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IMPREGNATING AGENT

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1 Claim. (Cl. 260—534)

The present invention relates to impregnating agents, especially for textile materials. Furthermore, it concerns a process which allows of rendering textile materials or the like hydrophobic.

According to the invention textile materials are impregnated with aqueous solutions or dispersions of carbamic acids containing higher aliphatic hydrocarbon radicals. The aqueous solutions or dispersions may also contain paraffins, waxes, fats, resin forming substances, salts of polyvalent metals, soaps of polyvalent metals or silicones. The textile materials treated with these solutions or dispersions are dried at temperatures above 100° C. The drying process may be carried out in two steps, viz. by pre-drying the textile materials at lower temperatures—at about 80–100° C.—and subsequently drying shortly at about 110° C. or at a higher temperature. Another embodiment of the invention consists in drying the impregnated textile materials, rinsing them with hot water and drying them once more, it being to be understood that the temperature must exceed 100° C. at least in one drying step.

The carbamic acids containing higher aliphatic hydrocarbon radicals, which are used according to the present invention, may be obtained in any suitable manner, e.g. by reacting the addition products of alkali metal bisulfites and higher aliphatic isocyanates with equivalent amounts of alkali metal hydroxides in an aqueous medium in the heat at temperatures not exceeding 90° C. The term "higher aliphatic hydrocarbon radicals" as herein used embraces aliphatic hydrocarbon radicals having an uninterrupted chain of at least eight carbon atoms as well as radicals whose carbon chain is interrupted by hetero atoms or groups of hetero atoms.

The impregnations obtained according to the present invention are fast to washing and solvents. Hydrophobic effects can be obtained by means of carbamic acids alone. The water repellent effect is improved by the complementary use of aqueous emulsions of paraffin and waxes; also soaps of polyvalent metals may be applied with equal success. As the soaps of polyvalent metals and the carbamic acids easily give rise to precipitates, the metal soaps are preferably applied after using the carbamic acids; in this case the drying step above 100° C. is performed after the application of the metal soaps. Alternatively, soap may be added to the carbamic acids or to the impregnating baths and the textile material is subsequently treated with water-soluble salts of polyvalent metals, for instance with aluminum salts such as aluminum acetate, preferably zirconium salts such as zirconium oxychloride adjusted to pH 4.0, zirconium acetate or zirconium formate. The treatment with salts of polyvalent metals results in an improved impregnating effect which is retained after washing with soap or with organic solvents.

The tendency of textile materials to wrinkle and shrink can be reduced by the addition of resin forming substances, such as methylol urea or melamine methylol

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ether, to the carbamic acids used according to the invention.

The following examples serve to illustrate the invention without, however, limiting it thereto; temperatures are in degrees of centigrade.

Example 1

25 g. of the addition product of sodium bisulfite and stearyl isocyanate are dissolved in half a liter of 85° hot water. After 10 minutes 100 cc. of water of 30° and, at 70°, 52 cc. of 1n sodium hydroxide solution are added; finally, after ten minutes, the emulsion is made up to 1 liter by adding water of 60°.

A rain coat material of non-mercerized cotton poplin is soaked with the above emulsion, squeezed so that the increase in weight is 60%, heated to 100° until it is dry and subsequently heated to 120° for a further 5 minutes. An excellent water repellent effect is obtained on the material which does not disappear even on repeated treatment with gasoline. The impregnated material has a soft feel.

Example 2

The emulsion prepared according to Example 1 is salted out by means of sodium chloride and the precipitate is washed with 5% sodium chloride solution and finally with water until the filtrate is free from sodium sulfite. The paste obtained is made into an emulsion at 85° by adding 400 cc. of hot water and the emulsion is filled up to ¾ liter by adding water of 40°.

Cotton poplin is soaked with the emulsion obtained, squeezed so that the increase in weight is 60%, dried first at 100° and afterwards heated to 120° for 5 minutes. The impregnated fabric is treated with hot water until it is completely wetted whereafter it is squeezed and finally dried at 110–115°.

The poplin possesses excellent water repellent properties which otherwise can only be obtained with impregnating agents containing paraffin. The water repellent effect is retained upon repeated washing with gasoline and carbon tetrachloride.

Example 3

An emulsion is prepared according to Example 2. Before salting out, however, 20 g. of an emulsion consisting of 4.85 g. of paraffin, 1.55 g. of an ester of montanic acid having the acid number 7.5, 0.95 g. of olein having the acid number 155, 0.95 g. of a 44% sodium hydroxide solution and 11.7 cc. of water are added to the starting emulsion.

Cotton poplin is soaked with this emulsion, squeezed so that the increase in weight amounts to 65%, shortly dried at 60° and thereafter wetted with an emulsion obtained by stirring a solution of 10 g. of zirconium oxychloride, 10 cc. of a 30% acetic acid and 8 g. of sodium acetate in 500 cc. of water into a solution of 5 g. of soap in 550 cc. of water, both solutions having a temperature of 60°. The cotton poplin is squeezed again and dried at 110° for 5 minutes.

The material possesses a water repellent effect which is fast to organic solvents. It is soft to the touch and fuller than material treated with the emulsion containing no zirconium soap.

The application of zirconium soap is particularly recommended for textile material of rayon, cellulose acetate and polyamides.

