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3,170,876

TEXTILE TREATING COMPOSITIONS

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This invention relates to novel compositions for treating textile fabrics and to concentrated aqueous dispersions of these novel compositions and the preparation of the dispersions. In particular the invention relates to compositions containing normally plastic or solid cationic textile softeners which compositions form concentrated aqueous dispersions possessing a high degree of freeze-thaw stability.

This application is a continuation-in-part of application Serial No. 4,872 filed January 27, 1960, now abandoned.

Cationic surface active agents have gained widespread acceptance as textile softeners. These cationic textile softeners possess many advantages, such as effectiveness in small amounts and resistance to washing, but they suffer from several disadvantages. Many of the cationic softeners are waxy or gummy in nature making them difficult to weigh or measure, to mix or disperse with other textile treating agents, and to place in a form, such as an aqueous dispersion, which may be readily applied to textiles. For these reasons, it is desirable to have a concentrated aqueous dispersion of a cationic textile treating composition, which concentrated dispersion is fluid, easily handled and readily dispersed in water.

It is an object of this invention to provide a textile treating composition which may be admixed with water to form a concentrated aqueous dispersion, said concentrated dispersion being fluid, easily handled and readily dispersed in water.

It is also an object of this invention to provide a concentrated aqueous dispersion of a textile treating composition which will remain stable throughout repeated freeze-thaw cycles.

Another object of this invention is to provide a concentrated aqueous dispersion of a textile treating composition which is readily dispersed in water to form a dilute solution of a textile treating composition which may be directly applied to textile fabrics.

A further object is to provide a cationic textile softener composition whose softening capability is substantially unimpaired by other components of the composition.

A still further object of this invention is to provide a cationic textile softener composition whose viscosity is easily controlled and which may be prepared as a concentrated aqueous dispersion characterized by fluidity even at high concentrations of cationic textile softener.

Still another object of the invention is to provide a method for preparing a concentrated aqueous dispersion of a cationic textile softener.

Other objects and purposes of this invention will be obvious to those skilled in the art in view of the description of the invention which follows.

It has been discovered that a textile treating composition consisting essentially of a cationic textile softener and a nonionic emulsifier selected from the group consisting of a polyoxyethylene ether of castor oil and a polyoxyethylene ether of hydrogenated castor oil can be dispersed in water to form a concentrated aqueous dispersion of the cationic textile softener. The resultant concentrated aqueous dispersion is fluid, easily handled and readily dispersed in water to form a dilute cationic treating solution which may be directly applied to textile fabrics.

The polyoxyethylene ethers of castor oil and hydro-

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genated castor oil which are useful as nonionic emulsifiers in the compositions and method of this invention may contain from about 20 to about 300 oxyethylene groups per mole, with a preferred range of from about 25 to about 50 oxyethylene groups per mole. It has been found that if the ether of the castor oil or hydrogenated castor oil contains less than 20 or more than 300 oxyethylene groups per mole of oil, the compound will have insufficient activity to act as a fluidizing agent for the cationic textile softeners useful in the practice of this invention or will be so costly as to be impractical for use as a fluidizing agent. These polyoxyethylene castor oil compounds are nonionic emulsifiers, and their preparation is disclosed in the prior art.

In order to convert a plastic or gummy cationic textile softener into a concentrated aqueous dispersion in accordance with this invention, it is necessary that enough nonionic emulsifier be added to the cationic component so that the mixture will form a fluid dispersion in water and yet not impair the cationic's textile softener properties. In general, the nonionic emulsifier may be employed in a ratio of from about 1:1 to about 1:3 based on the cationic softener component. If less than a 1:3 ratio is used, there may not be enough nonionic emulsifier to form a stable, fluid dispersion when the textile softener composition is admixed with water, whereas a greater than 1:1 ratio may impair the textile softening properties of the cationic component. It should be noted that ratios of nonionic to cationic outside of the above recited ranges may be operative and are included within the scope of this invention, though the 1:1 to about 1:3 ratios are generally preferred.

The cationic textile softeners used in accordance with this invention are useful in treating textile fabrics to give a soft and pleasing "feel" to fabrics which would otherwise tend to be harsh or stiff. The cationic softeners of the invention are also described in the art of textile finishing as particularly suitable in imparting richness of "hand" to the textiles treated therewith.

