

## UNITED STATES PATENT OFFICE

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## SOFTENING AGENT

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This invention relates to treating agents for fibrous materials and more particularly to agents for softening leather, paper and textile fibers. Still more particularly the invention relates to the combination of sulfamic acid or one of its salts with materials obtained by the sulfation of long-chain aliphatic alcohols as products for softening leather, paper and textile fibers and further to the use of these products in softening leather, paper and textile fibers.

This invention has as an object the preparation of novel softening agents and their use in softening leather, paper and textiles by combining the softening properties of long-chain alcohol sulfate ester salts or products based on long-chain alcohol sulfate ester salts with the hygroscopic properties of sulfamic acid or its organic and inorganic salts. The alcohol sulfate ester salts have the property of making fibrous materials, such as cotton, hemp, jute, linen, wool, silk, regenerated cellulose, cellulose acetate and wood pulp when in the form of filaments or sheets pliable so that they feel very soft when handled and in certain cases, to be described later, smooth to the touch.

It has been found that when sulfamic acid or its salts are used in combination with long-chain alcohol sulfate ester salts or softening agents based on long-chain alcohol sulfate ester salts, a novel finish is obtained on leather, paper and textile fibers which is in many cases more pleasing than the effect obtained without the use of sulfamic acid or its salts. The objects of this invention are accomplished by the addition of sulfamic acid or one of its salts to baths for treating leather, paper and textiles which contain long-chain alcohol sulfate esters or a softening agent based on long-chain alcohol sulfate esters or by mixing sulfamic acid or one of its salts with the long-chain alcohol sulfate esters or a softening agent based on the long-chain alcohol sulfate esters and then adding this mixture to the textile treating bath.

The invention comprises adding to the treating bath from 0.2 to 15 parts of sulfamic acid or one of its salts per part of long-chain alcohol sulfate ester or per part of a softening agent based on long-chain alcohol sulfate esters, as determined on a dry basis. The sulfamic acid or its salt may also be mixed with the long-chain sulfate ester or with the softening agent based on long-chain alcohol sulfate esters in a dry form or diluted with a small amount of water to form a concentrated paste which mixture may be later diluted to the desired extent to form a treating bath.

The long-chain alcohol sulfate ester salts may comprise the sulfuric ester salts of alcohols having from 8 to 20 carbon atoms or mixtures thereof. They may be produced by complete or by partial sulfation of the alcohol, and they may be neutralized with alkali metal bases or with organic bases. The preparation of these products is already described in the literature and is well known to the art. Products based on long-chain alcohol sulfate esters may comprise products in which hydrocarbons, such as the aliphatic hydrocarbons derived from petroleum, are mixed with the alcohol before sulfation. Examples of such products are described in the pending application of L. B. Arnold, Jr., Serial No. 137,374, filed April 16, 1937. Such products may also comprise products in which unsaturated long-chain alcohols are treated with acid anhydrides, such as acetic anhydride, prior to sulfation. Both of these classes of products may be neutralized either with alkali metal bases or with organic bases.

The following examples illustrate the use of sulfamic acid or its salts in conjunction with alcohol sulfate ester salts or softening agents based on alcohol sulfate ester salts in softening leather, paper and textiles.

*Example 1*

An aqueous solution is prepared containing 0.2% of sodium cetyl sulfate and 0.1% of sodium sulfamate. It is heated to 50° C. and applied to fabrics made from regenerated cellulose or cotton by passing them through the solution. After drying the fabrics are found to have a very smooth surface in addition to being pliable.

*Example 2*

An aqueous solution is prepared containing 0.5% by weight of the sodium salt of the product obtained by the conjoint sulfation of stearyl alcohol and petrolatum and 0.5% by weight of ammonium sulfamate. Taffeta fabrics woven from regenerated cellulose and broadcloth fabrics woven from cotton after being treated in this solution at 40–60° C. and dried are found to possess a very smooth velvety surface as well as being soft and pliable. Many other types of fabrics from all known textile fibers, synthetic or natural, may be treated in a similar manner. For example, crepe fabrics from artificial silk, crepe fabrics from artificial silk and cotton, printed dress fabrics from cotton and draperies from artificial silk and from cotton may be treated with sulfamic acid or its salts and alco-



hol sulfate esters or softening agents based on alcohol sulfate esters to enhance their finish.

#### Example 3

5 Paper is treated by padding onto it an aqueous solution containing 0.1% by weight of sodium cetyl sulfate and 2.0% by weight of potassium sulfamate. After drying the paper has a soft, smooth surface.

10 The following examples illustrate the preparation of alcohol sulfate ester salts and softening agents containing sulfamic acid or its salts.

