Research Article

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Design and characterization of flame resistant blended nondrip PA6/Lenzing FR[®]/PBI fiber nonwoven fabrics

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Abstract: In current thermal protective clothing systems, maximizing the personal protection performance against fire and heat is in great demand. Additionally, minimizing the manufacturing cost by using low-cost materials is also a critical factor. In previous studies [1–3], a new type of low-cost inherently flame resistant (FR) non-drip Polyamide 6 (PA6) nanocomposite fiber was developed. In this paper, this FR non-drip PA6 fiber was tested in blends with two commercially available inherently FR fibers to form FR nonwoven fabrics. Different formulations of varying blending ratios were processed into nonwoven fabrics. The fiber morphology was observed by Scanning Electron Microscopy (SEM). The fabric flammability and combustion properties were characterized using a Microscale Combustion Calorimeter (MCC), and a vertical flame tester, as well as Thermogravimetric Analysis (TGA). Tensile tests were conducted to characterize mechanical properties of these FR nonwoven fabrics. The water vapor permeability test was also performed to measure the wearability of the fabric. Results of several nonwoven blends were compared to find the one with optimum blend ratio which has the potential to be used as low-cost thermal protective fabric.

Keywords: High performance fibers; Blended fabric; Nonwoven fabric; Microscale Combustion Calorimeter; Flame resistance

1 Introduction

Fire is an ever-present danger in numerous professions. Apart from direct fire, exposure to heat sources, such as electric arcs, molten metals, and radiant heat, is also a significant danger in many industries [4]. Therefore, special protective clothing is required for fire and heat protection. In addition to flame resistance, the protective clothing should offer properties, such as lightweight, flexibility, and high moisture absorbency to provide comfort to the wearers.

Achieving effective thermal protection at low cost is a challenge. Indeed, inherently FR fibers with exceptional protective performance (*e.g.*, Nomex and PBI [5–8]) remain costly and prohibitive for large volume use, and in some cases lack the wear comfort if used as the only or majority component constituting the fabric.

Unlike traditional FR finishing for fabrics, the adoption of inherently flame and thermal resistant fibers would provide longer wear-life and durability. Polybenzimidazole (PBI, short for poly(2,2'-(*m*-phenylene)-5,5'-bisbenzimidazole)) is a synthetic fiber with high flame resistance and thermal stability. This kind of fiber has elongation of 27% and moisture regain of 15% (under the condition of 20°C and 65% RH), therefore, provides good overall comfort for the wearers [9]. Lenzing FR[®] is another inherently FR fiber made of natural cellulosic raw material. Its flame resistance properties were achieved by incorporating FR additives throughout the fiber mass [10]. Cellulosic fibers in protective clothing fabric can help reduce the danger of heat stress due to their excellent moisture management and breathability properties. Recently, a low-cost non-drip flame retardant polyamide 6 (FR PA6) fiber was developed by using a combination of non-halogenated flame retardants and nanoparticles [1–3, 11]. The non-drip

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FR PA6 fibers show significant reduction in heat release rate and offer the potential to achieve blends with higher nylon content than customary, thus resulting in significant cost reduction without compromising the FR performance of the fabric. Thus, the objective of this research is to develop and characterize a cost-effective FR nonwoven blend incorporating a newly developed non-drip FR nylon 6 material in conjunction with commercially available high-performance flame-retardant fibers to provide optimal performance.

Based on the above-mentioned considerations, the development of low cost FR nonwoven fabrics by blending PBI, Lenzing FR[®], and FR PA6 fiber is a novel and cost-saving technique for protective clothing. For the fiber blends, the blending ratios and the fabric structure design are the two main factors influencing final fabric properties. The nonwoven blending process was used as a screening tool in this study due to the fewer processing steps compared with traditional weaving and knitting, and to the availability of laboratory scale equipment. Herein, four different blending ratios were selected and compared. Microscale Combustion Calorimetry (MCC) results were used to investigate the blending ratio effect and the calculated and measured HRRs were compared in this study. Additionally, the flame resistance of the nonwoven fabric was tested.

