New fluorochemicals for protective clothing

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A new technology that allows fluorochemicals to be added to polypropylene during fiber extrusion provides the protective benefits associated with topical treatments while eliminating the disadvantages of post-processing.

Fluorochemicals have been used extensively in the nonwovens industry to impart repellent properties to nonwoven substrates utilized in areas such as the medical field. Their benefits are readily realized when considering the fluid resistance of a 2-oz/yd^2 polyester/cellulosic spunlace nonwoven. A 1% fluorochemical treatment typically provides fluid repellency and hydrostatic head barrier properties greater than 20 cm. An untreated sample fails to provide any barrier or repellent properties.

Unlike polyester/cellulosics, polypropylene nonwoven substrates are hydrophobic and, therefore, better barriers to water. Despite the inherent hydrophobicity, polypropylene nonwoven fabrics are not significant barriers to highly penetrating fluids commonly encountered in the medical field, such as isopropanol (IPA). Thus, overall repellent and barrier properties of polypropylene can be enhanced by applying a fluorochemical treatment. This fluorochemical treatment is typically applied as a topical finish through conventional techniques of padding, spraying, or foaming.

All of these aqueous application techniques involve some type of subsequent heating or drying process to remove solvent and to achieve coalescence. Since these application techniques are surface coatings, the performance is dependent upon a variety of coating parameters which are difficult to control and which may compromise ultimate barrier performance. For example, wetting of polypropylene substrates may require surfactants, which can interfere with barrier performance or produce substantial volatile organic emissions.

This paper describes a new technology developed to fluorochemically treat polypropylene nonwovens using a unique class of thermally stable fluorochemicals. This technology is predicated on applying the fluorochemical in the extrusion process. It imparts repellent and barrier properties to the substrate without any wet processing. We intend to demonstrate that upon fiber extrusion, the fluorochemical melt additive rapidly localizes to the fiber surface, imparting a low surface energy which results in enhanced barrier and repellent properties.

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I. Independent variable ranges

Independent variable	−a	−1	0	+1	+a
Fluorocarbon additive conc., wt%	NA	0	0.5	1.0	1.2
Die pressure, psi	100	107	125	143	150

Experimental

Surface analysis

Surface elemental analysis was conducted on a Surface Science SSX-100 ESCA spectrometer, using a focused monochromatic X-ray excitation source. Two polypropylene monofilaments were prepared by coextruding the fluorochemical additive at 2% and 0.5% by weight, respectively. The monofilaments were slant cut and analyzed for fluorine-to-carbon ratio.

Surface activity

Contact-angle measurements for advancing and receding contact angles were made on a Cahn dynamic contact analyzer using the Wilhelmy method. Monofilaments of 50–70 μm were extruded at 0% and 0.4% by weight of the fluorochemical additive and subsequently analyzed.

Resin molecular weight

Sixteen milligrams of the samples were cut into small pieces and combined with 10 mL of 1,2,4-trichlorobenzene (TCB). The TCB contained 0.1% BHT antioxidant to help prevent degradation of the sample. The sample solutions were heated to 160°C for 12 h and filtered through a 0.2-micron Gelman filter apparatus. One hundred ninety-five microliters of each solution were injected into a Waters Gel Permeation Chromatograph (GPC) at 140°C, with TCB as the mobile phase. A polystyrene calibration was used to obtain equivalent molecular weights of the sample.

III. Response values for all polypropylene resins

800 MFR

Row	Hydrostatic oil rating	20% Water rating	Head, cm	IPA, cm	Max. porosity, μm	$\overline{M}_w$, E^4	$\overline{M}_n$
1	0	2.0	44.0	12.3	44.69	3.96	1.18
2	0	2.0	34.6	9.3	56.47	4.03	1.14
3	6	10.0	38.3	20.3	56.47	4.05	1.17
4	2	9.0	31.6	17.0	63.87	4.11	1.21
5	2	9.0	35.3	18.6	62.50	4.02	1.20
6	1	9.0	35.0	18.6	59.94	4.14	1.20
7	1	8.0	37.0	19.3	56.47	4.09	1.15
8	1	7.0	35.6	18.6	61.20	4.08	1.25
9	1	6.3	42.7	20.3	48.11	4.16	1.23
10	1	8.7	32.7	16.3	61.20	4.08	1.14
11	0	2.0	36.7	9.3	53.38	4.28	1.16
12	0	2.0	35.0	6.6	58.74	4.07	1.22
13	6	10.0	32.5	16.8	66.79	4.23	1.20