Example 4

Before making up the emulsion according to Example 1 to 1 liter it is mixed with a solution of 5 grams of soap in 100 cc. of hot water and 20 grams of the paraffin wax-emulsion mentioned in Example 3.

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Textile materials are impregnated with the emulsion and squeezed. After that they are treated without drying with a solution of 15 grams of zirconium oxychloride in 1 liter of water, which is mixed with 15 cc. of 30% acetic acid and 12 grams of sodium acetate. The textile materials are again squeezed, dried at 100° and finally heated to 120° for 5 minutes.

The textile materials are soft and full to the touch and possess an excellent water repellent effect which is fast to washing with soap and organic solvents.

Example 5

A solution of 25 grams of the addition product of stearyl isocyanate and sodium bisulfite are dissolved in 300 cc. of water of 65° is mixed with 26 cc. of 2n sodium hydroxide solution. The mixture is cooled and the precipitated stearyl carbamic acid is filtered off and washed with water. Thereafter, the stearyl carbamic acid is added to 400 cc. of boiling water and the emulsion obtained made up to ¾ liter by the addition of water of 50°.

Cotton poplin is impregnated with the emulsion obtained, squeezed and dried shortly at 100° and at 115-120° for further 5 minutes. Thereafter the textile material is rinsed with boiling water until it is completely wetted and dried at 110°.

The poplin possesses a very good water repellent effect which is retained after washing five times with gasoline or carbon tetrachloride.

Example 6

An emulsion which contains per liter 25 grams of stearyl carbamic acid and 2 cc. of 30% acetic acid and which is heated to 60° for about 30 minutes is applied to cotton poplin.

The impregnated material is squeezed so that the increase in weight amounts to 60-70% and dried, first at about 100° and then at 120° for a further 5 minutes.

The impregnated cotton poplin which is soft and full to the touch possesses excellent water repellent properties which are fast to repeated washing with the gasoline or carbon tetrachloride.

By rinsing the textile material subsequently with boiling water until it is completely wetted, and drying at a temperature above 110°, the water repellent effect is still increased.

The stearyl carbamic acid can be obtained in the following manner:

50 grams of the addition product of sodium bisulfite and stearyl isocyanate are dissolved in 500 cc. of water at 85° and, after 10 minutes, mixed with 200 cc. of water of 20° within 1 minute, 125 cc. of 1n sodium hydroxide solution are added to the solution of 70° and the temperature is kept at 63° for 5 minutes. The emulsion obtained is mixed with 50 cc. of saturated sodium chloride solution and cooled to 25°. The precipitate formed is filtered off, washed first with diluted sodium chloride solution and then shortly with water. The paste obtained is digested with 6 times the amount of hot water and mixed at 65° in portions with 1n sodium hydroxide solution until the pH-value of the milky white emulsion is increased to 7.5 and is not lowered again. The emulsion is then cooled to 18°. The pulpy mass is coagulated with saturated sodium chloride solution, filtered and the residue is washed first with 3% sodium chloride solution until no sulfite can be detected in the filtrate any longer, and then with cold water until the filtrate becomes bluish

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turbid. The reaction product obtained contains 95% of stearyl carbamic acid.

Example 7

5 Cotton poplin is impregnated with the emulsion, described in Example 2, after addition of 40 grams of dimethylol urea and 2 cc. of 30% acetic acid. The poplin is squeezed so that the increase in weight amounts to 65%, dried at 100° and 135° for 5 minutes. The poplin is water repellent and distinctly less liable to wrinkling.

Example 8

15 50 grams of an aqueous paste, which contains 15 grams of stearyl carbamic acid and 2 grams of the addition product of sodium bisulfite and stearyl isocyanate, are heated with 200 cc. of water to 95° for 5 minutes, then diluted with water to 500 cc., and the emulsion is added slowly with good stirring at 30° to 500 cc. of a 2% solution of zirconium acetate prepared by dissolving zirconium hydroxide in acetic acid.

20 Rain coat material of cotton gabardine is impregnated with the emulsion obtained, squeezed and dried at 100° and at 120° for 5 minutes.

25 The fabric possesses a water repellent effect which is retained after repeated treatment with gasoline.

Example 9

30 40 grams of the aqueous paste containing stearyl carbamic acid of Example 8 are heated with 300 cc. of water to 98° and mixed with 160 cc. of an emulsion of 80° which contains 25 g. of the paraffin-wax-olein emulsion described in Example 3. The mixture is cooled to 30° and then slowly added with good stirring to a solution of 12 grams of zirconium acetate (prepared from zirconium hydroxide) in 500 cc. of water of 30°. The pH of the emulsion is brought to 4.0 by means of acetic acid and the emulsion is warmed at 40°.

35 A poplin fabric of pure cotton is soaked with the emulsion, squeezed and dried, first at 95° and then at 125° for 5 minutes.

40 The poplin possesses a water repellent effect which is substantially retained after washing five times with gasoline and ironing.

45 Instead of pure cotton fabrics also fabrics containing, besides cotton, 30% of rayon, or fabrics of cellulose acetate, wool or natural silk may be treated according to the present invention with equal success. Fabrics of polyamide fibers can also be impregnated with the new agents after rinsing them with hot water. All the impregnated fabrics are full and soft to the touch.

I claim:

50 An impregnating agent comprising stearyl carbamic acid in an aqueous medium.

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