2 Experimental

2.1 Materials

PBI staple fiber with the consistent size of 1.5 denier per filament was provided by PBI Performance Products, Inc., Charlotte, NC and Lenzing FR[®] fiber with the size of 1.5 denier per filament was supplied by Iovodex, Inc., Toronto, Canada. Non-drip FR PA6 fibers were produced using a twin-screw extruder. More details about the processing conditions of the FR PA6 fibers can be found in the previous publications [1–3, 11].

2.2 Materials and processing

Four nonwoven samples with different blending ratios are listed in Table 1. For the nonwoven fabric processing, the fibers were first fed into a laboratory carding machine for web formation and the obtained webs were combined on a laboratory needle punching machine for web bonding. Two blending methods were used to combine the three

Table 1: Blending ratios

Sample #	PBI (wt.%)	Lenzing FR [®] (wt.%)	FR PA6 (wt.%)
1	20	55	25
2	20	30	50
3	10	40	50
4	10	65	25
5	20	80	0

components: intimate blending, and multi-layer laminate structures.

It is anticipated that the multi-layer structure could offer more flexibility to optimize the performance of the fabric system. For instance, a cellulosic-rich layer close to the body would represent advantages in terms of wear comfort. A PBI-rich layer in the core would offer a high-performance FR barrier, while a PBI-free layer on the face of the fabric would allow potential print-ability given the inherent brown color of PBI fibers. Finally, as mentioned above, maximizing the FR nylon 6 nanocomposite fiber content in each of these layers will allow optimizing durability and cost. Thus, Lenzing FR[®] and non-drip FR nylon 6 fibers were blended intimately to constitute the outer layers of the laminate nonwoven fabric. PBI fibers were sandwiched in between the FR PA6/Lenzing blend layers (see Figure 1).

2.3 Characterization of the individual fiber components

2.3.1 Surface morphology of fibers

The fiber surface structures were observed by FEI Quanta 650 ESEM along the fiber axial direction. The samples were coated with gold to avoid charging in the microscope.

2.3.2 Flammability characterization

Thermal combustion properties were measured using a Microscale Combustion Calorimeter (MCC2, Govmark, Inc., Farmingdale, NY) according to ASTM D7309-2007. MCC directly measures heat release parameters of small amounts of materials in the size of milligrams. The heat release rate (HRR) during combustion and the total heat release are calculated using oxygen consumption rate and integration of the HRR versus time. The combustor temperature was held at 900°C. The heating rate of the pyrolysis zone was 1°C/sec. Each sample was tested in 3 repetitions to cal-

Figure 1: Representative photos of (a) the surface and (b) the cross section of the 20/55/25 PBI/Lenzing FR/FR PA6 nonwoven fabric

culate standard deviation value according to the recommended method [13, 14].

2.3.3 Char morphology characterization

The morphology of char residues forms all samples (individual fibers and nonwoven fabric) was examined by the FEI Quanta 650 ESEM. Char residues from MCC tests were transferred to the SEM sample holder and put into the sample chamber with Au/Pd coating.

2.4 Characterization of the nonwoven fabrics

2.4.1 TGA test

Thermogravimetric analyses (TGA) of all samples were performed using a TGA-50 from Shimadzu Scientific Instruments. The samples were heated in nitrogen environment from room temperature to 1000°C at a heating rate of 10°C/min. The nitrogen flow rate was 50 ml/min.

2.4.2 Vertical flame resistance test for the nonwoven fabrics

Flame resistance test for the nonwoven fabric was performed according to ASTM D6413. This test method is used to measure the vertical flame resistance of textiles.

2.4.3 Tensile properties

Tensile tests for the nonwoven fabrics were conducted according to ASTM D5035 standard 5.

2.5 Water vapor permeability test

Tests of water vapor permeability were conducted according to ASTM F1249.

3 Results and discussion

3.1 Physical properties of the nonwoven blends

Physical properties of nonwoven fabrics with 4 different blending ratios are listed in Table 2. The thickness and areal density were kept at a relatively constant value of 1.8-2.1mm and 4.2-5.0oz./sq. yd., respectively. Representative photos of the FR nonwoven fabrics are shown in Figure 1. All fabric blends show a clear sandwich structure: the core consists of pure PBI fibers whereas the surface layers are made of a mixture of LenzingFR® and FR PA6 fibers. Pictures were taken with DSLR camera.

