Developing Protective Textile Materials as Barriers to Liquid Penetration Using Melt-Electrospinning

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ABSTRACT: Electrospun polypropylene fiber webs and laminates were developed using melt-electrospinning, to explore an alternative way of manufacturing protective clothing materials for agricultural workers. Electrospun polypropylene webs were fabricated in two levels of thickness. To examine the effect of lamination on the protection/thermal comfort properties, the webs were laminated on nonwoven fabric substrates. Barrier performance was evaluated for the electrospun webs and laminates, using two pesticide mixtures that represent a range of surface tension and viscosity. Effects of web thickness and lamination on air permeability and water vapor transmission were assessed as indications of thermal comfort performance. Penetration testing shows that electrospun polypropylene webs provide excellent barrier performance against the high surface tension challenge liquid, whereas the laminated fabrics of electrospun polypropylene webs exhibited performance of 90–100% for challenge liquids with varying surface tension. Air permeability of electrospun polypropylene webs decreased by ~20% because of the lamination and web thickness, but was still higher than most of the materials currently in use for protective clothing. Water vapor transmission of electrospun polypropylene webs reduced by up to 12% from the lamination and web thickness as well, but was still in a range comparable to woven work clothing fabrics. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 102: 3430–3437, 2006

Key words: barrier; fibers; membranes; polypropylene (PP); nanotechnology

INTRODUCTION

Various protective clothing materials are used to reduce the dermal exposure of workers to pesticides, including nonwoven, woven, microporous, and monolithic materials. In a previous work,[1] protection and air/moisture vapor transport properties were examined on a broad selection of 36 available protective materials, including nonwovens, wovens, microporous membranes, and laminated fabrics. Figure 1 illustrates the chemical protection performance of those materials against a series of pesticide chemicals, relative to air permeability of materials. In general, a negative relationship exists between protection performance and air permeability. Nonwovens with high air permeability exhibit low barrier performance, whereas microporous materials and tightly constructed wovens offer higher level of protection but lower air permeability.

Obviously, the most important feature in protective clothing material is the effectiveness of material as a barrier to the chemicals of concern. Yet, protective clothing made of impermeable materials may in fact be a hazard in itself because of hyperthermia under conditions of high temperature and low evaporation rate.[2] Thus, breathability of material is another important factor to be considered in terms of wearer comfort for working in hot, humid conditions. To achieve an effective protective clothing system for such environments, protective clothing materials that can provide a combination of high barrier performance and thermal comfort is essential. Figure 1 shows the actual range of protection performance relative to air permeability of materials currently in use for protective clothing, indicating that there is a large "window of opportunity" for development of materials with both high barrier and comfort performance.

Electrospinning is an effective and promising technique for the production of fibers, with diameters in the submicron to micrometer range. This technique is of great interest, because not only can it produce polymer fibers with small diameters, but it also has the advantages of being simple and convenient compared with traditional fiber forming methods.[3] The basic mechanism of electrospinning involves applying an

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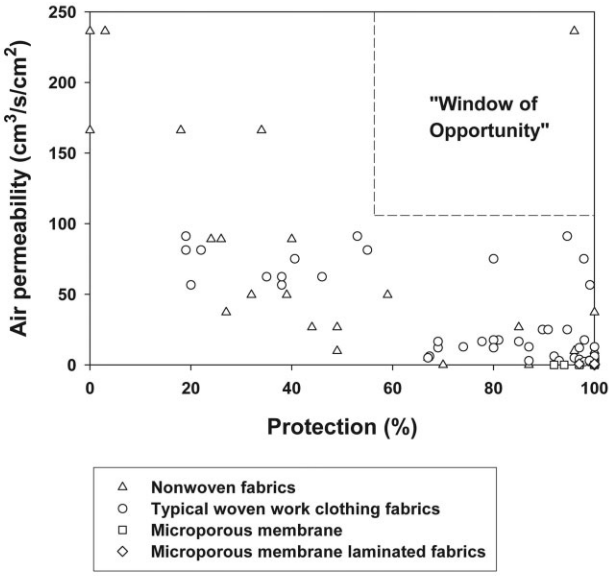

Figure 1 Relationship between protection performance and air permeability for nonwovens, wovens, microporous membranes, and laminates.

electric force between a suspended droplet solution or melt at a capillary tip and collector. When the intensity of the electric field overcomes the surface tension of the polymer solution or melt, a charged jet is ejected and travels to the grounded target, generating fibers typically in the form of a nonwoven mat.

