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Preparation Technique and Property Evaluation of Flame-Retarding/Thermal-Insulating/Puncture-Resisting PU Foam Composites

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ABSTRACT: In this study, flame-retarding property of PU foam was realized by compounding with flame-retarding nonwovens in order to stop flame burning and spread. Followed with global warming, thermal-insulating materials are also used inside of building for energy conservation. This article presents preparation technique and property evaluation of PU composite board composed of composite nonwovens and PU foam by integrating foaming process. By optimization of composite nonwoven, effects of foam density and nonwoven composition on flame-retarding and thermal-insulating properties were discussed. Puncture resistance property of composite board was also investigated for resisting against sharp-object impacts. The resultant composite board would be applied as partition materials in future building decoration. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2014**, *131*, 40463.

KEYWORDS: foams; thermal properties; polyurethanes; flame retardance

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INTRODUCTION

Thermal transmission inevitably occurs from the higher to the lower due to temperature difference between two objects. In building decoration design, thermal-insulating materials are considered to be used for preventing thermal transfer. In this way, energy conservation would be achieved especially in summer and winter. Porous materials are commonly used to insulate thermal conduction by method of containing more static air and decreasing thermal convection.[1,2] These contain fiber assembles, foaming material, and so on. Among them, PU foam is honored as an ideal thermal-insulating building material saving 65% energy due to its closed pores.[3]

PU foam is one of the most important categories of polymeric materials, and widely used as thermal insulation materials in building decoration. But cost, beauty, and practicability are commonly taken consideration during design and decoration, and the safety of the material is often ignored. Over the past years, uses of inflammable materials become primary cause for starting a fire.[4,5] Therefore, decoration materials used in inner building should have flame-retarding property. PU foam is a noninflaming retardant material, and easily triggers a big fire leading to potential safety hazards. Therefore, many researchers were devoted to studies on how to improve flame-retardant of PU foam by incorporation of expandable graphite,[6–12] nanoclay,[13] and flame retardants such as phosphorus-containing compounds,[14–18] melamine and its compounds,[19–21] polyhedral oligomeric silsesquioxane,[22] and alumina trihydrate and triphenylphosphate additives.[23]

On the other hand, fiber polymers ensure themselves with fire retardant property adopting fire-retardant agent. By absorption, deposition, and chemical-bonding effects, fire-retardant agent is fixed on fibers, yarns, or fabrics.[24] In this way, there is plenty of time for fire extinction and personal escape through restraining flame spreading when starting a fire. Currently, flame-retardant treatment on textiles focuses on using flame-retardant fiber. Therefore, we have applied flame-retardant fibers on surface of thermal-insulating PU foam in order to improve flame-retardant property of resultant composite board in this study. Moreover, puncture resistance property of composite board was also discussed for resisting against probe impact. After

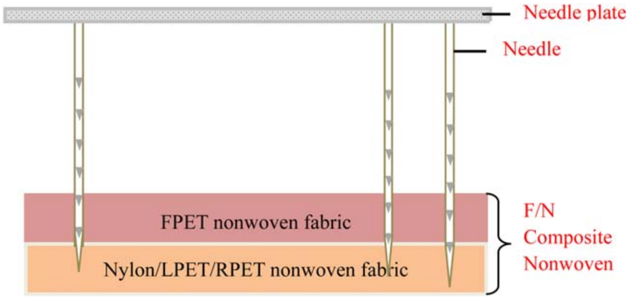

Figure 1. Manufacture diagram of F/N composite nonwoven. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

optimization of nonwoven process parameters, effects of foam density and nonwoven composition on flame-retarding, thermal insulating, and puncture resistance properties were investigated, respectively.

EXPERIMENTAL

Materials

Nylon 6 fibers (nylon) provided by Formosa Chemicals and Fibre Corporation, Taiwan, had 6 denier fineness, 51 mm length, 2.0 N/Tex tenacity, and 24.7% elongation. Low-melting PET fiber (LPET) provided by Far Eastern New Century Textile, Taiwan, possessed 4 denier fineness, 51 mm length, 0.69 N/Tex tenacity, and 120°C melting point. One thousand denier/192 filaments recycled high-strength PET selvages provided by Hsinnjy Nonwoven, Taiwan, were made into recycled high-strength PET fibers (RPET) after opening. Flame-retarding PET fibers (FPET) had 6 denier fineness, 64 mm length, 0.61 N/Tex tenacity, 32–35 limiting oxygen index (LOI) provided by Far Eastern New Century Textile, Taiwan. Faint-yellow polyol had volume density of 1.12–1.15 g/cm^3 and viscosity of 900 ± 100 cPs (25°C). Brownness methylene bisphenyl isocyanate (MDI) had density of 1.23–1.24 g/cm^3, viscosity of 200 ± 50 cPs (25°C). Polyol and MDI were used as two-agent PU foam agent, both provided by Chung-Hsing chemical, Taiwan.