3.2 Surface structure and morphology of the fibers

The fiber surface morphology along the fiber axis is shown in Figure 2. The cross section of PBI fiber is not circular which is due to the processing of fiber spinning [9]. The

Figure 2: The morphology of PBI fiber along the axial direction: SEM 5000× and (b) SEM 1000×

Table 2: Basic physical properties of the five types of nonwoven fabrics

Nonwoven Fabrics with Different Blending Ratios	Thickness (mm)	Areal Density (gsm)
20/55/25 PBI/Lenzing FR/FR PA6	2.07 ± 0.12	149.2
20/30/50 PBI/Lenzing FR/FR PA6	1.83 ± 0.09	143.8
10/40/50 PBI/Lenzing FR/FR PA6	1.87 ± 0.11	150.2
10/65/25 PBI/LenzingFR/FR PA6	2.11 ± 0.22	170.6
20/80 PBI/Lenzing FR	2.07 ± 0.18	151.6

fiber has a relatively smooth surface with the approximate diameter of 15 μm .

Similarly, Lenzing FR[®] is also an irregularly shaped fiber (Figure 3), with varying cross-sectional dimensions [10]. Unlike PBI fiber, some grooves along fiber axis can be seen in Figure 3. Using the scale bar, we can estimate that Lenzing FR[®] fiber has approximate diameter of 10 μm .

Unlike the PBI and Lenzing FR fibers, the FR PA6 have a rough surface with relative large fiber diameters around 30 μm (Figure 4). The protruding structures are in part due to the fact that so far, our inherently FR nylon has been processed in small-scale laboratory conditions [15].

Despite the protrusions, the fibers exhibited favorable flame-retardant and non-drip properties when compared to the commercial counterparts. These particles are non-halogenated FR additives and it is worth to notice that the FR PA6 fibers also yield deep grooves along the fiber axis. These grooves can be beneficial for sweat/water wicking purpose when such fibers are used for making protective clothing.

3.3 TGA results

The results of TGA and DTGA for the three pure fibers including PBI, Lenzing[®] FR, and FR PA6, and the four blended nonwoven fabrics are shown in Figure 5. It is clear that pure PBI fiber has the highest thermal stability with a char residue over 60%. It undergoes two decomposing stages, one slightly above 500°C and one between 700 and 800°C. Pure Lenzing FR[®] fiber have the lowest on-set degradation temperature of about 300°C. FR PA6 start to decompose at around 400°C and yield the lowest char residue of about 10%. All nonwoven blends exhibit two decomposition stages, one at lower temperature around 300°C attributed to the LenzingFR[®] component and one close to 500°C attributed to the FR PA6. Char residue of the blended nonwoven fabrics are slightly higher than the Lenzing FR[®] fiber.

Figure 3: The morphology of Lenzing FR[®] fiber along the axial direction: (a) SEM 5000× and (b) SEM 1000×

Figure 4: The morphology of FR PA 6 fiber along the axial direction: SEM 1000×

3.4 Flammability of individual fiber components

The comparison of HRR curves between, FR PA6, Lenzing FR[®], and PBI fibers is shown in Figure 6. Significant difference can be observed clearly between those three fibers. For Lenzing FR[®] fiber, the thermal decomposition started at approximately 220°C and peaked (400W/g) at 290°C. The decomposition process ended at about 400°C. On the other hand, PBI fiber started to decompose at ap-

proximately 550°C, and its HRR peaks (50W/g) at 787°C. The decomposition of PBI fiber ended at over 1000°C. The FR PA6 fiber starts decomposing at a higher temperature than Lenzing FR (350°C) and has the highest peak HRR among all three fibers at 531.4 W/g at the temperature of 478.5°C.

Heat release capacity (HRC) is an intrinsic material property independent of heating rate and sample size. Heat release capacities of the three types of fibers obtained from MCC are shown in Figure 7. In addition, Figure 7 shows HRC results for a control sample of neat nylon 6. The results summarized in Figure 5 show that the new inherent FR nylon fibers exhibit a substantial 37% decrease in heat release capacity compared to the control and compare more favorably to the Lenzing FR samples.

One major implication of the results above is that with the new non-drip FR nylon 6 fiber, it appears possible to achieve blends with higher nylon content than customary and not compromise the FR performance of the fabric. Indeed, experience shows that a typical neat nylon content in a FR fiber mix cannot exceed 10-12%, if one is to retain FR performance of the fiber. The new inherently FR nylon 6 fiber will allow significantly increased amount of the nylon ratio in the blend, thus resulting in sizeable cost savings and in significant gains in durability without compromising the FR protection.

