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FINISHING TEXTILE MATERIAL WITH POLYAMIDE DISPERSIONS

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8 Claims

ABSTRACT OF THE DISCLOSURE

Improved process for finishing textile material with an aqueous polyamide dispersion which comprises: impregnating the textile material with an aqueous polyamide dispersion in which the polyamide consists essentially of a cocondensate of caprolactam, the salt of hexamethylene diamine with adipic acid and the salt of 4,4'-diaminodicyclohexylmethane with adipic acid, the dispersion containing, as a protective colloid, about 0.5 to 5% by weight with reference to the weight of the polyamide of a water-soluble copolymer having a K value of at least 90 and derived essentially from acrylic acid, methacrylic acid or mixtures thereof and a mixture of acrylamide and methacrylamide; and then drying and heating the impregnated material to at least 100° C.

The present invention relates to an improved method of finishing textile materials of all types, particularly those which contain native or regenerated cellulose, with aqueous polyamide dispersions containing protective colloids.

It is known that aqueous polyamide dispersions which contain protective colloids may be used to finish textile material. For example dispersions have been described for this purpose which contain, as polyamides, those which contain condensed units of dicarboxylic acids having aliphatic side chains containing at least six carbon atoms and, as protective colloids, copolymers of methacrylamide, vinyl-pyrrolidone and if desired N-vinylimidazole. These dispersions give very good effects but they have the disadvantage that coatings obtained therefrom have a very small moisture absorptivity. Impregnated textile materials therefore do not soften enough in water; during washing they retain stiff edges which can easily cause damage to the material.

On the other hand if an attempt be made to increase the water absorptivity of these finishing agents, their resistance to washing is generally decreased at the same time. It has not yet been possible with completely satisfactory results to produce textile finishes with polyamide dispersions containing protective colloids which combine to the desired extent the opposed properties of good water absorptivity and high fastness to washing.

It is the object of this invention to finish textile material of any desired fibers by impregnation with a polyamide dispersion so that the polyamide is fixed washproof to the textile material but the textile material nevertheless retains good water absorptivity.

Another object of the invention is to produce textile material impregnated with polyamides which in the dry condition is stiff but on being wetted with water becomes soft and flexible and recovers its original stiffness when dried again.

These and other objects are achieved according to this invention by impregnating the textile material with an

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aqueous polyamide dispersion which contains as the polyamide a cocondensate of:

25 to 40% by weight of one or more lactams having six to twelve carbon atoms in the lactam ring, or equivalent omega-aminocarboxylic acids;

25 to 40% by weight of one or more salts of hexamethylene diamine and linear dicarboxylic acids having six to twelve carbon atoms;

25 to 40% weight of one or more salts of 4,4'-diaminodicyclohexylmethane and linear dicarboxylic acids having six to twelve carbon atoms and

0 to 10% by weight of other polyamide-forming substances, and as the protective colloid a water-soluble copolymer having a K value of at least 90 and derived from:

10 to 35% by weight of acrylic acid and/or methacrylic acid;

57.5 to 90% by weight of a mixture consisting of acrylamide and

20 5 to 40% by weight, on the total monomers, of methacrylamide; and

0 to 7.5% by weight of one or more other monomers.

Examples of lactams or omega-aminocarboxylic acids having six to twelve carbon atoms are caprolactam, oenantholactam, capryllactam, capric lactam, laurilactam, the related omega-aminocarboxylic acids and also omega-aminoundecanoic acid. Caprolactam is preferred because of its easy availability. Linear dicarboxylic acids having six to twelve carbon atoms are adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, undecane dicarboxylic acid and dodecane dicarboxylic acid. Particularly good results are obtained by using adipic acid.

In addition to these building blocks which have to be contained within the stated ranges in the polyamides used according to this invention, up to 10% by weight (on the whole polyamide) of other polyamide-forming substances may be present. Examples of these are dicarboxylic acids, omega-aminocarboxylic acids or diamines which are branched or contain a hetero atom in the chain, and C-substituted lactams.