Fabrication Process

The fabrication process was included by three steps, namely nonwoven fabrics preparation, composite nonwoven fabrication and PU composite board manufacture.

First, nylon fibers, LPET fibers, and RPET fibers were formed into nylon/LPET/RPET nonwoven fabric via processes including opening, blending, carding, lapping, and needle-punching. Weight fractions of nylon fibers were changed as 70, 60, 50, 40, and 30 wt %, and those of LPET fibers were 10, 20, 30, 40, and 50 wt % with constant 20 wt % RPET fibers. Effects of nylon fibers content and needle-punching speed on tensile, tear strengths were discussed for the optimal processing parameters. FPET staple fibers were fabricated into FPET nonwoven fabrics with changes of needle-punching speed. The optimal needle-punching speed was found based on tensile and tear strengths and LOI values.

Second, nylon/LPET/RPET nonwoven and FPET nonwoven, respectively, with 250 g/m^2 area weight were needle-punched into composite nonwoven as shown in Figure 1. Effect of hot-pressing on mechanical, inflaming-retarding, constant-rate puncture resistance properties was discussed by comparative study of composite nonwovens before and after hot-pressing (120°C). According to this, we determined composite nonwoven whether being hot-pressing or not.

Finally, composite nonwoven was compounded with PU foam, forming integrated nylon/PET/PU composite board. The process is displayed in Figure 2. After stirring for 10 s, foam agent with 1 : 1 polyol and MDI was injected into mold beforehand placed a layer of composite nonwoven. Then, another composite nonwoven was covered on the surface and sealed foaming and curing for 1 day at room temperature. Foam density was changed as 40, 50, 60, 70, and 80 kg/m^3 by controlling weights of foam agent. Composite nonwoven was divided into three different types. The first included FPET nonwoven on the surface and nylon/LPET/RPET nonwoven on the bottom, namely, F/N composite nonwoven. The second was comprised of two

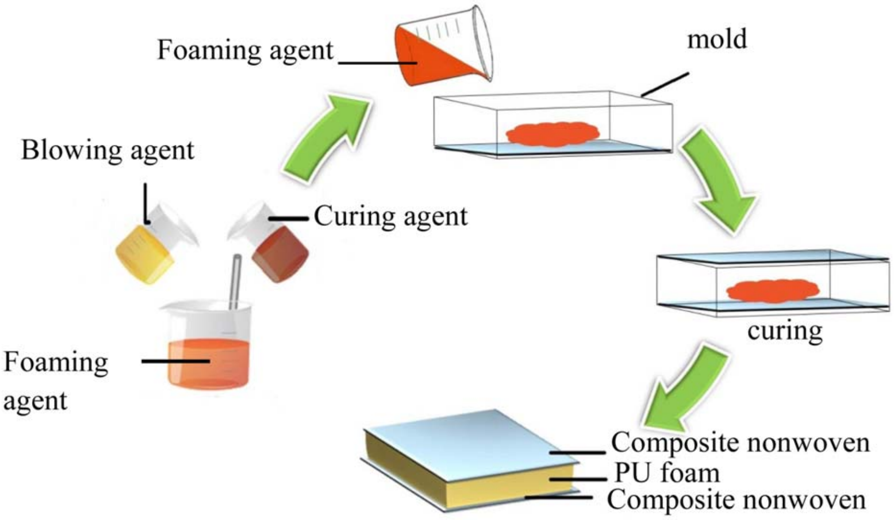

Figure 2. Preparation process of PU composite board. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

