June 20, 1967 V. C. SMITH ET AL 3,326,713

BREATHABLE AND WATERPROOF COATED FABRIC AND PROCESS OF MAKING SAME

Filed Jan. 29, 1964

Fig. 1.

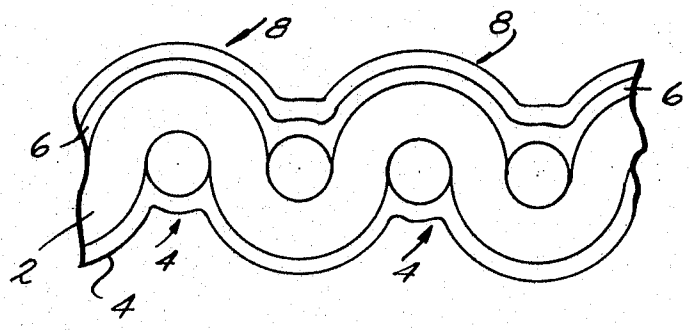
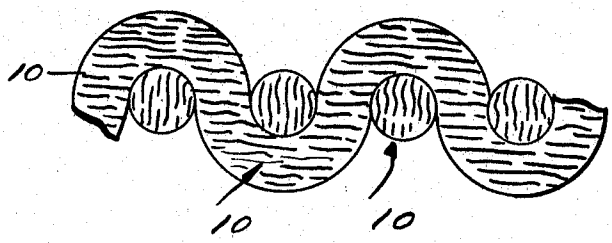


Fig. 2.



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United States Patent Office 3,326,713 Patented June 20, 1967

3,326,713

BREATHABLE AND WATERPROOF COATED FABRIC AND PROCESS OF MAKING SAME

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Filed Jan. 29, 1964, Ser. No. 340,893
8 Claims. (Cl. 117—76)

The present invention relates to coated fabric, e.g. rainwear or the like. More particularly, the invention is concerned with a coated fabric of the type indicated which is breathable and waterproof yet retains the desirable hand and appearance of an uncoated product.

Extensive prior efforts have been made to obtain rainwear fabrics which are not only waterproof and breathable, but also possess a soft and otherwise desirable hand. Despite these efforts, however, no completely satisfactory product has previously been developed. Thus, for example, while it is possible to prepare fabrics having desirable degrees of breathability and waterproofing using specialized and costly coating techniques or treatments, the resulting products usually demonstrate an unattractive hand. In particular, the coating may make the fabric stiff or boardy and even with a thin and soft type of coating, the product generally has an undesirable rubbery or waxy feel.

Obviously, it is not an easy thing to obtain an optimum combination of the desired characteristics since breathability and waterproofing, for example, are at least to some extent opposed to each other. Essentially the same is true in the case of hand since almost any coating material which is used in normal manner to waterproof a fabric will have some undesirable effect on the hand.

The principal object of the present invention is to obviate prior art difficulties and provide a coated fabric, suitable for use as rainwear or the like, which is breathable and waterproof and also demonstrates a highly desirable textile hand. A more specific object of the invention is to provide a coated fabric of the type indicated which has the appearance and hand of an uncoated product. Another object is to provide a process for preparing the indicated fabric. Other objects will also be apparent from the following detailed description of the invention and the accompanying drawings wherein:

FIGURE 1 is an enlarged vertical sectional view of a fabric processed according to the invention; and

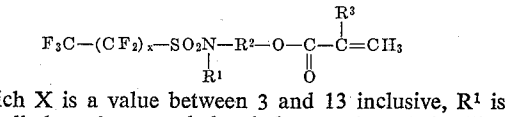
FIGURE 2 is a corresponding view of a similar product without the pretreatment of the invention.

Broadly stated, the objects of the invention are realized by pretreating the fabric with a fluorochemical compound having oil repellent properties and then coating the fabric with any conventional type of waterproofing resin or elastomer composition. Advantageously, the fluorochemical compound is applied to the fabric in the form of a dispersion or solution in water or organic liquid. The thus treated fabric is dried and cured and the waterproofing composition is then applied in an appropriate vehicle, e.g. an organic solvent, followed again by drying and curing. Without intending to be limited to any particular theory, it appears that the fluorocarbon prevents the solvent or liquid vehicle used for applying the waterproofing material from soaking into the fabric and carrying the elastomer or equivalent into the interstices of the fabric where breathability and hand might be detrimentally affected.

