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PROCESS FOR RENDERING FABRICS WATER REPELLENT

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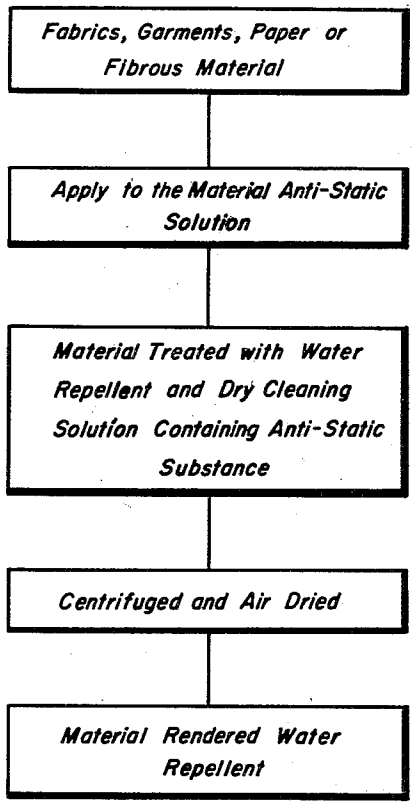

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PROCESS FOR RENDERING FABRICS WATER REPELLENT

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This invention relates to water-proofing and more particularly to a method of treating materials made from organic or inorganic fibers, for example fabrics, clothing, canvas, tents, draperies, paper and the like, to render the same water-repellent. The method of accomplishing this may be carried out employing a wet process, for example, where the article is wet with water and the water-repelling agent is water-borne, or utilizing a dry process wherein the article is treated in or with a non-aqueous liquid, such as dry cleaners' naphtha to which a water-repellent substance has been incorporated.

It is a principal object of the present invention to provide an improved method of treating such fibrous materials, especially garments, so as to render them water-repellent, and preferably utilizing a dry process. The chief benefits of the invention may, however, be obtained by employing either a dry or wet method, as will be readily apparent from the procedures hereinafter described and exemplified by the examples.

After conducting considerable research and experimental work in the treatment of fabrics to render them water-repellent, it has been observed that when applying water-repellent compositions comprising waxes, or the like, dissolved in an organic solvent, such as used in dry cleaning (e. g. Stoddard solvent) in accordance with prior known methods, that one fabric may be made substantially water-repellent and then later upon a subsequent treatment under like conditions and using the same or a similar fabric, the treatment failed to render the fabric water-repellent. It was further found that these results may be reversed. In other words, one operator using the same procedure and water-repellent composition in one instance would obtain good results and in another entirely unsatisfactory results as to water-repellency.

In accordance with the present invention, an improved method of carrying out the treatment of fabric materials and garments to render them water-repellent has been achieved so that the dry cleaning operation and water-proofing of fabrics can be satisfactorily accomplished under varying physical conditions of treatment as well as the article being treated. To this end it has been found that solutions of waxes and resins in solvents, such as dry cleaners' solvent and the like become charged with static electricity, and that these charges may be negative or positive and if not removed, bring about diverse results. The degree and character of electrification varies with the solutions used and, also, with the conditions under which the solutions are used. A solution, for instance, may normally carry a particular charge of electricity or the same may become electrified during use of the solution or mechanical handling of the fabric and/or solutions during the carrying out of the process. It has been found that even by simply pouring the solution which is used for treating the fabric from one container to another a solution may be caused to become electrified.

Furthermore, fabrics are easily electrified by the simple process of handling. For example, a woolen sweater or dress becomes charged with static electricity during the act of dressing or undressing; a nylon garment has been found to be highly electrified when it is drawn across a woolen blanket or similar kind of fabric. Similarly, garments that have been dry-cleaned and that have been placed or stored in a basket or hamper likewise attain some degree of electrification through the act of merely removing the garment from the others in the hamper.

It is also known that dry solvents and dry waxes (e. g. petroleum naphtha and wax, both of which are substantially free of water) form solutions which are very good insulating materials and that they do not readily conduct nor lead away static electricity. It is found that such a solution, for example one containing paraffin wax dissolved in naphtha without the presence of any appreciable amount of water or electrical conductive agent, and stored in a glass container, has retained a static electrical charge for a long period of time.