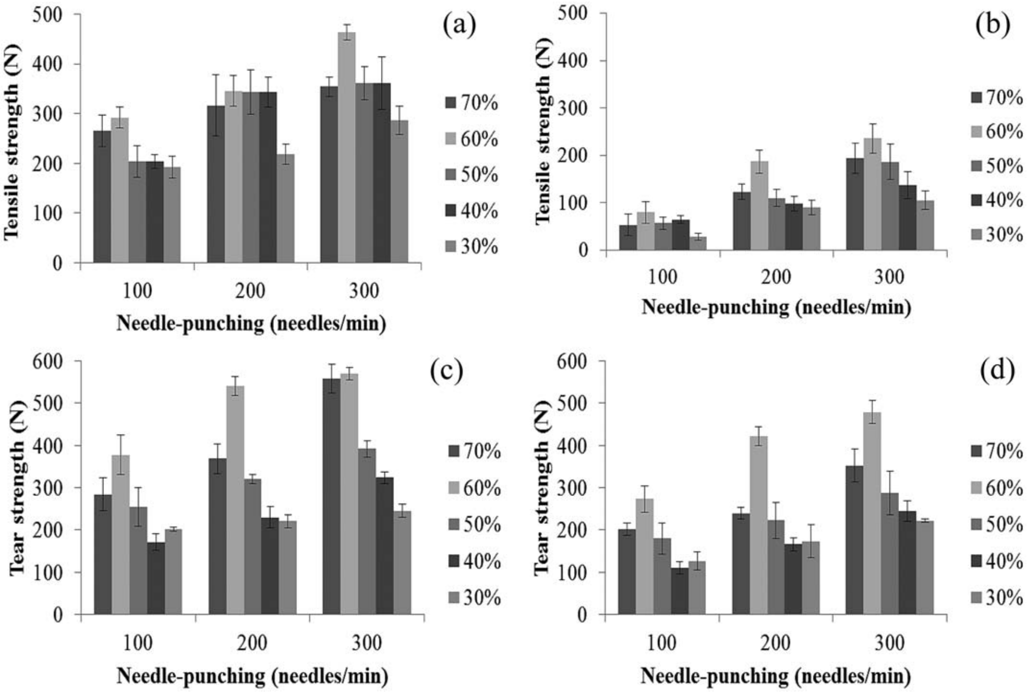

Figure 3. Tensile strengths along CD (a) and MD (b), as well as tear strengths along CD (c) and MD (d) of nylon/LPET/RPET nonwoven fabrics with different fractions of nylon fibers and needle-punching speeds.

layers of FPET nonwoven, named as F/F composite nonwoven. The third was made of double-layer nylon/LPET/RPET nonwovens, called as N/N composite nonwoven. Resultant PU foam composite was discussed in terms of burning duration, thermal conductivity, and constant-rate puncture resistance in order to found the best foam density and nonwoven composition.

Testing Methods

Mechanical Testing. Tensile strength and tear strength of nonwovens were tested according to ASTM D5035-11.[25] Testing speed was 305 ± 13 mm/min. Each sample was 180 × 25.4 mm^2, and gage distance of clamper was 76 mm. T-shape tear sample was 203.2 × 76.2 mm^2. The clamper distance for tear testing was 25.4 mm. Ten samples, respectively, along machine direction (MD) and cross direction (CD) were tested in each group for both tensile and tear strengths.

Flame-Retarding Testing. Flame-retarding property was expressed by LOI value and burning time. LOI value was tested by Limiting Oxygen Index Analyzer (Dynisco Plastics) according to ASTM D2863-08.[26] Testing sample was 150 × 10 × 10 mm^3. Three samples were repeatedly tested for each group. The minimum percent of oxygen and nitrogen that supports burning was LOI value. At constant nitrogen–oxygen ratio, burning time was recorded simultaneously.

Thermal-Insulating Testing. Thermal conductivity was measured by DRX-I-SPB Guarded-Hot-Plate Apparatus (Xiangtan Huafeng Equipment Manufacture, China) on the basis of ASTM C177-10.[27] The sample was 200 × 200 × 10 mm^3. Hot-plate temperature was 100°C. Sample was tested for three times and lasted for 6 h.

Constant-Rate Puncture Testing. Constant-rate puncture was measured by Instron 5566 Universal Tester (Instron, USA) according to ASTM F1342-05.[28] Two different probes (2 and 5 mm diameter, 0.25 mm shaft radius and 26° conical angle) were, respectively, fixed on the load cell and moved at 508 mm/min. Samples were in size of 100 × 100 mm^2, and placed between two circular plates with 10-mm-diameter hole in the center. Ten samples were measured repeatedly in each group.