As a typical illustration of the invention, a fluorochemical may be applied to nylon or cotton fabric after which the fabric is dried and cured. A polyurethane coating or the equivalent may then be applied to the fabric. A comparison of this fabric with the corresponding product where the fluorochemical pretreatment is omitted, shows that the dual treated fabric has a much softer hand and is otherwise more attractive. The polyurethane coating

on the fluorochemical pretreated product seems to ride on the surface of the fabric without significant penetration into the fabric due apparently to the action of the fluorochemical compound.

Any of the conventional fluorochemical compounds which have oil repellent properties can be used herein. For example, acrylates and methacrylates of hydroxyl compounds containing a highly fluorinated residue and their polymers and copolymers can be used. Fluorochemical compounds of this type are defined with greater particularity in U.S. Patents 2,642,416; 2,826,564; 2,839,513; 2,803,615. Other fluorochemical compounds which can be employed include the chromium coordination complexes of saturated perfluoromonocarboxylic acids of which the chromium complexes of perfluorobutyric acid and perfluorooctanoic acid are representative. Fluorochemical compounds suitable for use herein are available commercially, for example, those marketed under the tradename "Scotchgard" [1] such as Scotchgard FC 205 and FC 208 and "Zepel." [2] Of these compounds, the Scotchgard FC 205 and FC 208 products, which are available in emulsion form, may be described according to U.S. Patent 2,803,615 by the following general formula:



in which X is a value between 3 and 13 inclusive, R^1 is lower alkyl, such as methyl, ethyl, propyl, and the like, having 1–6 atoms, R^2 is alkylene containing 1–12 carbon atoms and R^3 is H, methyl or ethyl. The product "Zepel" is also available in emulsion form and while it is chemically different from the Scotchgard products, it is a fluorochemical oil repellent containing fluorocarbon tails composed of CF_2 groups which may end in a terminal CF_3 group.

Generally speaking, fluorochemical compounds of the type indicated (e.g. Scotchgard) are known to demonstrate a water repellent effect but they are not considered to be waterproofing materials. Nevertheless, in the present case, the fluorocarbon pretreatment followed by the treatment with a waterproofing material gives a waterproofing effect superior to that obtained when the pretreatment is omitted. This is highly unusual and points up the unexpected nature of the invention.

As noted, the fluorochemical compound may be applied to the fabric by padding the latter with an aqueous or organic system containing the pretreating compound. Usually, aqueous emulsions or like compositions containing from 0.5% to 5% by weight fluorochemical compound as supplied or compounds are used with from 0.01 to 1.0% of the compound or mixture of such compounds being deposited on the fabric, based on the weight of dry fabric.

The conditions which are used to dry and cure the fabric after application of the fluorochemical compound will vary depending on the fluorocarbon and liquid medium utilized as well as the fabric undergoing treatment. However, a temperature in the range of 300 to 375° F. for 1 to 5 minutes will usually be sufficient for this purpose.

As the waterproofing composition, there may be used any known water-insoluble film-forming, curable resin or polymer. Silastic or silicone elastomer coatings are preferred but other materials, e.g. acrylics, vinyls, polyurethanes and the like may be used. A typical silastic for use herein is that commercially available as Dow XT–40025 or Dow XT–40057 (30% solids as received, prod-

[1] Registered trademark of Minnesota Mining and Manufacturing Company (also known as 3–M).
[2] Registered trademark of E. I. Du Pont.

uct of Dow Corning Corp.). This and other suitable silastic of siloxane elastomers are described in U.S. Patent 3,076,726 and may be represented by the formula:

$$(CH_3)_xSiO_{4-x/2}$$

wherein x has an average value from 1.9 to 2, inclusive.

The waterproofing resin will generally be applied from a solution, dispersion or emulsion thereof in an inert organic liquid which does not affect the fabric or the fluorochemical compound. A preferred solvent is Varsol (a product of the Standard Oil Company), although other vehicles which may be used include xylene, toluene, heptane, trichloroethylene, perchloroethylene or any other solvent which is compatible with the elastomer being used.

The amount of waterproofing resin applied to the fabric will vary but, generally speaking, this will be in the area of 0.1 oz. to 2.0 oz. per square yard additional weight added to the fabric. Knife coating or other appropriate means may be used and the fabric may be so coated on what is to be its front side or back side only or both sides. Desirably, care is taken to insure that all, or substantially all, of the resin is just below the fabric surface so as to minimize the possibility of a rubbery or waxy feel and assure adhesion. At the same time, however, care must be taken not to press or force the material down too far into the fabric since this might have an undesirable effect on the hand.

