polymer as a ratio of 40 to 60. Melt-spun fibers exhibited smooth surfaces and oval cross-sectional shape. The internal polymer takes a middle region, which is the longitudinal axis that runs down the center of the internal fiber which has a cross-section of a winged-shape or amoeba-like shape. The longitudinal axis has a plurality of projections shown in Fig. 3b. The plurality of projections increases the surface areas and surface capillary portions along the length of the fiber that facilities the absorption of liquids within the fiber. Additionally, the channels allow particles, such as debris and dirt, to be picked-up and retained with the fiber. The surface area created by the internal fiber depends on the number of segments that are used during the manufacturing of the fiber. Figure 3b shows a core polymer with a backbone and 32 wings. The backbone exhibits a width of ~0.28 micron. The length and the width of wings were approximately 2.96 and 0.63 microns, respectively.

Table 1 Fiber properties of PLA/PBT fibers

Fiber morphology	Received fiber		Washed fiber	
	Minor	Major	Minor	Major
Cross-sectional fiber diameter (micron)				
Average	10.61	21.62	7.14	17.19
Standard deviation	1.01	2.15	0.66	2.38
CV (%)	9.52	9.94	9.30	13.84
Aspect ratio	0.49		0.42	

Table 2 Web properties of PLA/PP winged fibers with washing time

Washing time, t (min)	Weight reduction, ΔW (%)	Thickness (mm)	Bulk density (g/cm^3)
0	0	0.91	0.21
2	36.2	0.93	0.13
4	46.9	0.95	0.11
6	47.9	0.99	0.10
8	48.2	0.98	0.10

Table 1 summarizes the characteristics of winged fibers made up of PLA/PBT bicomponent fibers after removing PLA. The minor and the major of fibers washed were 7.14 and 17.19 micron, respectively. The fibers show uniform fiber diameter having a low coefficient of variance (CV). After removing PLA, it seems that the reduction of fiber diameter in the minor was larger than in the major. A round fiber would have an aspect ratio of 1; our winged fibers exhibited an oval cross section with an aspect ratio of 0.49 before removing the sheath and 0.42 after the sheath was removed.

The sheath polymer was removed at different washing time. Table 2 shows web properties of PLA/PP-winged fibers with a weight ratio of 50/50 after being washed. The weight of the web was reduced by 48.5% at 8 min of washing time. The web thickness also increased slightly due to the structure becoming looser after washing. Weight reduction was negligible beyond the initial 8 min indicating that the bulk of the PLA had been removed. The resulting fabric composed of only the core winged fiber became bulkier without the structure changing significantly. This is promising for aerosol filtration application in that we need to control solidity of the filter media for achieving a lower pressure drop without sacrificing the efficiency.

The most unique characteristics of area-enhanced winged fiber is its cross-sectional shape as shown in Fig. 4, which shows SEM images of various winged fibers consisting of 32 wings. The removal of sheath polymer has exposed the unique winged structure resulting in a structure with very high surface to volume ratio. The surface area in