The copolymers serving as protective colloids may contain, in addition to the abovementioned monomers, up to 7.5% (on the total weight of the monomers used) of units of other polymerizable monomers. Examples of these are other unsaturated carboxylic acids, such as α -chloroacrylic acid, phenylacrylic acid and crotonic acid, unsaturated dicarboxylic acids, such as maleic acid, fumaric acid, itaconic acid, citraconic acid and mesaconic acid, vinylsulfonic acid and derivatives of the said acids, such as anhydrides, salts and esters, the esters of acrylic acid and methacrylic acid themselves, such as methyl acrylate, ethyl acrylate, methyl methacrylate and ethyl methacrylate, as well as water-soluble compounds, such as vinylacetamide, N-dimethylacrylamide, N-methylacrylamide, N-ethylacrylamide and vinyl lactams. Particularly preferred protective colloids contain 0.005 to 1% (on the weight of all the monomers) of copolymerizable monomers containing N-methylol groups, such as derivatives of urea, melamine, dicyandiamide and guanidine containing N-methylol groups and polymerizable groups, and particularly N-methylolmethacrylamide or N-methylolacrylamide.

The copolymers serving as protective colloids should have a K value of at least 90, measured according to Fikentscher, Cellulosechemie 13 (1932) 60.

The K values of the copolymers serving as protective colloids are advantageously from 90 to 170, and copolymers having K values of from 125 to 155 are particularly suitable.

Particularly good results are achieved by using as the

protective colloid a water-soluble copolymer which has been prepared from: 20 to 22% by weight of acrylic acid and/or methacrylic acid; 53 to 60% by weight of acrylamide; 25 to 18% by weight of methacrylamide and, optionally, 0.01 to 1% by weight of N-methylol-methacrylamide. These preferred protective colloids may also contain up to 3% by weight of units of other monomers, particularly esters of acrylic acid and methacrylic acid with ethanol or preferably methanol.

The copolymers serving as protective colloids may be present in the dispersions in amounts of 0.5 to 5%, preferably 1 to 2.5% (on the weight of the polyamides).

Production of the polyamides may be carried out by conventional polycondensation of the abovementioned starting components. The protective colloids may also be made by conventional methods, for example by polymerization of a mixture of the abovementioned monomers in the presence of a mixture of the abovementioned monomers in the presence of water-soluble peroxides, such as potassium persulfate or ammonium persulfate as catalysts and triethanolamine, sodium thiosulfate, sodium sulfoxylate or ascorbic acid as activators.

The dispersions to be used according to the invention may be obtained for example by mixing a solution of the polyamide in an organic solvent with water which contains the protective colloid and if desired distilling off all or part of the solvent. An advantageous procedure is to dissolve the polyamide in a solvent, preferably a water-soluble alcohol or mixture of alcohols, such as ethanol, isopropanol, butanol or mixtures thereof with each other and/or with water, heating the solution to the boiling point of the solvent and distilling off the solvent, while at the same time the aqueous solution of the protective colloid and the amount of water or organic solvent for setting up the desired concentration are slowly added to the reaction mixture. Other possibilities for the production of the dispersions are explained in the examples.

Polyamide dispersions of the abovementioned type may be used for numerous finishing methods for textile material, particularly for coating fibers, filaments and threads, for finishing, coating, laminating and bonding textile sheet material and for bonding non-woven fabrics. The dispersion may contain additional constituents depending on the purpose for which they are to be used; examples of such constituents are dispersions of polyolefins or polyacrylates or other polymer dispersions or solutions, water repellents, dyes, pigments and emulsion thickening agents and particularly organic solvents and aminoplast-forming substances, as for example reaction products, optionally etherified with alcohols containing up to four carbon atoms, of formaldehyde with urea, melamine or cyclic urea derivatives, as for example with ethylenurea, trimethylenurea, 4,5 - dihydroxyimidazolidone - (2), triazinone-(2) and its 5-alkyl and 5-hydroxyalkyl derivatives having up to four carbon atoms in the alkyl or hydroxy-alkyl radical, 5-hydroxytrimethylenurea and urones. These aminoplast-forming substances may be monomeric substances or slightly condensed resinous products.