The conditions used to dry and cure the fabric after the application of the film-forming resin composition will also vary considerably depending on such conditions as the fabric structure and composition, the nature of the resins used, etc. Usually, however, the temperature of drying is in the range 200–300° F. for 1–10 minutes followed by curing at 250°–375° for 1–10 minutes. The temperature of the cure is limited only by the heat properties of the fiber in the fabric.

Conventional catalysts may be used in both the fluorochemical pretreatment and the subsequent resin or elastomer application. Additionally, it will be recognized that the invention is applicable broadly to any and all fabrics comprising natural and/or synthetic fibers, e.g. cotton, rayon, glass, nylon, polyester (polyethylene terephthalate), acrylics, etc.

The invention is illustrated, but not limited, by the following examples wherein parts are by weight unless otherwise stated:

Example A

A 100% cotton rainwear fabric was padded with 3% Scotchgard 205 and 1% Rhotex A–9, dried and cured two minutes at 350° F. to obtain an oil rating of 110. This Scotchgard treated fabric (specimen 1 below) was then knife coated with a mixture of 66% Dow XT–40025 (or Dow 23), 0.5% QZ–80925, 1.8% Catalyst 23A, 0.5% HAc and 31.2% Varsol 3. At the same time, a control sample (specimen 2) without Scotchgard was knife coated with the same silastic elastomer mixture. Both fabrics were knife coated two times on the back of the fabric. The results obtained were:

Specimen	Pretreatment	Oil Rating	Coating Weight, oz./sq. yd.	Hydrostatic Head
1	3.0% Scotchgard + 1% Rhotex A–9	110	.58	110
2	None	0	.57	35

The beneficial effect of the fluorochemical pretreatment is readily apparent from the above example. Thus, the hydrostatic head rating of the silastic rubber coated fabric is increased from 35 cm. for no pretreatment to 110 cm. with the pretreatment. Hydrostatic head is the best measure of the waterproofness of a fabric or film and the rating of 110 cm., for the pretreated specimen 1 indicates that the product is essentially waterproof, yet it retains a good amount of breathability or water vapor transmission. It was also found that the silastic elastomer is durable to laundering and dry cleaning, at least a major proportion of the silastic properties being retained through five commercial dry cleanings or even more. Furthermore, the product obtained using the fluorochemical pretreatment product was much softer and there was no evidence of silastic elastomer strikethrough whereas the silastic elastomer coated fabric obtained without the pretreatment was stiff and showed evidence of severe strikethrough to the face of the fabric. Additionally, with the product prepared according to the invention (specimen 1 above), no plastic or rubber film was evident on the fabric surface, the fabric retaining its textile appearance and feel. This is an extremely important advantage over prior waterproofing procedures where the rubber or plastic film is evident to the touch and gives the product a clammy or otherwise undesirable feel.

The following results were obtained on testing specimens 1 and 2 above for (a) waterproofness by the well known water resistance test or Slowinski rain test; and (b) conventional spray rating before dry cleaning and after five dry cleanings to determine durability of the treatment:

Specimens	Water Resistance Test Storm (3 ft. head, 5 min.), Grams Water Passing Through Fabric		Spray Rating	
	Original	After 5 Dry Cleanings	Original	After 5 Dry Cleanings
1	0.0	0.0	100	100
2	6.0	7.0	100	100

The dry cleaning durability of the finish applied to the fabric according to the present invention is readily apparent from the foregoing.

It is important to the success of the invention that the fluorochemical pretreatment have the proper oil rating. This is shown by the following example wherein the standard 3–M oil rating technique is used for evaluation purposes:

Example B

Specimens	Amount Fluorocarbon in pretreat	3–M Oil Rating	Elastomer Formulation	Results—Hydrostatic Head	
				Original	After 5 Dry Cleanings
3	0	0	None	0	0
4	0	0	20% in Varsol	33	10
5	½% Scotchgard 205	0	do	34	10
6	2% Scotchgard 205	70	do	36	10
7	2% Scotchgard 205 + 1% Rhotex A–9	100	do	48	15
8	2% Zepel + 1% A–9	100+	do	45	13
9	3% Scotchgard (Commercial fin.).	80–90	None—Control	37	

From the above data, the importance of a proper oil rating before application of the solvent/elastomer can be seen. Although the amount of elastomer applied to the fabrics was less than optimum, all samples were prepared under exactly the same conditions. It will be seen that, as the oil rating increases, the hydrostatic head increases. A minimum oil rating of at least about 70 is necessary for present purposes, the beneficial and unexpected action of the fluorochemical not occurring at significantly lower oil ratings.

