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WATER-REPELLENT FABRIC

William J. Thackston, Haddon Heights, N. J., and
Sivert N. Glarum, Ardmore, Pa., assignors to
Röhm & Haas Company, Philadelphia, Pa., a
corporation of Delaware

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This invention relates to a composition which imparts to textile fibers, yarns, and fabrics a high degree of water repellency which is retained on laundering or dry cleaning. The invention deals further with the application of the composition to textile materials.

Water repellency imparted to textile materials by waxes or metal soaps from solvent solutions or emulsion has generally a high initial value, but decreases rapidly when the materials are laundered or dry cleaned. This deficiency in retention of water repellency has been lessened, yet not overcome, by multi-bath procedures and by variations in formulas. Among such variations, it has been proposed that various resins be applied. None of the resins heretofore proposed for this use, however, has imparted to the water-repellent finish the necessary degree of retention of water repellency during the use of treated textile fabric or upon the cleaning thereof.

Improved retention of water repellency is obtained in so-called integral treatment in which a hydrophobic group is reacted with textile fabric. This treatment, on the other hand, is difficult to apply, causes loss in tensile strength, and frequently alters the shades of dyes. Good repellency often disappears on prolonged wear or exposure, even with this type of finish.

An object of this invention is the provision of compositions suitable for imparting to textile fibers, yarns, and fabrics a high initial water repellency and a high degree of retention of repellency. Another object is to produce water-repellent textile fabrics which retain their repellent nature after wear, exposure, and cleaning. Yet another object is to impart water repellency which is well retained during wear and cleaning and which avoids the difficulties heretofore encountered.

The composition which has been found particularly effective for rendering textile materials water repellent is a water-in-oil emulsion containing a condensate of solvent-soluble urea, formaldehyde, and a monohydric aliphatic alcohol of at least four carbon atoms, solvent-soluble cellulose ether, wax, an aluminum soap, suitable organic solvents, and water. This composition is applied to textile fibers, yarns, or fabrics by immersing, padding, calendering, or otherwise impregnating the textile material, and heating the impregnated material above about 200° F. but below the scorching point of the impregnated

material to insolubilize the urea-formaldehyde-alcohol condensate.

In preparing the water-repellent composition, the active ingredients, condensate, cellulose ether, wax, and aluminum soap, are first taken up in organic solvents to give a formulation which is emulsified with water prior to application to textile material. The proportions of active ingredients found necessary are as follows: organic solvent-soluble urea-formaldehyde-alcohol condensate, 10 to 35 parts; solvent-soluble cellulose ether, 0.1 to 1 part; wax, 5 to 35 parts; and aluminum soap, 5 to 37 parts.

The condensates of urea, formaldehyde, and monohydric aliphatic alcohol are well known in the resin art. In waterproofing textiles, only those condensates are effective which are soluble in organic solvents, such as excess of the alcohol used in the preparation of the condensate, benzene, toluene, solvent naphthas, etc. It is necessary that the alcohol have at least four carbon atoms, as in butanol, hexyl alcohol, capryl alcohol, etc. Condensates of this type are available in concentrated solutions which may be utilized as such or may be diluted with such solvents as butanol, toluene, naphtha, or the like, to any desired concentration. The term "urea-formaldehyde-alcohol condensate" is used to describe not only those condensates in which urea is the sole resin-forming carbamide, but also condensates based upon the joint use of urea and such compounds as thio-urea, melamine, thioameline, and the like.

An additional resin-like material which has been found of considerable value in the compositions of this invention is solvent-soluble cellulose ether, such as ethyl cellulose. This product may be dissolved in a solvent such as toluene, or xylene, or in the solution of urea-formaldehyde-alcohol condensate or in a solution of the other ingredients of the water-repellent composition.

As other necessary ingredients, a water-insoluble wax and an aluminum soap are taken up in organic solvent. Suitable waxes include paraffin, montan wax, beeswax, hydrogenated waxes, etc. The aluminum soap may be the aluminum salt of any long-chained fatty acid, such as oleic, palmitic, stearic, arachidic, cerotic, etc., the hard, higher fatty acids being preferred.

