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SULPHONATED CARBOXYLIC AMIDE

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13 Claims. (Cl. 260—124)

The present invention relates to the production of assistants for the textile and allied industries, such as wetting, cleansing and dispersing agents.

In the copending application Ser. No. 441,717, filed April 4, 1930, the present inventors and another have described a process for the production of assistants for the textile and allied industries, such as wetting, cleansing and dispersing agents, by converting organic carboxylic acids into amides and sulphonating the resulting products if they are free from sulphonic acid or sulphuric ester radicles. Thus for example fatty acids have been condensed with taurine to form the amide of the fatty acid concerned with the aminoethane sulphonic acid.

We have now found that valuable assistants for the textile and allied industries, such as wetting, cleansing and dispersing agents, are obtained by causing carboxylic acid anhydrides or halides to react with aminomethane sulphonic acid substances, i. e. the free acids and their salts, in the presence of an alkaline diluent the said components being so chosen that the sum of the carbon atoms in aliphatic chains thereof is at least six.

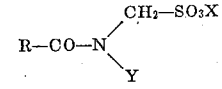
Anhydrides or halides of carboxylic acids which are suitable for the purposes according to the present invention may be chosen for example from those of saturated or unsaturated fatty acids, such as those of the series from acetic to stearic acids, or acids containing even more carbon atoms, of resinic and naphthenic acids, of mixtures of carboxylic acids obtained by the destructive oxidation of paraffin waxes, mineral oils, middle oils and the like, of acids of montan wax, of mixed aliphatic-aromatic carboxylic acids, such as of the type of phenyl-stearic or dodecyl-benzoic acids or other alkylated aromatic or arylated aliphatic carboxylic acids, or aromatic carboxylic acids, such as benzoic acid or naphthoic acid. The said carboxylic acid anhydrides or halides may also contain substituents, such as halogen, nitro, amino or hydroxy groups.

As aminomethane sulphonic acid substances are intended aminomethane sulphonic acid ($H_2N—CH_2—SO_3H$) or a salt thereof and derivatives of these compounds containing at least one hydrogen atom connected to the nitrogen atom as for example the conversion products from bisulphite compounds of formaldehyde (which are described in the literature in part as sulphurous acid esters) with ammonia or amines of aliphatic, aliphatic-aromatic or aromatic nature as for example methyl, butyl, cetyl, cyclohexyl or benzyl amines, aniline or toluidine, and also products containing the grouping

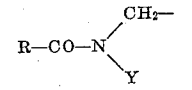
$$—NH—CH_2—SO_3X,$$

which are obtained by other methods and in which X is either hydrogen or the radicle of a base. Ammonia and aliphatic and aliphatic-aromatic amines yield after conversion with the bisulphite compounds of formaldehyde, products which are more reactive with reference to the carboxylic acid anhydrides or halides than are the products from purely aromatic amines.

The products obtained are carboxylic amides corresponding to the general formula



in which R—CO defines the acyl radicle of a carboxylic acid, Y defines hydrogen or an organic radicle, preferably an alkyl radicle, especially an aliphatic hydrocarbon radicle, and X defines hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base, such as K or Na or ammonia, pyridine, dimethyl aniline or alkylol amines such as ethanol amines, which latter salts may be obtained from the salts directly obtained by the usual conversion reactions. In any case the sum of the number of carbon atoms in



should be at least six, the components employed in the amidification being chosen correspondingly as mentioned above; if R—CO be for example $CH_3—CH_2—CO$, Y is at least $CH_3—CH_2—$.

The reaction is carried out in the presence of alkaline diluents, such as water containing an alkali as for example sodium or potassium hydroxides, or organic tertiary bases, such as pyridine, dimethyl aniline or triethylamine and the like, preferably in equimolecular quantities or also in lower or higher quantities. If desired the said organic bases may be employed as such in the absence of water provided they are liquid at the reaction temperatures. In all cases the said alkaline diluents serve as acid-binding agents and combine with the acids set free during the reaction. As a rule, in the presence of water, low temperatures i. e. those below 35° C. are employed, temperatures between 15° and 25° C. being usually preferred. If anhydrides of the carboxylic acids be employed the products will contain the

corresponding salts of the acids with the base employed and may be freed therefrom for example by means of an aqueous pulp of gypsum whereby the carboxylic acids are precipitated. In most cases, especially if anhydrides of the acids of vegetal, i. e. animal or vegetable, oils or fats have been employed, the soaps formed may be left in the products.