Example C

This example illustrates the use of the present process with various types of fabrics using the process of Example A except for the indicated pretreatment:

Specimen	Fabric	Pretreatment Amount of Fluorocarbon	Oil Rating	Hydrostatic Head Original, cm.
10	Polyester/Cotton	0	0	19
11	do	2% Scotchgard + 1% A-9	100	55
12	do	2% Zepel + 1% A-9	100	50

In the above, the hydrostatic test results clearly indicate the benefit of the fluorochemical pretreatment. Regardless of fiber content, the pretreatment with fluorocarbon always results in a higher hydrostatic head rating.

All of the above fabrics were knife coated two coats on the back side of the fabric with 20% solids mixture of Dow XT–40025 Elastomer and Varsol solvent.

Example D

This example illustrates the results obtained by variation in the knife coating applications of the elastomer:

Specimen	Pretreatment Amount Fluorocarbon	Oil Rating	Knife Coating Variation	Hydrostatic Head	Added weight of Coating, oz./sq. yd.
16	2.5% Scotchgard	100	2 coats backside	115	.42
17	0	0	do	33	.47
18	2.5% Scotchgard	100	1 coat each side	74	.69
19	0	0	do	34	.59
20	2.5% Scotchgard	100	2 coats backside	150+	.59
21	0	0	do	36	.49

The fabric in this case was 100% cotton raincoat fabric. A mixture of 20% solids Dow XT–40025 with Varsol was knife coated in all cases using a process as outlined in Example A above.

Specimens 20 and 21 differ from specimens 16 and 17 in that an attempt was made to apply a heavier knife coating to specimens 20 and 21. The results show that a heavier coating was applied and it will be noted that despite the weight of the coating, the hydrostatic head of the non-pretreated fabric did not improve beyond 36 cm. whereas the hydrostatic head for the pretreated fabric increased substantially.

Example E

The importance of having the appropriate oil rating for the fluorochemical treatment is further shown by this example using the process of Example A except for the changes indicated:

Specimen	Pretreatment		Elastomer Solids, Percent	Type Coating	Hydrostatic Head, cm.	Added Coating Weight, oz./sq. yd.
	Formula	Oil Rating				
22	2% Scotchgard + 1% A-9	90	20	2 coats each side	150+	.85
23	2.0% Scotchgard + 1% A-9 + 8% Zelan	0	20	do	38	1.12

The harmful effect of using too much Zelan, an oleophilic water repellent commercially available from Du Pont, in the fluorochemical pretreatment bath is shown by the above.

While this example shows the undesirable effects of using a relatively large amount of Zelan, it will be appreciated that this material or its equivalent may be advantageously used in conjunction with the fluorochemical materials as a water repellent extender or to otherwise enhance the properties of the fluorochemical for present purposes. For instance, if the elastomer is coated on the back side only, then the fluorochemical serves to provide a water repellent face. The fluorochemical will be a satisfactory water repellent without an extender such as Zelan, Phobotex, etc., but with addition of the proper amount of these extenders, a superior water repellent face can be obtained. The amount of Zelan which may be used for such purposes should be sufficiently low to give an oil rating of about 90 or above. More than this amount of Zelan, e.g. the 8% used in the example, has an undesirable effect on the hydrostatic head after the silicone elastomer is applied.

Example F

Approximately 100 yards each of the following fabric were processed full width on commercial coating equipment:

	Oz./yd.^{2}
Nylon ten fabric	4.2
Olive cotton raincoating	6.7
Gray cotton raincoating	7.2
Dacron/cotton poplin	5.4
Cotton sheeting	3.78

After suitable scouring to obtain clean fabric, the prepared fabric was padded with a bath containing

	Percent
Scotchgard FC–205	2.5
Rhotex A–9	1.0

on the weight of the bath. The padded fabric was dried and cured simultaneously at 350° at 15 y.p.m. The treated fabric was then knife coated on the back side with a formulation as follows:

	Percent
Dow XT–40025	66.0
Catalyst 23–A	1.8
QZ–80925 binder	0.5
Varsol 3 solvent	31.2
HAc	0.5

