SIZING COMPOSITIONS CONTAINING ACRYLAMIDE AND DIMETHYLOLATED ACETYLENE MONO-UREINE

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9 Claims

ABSTRACT OF THE DISCLOSURE

A cellulose textile dressing composition is provided comprising a condensate of one mole of dimethylolated acetylene-mono-ureine and 0.01 to 0.10 mole acrylamide, and a catalyst of glyoxylic acid.

This is a continuation-in-part of application Ser. No. 290,556.

The present invention relates to improvements in sizing compositions containing acrylamide and acetylene-mono-ureine.


In fact, the treatment of the above basic product with formaldehyde results in methylolated derivatives (AMU/AA-m), the sizing effects of which on textile are greater than those of known and valued sizing products such as dimethylolated acetylene-mono-ureine (AMU-m).

The methylolated products (AMU/AA-m) can be prepared in situ, for example by simultaneous condensation of urea with glyoxal (the two being constituents of acetylene-mono-ureine), acrylamide and formaldehyde.

Such in situ preparation may be considered as industrially advantageous since it reduces the cost of the AMU/AA-m product. However, acrylamide still remains a relatively costly product and the amount used in the reaction is large since one gram-molecule of acrylamide should be present for one gram-molecule of acetylene mono-ureine.

An object of the invention is to provide a highly effective yet inexpensive sizing for cellulose textile materials.

I have now found that the textile sizing properties of the dimethylolated acetylene-mono-ureine (AMU-m) can be much improved with acrylamide by applying the latter in an amount much lower than one gram-molecule of acrylamide for one gram-molecule of AMU-m.

The textile sizing product of my invention is consequently essentially dimethylolated acetylene mono-ureine modified by incorporating acrylamide in an amount less than one gram-molecule of acrylamide for one gram-molecule of dimethylolated acetylene mono-ureine.

In accordance with one aspect of the invention, the acrylamide associated with dimethylolated acetylene mono-ureine is in a free state or in a combined state.

In accordance with still another aspect, the amount of acrylamide used is advantageously between 0.08 and 0.1 gram-molecule per gram-molecule of the dimethylolated acetylene mono-ureine.

The sizing product according to the invention may be easily prepared in situ, by condensation of urea with glyoxal (constituents of acetylene-mono-ureine); followed by condensation with formaldehyde and with a suitable amount of acrylamide, for example, n/10 gram-molecule (moles) of acrylamide or less, for a gram-molecule (moles) of AMU-m; there is thus formed in situ a very low proportion (equal to or less than n/10 gram-molecule) of AMU/AA-m, the rest of the mixture being constituted by dimethylolated acetylene mono-ureine (AMU-m).

According to a preferred embodiment of the invention, the process for preparing a sizing product comprises condensing a molecular equivalent of urea with a molecular equivalent of glyoxal, then with 0.08 to 0.1 molecular equivalent of acrylamide and a sufficient amount of formaldehyde for obtaining a complete methylolation, formaldehyde being used in slight excess with respect to the amount theoretically necessary for obtaining such methylolation.

Alternatively, one molecular equivalent of urea is condensed with one molecular equivalent of glyoxal, then with a sufficient amount of formaldehyde for obtaining a complete methylolation, the formaldehyde being used in a slight excess with respect to the amount theoretically necessary for obtaining such methylolation. Dimethylolated acetylene mono-ureine is then obtained; the latter is then mixed at ambient temperature with a suitable amount of acrylamide, advantageously from 0.08 to 0.1 molecular equivalent.

A further object of the invention is to provide a process for treating cellulose textile materials with the above described sizing product, such process being essentially characterized by the fact that, in order to bind said sizing product, a catalyst constituted by glyoxylic acid or by a composition containing glyoxylic acid is used.

Examples of compositions which may be used include, besides solutions of glyoxylic acid itself, a mixture of glyoxylic acid with zinc nitrate and trichloro acetic acid or the like.