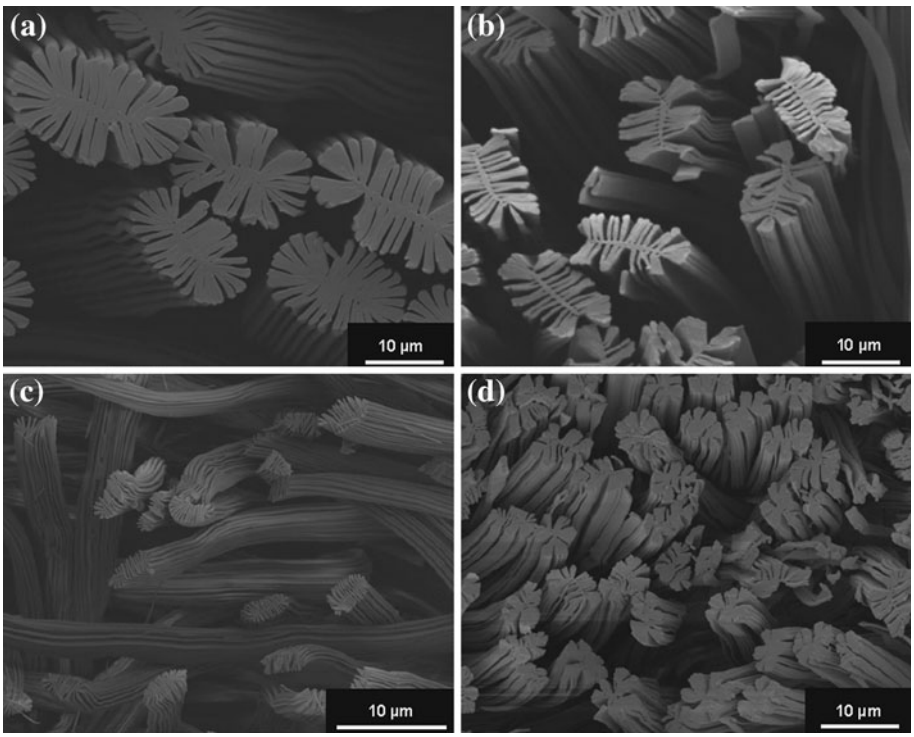

Fig. 4 SEM images of washed shaped, area-enhanced fibers: **a** PLA/PP, **b** PLA/PET, **c** PLA/PBT, and **d** EastONE^{TM}/PLA

conventional fabrics is typically very low, making them of little use in critical applications such as filtration and bio-separation which require significantly higher surface area.

Table 3 summarizes fiber morphologies of washed winged fiber consisting of different types of polymers with same sheath/core weight ratio of 50/50. The fiber diameter of washed fiber is affected by a polymer ratio and a polymer density of each polymer component. As expected, compared to PLA/PBT-winged fibers, PLA/PP fibers had a larger fiber diameter at a similar standard deviation due PP's lower density. PLA/PET fiber showed a similar fiber property to PLA/PBT. Meanwhile, the EastONE^{TM}/PLA fibers were closer to a round fiber, with an aspect ratio of 0.66.

Figure 5 shows the 4DG fiber and the winged fiber depicted from real fiber images. Each scale bar shows 15 micron, which exhibits well fiber dimension of each fiber. As mentioned previously, 4DG fibers are easily interlocking together due to its size and geometry shown in Fig. 5a. Meanwhile, new shaped fibers developed in this study can assemble closely together to form a web without interlocking when they are placed adjacent to each other. This is due to the fact that the fabric is formed when the wings are not exposed and the fibers are cylindrical in nature. After the structure is entangled, the sheath is removed to release the core. Consequently, there is little of no chance of the fibers interlocking. This is beneficial to developing a web having high surface area without losing the fiber properties in web forming processes.

Table 3 Fiber properties of washed winged fibers

Fiber morphology	PLA/PP		PLA/PET		EastONE^{TM}/PLA	
	Minor	Major	Minor	Major	Minor	Major
Cross-sectional fiber diameter (micron)						
Average	11.57	20.61	9.52	16.96	8.29	12.65
St. Dev.	1.11	3.30	1.08	2.01	1.21	1.44
CV (%)	9.63	16.03	11.32	11.88	14.59	11.40
Aspect ratio	0.56		0.56		0.66	

Figure 6 shows the potential of the new shaped fibers developed in this study for achieving high specific surface area. The specific surface area of shaped PET fibers was calculated from SEM images. Clearly, for a given fiber, the surface area is a function of its linear density (and therefore, the diameter) of the fiber. The round-shaped fiber such as melt-blown fiber and electrospun nanofiber shows above 10 m^2/g of specific surface area with submicron fiber. The specific surface area of 4DG fiber is typically up to 1 m^2/g even with 1 denier of fiber diameter that is a limit to produce this fiber. Meanwhile, winged fiber can reach 10 m^2/g of surface area with 1 denier of fiber diameter that is a typical diameter range of spunbond fiber as shown in Table 3.