RESULTS AND DISCUSSIONS

Nylon/LPET/RPET and FPET Nonwoven Fabrics Optimization

As shown in Figure 3(a,b), it is found that CD tensile strength is higher than MD. This is because web was lapped along CD after carding and thus more fibers were orientated in CD. As nylon fibers increase from 30 to 60 wt %, tensile strengths along CD and MD show an increase, which is attributed to higher tenacity of nylon fiber than LPET fibers. Besides, there is a small difference of fineness between nylon fibers and LPET fibers, presenting better entanglement among fibers in nonwovens. However, when nylon fibers contain 70 wt %, tensile strengths conversely decrease. This results from fact that fewer LPET fibers cannot provide entanglement with nylon fibers, thereby hindering from interfiber entanglement. In addition, a portion of RPET cannot open even through carding process due to its smooth surface and different width of recycled selvages. With these two effects, tensile strengths also have bigger standard deviation. While when nylon fibers add from 40 to 50 wt %, CD tensile strength almost maintains at the same needle-punching time. Even that fiber web strength increases when nylon fibers changes from 40 to 50%, discrepancy for fibers distribution between them is conspicuous. Therefore,

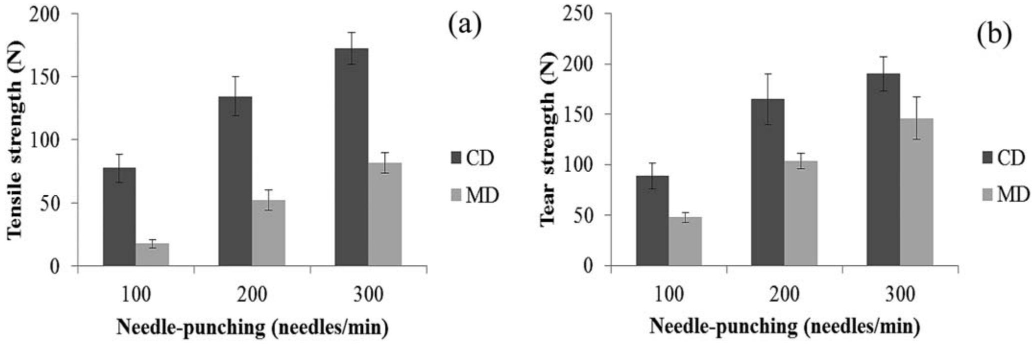

Figure 4. Tensile strength (a) and tear strength (b) along CD and MD of FPET nonwoven fabrics with different needle-punching speeds.

difference of maximum and minimum tensile strengths becomes larger with higher nylon fibers content. But these differences are also influenced by needle-punched times. Therefore, there is no improvement for tensile strength (CD) when increasing from 40 to 50 or to 60 wt % of nylon fibers for a speed of 200 needles/min or from 40 to 50 wt % of nylon for a needle speed of 300 needles/min.

Moreover, we also observe that tensile strengths along CD and MD rise with improvement of needle-punching speed in Figure 3(a,b). This can be explained that more fiber entanglements per unit area produced with increasing needle-punched speed, leading to stronger cohesive force among fibers. In addition, as displayed in Figure 3(c,d), tear strength in CD is higher than that in MD, similar to tensile strength. Moreover, tear strengths improve with needle-punching speed and nylon fibers (<60 wt %). But when needle-punched speed increases from 200 to 300 needles/min, tear strengths improve insignificantly. Therefore, based on tensile and tear properties, optimized nylon/LPET/RPET nonwoven is composed of 60 wt % nylon fibers, 20 wt % LPET, and 20 wt % RPET fibers, and needle-punched with 200 needles/min.

Tensile strengths and tear strengths of FPET nonwoven fabric are shown in Figure 4. Along with needle-punched speed, tensile and tear strengths both increase. This is explained that at multiple needle-punches, bottom fibers came back to surface, leading to stronger cohesion and entanglement as well as higher fiber volume density. Therefore, with higher needle-punching speed, nonwoven fabric becomes compact and, thus, tensile and tear strengths are improved. Alike former nonwoven fabric, FPET nonwoven has higher tensile and tear strengths in CD than those in MD.