Potential of electrospun mats for specialty textiles has been addressed in earlier studies,[4,5] including for use in filtration, membrane and protective clothing applications. Schreuder-Gibson et al.[6] has shown an enhancement of aerosol protection via a fine layer of electrospun fibers, focusing on barrier materials to the penetration of chemical warfare agents in aerosol form. They found that the electrospun webs of nylon 6,6, polybenzimidazole, polyacrylonitrile, and polyurethane provided good aerosol particle protection, without a significant change in moisture vapor transport of the system. One major advantage of electrospun webs for protective clothing use could be the direct application of electrospun webs to garment systems.[7] It may be sprayed directly onto three-dimensional forms, so that the thickness of the electrospun coating could be varied at various locations on a garment as needed. This could be useful in producing "zoned " materials in protective garments. Direct application of electrospun webs to garment systems would eliminate costly manufacturing steps, and solve seam-sealing problems that have been limiting factors in protective garments.[6,7]

In recent years, more than 40 different types of polymer fibers have been generated by solvent-based electrospinning.[8] Yet, relatively few studies have been reported on electrospinning from polymer melts.[9–12] This might be due to the difficulties in processing, such as a controlled thermal environment and high viscosity associated with polymer melts. However, melt-electrospinning has distinct advantages over solvent-based electrospinning, in that it does not require removal or recycling of organic solvents; thus, it is environmentally friendly as well as cost effective. Also, melt-electrospinning would be appropriate for polymers that do not have a proper solvent at room temperature, such as polyethylene, polypropylene, and polyester.[12]

For electrospun webs to work as an effective barrier against liquid penetration in personal protective equipment (PPE), surface chemistry is a critical factor to consider in the selection of polymer material for such applications, since the wetting mechanism depends on the surface energy of the material. Membrane hydrophobicity with its low surface energy plays a part in repellency of the challenge liquid. As shown in a previous work,[13] polypropylene is one of the widely used nonwovens for protective clothing on the market, because it has relatively low surface energy, and it is chemically inert, light-weight, and inexpensive. In this study, electrospun polypropylene webs are developed using melt-electrospinning to explore an alternative way of manufacturing protective textile materials as barriers to liquid penetration.

Polymeric membranes used as barriers to challenge liquids are laminated or adhered to a textile substrate, typically a nonwoven fabric, to provide strength and durability to the system.[14] The membrane is presumed to serve as the initial barrier to liquid penetration in the composite materials, but substrates supporting membranes could also influence the barrier/comfort performance of the material. It has been reported that generally electrospun webs have poor mechanical properties,[3] indicating that electrospun webs might also need to be used as a component in a laminated system for use in clothing. Thus, electrospun polypropylene webs are laminated onto nonwoven substrates, and the barrier performance and air/moisture vapor transport properties are evaluated on both electrospun polypropylene webs and laminated systems.

The aim of this study is to examine the feasibility of developing protective textile materials that could provide both high protection performance and thermal comfort, using melt-electrospinning. Barrier performance of electrospun polypropylene webs and laminates is evaluated against challenge liquids of different physical properties. Effects of lamination and web thickness on the air/moisture vapor transport properties are examined.

TABLE I
Pesticide Amounts, Surface Tension, and Viscosity of Pesticide Mixtures

Pesticide	Sample code	Pesticide amounts used in mixtures: Water (g)	Atrazine 90 WDG or Prowl 3.3 EC (g)	Oil (g)	Surface tension (dynes/cm)	Viscosity (mPa s)
Atrazine 90 WDG	P1	246.10	2.50	–	38.00	0.93
Prowl® 3.3 EC	P2	55.00	40.00	65.00	20.57	20.80

EXPERIMENTAL

Materials

Fiber-grade polypropylene pellets (P4C6Z-049) of melt flow index 35 were obtained from Huntsman Polymers Corp. (The Woodlands, TX). Molecular weight (M_w) was about 195,100 and polydispersity was of 4.3.

