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TEXTILE FINISHING PROCESS

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This invention relates to a textile finishing process, 15 more particularly to a process for imparting a durable, stiff finish to polyamide textile materials.

It has been proposed to impart stiff finishes to polyamide textile materials by applying thereto an aqueous solution of polyacrylic acid and then heating the textile 20 material so treated in order to render the finish resistant to washing. The aqueous solutions of polyacrylic acid used for this purpose, however, are strongly acidic and so cause considerable corrosion of the metal padding and drying equipment used in the process. Attempts to 25 neutralise some of the acidity prior to application to the polyamide, using ammonia, sodium hydroxide or organic amines, impair the durability of the finish, and beyond about 25% neutralisation it is difficult to obtain any fixation whatsoever. It has also been proposed to use 30 polyacrylic acid in conjunction with a polyhydric alcohol to obtain durable stiff finishes on polyamide textile materials, but the aqueous solutions used for this purpose are still acid, and moreover are not satisfactorily stable on storage, and possess a marked tendency to develop 35 mould-like growths.

Surprisingly, it has now been found that these disadvantages may be overcome, and that a durable, stiff, crisp finish can be imparted to polyamide textile materials by treating them with an aqueous solution of the 40 ammonium salt of an acrylic acid polymer and a polyhydric alcohol, optionally with an acid catalyst, and then drying and baking the material thus treated. Furthermore, it has been found that the stiff finish so produced is retained even after severe alkaline scouring 45 treatment.

Thus according to the present invention there is provided a process for finishing polyamide textile materials which comprises applying to the said textile materials an aqueous solution containing the ammonium salt of an 50 acrylic acid polymer, a polyhydric alcohol, and optionally an acid catalyst, and thereafter heating the textile material at an elevated temperature.

The textile materials for treatment by the process of the present invention may be made from any of the polyamide materials known to be useful for the manufacture of textile materials, for example polyhexamethylene adipamide and polymers from caprolactam, and may be in the form for example of filaments, fibres or yarns, which may be woven into fabrics.

The acrylic acid polymer may be made by polymerising acrylic acid, either by action of heat or catalytically, but there may also be used a polymer obtained by alkaline hydrolysis of polyacrylonitrile or an alkyl ester of polyacrylic acid followed by conversion of the alkali metal salt of the polymer of acrylic acid so formed into the free acid (for example by treatment with an ion exchange resin). Suitable polymers for use in the process of the present invention are those which form solutions having viscosities of from 10 to 250 centipoises, and preferably from 15 to 50 centipoises, at 20° C. when

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50 grams of the polymer are dissolved in sufficient 2 N ammonium hydroxide to give 1 litre of solution.

The ammonium salt of the acrylic acid polymer may conveniently be made by neutralising the acrylic acid polymer with ammonia gas or ammonium hydroxide. If desired, an excess of ammonia may be used, so that the pH of the solution may lie between 7.0 and 9.5, but is preferably between 7.5 and 8.0.

As examples of polyhydric alcohols which may be used in the process of the invention there may be mentioned diethylene glycol, glycerol, pentaerythritol, mannitol and polyvinyl alcohol.

Suitable acid catalysts include substances which become more acidic on heating at elevated temperatures, for example ammonium salts, such as ammonium dihydrogen phosphate, diammonium hydrogen phosphate, ammonium chloride and ammonium thiocyanate, or mineral acid salts of certain amines and amides, for example pyridine hydrochloride and urea nitrate.

The aqueous solution to be applied to the polyamide textile material may contain between 10 and 100 parts, and preferably between 15 and 50 parts, of the polyhydric alcohol for each 100 parts of the ammonium salt of acrylic acid polymer. The proportion of acid catalyst may be up to 20 parts, and preferably between 10 and 20 parts, per 100 parts of the ammonium salt of the acrylic acid polymer.