In accordance with applicants' improved method of treating fabrics and the like materials made from cellulose or animal fibers, which fabrics or the materials take on a charge of electricity, the electrostatic charge acquired by either the fabric or the solution applied thereto is regulated and preferably removed during or before the fabric is treated. In this connection it has been found that the response of fabric to treatment with water-repellent and dry solutions, for example, waxes or resins dissolved in cleaners' naphtha, varies with both the type of fabric and with the state of electrification. For example, a piece of cotton fabric that has been charged positively and then immersed into a negatively charged solution of a resin, then dried and ironed, may retain a good water-repellency rating, whereas on the other hand, when the same cotton fabric is negatively charged and then immersed in a positively charged solution of the same resin, it may exhibit no water-repellency whatever. Generally, when solution and fabric are similarly electrified, very poor water-repellency is obtained following the same procedure. Further, it has also been found that when the garment and the treating solution are oppositely charged, some fabrics obtain a degree of water-repellency whereas when treated in the same manner, and wherein the liquid and the fabric were electrified with the same kind of charge (positive or negative) the results are just the reverse. Evidence, however, has been established that in such cases where the water-repellency is not effective, there is indication that the degree of similar electrification charges are not equal and that one or the other is either more negative or more positive so that the degree of electrification becomes relative.

It has further been observed that where the fabric being treated and the treating solution are both positively charged but where the fabric is more highly positively charged than the treating solution, the treating solution may function similarly as if it were negatively charged with respect to the positively charged fabric.

Electrification of both the solvent solution of wax, resins, etc., and of fabrics or the like that are being treated can be determined by means of an electrometer. The circumstances under which the fabrics and treating solution acquire static charges are not easily regulated and such statically charged fabrics and solutions are hazardous in dry cleaning operations where combustible and inflammable liquids are used.

It is one of the objects of the present invention to provide a method of treating fabrics either during dry cleaning or separately so as to render the fabric water-repellent.

Another object is to provide a method for carrying out the process in such a manner that neither the solution containing the water-repellent composition nor the fabrics being treated have, or take on, any electrification whereby the beneficial results of the process are obtained.

Another object is to provide an improved method of

treating fabrics or the like so as to make them water-repellent by utilizing the novel step during the treatment of removing the static electrical charge if any is present, or preventing the same from developing by grounding both the material being treated and the treating solution so that the same has zero of ground potential, and thus avoid the effects produced where either one or the other carries an electrical charge.

Another object is to provide a novel water-repellent solution which is especially useful in carrying out the process and which solution contains an electrical conducting agent whereby the static electric charge is removed.

It is still another object to provide a water-repellent composition which is useful in conjunction with dry cleaning and which may be mixed with cleaning solvent such as petroleum naphtha (Stoddard solvent).

It is a further object to provide a novel method for eliminating the static electrical charge from both the material being treated and the solvent solution containing the water-repellent agent whereby enhanced water-repellency of the material treated is obtained.

In accordance with the present invention, the inhibitory effect of static electrical charges during the process of treating the article is overcome for all practical purposes by causing the fabrics, or other material being treated to become conductive of static electricity before or during application of the water-repelling solution. The solutions of water-repellent agent or agents employed function as electrolytes, and which are conductive of electric current, whereby any static electrical charges are nullified or discharged when the fabrics or the materials being treated are immersed in the water-repellent solution.

Where the material to be rendered water-repellent is to be treated before subjecting the same to the water-repellent agent or solution, the fabric or other article may be sprayed with or dipped into a solution which is conductive of static electricity. Compositions or compounds for accomplishing this conductivity are hygroscopic substances such as glycerol, propylene glycol, urea and calcium chloride. Other conductive organic salts such as quaternary ammonium compounds may be utilized as the electrical conductive substance. The fabric or material being rendered water-repellent is first sprayed with or dipped into a solution containing the electrical conductive substance and thereafter the fabric is dried but not dehydrated. It is thereafter immersed, sprayed or otherwise made wet with the solvent solution comprising the water-repellent composition. Articles thus treated are then allowed to dry by evaporation.

Where it is desired to carry out the process without first rendering the fabric free from electrical charge, this may be accomplished by utilizing the water-repellent composition which contains an electrical conductive substance, whereby the static electrical charge is conducted away from the solution as well as the fabric. In this instance the process may be carried out as substantially a single step or combined as an additive step to the dry cleaning process.