Although cationic textile softeners generally are useful in the practice of this invention, the invention is particularly applicable to cationic softeners which are normally plastic, waxy or gummy in nature. Representative of the cationic textile softener compounds which may be used to form concentrated aqueous dispersions in accordance with this invention are fatty carbamides, fatty amino amide compounds, acid salts of complex amino organic compounds, quaternary ammonium compounds, alkyl guanidine and distearyl dimethyl ammonium chloride. Further description of cationic softening agents useful in this invention may be found in British Patents 465,166, 490,637 and 531,691 and United States Patents 2,168,253, 2,185,427 and 2,195,194.

Compounds of the foregoing type may be found on the market under various trade names such as for example Ahcovel A and Ahcovel G manufactured by A. Hoffman, Arquad 2HT manufactured by Armour & Co., Soromine CS, Soromine BSA and Soromine BNS manufactured by Antara Chem. Co., Sapamine WL and Sapamine KWC manufactured by Ciba Chemical Co., Avitone SR manufactured by Du Pont Company, Aliquat H226 manufactured by General Mills and Adogen 442 manufactured by Archer Daniels Midland.

A particularly useful class of cationic textile softeners which may be employed in the practice of the present invention include the reaction product of a hexitol epihalohydrin condensate containing from about 1 to about 4 halohydrin radicals per hexitol residue and a C₁₂ to C₂₀ fatty acid diamide of diethylene triamine. If more than 4 halohydrin radicals are present per hexitol, it appears that the softening effect of the textile softening agent

it markedly reduced. A number of suitable compounds are disclosed in application Serial No. 654,472 filed April 23, 1959, by John D. Zech. Compounds within the scope of the present invention are exemplified by the following: (The ratio in parenthesis following the epihalohydrin indicates the molar ratio of hexitol to the epihalohydrin.) The reaction product of the condensate of sorbitol epichlorohydrin (1:2), and the tallow acids diamide of diethylene triamine, the reaction product of the condensate of mannitol epibromohydrin (1:4) and the stearic acid diamide of diethylene triamine, the reaction product of lauryl acid diamide of diethylene triamine and sorbitol epichlorohydrin (1:2), the reaction product of cotton seed oil acids diamide of diethylene triamine and dulcitol epichlorohydrin (1:1), the reaction product of lard fatty acids diamide of diethylene triamine and sorbitol epichlorohydrin (1:2), the reaction product of linoleic acid diamide of diethylene triamine and sorbitol epichlorohydrin (1:2), the reaction product of palmitic acid diamide of diethylene triamine and sorbitol epichlorohydrin (1:2).

The chemistry of the hexitol epihalohydrin condensation products and the amidation reaction products of diethylene triamine and the C_{12} to C_{20} fatty triglycerides and C_{12} to C_{20} fatty acids have been well elucidated in the prior art.

The cationic textile softeners useful in the practice of the present invention as hereinabove exemplified are prepared by condensing the hexitol derivative with the C_{12} to C_{20} fatty acid diamide at relatively low temperatures, usually about 100°C . to about 150°C . for a short period of time of about $\frac{1}{2}$ hour to about 4 hours. It is desirable however to employ a stoichiometric excess of the fatty acid diamide in preparing the compounds of the present invention in order to insure complete reaction of the halohydrin derivative.

The terms "halohydrin" and "epihalohydrin" as used in the specification and claims have a restricted meaning for those compounds containing only fluorine halogen are excluded. However, halohydrins containing chlorine, bromine, and/or iodine in addition to fluorine are contemplated herein. This limitation is imposed because of the lack of reactivity of fluorine in such compounds due to the relative stability of the fluorine-carbon bond.

As a cooperative agent for improving water dispersability of the cationic softeners, there may be employed along with the polyoxyethylene castor oil derivatives a glycol, alcohol, or polyglycol of low molecular weight, such as ethylene glycol, propylene glycol, tetraethylene glycol, isopropanol, etc., which multiplies the effect of the polyoxyethylene compound in attaining a lower and more consistent viscosity. The glycol may be added as such to the mixture of polyoxyethylene castor oil and the cationic textile softener; it may be added to the cationic textile softener or the polyoxyethylene castor oil alone prior to mixing; and it may be added to the cationic textile softener during its preparation. It is to be noted, however, that the addition of the glycol or polyglycol in an amount of from about 15% to about 35% by weight based on the cationic softener will make the cationic softener somewhat less viscous. However, the polyoxyethylene castor oil compound is the essential ingredient which permits the formation of concentrated aqueous dispersions of cationic softener. A more preferred range for the amount of glycol or polyglycol which may be added is about 20 to about 25% by weight based on the final composition.