For a round fiber, melt-blown and electrospinning processes can attain below 1 micron of fiber diameter. Typical fiber diameters of melt-blown fiber are in range of 1–10 micron. For achieving sub-micron fiber, polymer throughput in the process is considerably reduced compared to that for normal range of fiber. This is more severe

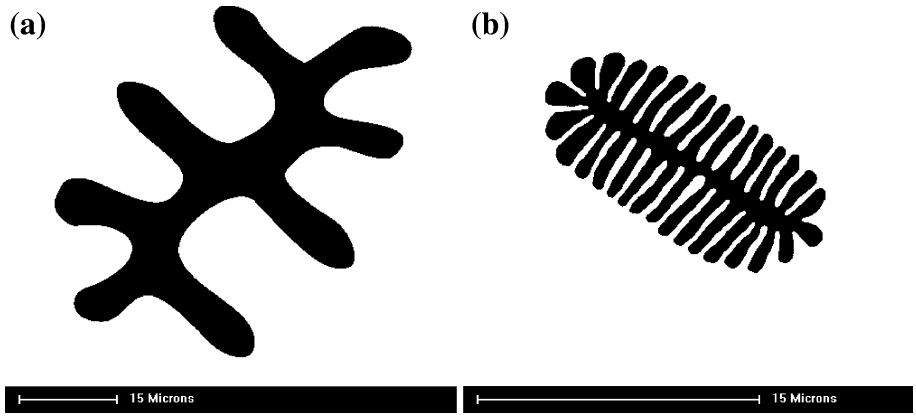

Fig. 5 Fiber shape and its scale: **a** 4DG fiber and **b** winged fiber

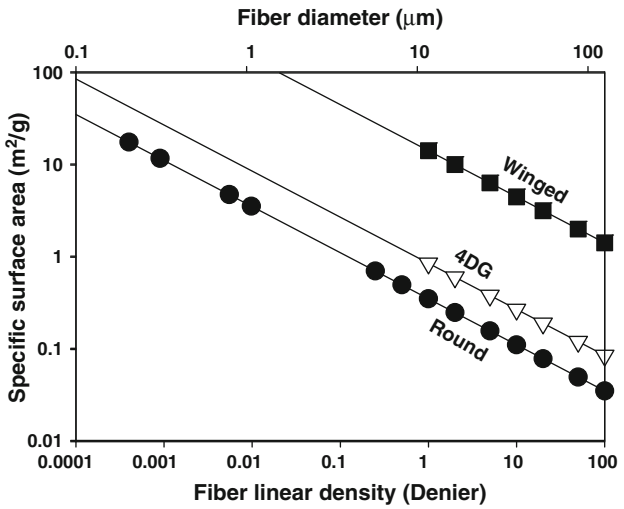

Fig. 6 Estimation of specific surface areas of different shaped PET fibers

for the electrospinning process in which for obtaining nanofibers with narrow fiber diameter distribution, electrospinning conditions such as applied voltage, polymer concentration, and electrode-to-collector distance need to be controlled carefully. The fiber diameter and productivity of electrospun nanofiber is determined by these conditions used in the process [18, 19]. And, as mentioned, 4DG fiber has a limitation to be spun below 6 denier of fiber diameter. The limitation in fiber diameter and the low productivity are significant drawbacks. The winged fiber was developed via a conventional spunbonding process with bicomponent spinning technology, which can be easily operated and controlled for the optimized fiber and web structures on a specific application at high throughput.

Conclusion

New shaped, area-enhanced fibers, and nonwovens were successfully developed with bicomponent spunbond technology consisting of a sacrificial sheath and a shaped core. The advantages of these newly developed fibers were high productivity, various polymer selections, and easy web formation. It is expected that these fibers can potentially achieve considerably higher specific surface areas compared to round fibers. These fibers have great potentials for such applications as filtration, tissue engineering, bioseparation, etc., due to their unique fiber shape and high surface area.

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