Compounds containing substantial amounts of nitrogen are known to be anti-static; and solutions containing organic salts, such as the sodium salt of sulfonated mineral oil together with a very small amount of moisture or water, have been found to be very effective. Hydrated quaternary ammonium compounds also are very effective and the same may be dissolved in or suspended in the solvent solution of a water-repellent agent. Such solutions have been found to eliminate static electrical charges and cause the fabric being treated to acquire better water-repellency than fabrics treated in a similar solution containing the water-repellent agent, but without the presence of these anti-static materials.

Methods illustrative of how the invention may be carried out are as follows:

Example I

Solution 1.—20 grams of paraffin wax, (M. P. of 130–135) were dissolved in 200 grams of Stoddard petroleum solvent and into this was dipped an eight inch square piece of khaki poplin.

Solution 2.—Then, 20 grams of the same paraffin wax were similarly dissolved in 200 grams of the same solvent and to the solution was added 0.020 gram of N-soya-N-ethyl morpholinium ethosulfate. A similar piece of khaki poplin was dipped in this solution. The two pieces of cloth were simultaneously and equally centrifuged and dried. Water-repellency tests made upon these pieces by the spray test method described in the Military Specification (National Military Establishment Specification JAN-C–1068 dated April 18, 1949) revealed that the cloth treated as described in Solution 1 had a repellency rating of 40% and that the cloth treated in Solution 2 had a repellency rating of 60%.

The N-soya-N-ethyl morpholinium ethosulfate used is the quaternary ammonium compound made by Atlas Powder Company and supplied commerically in a 35% aqueous solution.

Stoddard solvent is a standardized fraction of petroleum or naphtha, which is used in dry cleaning or as a solvent. It is water white in color, and has a flash point above 100° F., consisting in the distillation fraction of which 50% distills below about 350° F. and the remainder not over about 410° F.

Example II

Solution 3.—20 grams of a commercial synthetic resin, sold under the name Alphez resin (a condensation product of pinene and phenol) where dissolved in 180 grams of Stoddard petroleum solvent. An eight inch piece of khaki poplin was dipped into this solution.

Solution 4.—Another similar solution of Alphez resin (20 grams) dissolved in Stoddard solvent (180 grams) was further processed by adding 0.05% by weight of N-soya-N-ethyl morpholinium etho-sulfate. A piece of similar khaki cloth eight inches square was then dipped into this solution.

The two pieces of cloth as treated in Solution 3 and Solution 4 were then simultaneously centrifuged to expel excess solution and were then air-dried. Water-repellency tests of these treated cloths indicated a zero rating for the cloth treated in resin alone (Solution 3) and a better than 50% rating for water-repellency of the cloth treated in the resin solution which contained the etho-sulfate compound.

Example III

Solution 5.—6 grams of Amberol resin (phenol formaldehyde resin) and 18 grams of paraffin wax (melting point about 130° F.) were dissolved in 186 grams of Stoddard solvent. An eight inch square of khaki poplin, as described above, was immersed in this solution.

Solution 6.—Another similar solution of the Amberol resin and paraffin wax was made as in Solution 5 to which was added 0.05% by weight on N-cetyl-N-ethyl morpholinium etho-sulfate. Into this solution was dipped an eight inch square of khaki cloth. The two pieces of cloth as treated respectively with Solution 5 and Solution 6 were then simultaneously and equally centrifuged to remove excess solution and thereafter air dried. Water-repellency tests, as described in Example I, were then made upon these two cloths and it was found that the cloth treated in Solution 5 had a water-repellency rating of 70% while the cloth treated in the solution containing etho-sulfate had a water-repellency rating of 100%.

Example IV

An eight inch square of khaki cloth was immersed in a solution containing 6 grams of Amberol and 8 grams of paraffin (melting point 130 to 135° F.) and 186 grams perchloroethylene, to which was added and thoroughly stirred 0.200 gram of water and 0.010 gram of sodium salt of sulfonated petroleum oil (mahogany sulfonate). This small amount of electrolyte was found sufficient to make the perchloroethylene solution conductive of elec-

tricity so that the piece of cloth thus treated after centrifuging and drying was found to have acquired a 100% water-repellency rating.