All of the above ingredients are combined in one mixture to give a solvent solution, which may be further extended with a solvent such as an aromatic type of naphtha to yield, when mixed

with water, an emulsion of workable consistency. In mixing solvent solution and water, it is usually helpful to warm the solution, slowly add water, and stir until a homogeneous cream results.

A catalyst for hardening the urea-formaldehyde-condensate may be added to the solvent solution or preferably to the water. As catalysts there may be used small amounts of salts which yield acids, such as ammonium thiocyanate, ammonium phosphate, etc., or acids which will not tender the textile fibers to be treated, such as formic acid or acetic acid.

As a typical example of a composition found suitable for waterproofing, the following preparation is cited. 20 parts of paraffin wax, 20 parts of a petroleum naphtha, and 10 parts of butanol were warmed to 140° F. until the wax was taken up. The mixture was then cooled to 110° F. and 19 parts of aluminum stearate were added, followed by 6 parts of a 10% solution of ethyl cellulose in an aromatic hydrocarbon and 25 parts of a 50% solution of a urea-formaldehyde-butanol condensate in butanol and toluene. This mixture served as a stock paste.

In preparing an emulsion from this stock paste, 76.6 parts of the paste was placed in a mixing tank and stirred, while 104.4 parts of petroleum solvent was mixed therewith. Steam was then blown into the mixture to raise the temperature to 100° F. and a small amount of water was stirred in. There was then diluted with water 2.25 parts of acetic acid (56%), which was added to the mixture. In all, 230.4 parts of water were added.

Various types of cloth were then padded through the above emulsion and squeezed between wood and brass rolls to give a "pick up" of about 70% of their weight. In general, a pick up of 60% to 120% is satisfactory. The fabrics were then dried on a range dryer, cured at 320° F. for four minutes, washed in a soaper, rinsed, and dried on cans. Some of the fabrics were shrunk on a Palmer machine.

By the above procedure, water repellency was produced in the case of sulfur dyed mercerized 136 x 60, 4 yard broadcloth, a vat dyed mercerized twill, an all-rayon taffeta, a spun rayon and cotton fabric, a rayon warp satin, a mercerized poplin, and other fabrics, with production of high initial repellency and excellent retention of water repellency on cleaning.

All of these treated fabrics had an initial repellency of 100 on the scale recommended by the American Association of Textile Chemists and Colorists (American Dyestuff Reporter, vol. 36, No. 1, page P-6, 1941). After three wash tests made according to Federal Specification CCCT-191A, the repellency varied from 80 to 90 on the above scale, depending upon the construction and type of fabric. Other samples submitted to three dry cleaning tests all gave a repellency value of at least 80. It is of interest to note that pieces of the same fabrics treated with long-chained oxymethyl quaternary ammonium salts for integral water repellency gave values of 80 after three standard wash tests and of 70 after three dry cleaning tests.

In a modification of the above procedure, 25 parts of paraffin wax, 15 parts of aluminum stearate, and 20 parts of toluene were warmed together and 10 parts of amyl alcohol added thereto. There was then added 2 parts of a xylene solution of ethyl cellulose containing about 0.2-0.25 part of this ether and 28 parts of a 50% toluene-capryl-alcohol solution of urea-formal-

dehyde-capryl and butyl alcohol condensate. A portion of this preparation (42 parts) was diluted with 21 parts of solvent naphtha and emulsified with 84 parts of a 5% solution of a water-soluble urea-formaldehyde condensate and one-half percent of a catalyst mixture composed of 25% of hexamethylene tetramine, 10% of urea, 10% of sodium sulfate, and 55% of diammonium phosphate. A 184 x 96, 2.66 yards per pound, mercerized cotton twill was padded through the above emulsion, squeezed until 65% retention was obtained, dried, and heated at about 300° F. for about 5 minutes. The resulting fabric had a firm, full hand and exhibited high water repellency, both at the start and after repeated launderings.

When a piece of the above twill was treated with a similar emulsion of wax and aluminum stearate free from the urea-formaldehyde-alcohol condensate but containing the water-soluble urea-formaldehyde condensate, the resulting fabric was not fully water repellent at the start and completely lost all water repellency after three launderings.