As pointed out hereinabove, the cationic textile softener compositions especially useful in this invention are normally of a plastic or gummy nature and as such they are difficult to handle and difficult to apply to textile materials. It is known that very dilute aqueous solutions of cationic textile softeners can be prepared but with some difficulty, for it is hard to measure the gummy cationic compound and it is not easily dispersed in water.

For these reasons, textile chemical specialty houses

have found it desirable to sell a concentrated aqueous dispersion of cationic textile softening compositions which contains from about 15 to about 40% by weight of cationic softener nonionic emulsifier mixture, and known as a 15 to 40% active aqueous concentrate. The aqueous concentrate is fluid (pourable) and more easily handled than 100% active cationic softener composition. It is essential that a dispersing agent be used to form the aqueous concentrate, for otherwise only very small amounts of cationic compound can be dispersed in water. In the absence of a dispersant, a very small amount of gummy cationic softener will form a very viscous, non-pourable aqueous dispersion. In addition, it is essential that the dispersing agent which is used to form the aqueous concentrate not impair the softening properties of the cationic compound.

It has been found that fluid aqueous concentrates of cationic softeners can be prepared by using nonionic emulsifiers as dispersing agents as previously disclosed. The aqueous concentrate is prepared in accordance with this invention by heating the normally plastic cationic textile softener until it melts (generally about 75°C . will be sufficient), add the nonionic emulsifier to the melted cationic compound, add sufficient water with agitation to the liquid nonionic-cationic mixture until a smooth, creamy, fluid aqueous concentrate is obtained. The resultant aqueous mixture is stirred while cooling to room temperature.

In general, the fluidity of the paste formed by preparing an aqueous dispersion of the cationic textile softener and the nonionic emulsifier is affected by the pH of the undiluted cationic textile softener. It is preferred that the undiluted cationic textile softeners have a pH which will produce a smooth, creamy, fluid, 25% active aqueous concentrate rather than a very viscous, somewhat gelatinous composition. Depending upon the particular cationic textile softener used, the pH may vary from about 3 to about 9 and still give acceptable results with lower pH values generally giving more fluid concentrates. 25% active aqueous concentrates having the more desirable viscosities and fluidity and retaining other desirable textile softener properties such as scorch resistance, may be prepared from cationic softeners having a pH from about 3 to about 6. In particular, when using a hexitol-epihalohydrin condensate-fatty acid diamide as the cationic softener, it is preferred that the pH range from about 3 to about 5.

It is a relatively easy matter to adjust the pH of the cationic textile softener used in accordance with this invention to the desired pH value. If the pH of the cationic softener is too great to give the 25% active concentrate the desired viscosity, a buffering acid such as glacial acetic acid or phosphoric acid, may be added to the cationic softener to reduce the pH and thereby achieve the desired fluid viscosity. The buffering acid may be added to the cationic softener prior to, during, or after the formation of the 25% active concentrate, and in this manner, it is an easy matter to control the viscosity of the aqueous concentrate. However, it should be noted that the pH value of from about 3 to about 9 applies only to the cationic softener, and a different pH value may be necessary to obtain a fluid 25% active concentrate when adding the buffering acid to the already formed aqueous dispersion.

As previously stated, the cationic textile softener may have a pH from about 3 to about 9, though it should be noted that cationic softeners having a pH outside of this range are included within the scope of this invention.

As set forth above, the fluidity of the 25% active concentrate is affected by the amount of nonionic emulsifier (polyoxyethylene castor oil or hydrogenated castor oil) which is used, with greater amounts generally yielding greater fluidity. In general, the nonionic emulsifier may be used in a ratio of from about 1:1 to about 1:3 of the cationic textile softener, with the amount of nonionic emulsifier or fluidizing agent necessary to achieve a fluid

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25% active concentrate depending upon the particular cationic softener being used. If a hexitol-epihalohydrin condensate-fatty acid diamide is used as the cationic softener, in order to achieve good fluidity and retain softener properties, it is preferred to use a ratio of nonionic emulsifier to cationic softener ranging from about 31:69 to about 35:65, with a 33:67 ratio giving a particularly good, fluid 25% active concentrate.