The concentration of ammonium salt of acrylic acid polymer, polyhydric alcohol and acid catalyst may vary considerably, depending on the particular textile material being treated, the proportion of solution retained by the material, and the particular finish desired, but should be such as to provide between 0.5 and 10%, preferably between 2% and 5%, by weight of the ammonium salt of the acrylic acid polymer on the textile material.

The application of the solution to the polyamide textile material may conveniently be carried out by passing the textile material through the solution at atmospheric temperatures, though elevated temperatures may also be employed, and then removing the excess of solution by squeezing, and then drying the material and heating, for example at a temperature between 150° C. and 220° C. for a time which may be as short as 10 seconds or as long as 30 minutes, depending on the temperature employed. The finish may be fixed, for example under conditions similar to those used for heat-setting polyamide fabrics.

The finish obtained by the process of the present invention depends mainly upon the particular type of textile material employed. Thus, for example, when there are applied solutions containing the lower concentrations of the ingredients to continuous filament nylon fabric, a less slippery and fuller handle is imparted to the fabric so treated. The presence of an acidic catalyst to the solution enhances the stiffening effects obtainable, and also improves the durability of the finish.

When the process is used for the treatment of polyamide yarns, the solution may be applied to the yarn and dried thereon at normal drying temperatures for example from 50° C. to 110° C. and may thereby be utilised as a size for knitting or weaving the yarn into fabric. The fabric may then be heated for example at 150° C. to 220° C.

When the process is used for the treating of polyamide textile materials before they are dyed, the finish acts as a mordant for basic and onium dyestuffs. When the process is used after dyeing, little change in the shade of the dyeing occurs, though in certain cases the colour may appear a little stronger. In many instances the dyes on the treated fabric have improved fastness to washing and to light.

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Solutions of the ammonium salts of acrylic acid polymers are non-acidic in reaction, and remain non-acidic at normal drying temperatures, and therefore their application to textile materials by conventional methods does not involve the possibility of acid corrosion of metal equipment. Also, on storage, aqeuous solutions of the ammonium salts of acrylic acid polymers and, for example, glycerol do not develop mould growths as do solutions of free polyacrylic acid and glycerol.

The invention is illustrated but not limited by the following examples in which the parts are by weight.

Example 1

100 parts of 2 oz. nylon twill fabric is passed through a solution containing the ammonium salt of 4.5 parts of 15 an acrylic acid polymer and 1.5 parts of glycerol in 94 parts of water at 20° C. The fabric is squeezed until the amount of solution retained is 76% of the dry weight of the fabric, and then dried at 50° C. and baked in an oven at 150° C. for 30 minutes. The nylon fabric so treated 20 has a stiff, crisp finish which is retained after washing the fabric in an aqueous solution containing 5 parts of soap and 2 parts of soda ash per 1000 parts of solution for 15 minutes at 100° C. The fabric so treated and washed is found to have a flexural rigidity, measured at 20° C. and 25 55% relative humidity by use of a Shirley stiffness tester (British Standards Handbook, No. 11, page 125), 4.5 times that of the untreated fabric. The finish is retained after the treated fabric is dry-cleaned by tumbling in white spirit for 15 minutes.

The ammonium salt of the acrylic acid polymer, used in the process of the above example was made by catalytic polymerisation of acrylic acid (using ammonium persulphate as catalyst) and subsequent addition of sufficient 27% aqueous solution of ammonia to give a solution of pH 7.5. This ammonium salt formed a solution in 2 N aqueous ammonium hydroxide having a viscosity of 15.5 centipoises at 20° C. when 50 grams (calculated as acrylic acid polymer) are dissolved in sufficient 2 N ammonium hydroxide to give a total volume of 1 litre.

Example 2

100 parts of nylon twill fabric are passed through a solution containing 0.3 part of glycerol and the ammonium salt of 1 part of an acrylic acid polymer used in Example 1, in 98.7 parts of water at 20° C. The fabric is dried, heated and washed as in Example 1, and then has a stiff handle resembling taffeta, and a flexural rigidity 2.5 times that of the untreated material.