FPET nonwoven was used to resist against firing in this study. Flame-retarding nonwoven generated melting drops after burning, thus forming covering layer to prevent from oxygen entrance and achieving flame-retarding property. Therefore, flame-retarding property of FPET with different needle-punching speed is shown in Table I. The flame-retarding property is expressed by LOI value. At lower needle-punches, FPET nonwoven has higher LOI of 33, showing better flame-retarding property. This is because fibers were subjected to fewer damages with lower needle-punching speed, and less flame-retarding agent on fibers would be lost.

By above evaluations, optimal FPET nonwoven fabric is needle-punched at 100 needles/min. Although 300 needles/min has the best mechanical properties, optimized parameter of FPET nonwoven is chosen from highest LOI because FPET nonwoven aims to use for flame-retarding layer in this study.

Composite Nonwoven Property Evaluation

Composite nonwoven was comprised of optimal nylon/LPET/RPET and FPET nonwoven fabrics. In this part, F/N composite nonwoven was hot-pressed at 120°C in order to discuss hot-pressing effect on mechanical property, burning time, and puncture resistance by comparison with before and after hot-pressing composites.

As shown in Figure 5(a,b), tensile strength and tear strength go down after hot pressing. A part of LPET fibers was taken among FPET nonwovens after needle-punching, and forming bonding spots after melting. But these decrease elongation of composite nonwoven, thereby leading to declining mechanical strength.

When FPET nonwoven was compounded with nylon/LPET/RPET nonwoven, nonflaming retarding fibers was brought into surface layer of FPET nonwoven, which decreased flame-retardant effect. During flame-retardant testing, combustion source was placed on surface of composite nonwoven. FPET fiber presented molten state due to improved temperature, and became melting drops. After dripping, nonflaming retarding nylon/LPET/RPET nonwoven which was naked on the other side started conflagration. Therefore, FPET nonwoven took effect on delayed combustion, and burning time was used to characterize flame-retardation property at constant ratio of nitrogen and oxygen. When oxygen contains 26%, composite nonwoven extends burning time to above 5 s at the same condition as displayed in Figure 5(c). After hot pressing, interspaces among composite nonwovens became smaller and more

Table I. Limited Oxygen Index (LOI) of FPET Nonwoven Fabrics

Needle-punching speed	100 Needles/min	200 Needles/min	300 Needles/min
CD	35-37	34-36	32-35
MD	36-38	32-35	30-33