The following examples illustrate the preparation of a hexitol halohydrin-fatty acid diamide of diethylene triamine condensation product; and the preparation of a fluid, 25% active, textile softener concentrate by mixing the aforesaid condensation product, a cationic textile softener, with a polyoxyethylene castor oil nonionic emulsifier in accordance with one embodiment of this invention. The use of other cationic textile softeners and nonionic emulsifiers is also described.

Example I

913 grams of anhydrous sorbitol were heated to a reaction temperature of between 105° and 115° C.; 2.0 gms. of BF₃ etherate catalyst (45% BF₃) were then added.

Thereafter, 1040 grams of epichlorohydrin (molal ratio of sorbitol to epichlorohydrin of 1:2.25) were added dropwise over a period of 1 hour and 10 minutes with vigorous stirring and temperature control in order to maintain the temperature within the above-specified limits. The temperature was maintained with the use of additional heat to insure completion of the reaction.

100 grams of diethylene triamine and 571 grams of edible tallow were heated together at 135° C. for 3 hours. The residue in the flask consisted of a 99% yield of tallow acids diamide of diethylene triamine. The nitrogen content of the mixture was 6.3% and the apparent molecular weight was 700.

81 grams of the product of the above-prepared sorbitol epichlorohydrin and 664 grams of the above-prepared tallow diamide of diethylene triamine (molal ratio of sorbitol epichlorohydrin to tallow acids diamide of diethylene triamine of 1:4.7) were heated together for 2 hours at a temperature of from 90 to 126° C. The resultant condensate was a gummy plastic waxlike material dispersible in water and it possessed excellent textile softening and lubricating properties.

Example II

250 grams of the above prepared reaction product of Example I was melted at 75° C. and to this melt was added with stirring 250 grams of polyoxyethylene ether of hydrogenated castor oil containing 25 oxyethylene groups per mole of oil. This product was a paste concentrate which could further be diluted with water to provide a fluid textile concentrate which is readily dispersible in water and which possessed excellent textile and softening properties. 1500 grams of water at 75° C. was added to this paste concentrate and the resultant paste was allowed to cool to room temperature with stirring. The final product was readily dispersible in water and remained fluid and stable on storage.

Example III

250 grams of distearyl dimethyl ammonium chloride was heated to 75° C. and to this melted product was added 250 grams of polyoxyethylene ether of hydrogenated castor oil containing 25 oxyethylene groups per mole of oil. 1500 grams of water at 75° C. was added to this mixture and the resultant concentrate was allowed to cool to room temperature with stirring. The final product was readily dispersible in water and remained fluid and stable on storage.

Example IV

To the reaction product of Example I was added with stirring 250 grams of propylene glycol. To 500 grams of this mixture was added 250 grams of polyoxyethylene ether of hydrogenated castor oil containing 25 oxyethyl-

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ene groups per mol of oil and 2250 grams of water with stirring. The resultant product, a 25% active fluid concentrate, remained fluid after a storage of one week at room temperature and for the period of one week when stored at a temperature of 50° C. In addition, the product remained in a fluid state after 3 freeze-thaw cycles.

After evaluation of the fluidity of the concentrate and its stability to repeated freeze-thaw cycles, the 25% active fluid concentrate was dissolved in additional water to provide a 2.0% aqueous solution. The temperature of the solution was 50° C. Swatches of different fabrics including cotton, nylon, Orlon, rayon, Dacron, Dynel and Darvan were each immersed in 300 cc. of the 2.0% aqueous solution of the cationic textile softener. After 3 minutes, the fabrics were centrifuged for 30 seconds and allowed to dry. Each of the test samples exhibited a softness of "hand" or "feel" which was not found in swatches not so treated.