Example 3

100 parts of nylon twill are treated as described in Example 1, except that the fabric is baked in an oven at 175° C. for 7 minutes. The fabric has a stiff, paperlike handle, and after washing, the fabric has a flexural rigidity of 4.5 times that of the untreated material.

Example 4

100 parts of nylon twill are treated as described in Example 3, except that the solution of glycerol and the ammonium salt of the acrylic acid polymer contains in addition 1.0 part of diammonium hydrogen phosphate. The treated fabric has a stiff, paper-like handle, and after washing has a flexural rigidity 5.0 times that of the untreated material.

Example 5

100 parts of nylon twill are treated as described in Example 1, except that the fabric is heat-set on a hot plate at 200° C. for 20 seconds. The fabric, after washing, has a stiff, crisp handle, and has a flexural rigidity 3.8 70 times that of the untreated material.

Example 6

100 parts of nylon twill are treated as described in Example 1 except that the solution of glycerol and the am- 75

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monium salt of acrylic acid polymer used in Example 1 to impregnate the fabric is replaced by a solution containing 5 parts of glycerol and the ammonium salt of 10 parts of the acrylic acid polymer in 85 parts of water. The treated fabric has a stiff, crisp handle. After washing, the material has a flexural rigidity 3.0 times that of the untreated fabric.

Example 7

100 parts of nylon lace are passed through a solution containing 1.5 parts of glycerol and the ammonium salt of 4.5 parts of the polymer of acrylic acid described in Example 1. The fabric is squeezed until the amount of solution retained is 100% of the weight of the fabric, and is then dried at 50° C. and baked in an oven at 175° C. for 7 minutes. The treated lace has a stiff handle which is retained after washing.

Example 8

100 parts of a 1 oz. nylon marquisette are passed through a solution containing 2 parts of pentaerythritol and the ammonium salt of 6 parts of the acrylic acid polymer described in Example 1 in 92 parts of water. The fabric is squeezed until the amount of solution retained is 79% of the weight of the cloth, and is then dried at 50° C. and baked in an oven at 175° C. for 7 minutes. The treated fabric has a stiff, crisp finish which is retained after washing the fabric.

Example 9

100 parts of nylon plain weave fabric are passed through a solution containing 2 parts of diethylene glycol and the ammonium salt of 6 parts of the acrylic acid polymer described in Example 1 and 92 parts of water. The fabric is squeezed until the amount of solution retained is 79% of the weight of the fabric and is then dried at 50° C. and baked at 175° C. for 7 minutes. The treated fabric has a stiff, papery handle which is retained after washing.

Example 10

100 parts of nylon twill fabric are passed through a solution containing 1.5 parts of glycerol and 4.5 parts of the ammonium salt of an acrylic acid polymer in 94 parts of water. The fabric is squeezed until the amount of solution retained is 70% of the weight of the fabric, and is then dried, baked at 175° C. for 7 minutes. After washing the fabric, which has a crisp, papery handle, the flexural rigidity of the material is 4.8 times that of the untreated fabric.

The ammonium salt of the acrylic acid polymer used in this example was made by catalytic polymerisation of acrylic acid, using ammonium persulphate as catalyst, and subsequently adding sufficient 27% aqueous solution of ammonia to give a solution of pH 7.5. This ammonium salt formed a solution in 2 N ammonium hydroxide having a viscosity of 214 centipoises at 20° C. when 50 grams (calculated as acrylic acid polymer) are dissolved in sufficient 2 N ammonium hydroxide to give 1 litre of solution.

Example 11

Spun nylon yarn is slashed with a solution containing 0.7 part of glycerol and the ammonium salt of 2 parts of the polymer of acrylic acid described in Example 1. The yarn is dried by passage through a tube heated at 110° C. and is then knitted into fabric, which is baked for 4 minutes at 180° C. The fabric has a full, crisp handle, which is retained after washing.