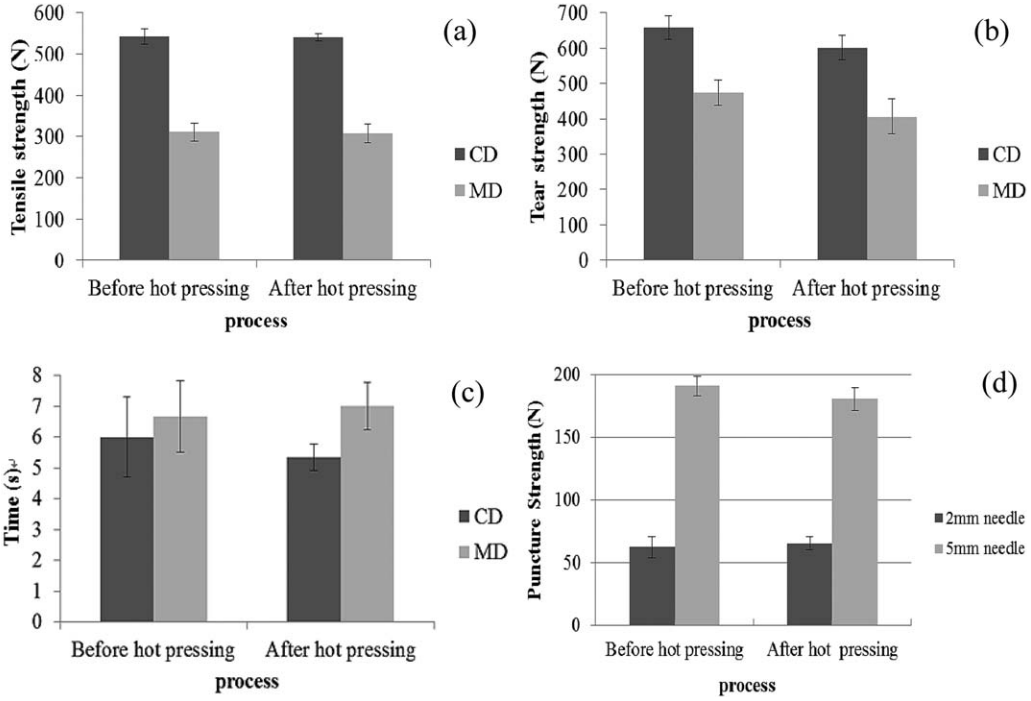

Figure 5. Property evaluations of F/N composite nonwoven including tensile strength (a), tear strength (b), burning time (c), and puncture resistance strength (d).

uniform, and a small amount of gas contacted with composite nonwoven. Therefore, burning time of hot-pressed nonwoven becomes slightly longer, and its deviation is smaller.

Figure 5(d) shows constant-rate puncture resistance strength of F/N composite nonwoven before and after hot pressing. 2-mm-diameter needle and 5-mm-diameter needle were both used for puncture testing. It is found that 2 mm-diameter needle produces lower puncture resistance strength. This is because nonwoven had porous structure, and finer needle penetrated more easily. After hot pressing, composite nonwoven resists against higher puncture strength. This can be explained that pore size became smaller after hot pressing, and pushing force of composite nonwoven to needle was larger. However, after hot pressing, composite nonwoven generates lower puncture resistance strength when subjected to 5-mm-diameter needle. This is due to the fact that porous structure nonwoven had pore size about 0.5 mm, which was small enough to resist against 5-mm-diameter probe penetration. By property evaluations, hot-pressing effect improves flame-retardant property. Therefore, all composite nonwovens were hot-pressed at 120°C in the following discussions.

PU Composite Board Property

Effect of Foam Density. Table II shows that with increase of foam density, burning time became longer up to about 6–12 s at oxygen content of 26%. This is due to characteristic of interface layer between surface composite nonwoven and PU foam. When foam density increased, interface layer became thicker and more compact. Thus, inner pores and air content among interlayer were smaller, which was hard to flame. But when flame spread to nonflame retardant PU foam layer, F/N PU composite was burned rapidly and triggered a big fire due to increasing air within cell. Therefore, at foam density of 80 kg/m^3, burning time of resultant F/N PU composite prolonged to 10 s, which eased threats from fire.

Table II also shows that nylon/PET/PU composite produced lowest thermal conductivity when foaming at 40 kg/m^3 density, indicating best thermal insulation property. PU foam contained closed cell, and had low density and less solid phase at this foam density. Interlayer between composite nonwoven and PU foam was thinner. As compared to 80 kg/m^3, cell opening is bigger than that with 40 kg/m^3. Inner air among cell opening was static, and had lowest thermal conductivity of 0.026 W/mK.

Table II. Flame-Retarding, Thermal-Insulating and Puncture Resistance Properties of F/N PU Composite Board with Different Foam Densities

Foam density (kg/m ^{3})		40	50	60	70	80
Burning time (s)		7.3 ± 1.3	8.3 ± 1.3	9.0 ± 0.7	9.5 ± 0.5	10 ± 1.1
Thermal conductivity (W/mK)		0.1397 ± 0.0068	0.1433 ± 0.0106	0.1563 ± 0.0163	0.1677 ± 0.0131	0.1861 ± 0.0189
Puncture strength (N)	2-mm needle	60.4 ± 8.91	90.5 ± 13.60	124 ± 17.70	92.6 ± 8.42	85.7 ± 8.89
	5-mm needle	237.8 ± 20.89	248.1 ± 17.11	259.97 ± 15.90	234.22 ± 10.58	210.99 ± 7.81

Table III. Flame-Retarding, Thermal-Insulating and Puncture Resistance Properties of PU Composite Board with Different Nonwoven Compositions

Composite nonwoven		N/N	F/N	F/F
Burning time (s)		4.3 ± 0.94	7.0 ± 0.70	9.5 ± 0.86
Thermal conductivity (W/mK)		0.1397 ± 0.0068	0.1433 ± 0.0106	0.1563 ± 0.0163
Puncture strength (N)	2-mm needle	124.3 ± 15.83	118.1 ± 11.68	52.29 ± 6.65
	5-mm needle	262.8 ± 18.86	259.1 ± 14.23	119.9 ± 14.37

Therefore, with lower foam density, thermal conductivity is lower, showing better thermal-insulating property. This also shows that thicker interface layer conducted conversely easily, and thus yielded higher thermal conductivity and worse thermal insulation.

