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AQUEOUS TEXTILE FINISHING COMPOSITION CONTAINING METHYLOL HIGHER FATTY ACID MONOAMIDES

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This invention relates to textile finishing compositions and particularly to compositions which impart water-repellency and dimensional stability to fabrics which are treated therewith.

There has been a demand in the textile trade recently for cellulosic fabrics which are water-repellent, crease-resistant, dimensionally stable and which have a firm hand. Water-repellency may be obtained by the use of a number of known hydrophobic materials, one of which is methylol stearamide. A variety of resins may be applied in order to secure the desired degree of dimensional stability. The various water-repellent agents are insoluble in water and must be applied either from non-aqueous solutions or from aqueous dispersions. The use of aqueous media is preferable from the standpoint of economy and convenience, but is often not possible because of the difficulty in preparing stable aqueous dispersions of the water-repellent agents.

It is an object of this invention to provide new compositions in the form of stable aqueous dispersions which are inexpensive and easy to apply to textile fabrics, and which produce durable water-repellency and dimensional stability in the fabric. A further object is to provide a process by which such stable aqueous dispersions are prepared. Further objects will appear from the detailed description which follows.

The compositions of this invention consist of aqueous dispersions of methylol fatty acid amides, polyvinyl alcohol and preferably a water-soluble heat-setting amide-formaldehyde resin. According to the process of this invention, these compositions are prepared by: (1) forming a dispersion of methylol fatty acid amides by heating together at 60 to 100° C. under vigorous agitation (a) a fatty acid amide mixture which melts between 90 and 100° C. and in which the total content of stearamide and palmitamide exceeds 80% by weight, (b) water and (c) a molecular excess of formaldehyde, during a portion of which heating step at least the fatty acid amides and water are agitated together at a temperature between the melting point of the amide mixture and 100° C. to emulsify the amides, and during a subsequent portion of which the dispersed fatty acid amides, formaldehyde and water are agitated at pH 7.5 to 9 and at 75 to 100° C.; (2) filtering off the methylol fatty acid amides and washing out the excess formaldehyde from the filter cake; (3) mixing the filter cake with from 10 to 30% polyvinyl alcohol in aqueous solution and with from 2 to 5% of a water-soluble long-chain alcohol sulfate containing from 12 to 18 carbon atoms,

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the percentages in each case being based on the dry weight of methylol fatty acid amides in the filter cake; and (4) mixing the resulting composition with from 1 to 15 parts by weight of a water-soluble heat-setting amide-formaldehyde resin per part of methylol fatty acid amides.

The fatty acid amide mixture which is employed as a starting material is preferably the mixture which is sold as "technical stearamide."

This product contains small amounts of free stearic acid and of stearonitrile, up to about 10%, from 50 to 95% stearamide and the balance palmitamide. Amide mixtures containing large amounts of the amides of acids having shorter chains do not provide a sufficient amount of water-repellency to be useful in this connection. Such materials may be present so long as more than 80% of the amide mixture consists of stearamide, palmitamide or a mixture thereof. In order to emulsify the fatty acid amide properly with water, it is necessary that the melting point of the amide mixture be less than 100° C. in water. Although pure stearamide is reported to melt at 109° C. and pure palmitamide at 106–107° C., the technical grade stearamide melts at about 93–98° C. in water.

A molecular excess of formaldehyde must be employed in order to obtain substantially complete reaction of the fatty acid amide. Some formaldehyde is lost by volatilization and some is decomposed by the disproportionation which takes place in alkaline media to give methanol and a formate. It is ordinarily suitable to use a 25 to 40% excess of formaldehyde over the theoretical requirement. A much larger excess does no harm to the product but produces no corresponding advantage.

The amount of water present during formation of the methylol fatty acid amide dispersion is not critical so long as enough is added to give a stirrable slurry. A convenient amount is from 3 to 5 parts of water per part of fatty acid amide.

The aqueous dispersion of methylol fatty acid amide is prepared by heating together at 60 to 100° C. under vigorous agitation the fatty acid amide mixture, the formaldehyde and the water. In an early portion of this step, the fatty acid amide mixture and water are heated together at a temperature above the melting point of the amide mixture and below the boiling point of water, in order to emulsify the amides. Formaldehyde may be present during this emulsification. There is no need to control the pH at this point.

After the fatty acid amide mixture is thoroughly dispersed, the mixture is heated with

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formaldehyde at a pH of 7.5 to 9, which is conveniently obtained by the addition of caustic soda. The mixture is heated under vigorous agitation until reaction appears to be complete and a smooth homogeneous dispersion is obtained. Alkalinities above pH 9 are operable but the loss of formaldehyde through disproportionation becomes substantial and economically undesirable. At least the latter part of this step is carried out at 75 to 100° C.