400 MFR

Row	Oil rating	Water rating	Hydrostatic head, cm	20% IPA, cm	Max. porosity, μm
1	0.0	2.0	66.3	13.0	22.90
2	0.0	2.0	37.0	7.3	61.20
3	1.0	6.7	64.0	29.6	30.49
4	4.7	10.0	30.6	15.6	67.80
5	1.0	6.0	41.0	18.6	48.90
6	2.0	9.0	35.0	16.6	61.20
7	1.0	7.3	42.3	18.6	56.47
8	2.0	9.0	40.0	18.6	54.37
9	1.0	5.0	54.7	22.0	38.24
10	1.0	9.0	34.0	16.6	64.84
11	0.0	2.0	40.3	8.0	48.2
12	0.0	2.0	42.0	7.0	49.19
13	5.3	10.0	40.6	19.3	54.22

70 MFR

Row	Oil rating	Water rating	Hydrostatic head, cm	IPA, cm				Max. porosity, μm
				10%	20%	30%	40%	
1	0.0	2.0	78.0	39.8	17.3	0.0	0.0	16.65
2	0.0	2.0	45.3	20.5	11.0	0.0	0.0	27.38
3	1.3	7.0	59.0	44.8	31.0	14.7	9.0	22.31
4	2.0	9.0	59.6	34.0	26.7	15.2	10.5	25.18
5	0.0	4.0	68.7	40.0	25.7	7.5	0.0	22.97
6	0.0	4.0	55.0	24.7	20.7	0.0	0.0	21.11
7	0.0	3.7	74.7	45.0	25.7	8.0	0.0	19.06
8	0.0	3.0	69.0	41.0	26.3	7.5	0.0	20.29
9	0.0	3.0	80.7	46.7	29.0	3.8	0.0	18.61
10	0.7	4.0	62.0	35.2	23.3	9.5	0.0	29.44
11	0.0	2.0	53.3	28.5	12.0	0.0	0.0	19.78
12	0.0	2.0	58.3	28.7	14.0	0.0	0.0	19.29
13	2.0	9.0	58.3	34.8	28.3	17.2	9.8	21.7

$\overline{M}_w$ = weight average molecular weight. $\overline{M}_n$ = number average molecular weight.

II. Independent variable settings

Row	Run order	Die pressure, psi	Conc., wt%	Die pressure, psi	Conc., wt%	ρ^2	C^2	PC
1	1	107	0.0	−1.0	−1.0	1.0	1.0	1
2	5	143	0.0	1.0	−1.0	1.0	1.0	−1
3	2	107	1.0	−1.0	1.0	1.0	1.0	−1
4	6	143	1.0	1.0	1.0	1.0	1.0	1
5	3	125	0.5	0.0	0.0	0.0	0.0	0
6	7	125	0.5	0.0	0.0	0.0	0.0	0
7	12	125	0.5	0.0	0.0	0.0	0.0	0
8	10	125	0.5	0.0	0.0	0.0	0.0	0
9	13	100	0.5	−1.414	0.0	1.999	0.0	0
10	11	150	0.5	1.414	0.0	1.999	0.0	0
11	4	125	0.0	0.0	−1.0	0.0	1.0	0
12	8	125	0.0	0.0	−1.0	0.0	1.0	0
13	9	125	1.2	0.0	1.414	0.0	1.999	0

Pore diameter

Pore size was determined using the Coulter Porometer instrument. This is an automated and extended version of the liquid displacement method widely used by the filter industry and reviewed in American Society for Testing and Materials Method F316-80 (*1*).

Repellent and barrier properties

Oil repellency test (*2*). Drops of standard test liquids, consisting of a selected series of hydrocarbons with varying surface tensions, were placed on the fabric surface and observed for wetting. Higher oil rating numbers represent better repellency, and the oil rating is the highest numbered liquid which did not wet the fabric surface.

Water/IPA repellency test (*3*). Specimens were challenged with increasing concentrations of isopropanol in water until wetting was observed. These ratings range from 0 to 10, with higher numbers representing better repellencies.

Hydrostatic head test (*4*). A test specimen, mounted under the orifice of a conical well, was subjected to water pressure increasing at a constant rate until three points of leakage appeared on its surface.

Test fluid preparations

Synthetic blood. A 47-dyne/cm solution was prepared consisting of 0.9 wt% NaCl in 6 vol.% IPA. The remaining balance consisted of deionized (D.I.) water.