Fabrics were dried at the temperature shown below at 30 y.p.m. The following results were obtained after curing at 375° F. at 10 y.p.m.:

Specimens	3-M Oil Rating	Elastomer Coating Weight, Avg.	Temp. Drying, ° F.	Fiber Content	Fabric Description	Color	Original Hydrostatic Head, Avg.
24	100	0.68	200	100% cotton	Rainwear	Olive	150+
25	100	*0.29	200	do	do	Gray	150+
26	100	0.67	240	Dacron/cotton	do	do	83
27	100	1.34	240	100% Nylon	Tentage	White	150+
28	100	0.96	200	100% cotton	Sheeting	Bleached	85

*Note, this particular gray raincoat fabric was thicker and heavier than the olive. Both fabrics passed under the knife at the same knife setting. The gray fabric being thicker received a thinner coat of silicone elastomer as evidenced by the elastomer coating weight. It will be noted that although the coating weight of 0.3 oz./sq. yd. was half that of the other samples, the gray raincoat fabric performed equally as well as a comparable fabric with a heavier coating.

The fabrics so produced were further tested for other repellent properties described below:

Specimens	Original			After 5 dry cleanings		
	Spray Face	Storm Test	Water Vapor Transmission, gms./sq. m./24 hrs.	Spray Face	Storm Test	Water Vapor Transmission
24	100	0	493	90	0	401
25	100	0	488	90	0	445
26	100	0	450	90	0	435
27	100	0	149			
28	90	0	535			

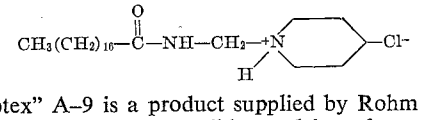
Example G

This example illustrates a modification of the present process wherein the silicone elastomer is appled by padding rather than by knife coating as in the other examples:

Specimen	Fabric	Pad	Pretreated	Suter Hydrostatic Head
29	Black cotton	15% solids	2% fluorocarbon	43
30	do	do	0	39
31	Gray cotton	do	2% fluorocarbon	36
32	do	do	0	26

After the fluorocarbon pretreatment, when used, the fabric was passed through a mixture of silicone elastomer/solvent as in Example A and the excess squeezed out by running through a nip roll. The padded fabric was then run continuously into a tenter frame for drying and curing. While the amount of silicone elastomer deposited from the dilute solution used did not make large changes in the hydrostatic head, in each case the presence of the fluorochemical increased the hydrostatic head significantly.

It is to be noted, with respect to the various products referred to by tradename in the above examples and not hitherto otherwise identified, that "Zelan" is a pyridinium acid chloride derivative of stearamide. It is available as a pre-emulsified solid which forms a stable emulsion on stirring in hot water. The following structure is typical of compounds of this type.



"Rhotex" A–9 is a product supplied by Rohm & Haas Company as a water dispersible emulsion of:

	Percent
Epoxidized soy bean oil	45
Polyvinyl alcohol	5
Water	50

"Phobotex" is a tradename for Ciba's water repellent product called a triazine fatty water repellent and is a mixture of various fatty oils and alcohols attached in some manner to a methylolmelamine nucleus.

Catalyst 23A (Dow Corning) is a product used as the catalyst for Dow XT–40025; while QZ–80925 (also Dow Corning) is a binder and crosslinker for the Dow XT–40025.

The following standard test methods are referred to in the foregoing description:

HYDROSTATIC PRESSURE TEST or hydrostatic head, AATCC standard test #18–1961 also known as ASTM–D583–58.

WATER RESISTANCE RAIN TEST—STANDARD TEST METHOD AATCC 35–1961

	Shower	Rain	Storm
Height of Column	2 ft.	2 ft.	3 ft.
Time of Exposure	30 seconds	2 min.	5 min.

Test fails if more than 1 gram penetrates (ASA L–22 standards #10.27–1960).

SPRAY RATING—AATCC standard test method 22–1961.

OIL RATING—Minnesota Mining and Manufacturing oil repellency test 3M Technical Bulletin, appendix A, dated June 19, 1962.

WATER VAPOR TRANSMISSION—Standard test method ASTM–697–42.

A modified test where a sample of fabric for testing is securely and tightly mounted over a dish containing water. Sample and dish placed in a test chamber where air is circulated at a rate not less than 500 feet/minute.