Considerable variation in the time of heating and the temperature schedule is permissible so long as the over-all heating period includes the emulsification period at a temperature between the melting point of the amide mixture and 100° and the subsequent reaction period under alkaline conditions at 75 to 100° C. Thus in one convenient and preferred embodiment of the process, the amide and water are heated together at about 93 to 100° C. until the amide mixture is thoroughly emulsified, the temperature is then allowed to drop to about 65°, formaldehyde is added, caustic soda solution is added until the pH is in the desired range, and the mixture is agitated at this temperature to effect a substantial degree of reaction. The temperature is then increased to about 80° and the mixture is agitated at this temperature until reaction appears to be complete. By adding the formaldehyde at the lower temperature, evaporation losses are reduced. On the other hand, the higher temperatures may be employed throughout and it is not necessary that the emulsification and the subsequent reaction with formaldehyde be carried out as separate steps. Thus a suitable dispersion is obtained by mixing the amide, water and formaldehyde together, adjusting the pH to the required range and heating at a temperature between the melting point of the amide and 100°, such as at about 95°, until reaction is complete.

Vigorous agitation of the mixture is essential during the formation of the dispersion in order that the particle size of the methylol amide shall be suitably small. The dispersion obtained by this process contains the methylol amide in the form of long slender needle-like crystals which range from about 1 x 5 microns to 2 x 10 microns.

The dispersion is dewatered by filtration or by any equivalent operation and the filter cake is then washed with water to remove any remaining excess formaldehyde. The filter cake ordinarily contains about 40 to 50% solids. The speed of filtration is improved markedly by adding a small amount of calcium acetate to the dispersion near the end of the heating step.

The filter cake is then mixed with polyvinyl alcohol and with a surface-active agent consisting of a water-soluble long-chain alcohol sulfate containing from 12 to 18 carbon atoms. There is added from 10 to 30% polyvinyl alcohol, based on the dry weight of methylol fatty acid amides in the filter cake, the preferred amount being from 10 to 20%. The polyvinyl alcohol can be in the form of a commercial partially saponified polyvinyl ester. Polyvinyl acetate which is from 80 to 95% saponified is particularly suitable. The polyvinyl alcohol is added in the form of an aqueous solution.

There is added from 2 to 5% and preferably from 4 to 5% of the long chain alcohol sulfate, based on the dry weight of methylol fatty acid amides. Higher amounts of the surface-active agent seriously interfere with the water-repellent

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properties of the final product. Any commercially available water-soluble long-chain alcohol sulfate, such as the sodium or triethanolamine salts of the sulfates of the long-chain alcohols obtained by reduction of coconut oil, sperm oil and the like, may be used.

The filter cake containing the methylol amides is mixed with the polyvinyl alcohol and the long-chain alcohol sulfate in any efficient mixer or colloid mill. The resulting mixture is useful directly as a textile finishing composition, producing water-repellent effects when applied to and heated on the fabrics. The effects are not as durable as when resins are also present, nor does the mixture of methylol amide and polyvinyl alcohol produce any significant degree of dimensional stability. These mixtures are stable dispersions which may be stored or transported without adverse effect, and which may subsequently be mixed with resins to form the preferred compositions of this invention.

The water-soluble heat-setting amide-formaldehyde resins which are useful in the practice of this invention include urea-formaldehyde resins, melamine-formaldehyde resins and 1,3-bis-(hydroxymethyl)-2-imidazolidone resins. They are most conveniently incorporated with the dispersion of methylol amide and polyvinyl alcohol by adding the resins in aqueous solution, followed by intimate mixing. The amount of resin to be added depends upon the nature of the fabric and of the resin and on the degree of stiffness and dimensional stability which is desired. About 1 part of resin per part of methylol amide represents the minimum amount. In treating viscose rayon, best results are obtained with compositions containing at least 4.5 parts of urea-formaldehyde resin, about 3.5 parts of melamine-formaldehyde resin or about 4 parts of 1,3-bis-(hydroxymethyl)-2-imidazolidone resin per part of methylol amide. With cotton, excellent results are obtained from compositions containing only 1 to 1½ parts of resin. Amounts of resin up to about 15 parts per part of methylol amide can be used, but at this point the compositions become undesirably expensive and water-repellency begins to decrease.

The preparation of the compositions of this invention is illustrated by the following examples:

Example 1

A mixture of 202 parts of technical grade stearamide and 808 parts of water is heated to 96° C. and agitated at 96-98° C. for five to ten minutes to convert it to a smooth, homogeneous paste. The charge is then cooled to 65° C. and 291 parts of a 37% formaldehyde solution are run in slowly over one to two hours at 60-65° C. with vigorous agitation. During this addition the charge is kept slightly alkaline to phenolphthalein paper (pH 8.5-9.0) by the addition of 30% sodium hydroxide solution. This pH is maintained while the charge is agitated vigorously for an additional hour at 60-65° C.

The mixture is then heated gradually to 80° C. during one hour and agitated at 78-82° C. for three more hours at the same pH. To aid in filtration of the methylol stearamide, 14 parts of calcium acetate are added, followed by an additional hour's agitation at 80° C. The charge is cooled to 50-60° C. with agitation and filtered. The filter cake is washed with water until substantially free of formaldehyde, and pressed until it has a methylol amide content of about 40%.