Synthetic urine. A 45-dyne/cm solution was prepared consisting of 10 g/L NaCl, 0.3 g/L $CaCl_2 \cdot H_2O$, 0.6 g/L $MgCl_2 \cdot 6H_2O$, and 0.15 g/L Triton X-100. The balance consisted of D.I. water (*5*).

Extrusion conditions

Polypropylene melt-blown webs were extruded on a 3/4-in. Brabender extruder with a 10-in. die. These 50 g/m^2 webs were formed through typical melt-blown conditions. All samples were subsequently calendered, before physical testing, on a 4% embossing roll at 200°F, 25 psi, and 12 ft/min.

Three polypropylene resins were used in this study. All polypropylene resins are from Exxon Chemical Co. and are designated with respective melt flows as follows: PP 3495G 800 melt flow resin (MFR), PP 3505 400 MFR, and PP 3435 70 MFR.

Statistical evaluation

A central composite factorial design experiment was utilized to determine the effects of varying melt-blown extrusion conditions on repellent and barrier properties. The independent variables of die pressure (extrusion temperature as a covariant) and fluorochemical additive concentration were statistically evaluated in three polypropylene resins as previously described. The independent variable ranges are described in **Table I**. The design format is described in **Table II**, with the uncoded independent variable settings provided on the left and the coded values listed on the right. A full description of response values is provided in **Table III** for all three polypropylene resins. Provided

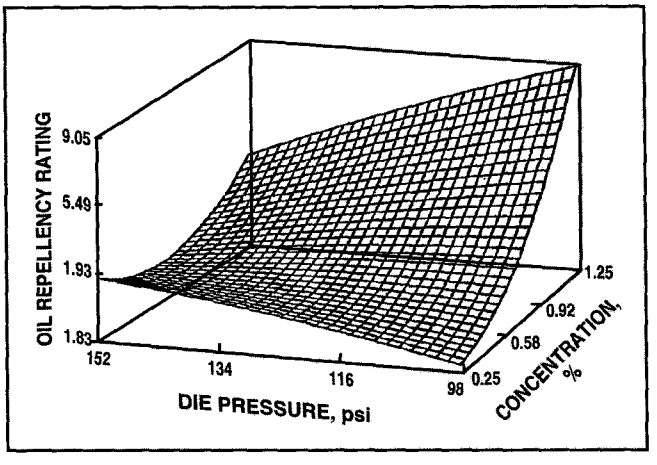
IV. Repellent properties of polypropylene melt-blown webs

Sample description	*MFR*	*Oil repellency rating*	*Water/IPA repellency rating*
1% Additive	800	6	10
1% Additive	400	5	10
1% Additive	70	2	9
Untreated	800	0	2
Untreated	400	0	2
Untreated	70	0	2

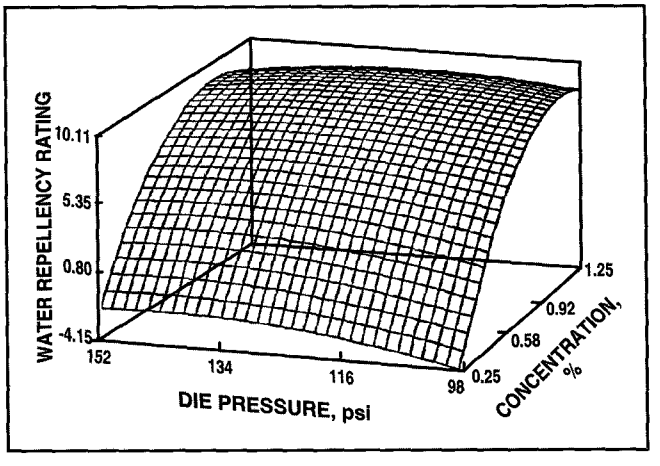
V. Repellency effects of annealing low MFRs

Sample description	*Unannealed repellencies*		*Annealed repellencies*	
	Oil	*Water/IPA*	*Oil*	*Water/IPA*
1% Additive	2	9	6	10
Untreated	0	2	0	2