The carboxylic amides obtained have a high stability to alkalines and acids and are not precipitated from aqueous solutions by the substances forming the hardness of water. They are even stable to alkalies when heated, while boiling with strong mineral acids may lead to partial saponification. The products have a very high wetting, cleansing and emulsifying power and may therefore be advantageously employed in many branches of daily and industrial use. They may be employed with especial advantage in the textile, leather and paper industries; they are suitable for cleansing, levelling, softening and penetrating, and also as assistants for many other improving treatments and as emulsifying and dispersing agents for emulsifying or dispersing water-insoluble substances such as fats, oils, waxes, hydrocarbons and the like, in water or aqueous solutions.

The amides may be employed for these purposes as such or in admixture with each other or together with other substances, such as soaps or saponaceous substances, bleaching agents, such as perborates or percarbonates, salts as for example Glauber's salt, sodium carbonate or bicarbonate, waterglass and like water-soluble salts, organic solvents such as trichlorethylene, benzyl alcohol or ethylene glycol mono-cresyl ether, glycerol, higher aliphatic alcohols such as from hexyl to cetyl alcohols and like solvents.

The following examples will further illustrate the nature of this invention but the invention is not restricted to these examples. The parts are by weight.

Example 1

40 parts of a methylaminomethane sulphonic acid ($CH_3—NH—CH_2—SO_3H$), obtainable by the interaction of formaldehyde-bisulphite with methylamine, are dissolved in 250 parts of water and 60 parts of the chlorides of the acids of coconut oil are added thereto. 60 parts of an aqueous caustic soda solution of 40° Bé. strength are caused to flow into the mixture at from 15° to 20° C. while cooling and stirring well. After stirring for some time, 410 parts of an approximately 20 per cent paste of the reaction product are obtained. This is soluble in water to give a clear solution and has a very high stability, at room temperature, to acids and to alkaline agents and is not precipitated from its aqueous solutions by the substances forming the hardness of water.

The reaction proceeds according to the equation

$$C_{11}H_{23}—\overset{\overset{O}{\|}}{C}—Cl+HN(\overset{CH_3}{|})—CH_2—SO_3Na+NaOH\rightarrow$$

$$C_{11}H_{23}—\overset{\overset{O}{\|}}{C}—N(\overset{CH_3}{|})—CH_2—SO_3Na+NaCl+H_2O$$

the acids of coconut oil being calculated as $C_{11}H_{23}COOH$.

10 parts of a 20 per cent paste of the coconut oil acid amide of methylaminomethane sulphonic acid are dissolved in 1000 parts of water. A solution having an extremely high wetting power is obtained which is also capable of scouring 20 parts of suint wool within ½ hour at from 40° to 50° C.; the solution may be employed for washing further batches of suint wool.

Example 2

40 parts of aminomethane sulphonic acid are dissolved in 250 parts of water while adding aqueous caustic soda solution until the solution is slightly alkaline, whereupon 50 parts of palmitic acid chloride are added at from 20° to 25° C. and the reaction mixture is kept alkaline by adding aqueous caustic soda solution. The reaction mixture is stirred for from 3 to 4 hours and then allowed to settle. A paste is obtained which constitutes an efficient softening agent for artificial silk such as viscose of cuprammonium silk. In the place of the palmitic chloride other carboxylic acid halides, such as halides of naphthenic acids may be employed.

The reaction proceeds according to the equation

$$C_{15}H_{31}—\overset{\overset{O}{\|}}{C}—Cl+HNH—CH_2—SO_3Na+NaOH\rightarrow$$

$$C_{15}H_{31}—\overset{\overset{O}{\|}}{C}—NH—CH_2—SO_3Na+NaCl+H_2O$$

If palmitic anhydride be employed the product comprises, besides the amide, an equivalent quantity of palmitic sodium salt.

Example 3

40 parts of an aminomethane sulphonic acid are dissolved in 250 parts of water while adding aqueous caustic soda solution until the solution is slightly alkaline, whereupon 50 parts of oleic acid chloride are added at from 20° to 25° C. and the reaction mixture is kept alkaline by adding aqueous caustic soda solution. The reaction mixture is stirred for from 3 to 4 hours. A product is obtained which is a highly efficient agent for washing piece goods. Instead of oleic chloride, oleic anhydride may be employed.

The reaction proceeds according to the equation

$$C_{17}H_{33}—\overset{\overset{O}{\|}}{C}—Cl+HNH—CH_2—SO_3Na+NaOH\rightarrow$$

$$C_{17}H_{33}—\overset{\overset{O}{\|}}{C}—NH—CH_2—SO_3Na+NaCl+H_2O$$

If oleic anhydride be employed the product comprises, besides the amide, an equivalent quantity of oleic sodium salt. Similar products are obtained if the aminomethane sulphonic acid be replaced by methyl-aminomethane sulphonic acid.

The term "acyl radicle" as used in the specification and claims should be considered as covering alkyl, aryl, aralkyl and alkyl aryl radicles.