For pesticide penetration testing, atrazine (2-chloro-4-ethylamino-6-isopropylamino-1,3,5-triazine) and pendimethalin (*N*-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine) were used for formulating pesticide mixtures. From a practical standpoint, commercially available pesticide formulations were used following the previous studies[13,15]: atrazine as wettable dispersible granules and pendimethalin as an emulsifiable concentrate. They were selected based on differences in chemical solubility. Atrazine 90WDG, from United Agri Products/Platte Chemical Company (Greeley, CO) contains 85.5% active ingredient. Prowl ® 3.3 EC, which consists of 37.4% active ingredients of *N*-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine, comes from American Cyanamid Company (Parsippany, NJ). Based on the previous studies, two pesticide mixtures, representing a range of viscosity and surface tension, were selected. Oil concentrate was added to the mixture to vary the surface tension and viscosity. Oil concentrate was All Seasons ® spray oil concentrate, which consists of 98.8% petroleum oil, manufactured by Bonide Products (Yorkville, NY). Pesticide concentrations, surface tension, and viscosity of selected mixtures are shown in Table I.

Electrospinning process

A schematic design of a melt-electrospinning setup is illustrated in Figure 2. It consists of a syringe with polymer melt, a precisely controlled syringe pump (PHD2000, Harvard Apparatus, Holliston, MA), a high voltage power supply capable of 0–30 kV (ES30P, Gamma High Voltage Research, Ormond Beach, FL), a grounded collector, and heating units. A shielded heating unit with a temperature controller and a guiding chamber with heating device, in which the needle temperature can be adjusted separately, were used to control the thermal environment in the process. The design results in four different temperature zones: syringe temperature (T_1), needle temperature (T_2), temperature in the guiding chamber (T_3), and collector temperature (T_4). The system was designed to control the temperatures separately.[12]

Polypropylene pellets were loaded into a syringe, and kept in the shielded heating unit for 30 min after T_1 reaches 230°C. T_1 was set at 230°C; T_2 between 280 and 290°C; T_3 between 100 and 140°C throughout the experiments. The collector temperature (T_4) was typically between 85 and 95°C. A high voltage of 10–20 kV was applied to a 20-gauged needle on a syringe through copper wiring. The syringe pump was used to

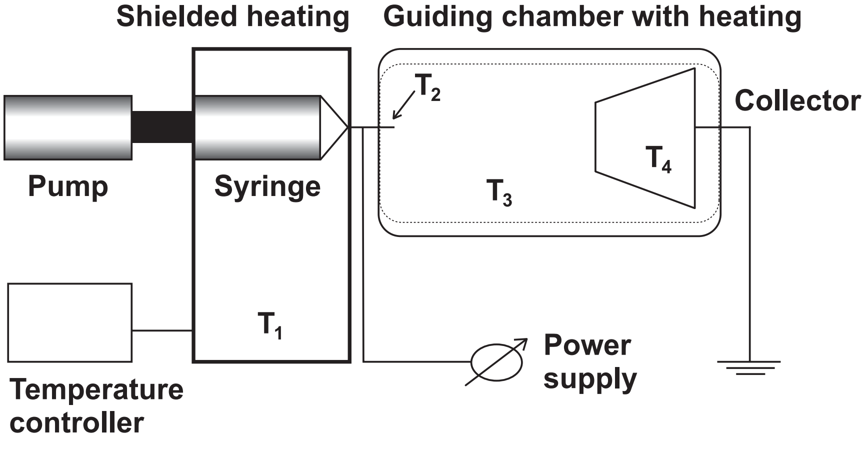

Figure 2 Schematic diagram of a melt-electrospinning setup.

control a constant volumetric feed rate, which ranged from 0.002 to 0.008 mL/min. As the applied voltage increases, a droplet at the needle tip deforms into a conical shape and, at sufficiently high voltage, an electrically charged jet is ejected from the tip. Fibers were laid down on the grounded copper collection plate, which was placed 5–7 cm from the tip, to form a nonwoven web.

Lamination of electrospun web onto fabric substrate

Electrospun polypropylene webs were laminated on a nonwoven substrate that has an adhesive on one side (fusible interfacing, No. 4400, 100% polyester, HTC, Wyckoff, NJ). The thickness of nonwoven substrate was 0.17 mm and the weight was 26 g/m^2. Electrospun webs were laminated onto the adhesive side of nonwoven substrate at 90°C for 15 s at a pressure of 15 gf/cm^2.