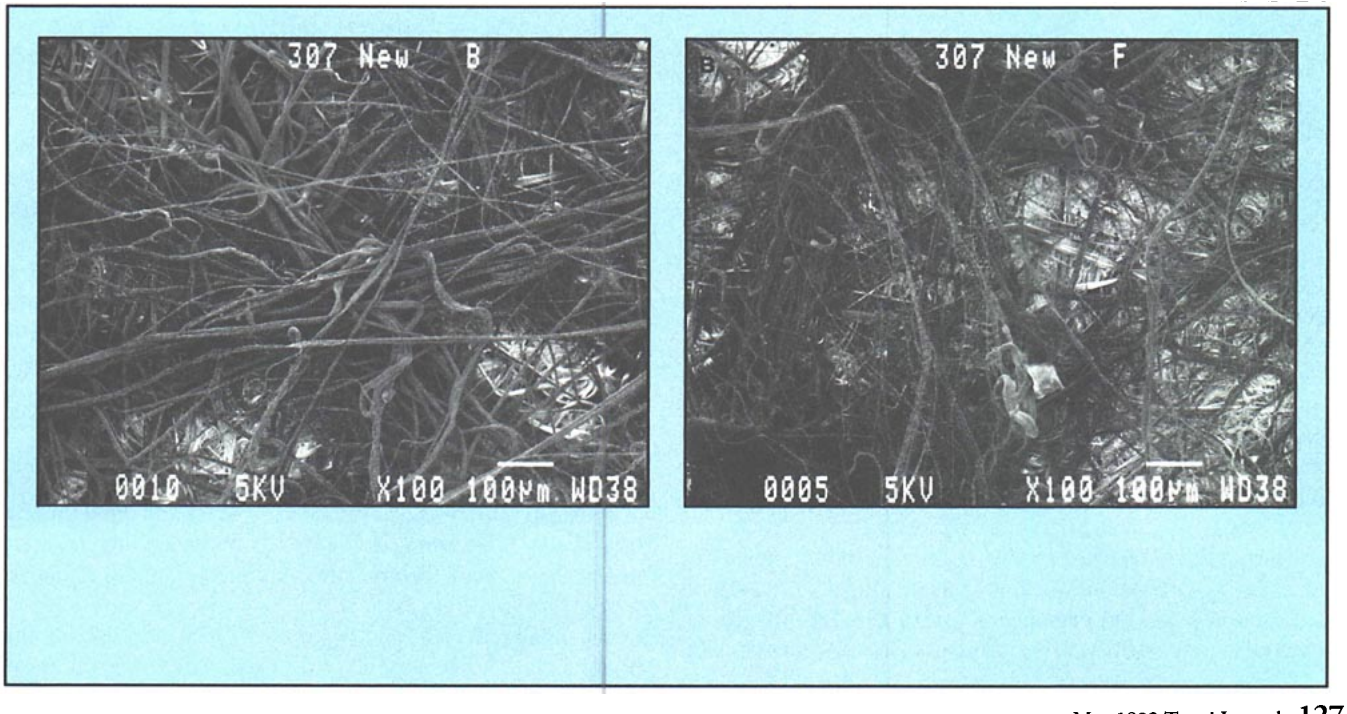
1. Oil repellency response surface for 800 MFR



2. Water/IPA repellency response surface for 800 MFR



3. SEM analysis of die temperature effects



the second-order models were statistically acceptable, the response surfaces were plotted to determine the region of optimal repellent and barrier performance properties within the experimental design space. Furthermore, the significant effects controlling a given response were also identified.

Results and discussion

This paper describes a new generation of fluorochemicals designed to withstand the conditions encountered in a polymer extrusion process. The complexities of designing fluorochemical melt additives include tailoring the molecular architecture for a given resin system. Once the fluorochemical is coextruded, it localizes at the fiber surface, imparting low surface energy and repellent properties.

Repellency on surfaces can be described by the spreading coefficient, S (*6*), as described in Eq. 1:

$$S = \gamma_{sv} - \gamma_{lv} - \gamma_{sl} \quad (1)$$

where

γ_{sv} = the solid/vapor interfacial energy

γ_{lv} = the liquid/vapor interfacial energy

γ_{sl} = the solid/liquid interfacial energy.

When $S < 0$, the surface is repellent to the liquid, and conversely, when $S > 0$, the liquid completely wets the surface.

The repellent condition pertinent in this study is given in Eq. 2:

$$\gamma_{sv} - \gamma_{lv} - \gamma_{sl} < 0 \quad (2)$$

By sufficiently decreasing γ_{sv}, the repellent condition is favored. Fluorochemicals effectively reduce γ_{sv} to the extent that only extremely low-surface-tension liquids will wet the surface. These principles are the basis for assessing repellency and barrier properties in this study.