What we claim is:—

1. A product comprising a sulphonated carboxylic amide corresponding to the general formula

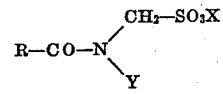
$$R—CO—N(\overset{CH_2—SO_3X}{\diagdown}\ \diagup Y)$$

in which R—CO is the acyl radicle of a carboxylic acid, Y is hydrogen or a hydrocarbon radicle, X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base, and in which amides the group

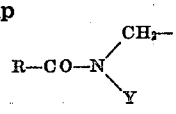
$$R—CO—N(\overset{CH_2—}{\diagdown}\ \diagup Y)$$

contains at least six carbon atoms.

2. A sulphonated carboxylic amide corresponding to the general formula

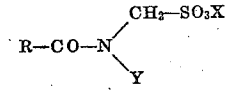


in which R—CO is the acyl radicle of a carboxylic acid, Y is hydrogen or a hydrocarbon radicle, X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base, and in which amides the group

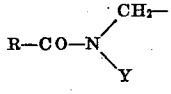


contains at least six carbon atoms.

3. A sulphonated carboxylic amide corresponding to the general formula

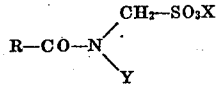


in which R—CO is the acyl radicle of a carboxylic acid, Y is hydrogen or an aliphatic hydrocarbon radicle, X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base, and in which amides the group

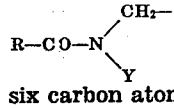


contains at least six carbon atoms.

4. A sulphonated carboxylic amide corresponding to the general formula

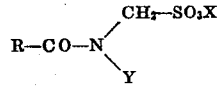


in which R—CO is the acyl radicle of an aliphatic carboxylic acid, Y is hydrogen or a hydrocarbon radicle, X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base, and in which amides the group

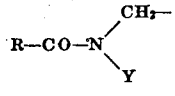


contains at least six carbon atoms.

5. A sulphonated carboxylic amide corresponding to the general formula

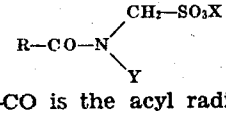


in which R—CO is the acyl radicle of a fatty acid, Y is hydrogen or a hydrocarbon radicle, X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base, and in which amides the group



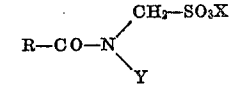
contains at least six carbon atoms.

6. A sulphonated carboxylic amide corresponding to the general formula



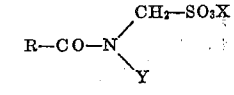
in which R—CO is the acyl radicle of a fatty acid of vegetal oils or fats, Y is hydrogen or an aliphatic hydrocarbon and X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base.

7. A sulphonated carboxylic amide corresponding to the general formula



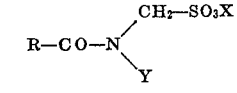
in which R—CO is the acyl radicle of a fatty acid of a vegetable oil or fat, Y is hydrogen or an aliphatic hydrocarbon and X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base.

8. A sulphonated carboxylic amide corresponding to the general formula



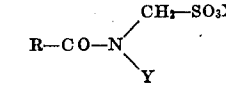
in which R—CO is the acyl radicle of an unsaturated fatty acid of a vegetable oil or fat, Y is hydrogen or an aliphatic hydrocarbon and X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base.

9. A sulphonated carboxylic amide corresponding to the general formula



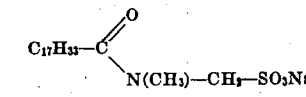
in which R—CO is the acyl radicle of a saturated fatty acid of a vegetable oil or fat, Y is hydrogen or CH_3 and X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base.

10. A product comprising a salt of an alkaline base with a fatty acid of a vegetal oil and a sulphonated carboxylic amide corresponding to the general formula



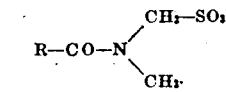
in which R—CO is the acyl radicle of a fatty acid of a vegetal oil, Y is hydrogen or an aliphatic hydrocarbon and X is hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base corresponding to that of said salt.

11. Oleic methylamidomethane sulphonic acid sodium salt corresponding to the formula



12. The process for the production of sulphonated carboxylic amides which comprises acting with an agent selected from the group consisting of carboxylic halides and anhydrides on an aminomethane sulphonic acid, containing at least one hydrogen atom connected to the nitrogen atom, in the presence of an alkaline diluent, care being taken that the sum of carbon atoms in said carboxylic halides or anhydrides and in said aminomethane sulphonic acid is at least six.

13. A sulphonated carboxylic amide corresponding to the formula



in which R—CO denotes the acyl radicles of the acids of coconut oil and X stands for hydrogen or the salt forming portion of an alkaline, metal or nitrogenous base.

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