Figure 5: The results of (a) TGA and (b) DrTGA of the pure FR fibers and their nonwoven fabrics

Figure 6: Comparison of average heat release rate curves between FR PA6, Lenzing FR®, and PBI fibers

Figure 7: Heat release capacity of non-drip FR nylon 6 fibers compared to control neat nylon 6 (PA6) and high-performance commercial inherently FR fibers

3.5 Nonwoven fabric flammability

Figure 8 shows photos of the five types of nonwoven fabrics after vertical flame resistance test (ASTM D6413). During the flame resistance test, all four types of nonwoven fabrics, showed non-dripping and self-extinguishing behavior. The after-flame time and afterglow time were both close to 0 second for all samples. The detailed char lengths of the five types of nonwoven fabric are listed in Table 3.

Table 3: The char lengths of the five types of nonwoven fabric

Nonwoven fabric with different blending ratios	Char length (mm)
20/55/25 PBI/Lenzing FR/FR PA6	4.05 ± 0.45
20/30/50 PBI/Lenzing FR/FR PA6	4.64 ± 0.12
10/40/50 PBI/Lenzing FR/FR PA6	3.47 ± 1.18
10/65/25 PBI/Lenzing FR/FR PA6	4.18 ± 0.13
20/80 PBI/Lenzing FR	3.8 ± 0.89

3.6 Char morphology

3.6.1 Fiber samples

Figure 9 shows the char morphology of PBI and Lenzing FR® fibers. Carbon-layer is formed on the fiber surface, which prevents the fibers from further burning. Both fibers

(a)

(b)

(c)

(d)

(e)

Figure 8: The photos after vertical flame resistance test of (a) 20/55/25, (b) 20/30/50, (c) 10/40/50, (d) 10/65/25 PBI/Lenzing FR/FR PA6 nonwoven fabrics, and (e) 20/80 PBI/Lenzing FR

Figure 9: Char morphology of (a) PBI (SEM 5000×) and (b) Lenzing FR fiber (SEM5000×)

Figure 10: Post MCC test specimen of non-drip FR nylon 6 nanocomposites (left) compared to the residual for neat nylon 6 (right)

keep their original shape very well indicating good thermal properties. Similar results have been reported in the work of other researchers, where the residues of Lenzing FR fibers were observed to retain in shape after combustion by SEM.

3.6.2 Fabric samples

After vertical flammability tests, char morphology of sample 10/40/50 and its control was compared under SEM. Figure 11 clearly shows that after burning the FR nylon maintains its fiber shape. A swelled hollow structure is formed due to the intumescent FR additive. Lower magnification image in Figure 11 confirms that the overall structural integrity is well preserved.

Figure 11: SEM char morphology of sample 10/40/50 with FR nylon fibers

Figure 12: SEM char morphology of sample 10/40/50 with neat nylon fibers

A control sample with the same composition but with neat non-FR nylon fibers was prepared and tested for flammability. After burning, the LenzingFR® and PBI fibers appear embedded into the melt nylon fibers as shown in Figure 12. Low magnification image indicates the neat nylon fibers melted into pools on the surface of the fabric. The nylon melt tends to stick to Lenzing FR® and PBI fibers causing a wick effect, thus making the flame spread more quickly.

From the comparison above, the difference in char morphology between the FR nylon and neat nylon is obvious. The blend with FR nylon have much better char integrity as compared to the blend with neat nylon fibers.

3.7 Tensile properties test

The typical load-extension curves for the nonwoven fabrics are shown in Figure 13. The comparison of stress nor-

Figure 13: The Load-extension curves in the tensile property test for 20/55/25 PBI/Lenzing FR/FR PA6 nonwoven fabric

Figure 14: The comparison of load for the four blending nonwoven fabrics and pure Lenzing FR fabric

malized by their thickness for the four types of nonwoven fabrics and pure Lenzing® FR fabric is shown in Figure 14.