Oil and water/IPA repellencies

The oil and water/IPA repellencies are given in **Table IV** for the three different melt flow resins. These results indicate incorporation of the fluorochemical (FC) melt additive greatly increases repellent properties.

The response surface for oil repellency is shown in **Fig. 1.** The response surface serves as a road map to predict where within the design space the optimized repellency should occur. In all three resins, the oil repellency models predict that increasing the fluorochemical concentration will increase the repellency performance. Furthermore, with the exception of the 70 MFR, die pressure has no statistical effect on repellency performance. In the 70-MFR response, die pressure has a positive effect. In the experimental design, the objective was to maintain constant die pressure from resin to resin. In the 70-MFR case, this required us to operate at impractically high extrusion temperatures (350°C). This may have resulted in degradation and reduction in performance.

In the 800 MFR, the response curves predict a maximum rating of 6 is easily obtained at concentrations from 1.0% to 1.25%, and when die pressure is within 100–125 psi. Under our extrusion conditions, this die pressure range corresponds to a die temperature of 245°C.

The response surface for water/isopropyl alcohol mixtures are similar to the oil response surface (**Fig. 2**). Increasing the concentration of the additive increases repellency, and very high ratings of 9 and 10 can be obtained at 1% concentration of the fluorochemical additive. Analogous to the oil repellency results, the higher die pressures (lower extrusion temperatures) in the 400 and 70 MFRs provide the best water/IPA repellency. Within this design space, there is no statistical effect of die pressure on repellency in the 800 MFR.

Further comparisons can also be made among the three resins. Specifically, the higher-melt-flow resins provide better repellency properties as compared to the lower-melt-flow resins extruded under the same conditions. The repellency properties in the lower-melt-flow resins can be enhanced and made comparable to the higher-melt-flow resins through some form of annealing. As shown in **Table V**, the 70-MFR samples were annealed at 250°F for 3 min, and the repellencies were compared to the original values. Significantly improved repellencies were observed.

Thus, Figs. 1 and 2 and Table V demonstrate that by incorporating 1% of the melt additive significantly higher repellencies can be achieved on polypropylene nonwovens without the added cost and processing difficulties associated with wet finishing.

Effects of processing conditions on web geometry

Since web geometry is a significant variable in evaluating repellent and barrier properties, efforts were made to understand and control the influence of processing conditions. Increasing extrusion temperature on a given melt flow resin will reduce melt viscosity and result in a lower denier and a more dense web, as compared with the same resin extruded at lower temperatures. This was confirmed by scanning electromicrograph (SEM) analysis as shown in **Fig. 3.** This effect is due to variations in viscosity at the die.

Consequently, die pressure (which can be related to melt viscosity) and fluorochemical additive concentration are two significant independent variables which can be used to understand the effects of extrusion process conditions and resin melt flow on repellent and barrier properties. Changes in die pressure were obtained by manipulating extrusion temperature.

The first four experiments within each of the nonwoven extrusion designs consisted of a low and a high die pressure setting. These settings were combined with and without the incorporation of the fluorochemical additive. Through this approach, we were able to assess the effect of fluorochemical treatment on repellent and barrier properties, independent of web geometry.

To achieve the same die pressure in the presence of the fluorochemical, it was necessary to reduce the extrusion temperature. Specifically, in the 70 MFR without the FC, a die pressure of 107 psi was obtained at a barrel and die temperature of 370°C. Whereas, addition of 1% of the FC melt additive provided the same die pressure at a barrel and die temperature of 338°C.

Since die pressure decreases with the addition of the fluorochemical melt additive, GPC analyses were run to de-

VI. GPC versus processing conditions

Sample description	Die pressure	Die temp., °C	MFR	Molecular weight $\bar{M}_w$, E^4	Molecular weight $\bar{M}_n$, E^4	ρ
Untreated	107	245	800	3.96	1.18	3.35
1% Additive	107	245	800	4.05	1.17	3.46
Untreated	143	223	800	4.03	1.14	3.55
1% Additive	143	223	800	4.11	1.21	3.40
Untreated	107	313	400	4.07	1.22	3.33
1% Treated	107	293	400	4.18	1.12	3.72
Untreated	143	262	400	4.43	1.17	3.78
1% Treated	143	262	400	4.63	1.20	3.85
Untreated	107	370	70	4.12	1.31	3.15
1% Treated	107	338	70	4.36	1.39	3.14
Untreated	143	330	70	5.69	1.44	3.94
1% Treated	143	320	70	5.16	1.48	3.48

$\bar{M}_n$ = number average molecular weight. $\bar{M}_w$ = weight average molecular weight. r = polydispersity. MFR = melt flow resin.

termine whether the additive affected the resin's molecular weight. Results are summarized in **Table VI.**

Although extrusion temperature appears to affect molecular weight, statistical analysis of the 800 MFR indicates that neither die pressure nor FC melt additive have an effect on molecular weight.