According to an advantageous manner for carrying out this treatment process, the solution containing the active sizing compound is brought up to the desired dilution by adding water after which the catalyst is dissolved therein together with other desired additives. The textile material is then impregnated with the thus obtained solution and then the remainder process treatment is carried out in a conventional manner.

In order to make the invention more clearly understood, some examples of its application will be given below by way of illustration but not in any limitative sense.

Example 1 as given below concerns the preparation, in situ, in accordance with the invention of a size constituted by dimethylolated acetylene mono-ureine chemically combined with a small amount of acrylamide.

Example 2 as given below concerns the preparation, in situ, and in accordance with the invention, of a size comprising dimethylolated acetylene mono-ureine and a small amount of free acrylamide.

Example 3 concerns the preparation of a control size constituted by dimethylolated acetylene mono-ureine without any acrylamide.

Example 1

In 400 gms. (2.0 mols.) of a solution of glyoxal at 30%, its pH being brought up to 6.1 by the addition of an alkaline solution, there is dissolved, while stirring, 124 gms. (2.0 mols.) of urea, the temperature being maintained between 18° and 20° C. The reaction medium is allowed to stand for from 8 to 12 hours at ambient temperature. To the resulting solution 460 gms. of a solution of formaldehyde (40% by volume—36.5% by weight) are added. The pH of the solution is brought up to 8.3—8.4 by the addition of hydrated barium hydroxide. The temperature is from 18° to 20° C. Next 12.25 gms. (0.172
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mols) of acrylamide and 4.5 gms. of triethanolamine are added. The mixture is heated to 60° C. and this temperature is maintained for 4 hours. During the reaction the pH must be 8.3—8.4. The mixture is then cooled to 20° C. and is then neutralized with a stream of CO₂. The pH, after neutralization, is from 7.9—8.0. The mixture is then filtered.

1000 gms. of a clear, substantially colourless, liquid are obtained having a content of 40% of dry extract; density; 1.17—1.18; free formaldehyde content; 5 to 6%.

Example 2

Example 1 is repeated except that acrylamide is not condensed by heating with dimethylolated acetylene-mono-urea. It is simply introduced at the end of the run.

Example 3

Example 1 is repeated but in the absence of acrylamide.

TEXTILE TREATING TESTS

Test series I

(A) A bleached cotton poplin, double twisted, is impregnated by padding with an aqueous solution containing 125 gms. per litre of the condensation product of Example 1. Then 18% by weight (i.e., 18 gms. per 100 gms. of dry resin) of a catalyst having the following formula is added:

Catalyst I

Zinc nitrate .............................................................. 39.2
Acetic acid ............................................................... 0.8
Water ................................................................. 60.0

70% of the liquid is then wrung from the textile. It is frame-dried at 120° C. and the condensation is carried out over a period of 3 minutes at 150° C. in a highly ventilated wind-tunnel.

Table I below gives the results obtained with tests carried out on this textile.

The "non-creasing" of the fabric is determined with the DAM apparatus in accordance with I.T.F. specifications which are quite similar to those of AATCC 66—1959T specifications. Generally speaking, this property is expressed by the value of a residual angle of the fabric previously folded under standard conditions.

The washings are effected in a washing machine provided with a reciprocating drum over a period of 10 minutes at 80° C. For this purpose a mixture of soap and sodium carbonate is used.

The mechanical strength measured according to French Standard Specifications AFNOR G 07—001 and is expressed by the force in kg. required to break a test sample of fabric of 5 cm. in width.

For determining the chlorine-resistance the scorch tester apparatus is used in accordance with the AATCC92 specifications. In accordance with this test samples are treated with hypochlorite then, after drying, they are heated between the two plates of the scorch tester at 180° C. The loss of mechanical strength is then measured and is expressed as % loss with respect to treated samples not submitted to the scorch tester.

These results show that the addition of acrylamide improves performance and that a condensation of the amide with acetylene-mono-urea and formaldehyde is required in order that addition plays its beneficial role to the maximum. On the other hand these results show the superiority of Catalyst II over Catalyst I.