Fiber morphology

Morphology of electrospun polypropylene fibers was examined using a scanning electron microscope (Leica 440 scanning electron microscope, Cambridge, UK) after sputter-coating with Au/Pd, to minimize charging.

Protection performance

Pesticide repellency, retention, and penetration were measured according to ASTM F 2130−01, standard test method for measuring repellency, retention, and penetration of liquid pesticide formulation through protective clothing materials, using 0.1 mL of contamination load. For collector layers, absorbent paper, backed with polyethylene film (Whatman ® Benchkote ™ Plus with polyethylene backing, Whatman 3 mm cr, Whatman plc, Whatman House, Kent, UK) was used. HPLC-grade acetone (AlliedSignal, Burdick and Jackson, Muskegon, MI) was used for extraction. Tests were performed in triplicate for each combination of pesticide mixtures and electrospun web/laminates.

Hewlett–Packard model 5890 gas chromatograph (Hewlett–Packard Company, Wilmington, DE) equipped with a nitrogen–phosphorus detector and automatic injector was used for pesticide analysis. Separation was achieved on a 30 m × 0.25 mm i.d. capillary column (5% phenyl substituted methyl-poly-siloxane, HP-5, Hewlett–Packard Company, Wilmington, DE), with a nitrogen flow of 1.7 mL/min. Column temperature was maintained at 50°C for 1 min, then programmed at 25°C/min to 260°C and held 1 min. Injector port and detector temperatures were 250°C.

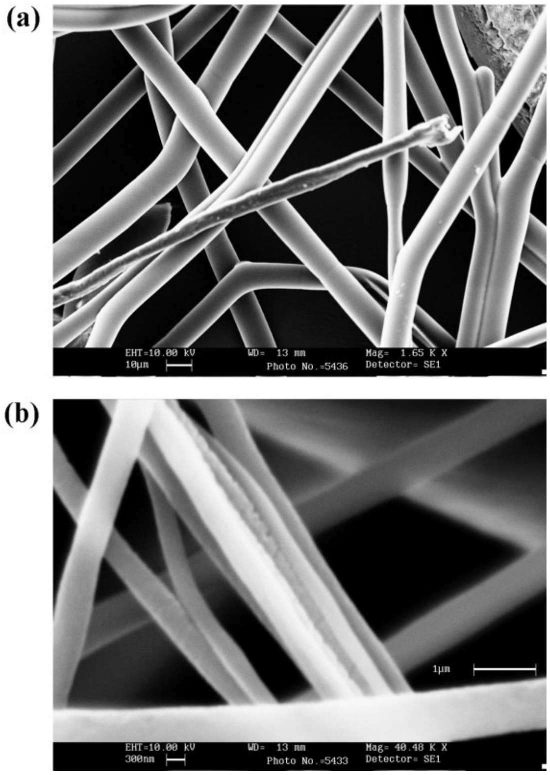

Figure 3 Surface view of electrospun polypropylene fibers (a) at 1.65 × 10^3 magnification and (b) 40.48 × 10^3 magnification.

Air and moisture vapor transport properties

Air permeability

Air permeability of electrospun web/laminates was measured according to ASTM D 737–96, standard test method for air permeability of textile fabrics, using a Frazier air permeability tester with reduced area (D = 3 cm) for four samples.

Water vapor transmission

Water vapor transmission rate was measured according to ASTM E 96−00, standard test method for water vapor transmission of materials, using a dish assembly (Vapometer, Thwing-Albert Instrument Company, Philadelphia, PA) with reduced area (D = 3 cm) for three samples.