Therefore, reductions in die pressure appear to be due to an extrusion-processing-aid effect of the additive and not to reductions in molecular weight caused by the melt additive. This processing-aid effect was not observed with the 800 MFR.

As previously discussed, die pressure had a strong effect on web geometry, as defined by Coulter porosity measurements. Additionally, the fluorochemical had the effect of increasing the average and maximum pore diameter. **Figure 4** depicts the relationship of die pressure and fluorochemical concentration on maximum pore diameter for the 800-MFR design.

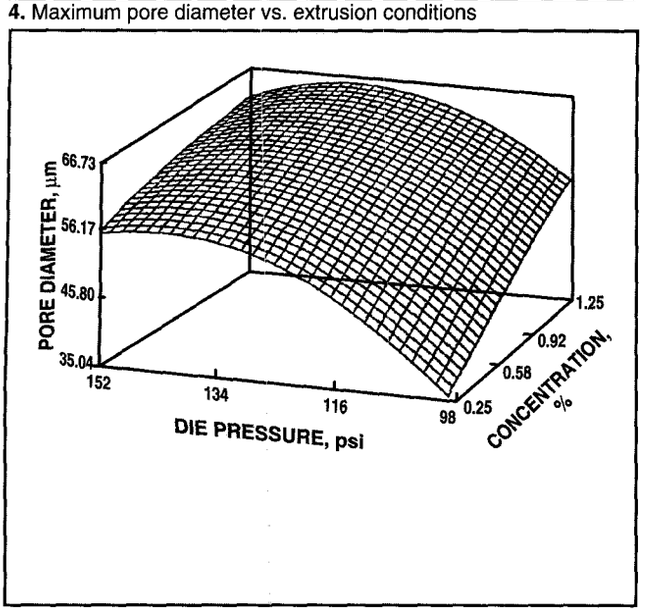
The response surface depicts that the largest pore diameter is obtained by increasing die pressure (decreasing temperature) and increasing the concentration of the fluorochemical. Specifically, in the 800 MFR, a pore diameter of 65 microns is obtained at die pressures ranging from 125 psi to 143 psi and at 1.0–1.25% additive. Conversely, small pore diameters conducive to barrier properties are obtained by decreasing die pressures through increasing extrusion temperatures.

Barrier properties

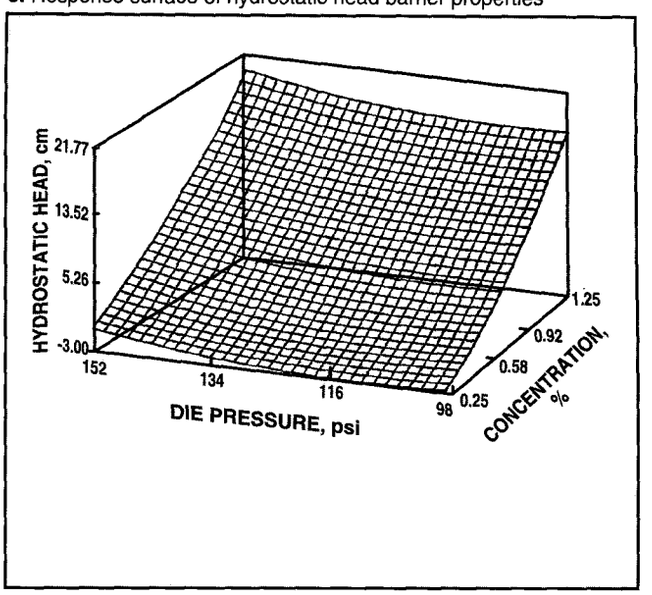
After determining the influence of processing conditions on web geometry, comparable geometries of treated and untreated webs could be obtained. Subsequently, barrier properties were statistically evaluated through hydrostatic head measurements. While water resistance was not enhanced by the addition of the fluorochemical melt additive, tests with lower-surface-tension fluids show that the fluorochemical melt additive significantly improved barrier properties. Samples from the three resins were evaluated for increasing head barrier performance by increasing the proportion of IPA in water. **Table VII** describes the barrier properties of the 70 MFR, and **Fig. 5** illustrates the corresponding response surface for the 30% IPA test liquid.