An emulsion was prepared having the following final composition: 3.7% wax, 3.4% aluminum stearate, 2.3% urea-formaldehyde-butanol condensate, 3.9% butanol, 0.11% ethyl cellulose, 0.25% xylene, 29.8% solvent naphtha, 0.3% acetic acid, and the balance water. A 2.5 pound per yard cotton twill was padded through this emulsion with 65% pick up, dried, and heated at 280° F.-300° F. to insolubilize the condensate. The initial repellency value was 100, and the repellency value after three washes of 40 minutes each in a running suds of soap and soda ash at the boil was between 90 and 100. A sample of fabric soaked in a 10% solution of sodium hydroxide for one hour at room temperature and well rinsed retained its water repellency value of 100, while a piece of the same twill integrally waterproofed dropped in repellency to a value of 80.

Samples of the above twill rendered water repellent with the compositions here disclosed and a sample of integrally waterproofed twill were exposed in a frame to the weather over a period of several weeks, during which time they were subjected to snow, rain, smoke, and sunshine. The two types of samples were then laundered to remove soot and dirt and tested for repellency. The samples made according to this invention had repellency values of 80 and 90, while the integrally treated fabric had a value of 50.

Comparison was also made of the two types of water-repellent twills after abrasion in the Duffee Crockmeter. Water repellency values for the integrally treated piece were determined after 25, 50, 75, and 100 cycles to be 50, while the twill finished according to this invention gave values of 90, 85, 80, and 75 after 25, 50, 75, and 100 cycles, respectively. Abrasion resistance was also determined by the "T. B. L." abrasion test, which gave a value of 35,000 for the twill treated according to this invention and 24,000 for the integrally treated sample.

A piece of 80 x 80 cotton percale was finished by the method above described in connection with twill. Its initial repellency value was 100, which was retained for two wash tests, dropping to 90 after the third wash test.

An important advantage resulting from the use of the compositions here disclosed is the lack of action on dyes. Many types of water-

proofing treatments, particularly those in which amines or quaternary ammonium salts are involved, "throw" certain shades of naphthol dyes, vat dyes, and "salt" colors. Such severe alterations in shade are not encountered with the emulsions of this invention. Another advantage results from the ease with which the disclosed process is carried out. This process is effective on all types of textile fibers and fabrics, whether of vegetable or animal origin and whether natural or synthetic.

We claim:

1. A composition suitable for imparting water repellency to textile fibers, yarns, and fabrics which comprises a water-in-oil emulsion containing in the oil phase 10 to 35 parts of an organic solvent-soluble condensate of urea, formaldehyde, and a monohydric aliphatic alcohol of at least four carbon atoms, 0.1 to 1 part of organic solvent-soluble ethyl cellulose, 5 to 35 parts of a water-insoluble wax, 5 to 37 parts of an aluminum soap, and organic solvent therefor.

2. A composition suitable for imparting water repellency to textile fibers, yarns, and fabrics which comprises a water-in-oil emulsion containing in the oil phase 10 to 35 parts of a condensate of urea, formaldehyde, and butanol, 0.1 to 1 part of organic solvent-soluble ethyl cellulose, 5 to 35 parts of paraffin wax, 5 to 37 parts

parts of aluminum stearate, and organic solvent therefor.

3. The process of imparting to textile fabrics a high degree of water repellency which is retained on cleaning, which comprises padding textile fabric through a water-in-oil emulsion containing in the oil phase 10 to 35 parts of an organic solvent-soluble condensate of urea, formaldehyde, and a monohydric aliphatic alcohol of at least four carbon atoms, 0.1 to 1 part of organic solvent-soluble ethyl cellulose, 5 to 35 parts of a water-insoluble wax, 5 to 37 parts of an aluminum soap, and organic solvent therefor, removing excess emulsion from the padded fabric, drying it, and heating it to insolubilize the condensate thereon.

4. The process of imparting to textile fabrics a high degree of water repellency which is retained on cleaning, which comprises impregnating textile fabric with a water-in-oil emulsion containing in the oil phase 10 to 35 parts of a condensate of urea, formaldehyde, and butanol, 0.1 to 1 part of organic solvent-soluble ethyl cellulose, 5 to 35 parts of paraffin wax, 5 to 37 parts of aluminum stearate, and organic solvent therefor, and heating the impregnated fabric to insolubilize the condensate thereon.

WILLIAM J. THACKSTON.
SIVERT N. GLARUM.