In order to determine the ratio of the polyoxyethylene ether of hydrogenated castor oil to the cationic softener prepared in Example I necessary for obtaining the best combination of fluidity of 25% active concentrates and textile softener properties, various ratios were evaluated. Samples with nonionic fluidizing agent to cationic textile softener ratios of 29:71, 31:69, 33:67, 35:65 and 37:63 were formulated into 25% active aqueous dispersions and were checked for fluidity and retention of textile softener properties. It was determined that samples containing 69% or more of the cationic softener tended to become slightly gellatinous and therefore are not preferred. Samples containing 65% or less of the cationic softener were very fluid but their softening activity was impaired to a certain degree. The 33:67 (nonionic:cationic) formed very good fluid, 25% active concentrates and retained excellent textile softening properties.

The textile softening compositions of the present invention may also be applied to textile fabrics by padding from a padder, quetch or mangle or a substantive application may be made by a jig, beck or package machine. If the application is made by padding, the flat goods can be padded at 125° F. from the padder, mangle or quetch with a 1% to 2% solution of the 25% active fluid concentrate using a wet pick-up of about 100%. Yarns and textile fabrics can also be effectively softened in a package machine using 2 to 3% of the 25% active fluid concentrate based on the weight of the yarn at a temperature of 125° F.

Thus, having described the invention, it may be construed in accordance with the following claims. What is claimed is:

1. A fluid textile softening composition which is readily dispersible in water consisting essentially of about 1 part by weight of a nonionic emulsifier selected from the group consisting of polyoxyethylene ethers of castor oil and polyoxyethylene ethers of hydrogenated castor oil, said ethers containing from 20 to 300 oxyethylene groups per mole of oil, and from about 1 to 3 parts by weight of the reaction product of a hexitol epihalohydrin condensate containing from 1 to 4 halohydrin radicals per hexitol residue reacted with a stoichiometric excess of an acyclic C₁₂ to C₂₀ fatty acid diamide of diethylene triamine.

2. The composition of claim 1 in which the polyoxyethylene ethers contain from 25 to 50 oxyethylene groups per mole of oil.

3. A composition as in claim 1 wherein said polyoxyethylene ether is the ether of castor oil.

4. A composition as in claim 2 wherein said polyoxyethylene ether is the ether of hydrogenated castor oil.

5. A composition as in claim 1 wherein said polyoxyethylene ether is present in an amount of from about 31:69 to about 35:65 of said reaction product.

6. A fluid textile softening composition which is readily dispersible in water consisting essentially of about 1 part by weight of a polyoxyethylene ether of hydrogenated castor oil containing 25 oxyethylene groups per mole of

castor oil and from about 1 to 3 parts by weight of the reaction product of sorbitol epichlorohydrin condensate containing about 2 chlorine radicals per sorbitol residue reacted with a stoichiometric excess of an acyclic tallow acids diamide of diethylene triamine.

7. A fluid textile softening composition which is readily dispersible in water consisting essentially of about 1 part by weight of a polyoxyethylene ether of hydrogenated castor oil containing 25 oxyethylene groups per mole of castor oil, and from about 1 to 3 parts by weight of the reaction product of sorbitol epichlorohydrin condensate containing about 2 chlorine radicals per hexitol residue reacted with a stoichiometric excess of an acyclic tallow acids diamide of diethylene triamine, and from 15 to 35% propylene glycol based on the weight of the reaction product.

8. A fluid textile softening composition which is readily dispersible in water consisting essentially of about 1 part by weight of a polyoxyethylene ether of hydrogenated castor oil containing 25 oxyethylene groups per mole of castor oil, and from about 1 to 3 parts by weight of the reaction product of sorbitol epichlorohydrin condensate containing about 2 chlorine radicals per hexitol residue reacted with a stoichiometric excess of an acyclic tallow acids diamide of diethylene triamine, and 25% propylene glycol based on the weight of the reaction product.

9. A fluid textile treating composition which is readily dispersible in water and possessing freeze-thaw stability consisting essentially of about 1 part by weight of a polyoxyethylene ether of hydrogenated castor oil containing 25 oxyethylene groups per mole of castor oil, and from about 1 to 3 parts by weight of the reaction product of sorbitol epichlorohydrin condensate containing about 2 chlorine radicals per hexitol residue and a stoichiometric excess of tallow acids diamide of diethylene triamine, and 25% propylene glycol based on the weight of said reaction product and sufficient water to provide an aqueous concentrate in which the polyoxyethylene ether plus the reaction product content is about 25% by weight.

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