

# UNITED STATES PATENT OFFICE

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## LIME, MAGNESIA- AND ACID-RESISTING SULPHONATES PRODUCED FROM UNSAT- URATED FATTY ALCOHOLS

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The present invention relates to wetting and dispersing agents and has for an object to provide an improved process of producing sulphates and/or sulphonates of fatty alcohols.

5 The nature and objects of the invention will be understood from the following description:

It has been found that by the sulphation and/or sulphonation of oleyl alcohol and its homologues, products are obtained with properties varying according to the method of production employed. Depending upon whether the sulphation and/or sulphonation is performed at high temperatures or under substantial cooling, the sulphates and/or sulphonates possess different degrees of stability against inorganic products such as lime- and magnesia-compounds, further against acids, salts with salting out effects, etc. Products of astonishing resistance against the aforementioned substances are obtained if unsaturated fatty alcohols are sulphated and/or sulphonated at temperatures lower than or only slightly above the melting-point of fatty alcohols. Indeed, complete sulphation and/or sulphonation to an extent adapted to obtain clear solubility in water may not be successfully achieved by the methods employed up to the present time, the reason being that the fatty alcohols solidify at the temperatures desirable for the reaction and only an incomplete reaction takes place with sulphuric acid. It was found, however, that these difficulties can be eliminated either by thorough pre-cooling of the sulphuric acid and then gradually adding the fatty alcohol to the pre-cooled acid, or by adding agents which reduce the melting-point of the fatty alcohol thereby admitting a reduction of the temperature to 5° without involving solidification. The products produced are of great capillarity, and may be used as agents for wetting, steeping, dispersing, levelling and washing purposes. They may be employed in acidic or alkaline washing liquors, as well as in baths containing for instance bitter salt or alum. The use of the aforementioned products also prevents the formation of lime deposits and detrimental effects due to the precipitation of such lime-soap deposits on the fibre.

### Examples

1. 75 kg. of sulphuric acid (about 96–98%) are cooled down to –6° and 100 kg. of oleyl alcohol (iodine number of about 85) are gradually added in such a manner as not to raise the temperature of the reaction mixture above 0°.

The product neutralized by ordinary means is completely soluble in a 50% solution of epsom

salts and in water of 50% hardness. It is neither salted out by a 12% solution of Glauber's salt nor by a 20% solution of sodium chloride, and withstands boiling with sulphuric acid of 1% strength. Under the same circumstances a sulphate and/or sulphonate produced in the same manner, though at 30°, is salted out or decomposed.

2. To common oleyl alcohol (100 kg.), solidification point of 19°, there is added an equal quantity of carbon tetrachloride and the mass is sulphated and/or sulphonated by addition of 60 parts of sulphuric acid monohydrate at 5° and then at 0°. After being stirred for three hours at the same temperature and subsequent neutralization, the mixture is evaporated to a salve-like consistency.

The reaction product possesses the same properties as the products obtained in Example 1, and may be produced free of sodium sulphate by dissolving in spirit, butyl alcohol and the like and subsequent further vaporization.

The pure product may also be obtained by extracting the non-neutralized sulphation and/or sulphonation mixture without previous concentration by vaporization.

The expression "unsaturated fatty alcohols" is used in the claims to define the oleyl alcohol and its homologues hereinbefore mentioned and with which the present invention is particularly concerned.

We claim:—

1. A process for the production of soap-like products, comprising the gradual addition of unsaturated fatty alcohols to concentrated sulphuric acid which is pre-cooled to a temperature only slightly above the melting-point of the alcohols.

2. A process, according to claim 1, comprising the sulphonation of unsaturated fatty alcohols in presence of additional indifferent agents lowering the melting-point of said alcohols.

3. The process for the production of sulphuric acid reaction products of oleyl alcohol which comprises cooling concentrated sulphuric acid to a temperature approximating –6° C. and gradually adding thereto oleyl alcohol while maintaining the temperature below 0° C.

4. The process for the production of sulphuric acid reaction products, as defined in claim 3, wherein the proportion of alcohol and sulphuric acid approximates 100 to 75.

5. The process for the production of sulphuric acid reaction products which comprises mixing oleyl alcohol and carbon-tetra-chloride in a proportion of the order of 1 to 1 and reacting with

sulphuric acid at a temperature approximating 0 to 5° C. for approximately three hours.

6. The process for the production of sulphuric acid reaction products of unsaturated fatty alcohol which comprises cooling concentrated sulphuric acid to a temperature below the melting point of the alcohol and gradually adding thereto the alcohol while maintaining the materials at approximately the same temperature.

7. A process for the production of soap-like products comprising gradually adding unsaturated fatty alcohols to a concentrated sulphating agent which has been pre-cooled to a temperature substantially below the melting point of the alcohols but insufficiently low to cause solidification of the alcohols before contact and reaction with the acid occurs.

8. A process for the production of soap-like products comprising mixing concentrated sulphuric acid with unsaturated fatty alcohols in the presence of an inert diluent which produces a mixture having a melting point below that

of the alcohols, and reacting the acid and the alcohols at a temperature only slightly above the melting point of the alcohol-diluent mixture.

9. In the production of sulphated unsaturated fatty alcohols, the steps comprising cooling a concentrated sulphating agent to a temperature below the melting point of the unsaturated fatty alcohol employed and gradually adding the alcohol to the cooled mass while maintaining the mass at approximately the same temperature, said temperature being sufficiently high to prevent solidification of the alcohol being introduced.

10. In the production of sulphates of unsaturated fatty alcohols, the step comprising gradually adding an unsaturated fatty alcohol to concentrated sulphuric acid which has been pre-cooled to a temperature adapted to cause the alcohol to enter the solution at a temperature within the range below or only slightly above the melting point of the alcohol.

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