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The filter cake amounting to about 550 parts is charged into a kettle containing 440 parts of a 10% aqueous solution of polyvinyl alcohol (86-89% saponified polyvinyl acetate), 10 parts of the sodium sulfate of a mixture of long chain alcohols containing from 10 to 16 carbon atoms, predominantly lauryl and myristyl alcohols, and 5-10 parts of water. The charge is agitated at 40-50° C. to a smooth, homogeneous paste and passed through a colloid mill to complete the mixing.

The 1000 parts of dispersion resulting from this procedure are mixed with 1500 parts of a commercially available urea-formaldehyde textile resin in the form of a 20% aqueous solution. Viscose-acetate rayon gabardine treated with this mixture possesses good water-repellency, crease-resistance and dimensional stability, and has a firm, crisp hand.

When 1000 parts of 1,3-bis-(hydroxymethyl)-2-imidazolidone are used in place of the urea-formaldehyde resin, similar effects are obtained, except that the treated fabric is not as stiff.

Similarly, 850 parts of a commercial methylated methylol melamine are mixed with 1000 parts of the methylol amide-polyvinyl alcohol dispersion described above. When the resulting dispersion is applied to rayon, it imparts to the fabric a high degree of water-repellency together with satisfactory dimensional stability and a firm hand.

Example 2

The general procedure of Example 1 is repeated using less formaldehyde and different temperatures.

A mixture of 808 parts of water, 202 parts of technical grade stearamide and 81 parts of a 37% aqueous formaldehyde solution is heated to 96° C. and agitated for five to ten minutes at 96-98° C. to convert it to a smooth, homogeneous paste. The charge is cooled to 80° C. in half to three-quarters of an hour with agitation. The pH is adjusted to about 7.5 by the addition of 30% sodium hydroxide solution and the charge is agitated vigorously for two hours at 79-83° C., maintaining the same pH. During this period the charge changes from a light brown, non-homogeneous mass to a smooth, homogeneous white paste.

After the charge is cooled to 70° C., 14 parts of calcium acetate are mixed in by agitating for about thirty minutes. The charge is further cooled to 60° C. and filtered. The filter cake is washed free of formaldehyde with water and pressed to 550 parts, having a 40% solids content. This produces a methylol amide mixture having a melting range of about 108-110° C.

The filter cake is mixed with polyvinyl alcohol and sodium alcohol sulfate as described in Example 1. When portions of the resulting dispersion are mixed with the urea, melamine and 1,3-bis-(hydroxymethyl)-2-imidazolidone resins as in Example 1 and applied to a rayon fabric, the treated fabrics possess good water-repellency, dimensional stability and firm hand.

The compositions of this invention are prepared for application to the fabric simply by mixing them into water with a small amount of any conventional mildly or potentially acidic catalyst for curing the resin. Examples of suitable catalysts include tartaric acid, the ammonium acid phosphates, zinc nitrate and the like,

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The aqueous dispersion is then applied to the fabric by padding or by any other convenient method. The fabric is dried and baked for about 3 minutes at 325 to 375° F. to cure the resin. The treated fabric displays good water-repellency and dimensional stability. The hand of the fabric is improved by becoming firmer and more resilient. These effects are quite durable and survive washing and dry-cleaning of the fabric. The resin content of the composition serves not only to contribute crease-resistance and dimensional stability but also to bind the methylol amide to the fabric so as to give a more durable water-repellent effect.

An important advantage of these compositions is that they are quite stable without close control of the pH. This is not true of many other water-repellent compositions, as for example dispersions of methylol stearamide in soap solution, which are extremely sensitive to changes in pH.

I claim:

1. A process of preparing a textile finishing composition which comprises: (1) forming a dispersion of methylol fatty acid amides by heating together at 60 to 100° C. under vigorous agitation (a) a fatty acid amide mixture which melts between 90 and 100° C. in water and in which the total content of stearamide and palmitamide exceeds 80% by weight, (b) water, and (c) a molecular excess of formaldehyde, during a portion of which heating step at least the fatty acid amides and water are agitated together at a temperature between the melting point of the amide mixture and 100° C. to emulsify the amides, and during a subsequent portion of which the dispersed fatty acid amide, formaldehyde and water are agitated at pH 7.5 to 9 and at 75 to 100° C.; (2) filtering off the methylol fatty acid amide and washing out the excess formaldehyde from the filter cake; and (3) mixing the filter cake with from 10 to 30% polyvinyl alcohol in aqueous solution and with from 2 to 5% of a water-soluble long-chain alcohol sulfate containing from 12 to 18 carbon atoms, the percentages in each case being based on the dry weight of methylol fatty acid amide in the filter cake.

2. A textile finishing composition prepared by the process of claim 1.

3. A textile finishing agent consisting of a composition prepared by the process of claim 1 in admixture with from 1 to 15 parts by weight of a water-soluble heat-setting amide-formaldehyde resin per part of methylol fatty acid amide.

4. A composition according to claim 3 in which the resin is a urea-formaldehyde resin.

5. A composition according to claim 3 in which the resin is a methylated methylol melamine resin.

6. A composition according to claim 3 in which the resin is a 1,3-bis(hydroxymethyl)-2-imidazolidone.

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