Results reported as grams (water vapor transmitted) per sq. meter (of fabric area) per 24 hours (grams/sq. meter/24 hours).

On the basis of the foregoing description, the advantages of the present invention over prior techniques will be apparent to those skilled in the art. For example, in one existing prior procedure which is used on a commercial basis, the fabric is first coated with a definite thickness of elastomer which may be vinyl, rubber or the like. The coating, even if colored to match the fabric color is nevertheless visible and has the feel of conventional rubber coated raincoat. In said prior process, the coating is filled with starch and this starch is subsequently expanded by heat and then removed with acids or enzymes so as to leave many microporous interconnecting holes through the thickness of the fabric. This leaching technique makes the film breathable, or at least allows the passage of water vapor through the film. However, it will be recognized that such an operation is much more complicated than that contemplated herein. Another prior

technique utilizes salt which is then leached out to leave microporous holes. The steps in this alternate process involve (a) grinding together the elastomer, color, salt and any other ingredient; (b) knife coat the resulting elastomer composition; (c) dry and cure; (d) leach out the salt (or starch if the latter is used); (e) dry; and (f) apply water repellent to the uncoated surface. In contrast, the present process eliminates the leaching operation and gives the desirable characteristics noted above by the controlled coating of the elastomer which results from the preliminary application of the fluorochemical compound. With the present method, the natural voids formed by the interstices of the fabric and by means of the controlled coating fill these voids until submicroscopic voids are achieved. It is believed that without the fluorochemical treatment, the elastomer utilized will not fill up the natural voids present in fabric. Rather, the solvent or liquid vehicle penetrates into the accessible regions between the fibers and around the yarn bundle, since from visual observation, there is very little change in the size of the natural voids before or after coating the elastomer. The fluorochemical or fluorocarbon pretreatment on the other hand, does not permit the solvent to penetrate into the accessible region between the fibers and around the yarn bundle. Accordingly, the solvent/elastomer system coated on the fabric must remain at or near the surface. The only accessible region for the elastomer is the fiber bundle near the surface and the natural voids are filled to give the submicroscopic voids which give breathability while retaining the desired waterproofness and hand.

The drawings herein further illustrate the manner in which the fluorocarbon pretreatment functions to give the desired results. Thus, FIGURE 1 shows the fabric 2 with one surface (e.g. the outer face) coated with a fluorocarbon barrier layer 4. The opposite surface of the fabric carries a similar fluorocarbon barrier layer 6 with the waterproofing elastomer coating 8 thereover. FIGURE 2 illustrates the case where no fluorocarbon pretreatment is used. In this situation, the entire fabric 10 is deeply penetrated and more or less saturated with elastomer as shown by the shading resulting in a product having poor hand.

It will be appreciated that various modifications may be made in the invention described herein while remaining within the scope of the invention as defined in the following claims wherein we claim:

1. A coated fabric suitable for rainwear which is breathable and waterproof and having substantially the appearance and hand of an uncoated fabric, which comprises a fabric, a first coating on said fabric of a fluorochemical having an oil rating of at least 70, a second coating thereon of a waterproof curable polymer, said first fluorochemical coating being at least partially disposed within the interstices of the fabric whereby the said second waterproof coating is prevented from substantially penetrating the said interstices and whereby said second coating lies substantially at or near the surface of the fabric.

2. The fabric of claim 1 wherein said curable polymer is a siloxane elastomer.

3. The fabric of claim 1 wherein said fluorochemical is selected from the group consisting of acrylates and methacrylates of hydroxyl compounds containing a highly fluorinated residue and their polymers and copolymers.

4. The coated fabric of claim 1 wherein the base layer of fabric has fluorochemical on both its sides and the waterproofing elastomer is on only one side.

5. The fabric of claim 1 wherein said fluorochemical coating has an oil rating of from 90–100.

6. A process for producing a textile fabric suitable for rainwear which is breathable and waterproof and having substantially the appearance and hand of an uncoated fabric, comprising depositing at least partially within the interstices of the fabric a fluorochemical having an oil rating of at least about 70, curing the fluorochemical, applying to the fabric a curable waterproofing polymer composition and curing said polymer composition, whereby the cured fluorochemical prevents substantion penetration of the uncured polymer into the interstices of the fabric.

7. The process of claim 6 wherein said composition is an elastomer in liquid vehicle, said composition being applied by knife coating.

8. The process of claim 6 wherein said composition is an elastomer in a liquid vehicle, said composition being applied by padding.

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