The polyamide dispersions to be used according to this invention are applied to the material to be finished in the conventional way, for example by dipping, spraying, pouring or brushing. The concentration and rate of application of the dispersions is chosen in the conventional way so that the amount of polyamide applies to the textile material conforms to the desired purpose. The amount of polyamide applied is in general 2 to 5% by weight (on the weight of the dry textile material) for stiff finishes; it may be higher, for example 8 to 15% by weight, for the production of laminations. The material thus treated is dried and heated to at least 100° C., preferably to 100° to 150° C. Finishes of unusual resistance to the stresses encountered in use and extremely high resistance to water are obtained in this way.

Water resistance is not however obtained at the ex-

pense of an inadequate water absorptivity and the associated disadvantages of the kind described above.

The invention is illustrated by the following examples in which parts and percentages are by weight.

EXAMPLE 1

Preparation of the dispersion:

2000 parts of a polyamide (prepared by conventional polycondensation of a mixture of equal parts of caprolactam, hexamethylene diamine adipic acid salt and 4,4'-diaminodicyclohexylmethane adipic acid salt) is dissolved in 2000 parts of ethyl alcohol. Then, while stirring, a solution of 26 parts of a copolymer of 60 parts of acrylamide, 18 parts of methacrylamide and 22 parts of acrylic acid having a K value of 125 and 1 part of triethanolamine in 104 parts of water and then 2200 part sof water are added separately.

Another 2500 parts of water is added in the course of about five hours while heating at 80° to 100° C. and at the same time about 3000 parts of a mixture of ethanol and water is distilled off.

This dispersion which contains about 40% of the polyamide and about 0.5% of the protective colloid is diluted to a polyamide content of 20 to 25% by stirring in butanol.

800 parts of water is added to 200 parts of this dispersion. Cotton cloth having a shrinkproof finish is padded with this dilute dispersion, the liquor retention being about 60%. The cloth is dried at 90° to 100° C., to a residual moisture content of 7% and then heated for five minutes at 130° C. A cloth is obtained which has a wash-fast stiff finish which is such that the cloth becomes soft during washing but regains its original stiffness after drying.

Even better results are achieved when the protective colloid used is a copolymer which contains, in addition to the abovementioned components, 0.01% by weight (on the whole of the monomers) of units of N-methylolmethacrylamide.

EXAMPLE 2

A dispersion prepared as described in the first two paragraphs of Example 1 is applied by means of a floating knife to a cotton cloth having a shrinkproof finish, so that the amount applied (reckoned as dry material) is about 10% of the weight of the cloth. While the coating is moist, a second strip of cloth is applied and the doubled material is pressed onto a steel calender whose rolls are heated to 100° C.

After the cloth material thus treated has been heated, a laminated material is obtained whose stiffness and bond has high resistance to washing.

EXAMPLE 3

The procedure of Example 2 is followed but the polymeric dispersion is replaced by one which has been prepared by the following method:

2000 parts of polyamide (prepared by conventional polycondensation of a mixture of 40% of caprolactam, 35% of hexamethylene diamine adipic acid salt and 25% of 4,4'-diaminodicyclohexylmethane adipic acid salt) is dissolved in 2000 parts of ethyl alcohol. Then while stirring at 75° to 80° C., a solution of 26 parts of a copolymer of 57.5% of acrylamide, 0.5% of methylol-methacrylamide, 20% of methacrylamide, 2% of methacrylic acid and 20% of acrylic acid which has a K value of 125 in 2304 parts of water is added during about one hour. While heating at 80° to 100° C., another 2500 parts of water is added in the course of about five hours and at the same time about 3000 parts of a mixture of ethanol and water is distilled off. And about 40% polyamide dispersion is obtained which is adjusted to a content of 20 to 25% by stirring in 2500 parts of n-butanol and if necessary more water.

Material laminated with this dispersion has very good resistance to washing.