RESULTS AND DISCUSSION

Fiber morphology

Surface morphology of the electrospun polypropylene fibers obtained at the feed rate of 0.002 mL/min, the voltage of 15 kV, and the collecting distance of 5 cm is shown in Figure 3. Smooth cylindrical fibers with diameters in the micrometer range were collected from melt-electrospinning [Fig. 3(a)]. Compared with

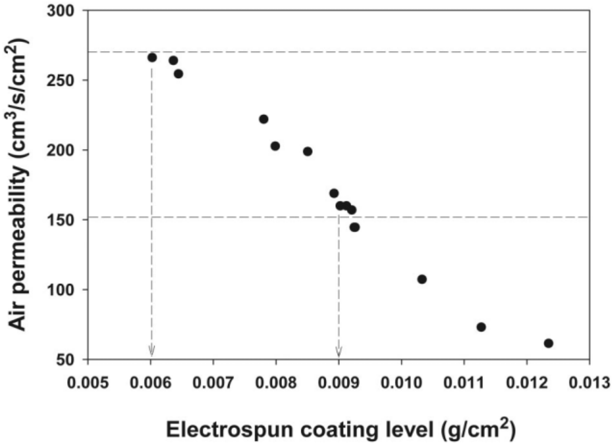

Figure 4 Air permeability as a function of electrospun coating level of electrospun polypropylene webs.

solvent-based electrospinning, in which further decrease in fiber diameter is achieved by solvent evaporation, thicker fibers from pure melt-electrospinning are expected, since no solvent evaporation is involved in the process. While the majority of fibers were in micrometer range, some fibers close to nanometer range were also observed, as shown in Figure 3(b). This is somewhat consistent with a previous study,[11] in which the effect of molecular weight of polymers on fiber diameters was shown in melt-electrospinning. They found that the higher the molecular weight of the polymers used, the larger the diameter of the fibers formed, but even with the lowest molecular weight polypropylene polymer they used (M_w = 12,000), a majority of fibers obtained were above 1 μm, with some nanometer fibers observed as branches from larger fibers. In traditional spunbonded nonwovens, fiber diameters range typically from 15 to 40 μm.[16]

A general tendency is that fiber diameter increases with increasing feed rate, but there was no notable change in fiber diameter from the applied voltage for the range we used in the study.

Selection of web thickness

In electrospinning, electrospun coating level, i.e., web thickness, can be controlled by direct spray time. A previous study on transport properties of electrospun fiber mats demonstrated that air flow resistance of electrospun webs correlates well with the electrospun coating level.[7] To determine a range of web thickness that could provide acceptable thermal comfort properties, electrospun polypropylene webs were produced in a range of density, and the relationship between the web thickness and air permeability was examined (Fig. 4). The scatter plot of electrospun coating level against air permeability of electrospun polypropylene webs shows that air permeability decreases with increasing web thickness.

The overall plot of air permeability versus protection performance of currently available protective fabrics (Fig. 1) was also used as a guideline to find a target zone, which is not covered by currently available materials and that can be used for the development of a new material for better protection and comfort. The plot reveals that a large "window of opportunity" exists in the air permeability range between 100 and 250 cm^3/s/cm^2. From the previous experimental data, we found one data point from a nonwoven fabric in the so-called "ideal zone," showing high air permeability as well as high protection performance of over 90% against a challenge liquid of high surface tension, but it failed to show such high barrier performance for other challenge liquids.[13] Thus, a target zone was carefully chosen in the air permeability range between 100 and 250 cm^3/s/cm^2, with an aim of developing a material that fits into the air permeability range, and could also provide high protection for a range of challenge liquids with less deviation.

Based on the scatter plot between the air permeability and web thickness of electrospun polypropylene webs established before (Fig. 4), web thickness, to be produced and examined hereafter, was chosen corresponding to the air permeability range selected. Two levels of web thickness were selected to examine the effect of web thickness on protection and air/moisture vapor transport properties: 0.006 g/cm^2, which corresponds to about 270 cm^3/s/cm^2 in air permeability, and 0.009 g/cm^2, which corresponds to around 150 cm^3/s/cm^2 in air permeability.

Protection performance of electrospun polypropylene web/laminates

Effects of web thickness and lamination on protection performance were evaluated against challenge liquids, representing a range of surface tension and viscosity. Percentage repellency, pesticide retention, and penetration against pesticide mixtures P1 and P2 are presented in Table II. A distinct difference was noted in penetration behavior between the two challenge liquids. For the challenge liquid P1, the mixture of high surface tension, no penetration was observed, regardless of thickness or lamination. On the other hand, a range of pesticide penetration was observed for the challenge liquid P2, which confirms the significance of liquid parameters in determining barrier performance of protective material. It also shows that more repellency was observed for the mixture P1 than mixture P2, across the materials.