Example 12

100 parts of continuous filament nylon twill fabric is treated as described in Example 1, except that the solution of glycerol and the ammonium salt of acrylic acid polymer used is replaced by a solution containing 0.03 part of glycerol and the ammonium salt of 0.1

part of the acrylic acid polymer in 100 parts of water. The treated fabric has an improved handle, in that the "slippery" feel of the fabric is reduced.

What I claim is:

- 1. A process for imparting a durable, stiff finish to 5 polyamide textile materials which comprises treating said materials with an aqueous solution consisting essentially of the ammonium salt of an acrylic acid homopolymer, water, and a polyhydric alcohol, removing excess solution from the fabric, and drying and heating 10 the impregnated material at an elevated temperature above 150° C.
- 2. A process for imparting a durable, stiff finish to polyamide textile materials which comprises treating said materials with an aqueous solution having a pH between 7.0 and 9.5 and consisting essentially of the ammonium salt of an acrylic acid homopolymer, water, and a polyhydric alcohol, removing the excess solution from the material, and drying and heating the impregnated material at an elevated temperature above 20 150° C.
- 150° C.

 3. A process for imparting a durable, stiff finish to polyamide textile materials which comprises treating said materials with an aqueous solution consisting essentially of the ammonium salt of an acrylic acid homopolymer which forms a solution having a viscosity of from 10 to 250 centipoises at 20° C. at a concentration of 50 grams per liter of solution in 2 N ammonium hydroxide, water, and a polyhydric alcohol, removing excess solution from the material, and drying and heating the impregnated material at an elevated temperature above 150° C.
- 4. The process of claim 3, wherein said viscosity is within the range from 15 to 50 centipoises.
- 5. The process of claim 4, wherein the pH of said solution is between 7.0 and 9.5.
- 6. The process of claim 5, wherein said pH is between 7.5 and 8.0.
- 7. A process for imparting a durable, stiff finish to polyamide textile materials which comprises treating said materials with an aqueous solution consisting essentially of the ammonium salt of an acrylic acid homopolymer, water, and a polyhydric alcohol, wherein the

weight ratio of said polyhydric alcohol to said acrylic acid homopolymer is from 1:10 to 1:1, removing the excess solution from the material, and drying and heating the impregnated material at an elevated temperature above 150° C.

8. The process of claim 7, wherein said ratio is between 1.5:10 and 5:10.

9. A process for imparting a durable, stiff finish to polyamide textile materials which comprises treating said materials with an aqueous solution consisting essentially of the ammonium salt of an acrylic acid homopolymer, water, a polyhydric alcohol and an acid catalyst, wherein the weight ratio of said polyhydric alcohol to said acrylic acid homopolymer is within the range of 1:10 and 1:1 and the weight ratio of said acid catalyst to said acrylic acid polymer is within the range of from 1:10 to 2:10, removing excess solution from the material, and drying and heating the impregnated material at an elevated temperature above 150° C.

10. The process of claim 9, wherein the finish obtained has a weight between 0.5% and 10% of the weight of said taylile materials.

of said textile materials.

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11. The process of claim 10, wherein said weight of said finish is between 2% and 5%.

12. As an article of manufacture, a dry polyamide textile material coated with a durable stiff finish, retained as a coating on said textile after alkaline scouring treatment, which coating consists essentially of from 0.5 to 10% by weight of said textile material of the ammonium salt of an acrylic acid homopolymer deposited from an aqueous solution consisting essentially of said ammonium salt of said acrylic acid homopolymer, water, and a polyhydric alcohol, and heat treated at an elevated temperature above 150° C.

References Cited in the file of this patent UNITED STATES PATENTS

	1,976,679	Fikentscher et al Oct. 9,	1934
•	2,651,587	Rossin Sept. 8,	1953
	2,692,182	Nuessle et al Oct. 19,	1954
	2,715,590	Brockman et al Aug. 16,	1955

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