#### Example 4

15 19.3 parts by weight of cetyl alcohol are sulfated with 7.6 parts by weight of chlorosulfonic acid with agitation of the mass over a period of from 30 to 40 minutes with the temperature maintained between 55° C. and 65° C. The mass is then stirred for 4 or 5 minutes more to allow the reaction to come to an end. 60.3 parts by weight of 5.4% aqueous sodium hydroxide at 30° C. are then added to the reaction mass slowly during stirring. 20 parts by weight of ammonium sulfamate are then added, the temperature is raised to between 45° C. and 50° C., and the mass is then stirred until it is homogeneous. The product is a homogeneous, white paste in appearance. It emulsifies readily in warm water with simple agitation.

30 The sulfation may be also carried out to obtain still less complete sulfation of the aliphatic alcohol in order to change the finishing effect of the product. Textiles, leather and paper may be treated with this product following the procedure of Examples 1, 2 and 3.

#### Example 5

40 19.3 parts by weight of stearyl alcohol and 12.8 parts by weight of refined petrolatum (M. P. range 40-50° C.; Iodine No. 10-20) are melted together and 7.6 parts by weight of chlorosulfonic acid (92% by theory on weight of stearyl alcohol) are added during agitation of the mass over a period of from 30 to 40 minutes with the temperature maintained between 55° C. and 65° C. The mass is then stirred for 3 or 4 minutes to allow the reaction to come to an end. 60.3 parts by weight of 5.4% aqueous sodium hydroxide at 30° C. are added to the reaction mass slowly while stirring. 45 parts by weight of ammonium sulfamate are added. The temperature is brought to from 50° C. to 60° C., and the mass is stirred until it is homogeneous.

55 The product is a homogeneous, white paste in appearance. It emulsifies readily in warm water with simple agitation and dilute emulsions even up to 5% will not separate after from 10 to 20 minutes boiling or even while cooling from the boiling point.

60 The fatty alcohol which is sulfated may also be lauryl, myristyl, cetyl, oleyl or mixtures of these alcohols. Mineral oil or paraffin wax or mixtures of these materials with petrolatum may be used instead of the petrolatum.

65 Sulfamic acid itself and other of its salts than those shown in the above examples may be used

with somewhat less effective results in most cases. Suitable salts are the calcium, magnesium, triethanolamine, trimethyl amine, stearyl trimethyl ammonium, diethanol stearamide, etc. In the claims we use the term "sulfamate" to cover sulfamic acid (hydrogen sulfamate) as well as its salts.

If a dry product is required, the sulfation product may be dried immediately after neutralization, and the dry product ground with the dry salt of sulfamic acid, or the finished product may be dried after addition of the salt of sulfamic acid and ground to a stable powder form. The product may also be dried in flake form on such apparatus as a drum drier. The products of Examples 4 and 5 may be applied to leather, paper or textiles at concentrations varying over a wide range but more particularly at concentrations varying from 0.05 to 5% in solution under the conditions illustrated in the first three examples.

The above description and examples are intended to be illustrative only and not to limit the scope of the invention. Any departure therefrom which conforms to the spirit of the invention is intended to be included within the scope of the appended claims.

We claim:

1. A process of imparting an improved softened finish to a fibrous material selected from the group consisting of textile fabrics, paper, and leather which comprises applying to said material an aqueous medium containing a sulfamate having the general formula:  $H_2NSO_3X$ , wherein X is a substituent selected from the group consisting of hydrogen and a salt forming radical, and a long chain alcohol sulfate ester salt containing 8 or more carbon atoms.

2. A process of imparting an improved softened finish to a textile fabric which comprises treating said fabric with an aqueous medium containing an alkali metal or ammonium sulfamate having the general formula:  $H_2NSO_3M$ , wherein M stands for alkali metal or ammonium, and a long chain alcohol sulfate ester salt containing from 8 to 20 carbon atoms.

3. A textile finishing agent which is capable of imparting an improved softened finish to a textile fabric when it is applied thereto from an aqueous medium comprising a mixture of a sulfamate having the general formula:  $H_2NSO_3X$ , wherein X is a substituent selected from the group consisting of hydrogen and a salt-forming radical, and a long chain alcohol sulfate ester salt containing 8 or more carbon atoms.

4. A textile finishing agent which is capable of imparting an improved softened finish to a textile fabric when it is applied thereto from an aqueous medium comprising a mixture of an ammonium or alkali metal sulfamate having the general formula:  $H_2NSO_3M$ , wherein M stands for alkali metal or ammonium, and a long chain alcohol sulfate ester salt containing from 8 to 20 carbon atoms.

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