The nonwoven fabric with blending ratio of 20/55/25 PBI/Lenzing FR/FR PA6 presented high extension properties with the highest value at the yield point of about 70%, indicating that these nonwoven fabrics can withstand large deformation, thus possibly leading to good wearability. The dispersion in stress values could be caused by the unevenness in the density of the lab-prepared nonwoven due its layered sandwich structure.

From Figure 14, pure Lenzing FR could withstand a load of 32N. The four types of blended nonwoven fabrics showed different performances. The load for the nonwoven fabric with blending ratio of 10/65/25 PBI/Lenzing FR/FR PA6 presented high extension properties with the highest value nearly 37N. The blended nonwoven fabrics

with blending ratio of 10/40/50 and 20/30/50 PBI/Lenzing FR/FR PA6 had similar load value around 27N. Among the four types of fabrics, the nonwoven fabric with blending ratio of 20/55/25 PBI/Lenzing FR/FR PA6 have lowest tensile strength of 18N.

3.8 Water vapor permeability test

Test results of water vapor permeability conducted according to ASTM F1249 are listed in Table 4. The water vapor permeability of 20/55/25 PBI/Lenzing FR/FR PA6 nonwoven is $3941.3 \pm 18.1 \text{ g/m}^2 \cdot 24\text{h}$. The water vapor permeability of 20/30/50 PBI/Lenzing FR/FR PA6 blended fabric is $4293.8 \pm 50.2 \text{ g/m}^2 \cdot 24\text{h}$, while the value for 10/65/25 PBI/Lenzing FR/FR PA6 is $4204.3 \pm 51.8 \text{ g/m}^2 \cdot 24\text{h}$. The value of 10/40/50 PBI/Lenzing FR/FR PA6 is the highest among the four blends, at $4393.4 \pm 115.2 \text{ g/m}^2 \cdot 24\text{h}$, indicating the higher water vapor permeability for fabrics with 50% FR PA6. The relatively large fiber diameter of the FR PA6 fibers may account for the high permeability value. The average denier of the lab-made FR PA6 fibers used in this study are close to 11 which is about seven times higher than the other two fibers [12].

Table 4: Water vapor permeability data for four types of nonwoven fabric

Nonwoven fabric types with different blending ratios	Water vapor permeability value ($\text{g/m}^2 \cdot 24\text{h}$)
20/55/25 PBI/Lenzing FR/FR PA6	3941.3 ± 18.1
20/30/50 PBI/Lenzing FR/FR PA6	4293.8 ± 50.2
10/40/50 PBI/Lenzing FR/FR PA6	4393.4 ± 115.2
10/65/25 PBI/Lenzing FR/FR PA6	4204.3 ± 51.8

4 Summary and Conclusions

PBI and Lenzing FR® fibers were selected as the materials for forming the flame resistant nonwoven fabrics. The fiber surfaces were observed by SEM showing different structures for the two types of fibers. Flammability of the two

fibers tested by MCC presented high flame and heat resistance of the fibers.

In this paper, four types of PBI/Lenzing FR[®]/FR PA6 flame-retardant nonwoven fabrics have been fabricated and characterized. The properties including the flame resistance, tensile properties, TGA, and water vapor permeability tests as well as the surface morphology of the fabrics before and after the flame test have been discussed.

All FR blends showed non-drip and self-extinguishing properties. Compared to blends with neat nylon fibers, the blend with FR PA6 fibers exhibited self-extinguishing property. The char morphology of the FR PA6 fibers shows individual fibers whereas the neat nylon blend forms melt pools. The 10/65/25 PBI/Lenzing FR/FR PA6 has the highest tensile load of 37N. All FR nonwovens start to decompose at around 300°C which is attributed to the Lenzing FR[®] fibers. The char yield of the nonwovens is between 30-40 wt.%. Among the four nonwoven blends, 10/40/50 PBI/Lenzing FR/FR PA6 has the highest water vapor permeability value. One major implication of the results obtained in this research is that with the new non-drip FR nylon 6 fiber, it was possible to achieve blends with higher nylon content than customary and not compromise the FR performance of the fabric. Indeed, experience shows that a typical neat nylon content in a FR fiber mix cannot exceed 10-12%, if one is to retain FR performance of the fiber. Our new FR nylon 6 fiber allow significantly increased amount of the nylon ratio in the blend, up to 50%, thus resulting in sizeable cost savings without compromising the FR protection.

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