For the challenge liquid P2, which has low surface tension and high viscosity, lamination significantly lowered pesticide penetration through the material, as illustrated in Table II, indicating that the barrier per-

TABLE II
Percentage of Pesticide Repellency, Retention, and Penetration of Electrospun Polypropylene Web/Laminates Against Pesticide Mixtures P1 and P2

	Mixture P1			Mixture P2		
	Repellency (%)	Retention (%)	Penetration (%)	Repellency (%)	Retention (%)	Penetration (%)
Web*thin	42	58	0	6	21	73
Web*thick	78	22	0	3	26	71
Lam*thin	59	41	0	17	75	8
Lam*thick	65	35	0	14	83	3

formance could be improved considerably by lamination. This might be because chemicals are more retained within the material rather than penetrate through when material is laminated, as shown in Table II. The substrate used for the lamination and adhesive on the substrate could be contributing factors for the improved barrier performance. To gain more insight on the contributing factors, we laminated a conventional polypropylene nonwoven fabric, which has similar weight and thickness as the electrospun polypropylene web, with the same substrate material, and conducted a penetration testing. The barrier performance of the conventional nonwoven increased after the lamination, but not as significantly as the electrospun polypropylene web, indicating that the fibers close to nanometer range in the electrospun web might also play a role and contribute to the improved barrier performance, although they make up a small portion.

On the other hand, there was no such significant effect from web thickness on barrier performance. This might be because the web thickness range selected is not wide enough to show the contribution to protection performance.

Air and moisture vapor transport properties of electrospun polypropylene web/laminates

To examine how web thickness and lamination affect thermal comfort properties of material, air permeability and water vapor transmission rate were assessed for electrospun polypropylene webs and laminates as indications of thermal comfort performance. Table III shows the effects of web thickness and lamination on air permeability of electrospun polypropylene web/laminates. A statistical difference was observed in air permeability because of the increased web thickness and lamination. As shown in Table III, the increased thickness of electrospun webs lowered the air permeability by around 20%. Lamination also reduced the air permeability of electrospun polypropylene webs by ~20%. However, the reduced values of air permeability were still above 100 $cm^3/s/cm^2$, which is higher than most of the protective clothing materials currently in use (Fig. 1).

Water vapor transmission of the material was also reduced by increased web thickness and lamination to a certain degree (Table IV). A statistical difference was observed in water vapor transport because of the increased web thickness and lamination. As presented in Table IV, moisture vapor transport was reduced by the increased web thickness by 2–9%. Lamination lowered water vapor transport of material by 6–12%. The reduced values of water vapor transmission were comparable to those of typical woven work clothing fabrics from our previous study,[1] and we could say they are still in an acceptable range.

Air permeability versus protection performance of electrospun polypropylene web/laminates

To examine how electrospun polypropylene webs and laminates perform compared to existing materials for PPE and find out if the goal of developing a material providing high protection as well as an acceptable level of comfort has been met, air permeability and protection property of electrospun polypropylene web/laminates were plotted on the overall plot of PPE

TABLE III
Effects of Web Thickness and Lamination on Air Permeability of Electrospun Polypropylene Web/Laminates

	Air permeability ($cm^3/s/cm^2$)		% Average decrease in air permeability due to lamination
	Web	Laminate	
Thin	203 (3)[a]	158 (10)	21.9
Thick	159 (2)	129 (6)	18.8
% Average decrease in air permeability due to increased thickness	21.7	18.6	

[a] Standard deviations given in parentheses.

TABLE IV
Effects of Web Thickness and Lamination on Water Vapor Transmission of Electrospun Polypropylene Web/Laminates

	Water vapor transmission rate (g/h/m^2)		% Average decrease in water vapor transmission rate due to lamination
	Web	Laminate	
Thin	21.0 (0.8)[a]	19.7 (0.1)	6.2
Thick	20.5 (0.1)	18.0 (0.5)	12.17
% Average decrease in water vapor transmission rate due to increased thickness	2.38	8.57	

[a] Standard deviations given in parentheses.