The 30% IPA response surface indicates that the barrier properties increase with increasing FC melt additive concentration. Furthermore, die pressure is not a statistical factor once the IPA concentration exceeds 10%, or 0.1 IPA volume fraction. Below 10% IPA, reducing die pressure (by increasing extrusion temperatures) improves barrier properties. This

4. Maximum pore diameter vs. extrusion conditions



5. Response surface of hydrostatic head barrier properties



VII. Hydrostatic head barrier properties of 70 MFR

Sample	FC conc., %	Die pressure, psi	Water, cm	Fraction IPA in water, cm 0.1	0.2	0.3	0.4	CH_2I_2 cm
Untreated	0	107	78.0	39.8	17.3	0	0	0.7
Treated	1	107	59.0	44.8	31.0	14.7	9.0	11.0
Untreated	0	143	45.3	20.5	11.0	0	0	0.6
Treated	1	143	59.6	34.0	26.7	15.2	10.5	10.0

is due to geometrical effects in the web.

Upon comparing the barrier properties to water in Fig. 1, it appears that the fluorochemical has an inconclusive effect on barrier properties. However, normalizing for pore size, water barrier properties do not appear to be enhanced. This is not a practical consideration since polypropylene water barrier properties are sufficient. The important issue is the enhancement of barrier properties to highly penetrating fluids (low surface tension) imparted through fluorochemical melt addition.

Also reported in Table VII are increasing head results for diiodomethane, a nonpolar high-surface-tension test fluid. A significant improvement in barrier properties is noted for the fluorochemically treated samples. Due to the high density of diiodomethane (3.33 g/cm^3), the reported increasing head results represent a proportionately higher static head performance.

The fluorochemical melt additive is effective in enhancing barrier properties to highly penetrating fluids, both polar and nonpolar. While this is characteristic of conventional topical fluorochemical treatments, the enhanced barrier properties provided from this technology can be achieved directly from the melt.

Surface analysis

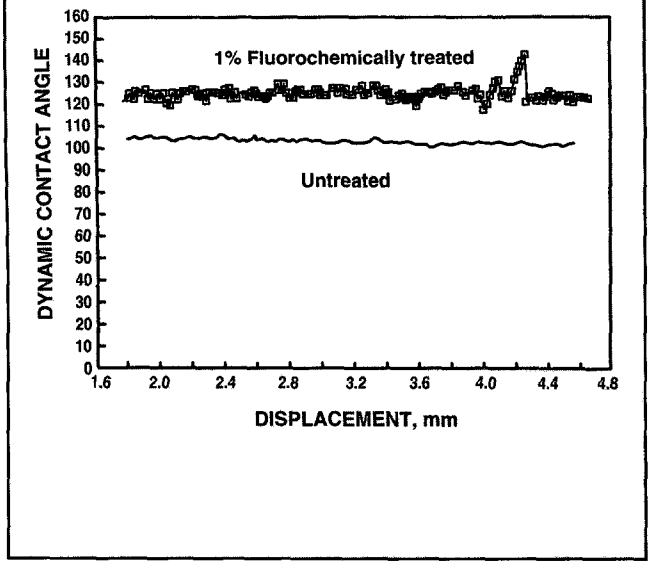
ESCA spectrometer and dynamic contact angle measurements were performed to corroborate enhanced repellent and barrier properties. Nonwoven melt-blown samples were analyzed by ESCA, indicating that all samples treated at a 1% level of the FC additive contain at least 40 atom% fluorine on the surface. Based upon total available fluorine, this indicates effective surface localization.

Dynamic contact angles

The findings from the statistical modeling for barrier properties to the test fluids correlate well with measurements of advancing contact angles. **Figure 6** shows advancing contact angle measurements for a 1.0% fluorochemically treated sample, as compared with an untreated sample.