Example V

In this instance 0.20 gram of water and 0.005 gram of alkyl (C_9 to C_{15}) tolyl methyl trimethyl ammonium chlorides were stirred into a xylol solution of 5 grams of Amberol resin and 8.0 grams of penta erythritol tetra stearate, forming a treating solution. When khaki cloth pieces were dipped into this solution and centrifuged and dried as described above, it was found that the water-repellency was approximately 90% as compared with the repellency of 70% when a similar solution was used which did not contain the added chloride electrolyte. It was thus found that a cationic compound was effective when added to the dry cleaning solution, together with a very minute amount of water to render the solution of water-repellent material conductive to electricity and thus greatly enhance the water-repellency rating of cloths or fabric treated with such a solution containing the electrolyte.

In the preferred practice of the invention a combination of waxes and resins are used with the cleaners' solvent, such as Stoddard petroleum fraction. However, the particular wax and resin to be used in any given case may vary, depending upon the material being treated and the color and characteristic fiber of the material being made water-repellent. Further, for economic reasons the waxes and resins employed will necessarily be those commercially available and which are of low cost.

As examples illustrative of a suitable water-repellent composition, the following are given:

Example VI

	Parts by weight
Resin (a phenol-pinene condensate) (Alpha S)	10.6
Paraffin wax, 133–135° F., M. P.	8.0
Xylol	18.7
Petroleum solvent distillate (140° F. flash)	16.6
N-soya-N-ethyl-morpholinium etho-sulfate	0.1

The above composition represents a concentrated water-repelling solution which may be used by the dry cleaner for dry cleaning and rendering the cloth or fabric water-repellent by mixing one part of the above composition with six parts of Stoddard solvent.

Another example which is illustrative of a concentrated water-repelling solution for similar use with Stoddard solvent or cleaners' naphtha is as follows:

Example VII

	Parts by weight
Amberol (phenol formaldehyde condensation product)	10.0
Paraffin (133–135° F., M. P.)	8.5
Xylol	18.7
Petroleum solvent (140° F. flash)	16.0
N-soya-N-ethyl-morpholinium etho-sulfate	0.1

In the above formulations, instead of the straight phenol formaldehyde resin, similar synthetic resins of the phenol modified type may be employed such as those sold commercially and consisting of a chemical combination of phenol, formaldehyde, resin and glycerin.

In compounding the water-repellent solutions as described for use in treating fabrics and the like to render them water-repellent, it has been found that various types of surface active agents may be utilized as static conductive agents, but it is essential to be careful not to introduce surface active agents which possess such a high degree of wetting action that their effect is to make the fabric, or other material being treated, readily wet with water which would obviously defeat the purpose of this invention. In this connection it has been found that surface active agents suitable for the purpose of this invention and which render the solution conductive of static electricity without causing the fabrics to become sensitive to wetting with water are the quaternary ammonium compounds such as para di-isobutyl phenoxy ethoxy ethyl dimethyl benzyl ammonium chloride, and N-soya-N-ethyl morpholinium ethosulfate as aforementioned. N-cetyl-N-ethyl morpholinium ethosulfate (supplied commercially in a 35% aqueous solution) may likewise be used. Also, urea, propylene glycol, glycerol mono-laurate, and mono-sulfonated glycerol compounds are effective antistatic addition agents.

The quantities of these wetting agents which are needed for satisfactorily overcoming the effect of static electricity are of very small percentages in the composition, and the most practical range of their concentration has been generally found to be between about 0.001% to 0.050% of the weight of the treating solution. It has also been found that the usefulness of these antistatic additives or wetting agents is enhanced by the presence of small amounts of moisture. The solvent solution may be rendered saturated or partially so with moisture in order to have sufficient water present to enhance the results or the amount of the moisture in the electrolyte or wetting agent may be predetermined so as to have enough water present to increase the efficiency of the process. Moisture in the amount of as low as 0.001% has been found to be satisfactory for this purpose.