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EXAMPLE 4

The procedure of Example 1 is followed but the polymer dispersion used is prepared by the following method:

1775 parts of polyamide (prepared by a conventional polycondensation from a mixture of 30 parts of caprolactam, 40 parts of hexamethylene diamine adipic acid salt and 30 parts of 4,4'-diaminodicyclohexylmethane adipic acid salt) is dissolved in a mixture of 1050 parts of n-butyl alcohol, 1250 parts of isopropyl alcohol and 500 parts of water while stirring intensely at 75° to 85° C. Then at about 70° to 75° C. in the course of forty-five to sixty minutes a mixture of 3665 parts of water and 240 parts of a 15% aqueous solution of a copolymer having a K value of 150 and derived from 22% of acrylic acid, 18% of methacrylamide, 59.3% of acrylamide and 0.7% of N-methylolmethacrylamide is added. The dispersion obtained is cooled to about 25° C. and a mixture of 590 parts of an aqueous 30% dispersion of a resinous reaction product from 1 mole of melamine and 6 moles of formaldehyde, which has been etherified with butanol in the molar ratio of 1:6 (as aminoplast-forming substance), 500 parts of water, 80 parts of a copolymer of 50% of acrylonitrile and 50% of methylacrylate (which is incompletely hydrolyzed with ammonia) and 350 parts of n-butyl alcohol is stirred in.

Cloth treated with this dispersion has a highly water-resistant boilproof stiff finish which is not lost even after repeated washing in a washing machine followed by drying.

EXAMPLE 5

The procedure of Example 2 is followed but a dispersion is used which has been prepared as follows:

300 parts of a polyamide (prepared by conventional polycondensation of a mixture of 28% of caprolactam, 32% of hexamethylene diamine adipic acid salt and 40% of 4,4'-diaminodicyclohexylmethane adipic acid salt) is dissolved in a mixture of 150 parts of isopropyl alcohol, 90 parts of n-butyl alcohol and 60 parts of water. While stirring at a temperature of 75° to 85° C., a solution of 337 parts of water and 40 parts of a 15% aqueous solution of a copolymer of 53.2 parts of acrylamide, 24 parts of methacrylamide, 20 parts of acrylic acid, 1 part of methyl acrylate, 1 part of methyl methacrylate and 0.8 part of N-methylolmethacrylamide, having a K value of 107, is added in the course of about thirty minutes.

315 parts of water is then added within thirty minutes. The whole is cooled to 25° to 30° C. Finally 210 parts of n-butyl alcohol is slowly stirred in.

Cloth treated with this dispersion has a good stiff finish with good resistance to washing.

We claim:

1. In a process for finishing textile material with an aqueous polyamide dispersion as the finishing agent, the

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improvement comprising impregnating the textile material with an aqueous polyamide dispersion containing, as the polyamide, a cocondensate of:

25 to 40% by weight of caprolactam,

25 to 40% by weight of the salt of hexamethylene diamine with adipic acid, and

25 to 40% by weight of the salt of 4,4'-diaminodicyclohexylmethane with adipic acid, and

as a protective colloid, 0.5 to 5% by weight, with reference to the weight of the polyamide of a water-soluble copolymer having a K value of at least 90 and derived from:

10 to 35% by weight of a member selected from the group consisting of acrylic acid, methacrylic acid and mixtures thereof,

57.5 to 90% by weight of a mixture consisting of acrylamide and 5 to 40% by weight, on the whole of the monomers, of methacrylamide, and 0 to 7.5% of other monomers, and drying and heating the impregnated material to at least 100° C.

2. A process as claimed in claim 1 wherein the protective colloid used contains 0.005 to 1% by weight of units of N-methylolacrylamide or N-methylolmethacrylamide.

3. A process as claimed in claim 1 wherein the protective colloid used has a K value of 90 to 170.

4. A process as claimed in claim 1 wherein the protective colloid used has a K value of 125 to 155.

5. A process as claimed in claim 1 wherein the protective colloid used contains as other monomers units of methyl acrylate, methyl methacrylate and N-methylolmethacrylamide.

6. A process as claimed in claim 1 wherein the aqueous polyamide dispersion contains an aminoplast-forming substance.

7. Textile material which has been finished by the process as claimed in claim 1.

8. Cellulosic textile material which has been finished by the process as claimed in claim 1.

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