materials currently in use. The protection property was calculated from the percentage of pesticide penetration as follows:

$$\text{Protection}(\%) = 100 - \text{penetration}\ (\%) \qquad (1)$$

As presented in Figure 5, most of the developed materials located in the target zone of both high barrier performance and air permeability discussed earlier, except the two data points showing low protection performance of electrospun webs against the challenge liquid of low surface tension. It again illustrates that electrospun polypropylene webs would provide different level of barrier performance depending on the property of challenge liquids, whereas the laminates would offer high protection for a range of challenge liquids with less deviation. Electrospun polypropylene webs exhibited relatively high air permeability compared to existing materials, and even laminated materials, which sacrificed some degree of breathability for improved protection, and showed higher air permeability than most of the PPE materials available. The results clearly indicate that protective materials that cover the gap in protection/comfort performance of existing materials can be successfully developed from polypropylene nonwoven webs and laminates, using melt-electrospinning technique.

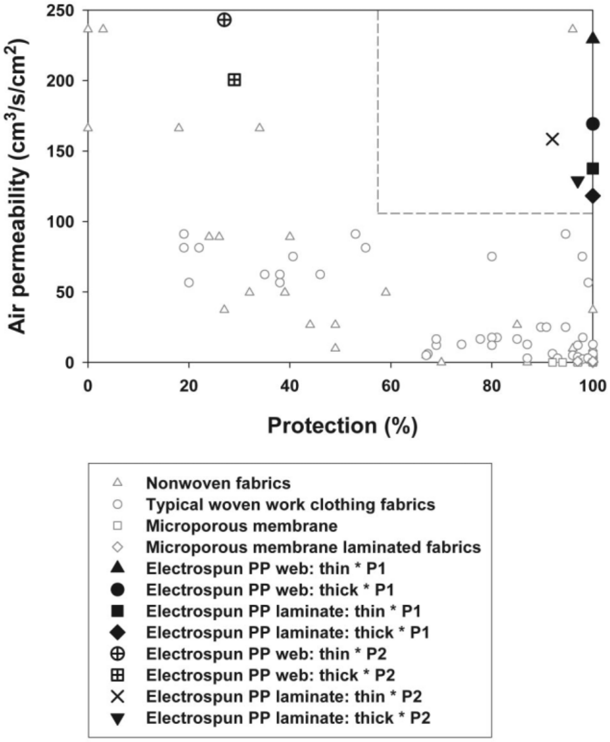

Figure 5 Air permeability and protection performance of electrospun polypropylene web/laminates compared with existing PPE materials (PP = polypropylene; P1 = pesticide mixture 1; P2 = pesticide mixture 2).

In this study, electrospun polypropylene webs consisting of fibers with diameters mostly in the micrometer range were generated, and the webs and composites exhibited generally high air permeability as well as barrier performance. For future work, it would be interesting to fabricate polypropylene webs of nanometer scale fibers, possibly from melt-electrospinning, using much lower molecular weight polymers or solvent-based electrospinning at elevated temperature and assess the barrier/comfort performance. Also, assessing wicking effects of electrospun webs and composites would be helpful in further understanding interactions between the material and challenge liquids.

CONCLUSIONS

The aim of this research was to develop protective textile materials that could provide a combination of high barrier performance and thermal comfort properties, which is not attainable with PPE materials available, using melt-electrospinning. Protection performance of electrospun polypropylene webs and laminates was evaluated against two challenge liquids with a range of surface tension and viscosity at different levels of web thickness. Effects of lamination and web thickness on air permeability and water vapor transmission were assessed as indications of thermal comfort performance.

Electrospun polypropylene webs exhibited a range of barrier performance depending on challenge liquids: no penetration against the challenge liquid of high surface tension, whereas quite high penetration for challenge liquid of low surface tension. However, the laminated fabrics exhibited higher barrier performance with less deviation against a range of challenge liquids. Air and water vapor transport properties were reduced to some degree by the increased web thickness and lamination, but still in acceptable ranges.

Compared with PPE materials currently available, electrospun polypropylene webs and laminates exhibited a combination of high protection performance and an acceptable level of air/vapor transport properties. More specifically, electrospun polypropylene webs with low level of thickness would be ideal to be used for pesticide mixtures of high surface tension, which would provide excellent protection performance as well as high level of air permeability. For pesticide mixtures of low surface tension, the laminated fabrics with high level of thickness would be better, since it would offer over 95% protection performance with an acceptable level of air permeability, which is still above 100 $cm^3/s/cm^2$.

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