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PROCESS OF IMPARTING HYDROPHOBIC PROPERTIES TO CELLULOSE FIBERS

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The present invention relates to a process of imparting hydrophobic properties to cellulose fibers.

This application is a continuation-in-part of our co-pending U. S. patent application Serial No. 81,122, filed May 21, 1936.

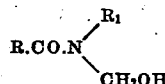
It is known that fibers of cellulose or hydrated cellulose, such as cotton, artificial silk or staple fiber are extraordinarily hydrophil, that is to say they are very quickly wetted when in contact with water. This property is a great disadvantage for many of the applications of this material.

This invention consists in a process for making cellulose fibers hydrophobe by applying to the fibers or incorporating with them a compound which contains at least one aliphatic or cycloaliphatic residue having at least 4 carbon atoms and capable of reacting with aldehyde and exposing the fibers thus treated simultaneously or subsequently to the action of an aliphatic aldehyde or dialdehyde, for instance, formaldehyde or glyoxal.

Suitable compounds are, for example, fatty amines and fatty acid amides such as dodecylamine, hexadecylamine octadecylamine, octadecenylamine, the amides of carboxylic acids, for example, abietic acid amide, lauric acid amide, stearic acid methylamide, stearic acid butylamide, stearic acid octadecylamide, stearic acid amide, montanic acid amide, paraffin carboxylic acid amide, dichlorostearic acid amide, phenylstearic acid amide, furthermore alkylbenzoic acid amides and the nuclear hydrogenation products thereof, for instance, p-dodecylbenzoic acid amide and dodecylcyclohexylcarboxylic acid amide. Moreover, there may be named such acid amides wherein the aliphatic chains are interrupted by aromatic or hydroaromatic radicals, for instance, dodecylphenylbutyric acid amide, dodecylcyclohexylbutyric acid amide, furthermore octylphenoxyacetic acid amide and dodecylphenoxypropionic acid amide; further compounds of the character of alkyl-substituted ureas such as mono-isobutyl urea, monododecyl urea, mono-octadecyl urea, stearyl urea; further fatty acid imino ethers or amidines. There also come into consideration compounds containing hydroxyl groups, such as, for instance, dodecylphenol, di-isohexyl-phenol.

Instead of the products named above there may also be used with advantage their products of reaction with aliphatic aldehydes, for example, their methylol compounds. If these latter are used the separate after-treatment with formaldehyde can be omitted in many cases, a simple after-heating operation sufficing.

The reaction products of carboxylic acid amides with formaldehyde, for instance, have the general formula:



wherein R stands for the radical attached to the carboxylic acid amide group and containing at least one aliphatic or cycloaliphatic residue having at least 4 carbon atoms, R₁ stands for a member of the group consisting of hydrogen and aliphatic hydrocarbon radicals.

The process may be conducted, for example, as follows: the cellulose fibers are first saturated with a solution of the aliphatic compound in an organic solvent, for instance, pyridine, acetone or in an aqueous emulsion of the aliphatic compounds. They are then dried and exposed to the action of, for instance, formaldehyde. The formaldehyde may be added to the impregnating solution so that a subsequent treatment with formaldehyde becomes unnecessary. In this case the fiber needs only subsequent heating to ensure sufficient reaction of the formaldehyde with the impregnating compound and the fiber.

In many cases the hydrophobe effect is essentially enhanced if the impregnation is carried out in the presence of an agent having a feebly acid action, for example, acetic acid, lactic acid or agents of acid reaction such as sodium bisulfite. The impregnation may be carried out in an aqueous medium as well as in an organic solvent, such as benzene, carbon tetrachloride, pyridine and the like. An agent having a feebly acid action may be added during the impregnation, or it may be incorporated with the fiber by a pre- or after-treatment.

Fibers treated in this manner are characterized by a particularly high stability towards water and even hot soap solution. For example, such material can be subjected to the usual fulling treatment, that is to say heating for ½

hour in a solution of 50 grams of soap and 5 grams of sodium carbonate per liter at 50° C. without loss of the property of repelling water. By suitable combination of the parent material and working conditions an effect can be obtained which is not notably diminished by several washings with boiling soap solution.

An alternative method of incorporating the material in the fibers consists in adding the compound in question as such or its condensation product with aldehyde to the spinning solution which is to serve as the parent material for making artificial cellulose threads. The fibers produced from solutions containing such an addition may, if desired, be subsequently treated with an aliphatic aldehyde.

The following examples serve to illustrate the invention, but they are not intended to limit it thereto, the parts being by weight:

(1) Unsoaped, dry viscose silk is immersed for 5 minutes in 5 times its weight of a solution of 5 to 10 per cent. strength of methylol-stearinamide in pyridine. The goods are centrifuged, dried and heated for 15 hours at 110° C.

(2) Dry viscose artificial silk is treated for 15 minutes with a solution of 5 grams of a condensation product, obtainable from stearic acid amide and aqueous formaldehyde, in one liter of carbon tetrachloride. The material is squeezed and dried at the air. Thereupon it is after-treated at room temperature for 15 minutes with a solution of 5 grams of lactic acid in 1 liter of water, squeezed, dried in a current of air and heated for 2 hours at 110° C. The material is finally well rinsed and dried.