When the process is applied, for example, to cloths, fabrics, etc. the materials may be first rendered conductive to static electricity, as by treating the same with an electrically conductive substance as heretofore described, and prior to immersion or treatment with an organic solvent solution containing water-repelling agents to render the material water-repellent, as described. The water-repelling solutions which contain small percentage amounts of the static conductive compounds, and which are generally hygroscopic, are then utilized in wetting, spraying or otherwise treating the fabric or the like whereby the same is rendered water-repellent. On the basis of the weight of the treated fabric or other material being made water-repellent, it has been observed that additions of from about 0.001% to 0.10% by weight of the treating solution to the fabric is generally sufficient to provide the fabric with a satisfactory water-repellency. Additions of higher or lower amounts of the water-repellent solution, of course, may be required where the kind of fabric being treated necessitates it.

As a modification of the above process, it has been found that static electrical charges can be substantially removed or caused to be discharged from both the fabrics or other fibrous materials being treated, as well as the solvent solution of the water-repellent material by merely exposing the same to radiation from radio-active elements. In a like manner, it has also been noted that these static electrical charges may be eliminated by exposing the materials being treated as well as the solution during treatment to ultra-violet light. Accordingly, static electrical charges on fabrics as well as the treating solution may be eliminated or inhibited together with their undesirable effects by carrying out the process while subjecting the materials being treated and the treating solution to either radio-active radiation or ultra-violet light or to both concurrently, to further enhance the water-repellency of the fabrics or other materials being treated.

The invention is particularly useful in the treatment of garments during or after the dry cleaning treatment whereby the same are rendered water-repellent. The invention makes it possible for the first time to treat fabrics or similar fibrous materials so as to make them water-repellent. Further, by employing different electrolytes, the degree of water-repellency may be varied for different materials or pieces of cloth as desired. This has never been accomplished heretofore, insofar as known, in the prior processes.

In view of the foregoing description of this invention and the advantages set forth, it will be seen that there is provided a new method and new compositions for treat-

ing materials to render the same water-repellent. Further, it will be understood that various changes and substitutions may be made in the above methods and treating compositions described without departing from the spirit and scope of this invention. Such obvious variations, modifications and changes are contemplated as being within the scope of the present invention which is more particularly set forth in the appended claims.

What is claimed is:

1. A process for rendering articles made from fibrous materials water-repellent, said process comprising the steps of first treating said articles so as to render the same conductive of static electricity by applying thereto a solution which is conductive of electricity, said solution comprising a quaternary ammonium compound which does not render the fibrous articles readily wettable by water, dissolved in petroleum naphtha, and then applying to the thus treated fibrous material an organic solvent solution containing a water-repellent, said water-repellent comprising resin, wax and petroleum solvent.

2. A process for rendering garments, fabrics, paper, and articles made of fibrous material water-repellent which consists of the steps of applying to said articles a solution containing a substance which makes the solution conductive of static electricity and comprising a quaternary ammonium compound which does not render the fibrous articles readily wettable by water, thereafter applying to said articles which have been made conductive to static electricity a water-repelling solution, said water-repellent comprising resin, wax and petroleum solvent.

3. A process for rendering garments, fabrics, paper, and articles made from fibrous material water-repellent by the successive steps of (*a*) rendering said articles to be treated conductive of static electricity by the application thereto of a solution of an electrically conductive material, said solution comprising a quaternary ammonium compound which does not render the fibrous articles readily wettable by water, dissolved in petroleum naphtha, (*b*) immersing said treated material which has been made electrically conductive into a solution containing organic solvent and a water-repellent substance, said water-repellent comprising resin, wax and petroleum solvent.

4. A process for rendering garments, fabrics, paper, and articles made from fibrous material water-repellent by the successive steps of (*a*) rendering said articles to be treated conductive of static electricity by the application thereto of a solution of an electrically conductive material, said solution comprising a quaternary ammonium compound which does not render the fibrous articles readily wettable by water, dissolved in petroleum naphtha, (*b*) immersing said treated material which has been made electrically conductive into a solution containing petroleum organic solvent, paraffin wax and resin.

5. A process for rendering garments, fabrics, paper, and articles made from fibrous material water-repellent by the successive steps of (*a*) rendering said articles to be treated conductive of static electricity by the application thereto of an electrically conductive solution, said solution comprising a quaternary ammonium compound which does not render the fibrous articles readily wettable by water, dissolved in petroleum naphtha, and (*b*) immersing said treated material which has been made electrically conductive into a solution containing petroleum organic solvent, paraffin, resin and N-soya-N-ethyl morpholinium etho-sulfate.

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