With improvement of foam density (40–60 kg/m^3), puncture resistance strengths with 2 and 5 mm diameter both increase due to lower air-contained content and relatively proper foam cell. Moreover, foaming agent amount was close to saturation condition inside the mold, thereby producing compact interface layer between composite nonwoven and PU foam. Therefore, as foam density was higher, interface layer became thicker and whole composite got rigid. As found from Table II, puncture resistance strength tends down at foam density of 70 and 80 kg/m^3. This is because harder composite produced cracks easily when penetrated by needle tip. By above discussions, optimal foam density is 60 kg/m^3, and meanwhile resulting nylon/PET/PU composite has puncture resistance strength of 260 and 119 N, respectively, with penetration by 2-mm and 5-mm diameter needles.

Effect of Nonwoven Composition. In this part, composite nonwovens had three kinds, named as N/N, F/N, and F/F, respectively. These three nonwovens were compounded with PU foam, forming different PU composite boards. The foam density was constant as 60 kg/m^3. Flame-resisting, thermal-insulating and puncture resistance properties of PU composite boards with different nonwoven compositions are shown in Table III.

Comparatively, we have found that N/N PU composite board has shortest burning time at 26% oxygen because composite nonwoven has nonflame retarding effect. But when FPET nonwoven is contained in composite nonwoven, burning time extends to 7 s. Because FPET nonwoven slows down burning time, F/F PU composite possesses longest burning time up to 9 s. Visually, surface nonwoven composition affects flame-retardant properties of whole PU composite board. Using flame-retardant nonwoven is an effective measure to improve flame-retardation of PU composite board. Also, F/F PU composite board has lowest thermal conductivity of 0.0714 W/mK because thermal conductivity of FPET fiber was three times lower than that of nylon fiber. However, F/F composite nonwoven had larger opening, and thus PU agent easily permeated into composite nonwoven. These results in higher fiber content among interface layer. Therefore, when probe penetrated F/F PU composite board, more cracks generated resulting in lower puncture resistance strength. N/N and F/N composite nonwoven had compact structure and thus PU agent was difficult of permeation. Consequently, interlayer was harder and results in higher constant-rate puncture resistance property. Comparatively, N/N PU composite board provides best constant-rate puncture resistance strength of 124.3 N (2-mm needle) and 262.8 N (5-mm needle).

CONCLUSIONS

This study had successfully prepared flame-retarding/thermal-insulating/puncture-resisting composite board by combinations of composite nonwovens and PU foam. FPET nonwoven with LOI of 37 which was punched at 100 needles/min was used as surface layer to retarding fire. 60/20/20 wt %/wt %/wt % nylon/LPET/RPET nonwoven via 200 needles/min aimed to resist against better puncture damage. Compounding with two-layer nonwovens, hot pressing was conductive to improve flame-retarding and puncture resistance property (2-mm needle), and improved tensile and tear strengths (in CD), respectively, by 0.37 and 9.5%. After foaming with PU agent, the optimal foam density of resultant PU composite board was 60 kg/m^3 whose puncture resistance was improved by 86% (2-mm needle) and 45% (5-mm needle) as compared to that with 40 and 80 kg/m^3. Moreover, with foam density, their flame-retarding became better but thermal insulation turned into worse. For composite board with 60 kg/m^3 foam density, burning time was 9 s and thermal conductivity was 0.1564 W/mK.

Diversified applications aspects could be chosen based on property evaluations. For environmental protection, F/N PU foam composite board was an alternative. In thermal-insulating and flame-retardation application, F/F PU composite board became a better candidate. N/N PU foam composite board had superiority of durability and strength. Nonwoven compositions were determined on basis of using requirements in building. Different kinds of nylon/PET/PU composite board had an integrated structure, which gives convenience for construction in building partition.

Actually, burning time of our resultant PU foam composite board needs to be prolonged for improving combustion time. In the following study, flame-retardant fibers would be incorporated in PU foam to slow down spreading of flame further.

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