(3) Non-dyed or dyed viscose artificial silk is treated with a solution which contains per liter of water 5 grams of lactic acid or glycolic acid, and is then dried. This pre-treated material is treated for 10 minutes at 60° C. to 70° C. with a solution of a condensation product, obtainable from montanic acid amide and aqueous formaldehyde, in carbon tetrachloride. The material is squeezed, dried and heated for some minutes at a temperature of 140° C. There is thus obtained a hydrophobe tissue having a very good water-repellant effect.

(4) A cotton fabric is treated for a short time with an aqueous lactic acid solution of 1 per cent. strength and then dried at 50° C. to 60° C. It is subsequently impregnated for 10 minutes with a solution of 1 per cent. strength of 9.10-dichlorostearic acid methylolamide in cyclohexane, centrifuged and heated at 135° C. to 140° C. for 30 minutes.

(5) Artificial silk crepe is impregnated with an alcoholic solution of 2 per cent. strength of dodecylbenzoic acid methylolamide. It is then squeezed, dried at 50° C. and heated for 1 hour at 140° C.

(6) Artificial silk crepe is treated for some minutes in an aqueous emulsion of p-dodecylphenylbutyric acid methylolamide, containing per liter 10 grams of the methylolamide and 5 grams of lactic acid. It is subsequently squeezed, dried at 50° C. to 60° C. and heated for 30 minutes at 130° C. to 140° C.

(7) Cotton calico is impregnated for a short time with a solution of 2 per cent. strength of the condensation product of dodecylcyclohexylbutyric acid amide and formaldehyde in carbon tetrachloride. Then it is squeezed and heated for 1 hour at 140° C.

(8) Dyed artificial silk is impregnated with an alcoholic solution containing per liter 5 grams of

maleic acid and 20 grams of isooctylphenoxyacetic acid methylol amide. After centrifuging the whole is heated for 1 hour at 120° C. to 130° C.

(9) 15 grams of beta-naphthoxyacetic acid-methylolamide are dissolved in 1000 grams of alcohol and 10 grams of lactic acid are added to this solution. Artificial silk fabric is treated in the bath so prepared for ½ hour at 60° C. to 70° C. Then it is squeezed, dried at about 50° C. and subsequently exposed to a temperature of 140° C. for 60 minutes in a drying cylinder or in a tentering frame or in a drying oven. The fabric has become hydrophobe. The effect is enhanced by washing the fabric in a soap solution rendered alkaline by means of sodium carbonate and subsequent drying.

With a still better success there may be used a bath prepared in an analogous manner and containing 10-15 grams of iso-octyl-beta-naphthoxyacetic acid-methylolamide.

Instead of viscose artificial silk, cellulose fibers of all kinds, for instance, copper artificial silk, artificial spinning fibers, cotton, also mixtures of wool and cellulose fibers as well as fabrics made from the said fibers and mixtures of fibers may be used. In some cases a preliminary cleansing or desizing of the fibers or fabric is advisable.

We claim:

1. The process of imparting hydrophobic properties to cellulose fibers by incorporating with them an N-methylol compound of an acid amide of a monocarboxylic acid and ammonia which contains at least one radical with at least 4 carbon atoms selected from the group consisting of aliphatic and cycloaliphatic radicals and subsequently heating them.

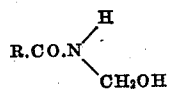
2. The process of imparting hydrophobic properties to cellulose fibers by impregnating the fibers in a bath which contains an N-methylol compound of an acid amide of a monocarboxylic acid and ammonia having at least one radical with at least 4 carbon atoms selected from the group consisting of aliphatic and cycloaliphatic radicals and heating them after the impregnation.

3. The process of imparting hydrophobic properties to cellulose fibers by impregnating the fibers in a bath which contains an N-methylol compound of an acid amide of a monocarboxylic acid and ammonia having at least one radical with at least 4 carbon atoms selected from the group consisting of aliphatic and cycloaliphatic radicals, and an agent of acid action such as a feeble acid, and heating them after the impregnation.

4. The process of imparting hydrophobic properties to cellulose fibers by impregnating the fibers with a condensation product from formaldehyde and an acid amide of a monocarboxylic acid and ammonia which contains at least one radical with at least 4 carbon atoms selected from the group consisting of aliphatic and cycloaliphatic radicals, and heating them after the impregnation.

5. The process of imparting hydrophobic properties to cellulose fibers by impregnating the fibers with a condensation product from formaldehyde and an acid amide of a monocarboxylic acid and ammonia which contains at least one radical with at least 4 carbon atoms selected from the group consisting of aliphatic and cycloaliphatic radicals, in the presence of an agent of acid action such as a feeble acid and heating them after the impregnation.

6. The process of imparting hydrophobic properties to cellulose fibers by impregnating the fibers with a compound of the general formula:



10 wherein R stands for an aliphatic radical with at

least 4 carbon atoms, in the presence of a feeble acid and heating them after the impregnation.

7. Waterproof cellulose fibers obtained by the process defined in claim 1.

8. Waterproof cellulose fibers obtained by the process defined in claim 6.

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