Test series III

The resistance of the sizes to acidic hydrolysis was studied using the following method:

The fabrics are passed in baths containing 160 gms. per litre of the methylated derivatives corresponding to the above examples in the presence of catalyst I or catalyst II.

For washing, various buffered aqueous solutions were used such as—

\[ pH \, 1.9: \]

\[ \text{Urea} \] .................. 50

\[ \text{Phosphoric acid} \] .................. 15

\[ \text{pH} \, 3: \]

\[ \text{Sodium dihydrogen phosphate plus phosphoric acid} \] .................. 20

\[ \text{pH} \, 5: \]

\[ \text{Concentrated acetic acid plus sodium hydroxide solution} \] .................. 20

The treatment is carried out at 80° C. during a period of 30 minutes in the above acidic solutions.

Table II gives the angles of crease in the dry state of fabrics which have been sized and submitted to said acidic treatment together with the loss of the non-creasing properties expressed in % with respect to the gain of the non-creasing properties obtained with the size without acidic treatment (as such).

\[ \text{Loss} \% = \frac{\text{angle as such} - \text{angle after washing}}{\text{angle as such}} \times 100 \]

\[ \text{angle for the untreated fabric} \]
This shows, in a general manner, the superiority of the condensation product containing acrylamide added as in Example I associated with catalyst II.

Test series IV

The action of chlorine on these fabrics which have been submitted to an acidic washing step under the conditions specified was applied in accordance with the AATCC92--1962 specifications and evaluated in the scorch tester. The results obtained are given in Table III.

Here again, a lower loss of resistance is noted in the presence of acrylamide and a marked improvement noted with catalyst II.

Example 4

Example 1 is repeated except that 0.6 mole of acrylamide is used instead of 0.172 mole. Results similar to those shown in Tables I, II, and III for the product of Example 1 are obtained.

Example 5

Example 2 is repeated except that 0.6 mole of acrylamide is used instead of 0.172 mole. Results similar to those shown in Tables I, II and III for the product of Example 2 are obtained.

What is claimed is:

1. A dressing composition for textiles consisting essentially of dimethylated acetylene-mono-urea and acrylamide, said acrylamide being present in an amount between 0.01 and 0.10 moles per mole of dimethylated acetylene-mono-urea.

2. A composition in accordance with claim 1 wherein said acrylamide is present in an amount between 0.08 and 0.10 moles per mole of dimethylated acetylene-mono-urea.

3. A dressing composition for textiles consisting essentially of a condensate of one mole of acetylene-mono-urea, a molar excess of formaldehyde and 0.01 to 0.10 of acrylamide, and a catalyst of glyoxylic acid.

4. The method of making a dressing composition comprising condensing urea with glyoxal to provide acetylene-mono-urea, condensing from 0.01 to 0.10 mole of acrylamide with each mole of acetylene-mono-urea, and then condensing the reaction product of acrylamide and acetylene-mono-urea with a molar excess of formaldehyde.

5. The method of making a dressing composition comprising condensing glyoxal with urea and then with a molar excess of formaldehyde to form dimethylated acetylene-mono-urea, and then adding 0.01 to 0.10 moles of acrylamide to each mole of condensed product at room temperature.

6. A method in accordance with claim 4 wherein the molar proportion of acrylamide is 0.08 to 0.10.

7. A method in accordance with claim 5, wherein the molar proportion of acrylamide is 0.08 to 0.10.

8. The method of treating cellulose textile material consisting essentially of contacting said textile material with a bath comprising a molar proportion of dimethylated acetylene-mono-urea, 0.01 to 0.10 moles of acrylamide, and a catalyst comprising glyoxylic acid.

9. A method in accordance with claim 8, wherein said acrylamide is present in an amount between 0.08 and 0.10 moles per mole of dimethylated acetylene-mono-urea.

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