These samples are polypropylene monofilament fibers extruded on a conventional melt-spinning fiber line and have

6. Advancing water-contact-angle polypropylene fiber scans



VIII. Advancing contact angles for test fluids

Test fluid	Fluid surface tension, dynes/cm	Advancing contact angle, ° 1% Treated	Untreated
Water	72	125	107
10% IPA	51	115	94
20% IPA	34	98	63
30% IPA	29	84	41
40% IPA	27	77	31
Synthetic urine	41	81	59
Synthetic blood	49	94	85
CH_2I_2	45	118	62

been annealed at 150°C for 15 minutes. The graph shows a contact-angle scan along the length of the fiber with water. The high contact angle of approximately 125° indicates that the fluorochemical treatment is providing a low-energy surface in comparison to an untreated polypropylene sample with a contact angle of 107°. The fluorochemical is imparting a low-energy surface to provide repellent and barrier properties to polypropylene nonwovens.

In support of the increasing head measurements, dynamic contact-angle measurements were conducted using the same test fluids. **Table VIII** shows the surface tension of the test fluids, with the corresponding advancing contact angles, for 1% fluorochemically treated and untreated filaments.

In all cases, the advancing contact angles are substantially greater for the fluorochemically treated fibers. While a contact angle greater than 90° is presumably required for nonwetting, recent literature suggests that angles substantially below 90° can still provide repellency on porous, low-surface-energy materials (*7*). This information may explain barrier properties realized on those test fluids with advancing contact angles less than 90°.

Theoretically, barrier properties can be related to advancing contact angles through the Washburn equation (*8*):

$$\rho gh = (2\sigma \cos \Theta \text{ adv})/r \qquad (3)$$

where

ρgh = the static head

σ = the liquid surface tension

r = the pore radius.

The theoretical relationship of advancing contact angle and pore radius for a given web can be graphically illustrated (**Fig. 7**).

For a given pore radius, any increase in advancing contact angle should provide an increase in static head barrier properties. Furthermore, barrier enhancement is particularly significant when the pore sizes are small.

In summary, the advancing contact angles and the maximum pore radius of the nonwoven web are two key independent variables to address in enhancing repellent and barrier properties.

Summary and conclusions

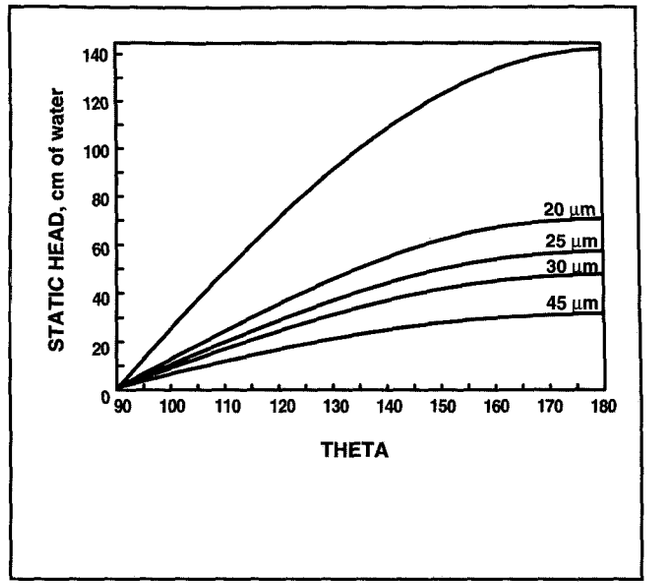
While fluorochemicals have been extensively utilized in the nonwovens industry to impart repellent and barrier properties, the requirement of added cost for wet processing and the associated problems of application can be eliminated through fluorochemical melt-additive technology. This technology permits integrated processing of polypropylene nonwovens, and a related cost savings to the manufacturer. Commercially available, topically applied fluorochemicals are commonly not stable to extrusion conditions and should not be coextruded with thermoplastic resins.

We have demonstrated, through statistical web characterization, that the fluorochemical effectively localizes to the surface and reduces surface energy. The reduced surface energy imparts superior repellent properties and enhances barrier properties to highly penetrating fluids, compared to conventional polypropylene nonwovens. This enhancement of protective properties can be achieved in a wide range of melt flow resins.

Surface analysis using ESCA indicates a high level of fluorine at the surface of the nonwoven web. Further surface analysis, involving advancing contact angle measurements, corroborates repellency and barrier results. Specifically, contact angles are consistently higher for the fluorochemically-melt-additive treated fibers.

These fluorochemicals also function as a processing aid through die pressure reduction. Furthermore, increases in fiber pore size were observed, which can be controlled through extrusion temperature adjustments.

7. Static head vs. advancing water contact angle (R = 10 μm)



In the nonwovens industry this technology should be applicable to a variety of products in both the industrial and consumer markets. These products could find uses in diapers, protective clothing, filters, breathable membranes, and a long list of other diversified products. □

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