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F. W. MUNCIE

DETERGENT AND METHOD OF PREPARATION

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COCOA OIL

SODIUM HYDROXIDE

GLYCERENE

INTERMEDIATE PRODUCT

SULPHURIC ACID

SULPHURIC ACID ESTER

SODIUM HYDROXIDE

FINAL PRODUCT

Inventor

FWMuncie

By Richard K. Stevens

Attorney
This invention relates to the art of deterging, washing, cleaning and the like and particularly to a new type of chemical product useful in such processes and to a process for its manufacture.

The new products formed and used according to this invention are the result of the interaction of fatty oils, such as are commonly used in soap making, with glycerine and sulphuric acid. Such products are well adapted to replace soap for domestic use.

Ordinary soaps, that is, the alkali salts of the fatty acids, have been in use for many, many years and consequently, the commonly used soaps and the processes of manufacturing them have become quite standardized. To supply the demand, requires large soap manufacturing plants involving large initial investments and large up keep expenditures, this being mainly due to the fact that the saponification process in large scale production requires kettles of enormous size and power sources of commensurate size to furnish the necessary heat for the reaction.

The merchandizing of soap, however, has developed into a highly competitive field, thus reducing the retail selling price of soap and soap products to a hazardous level from the standpoint of the legitimate manufacturers with their large plants. The cost price to the manufacturers of good quality basic materials used in soap production is quite uniform and the processes of manufacture are all very much the same.

Soap, in addition, regardless of the quality of the fats and oils employed therein, is well known to have certain objectionable characteristics, and some manufacturers have attempted to meet the keen price competition by eliminating some of these characteristics from the products which they produce, thus to win for them by merit a price which will give a reasonable profit. Research to this end has been intensified during recent years and a large number of new products suggested for use.

One of the main objections to ordinary soaps is that they form insoluble curds with some of the metals that are commonly present in hard water or the materials to be washed. They also have other disadvantages, such as being alkaline and having insufficient detergent power when used in hard water. The compounds which have been suggested to replace soap, and which have to a certain extent already replaced it, are, most of them, sulphonates or sulphates of organic compounds. An examination of the compounds reveals that they differ widely in their wetting and detergent power, and also in their stability, foam producing ability, solubility and in the solubility of their salts. They differ also, of course, in the difficulty of their manufacture, the cost of the raw products necessary to make them, and in their structural formulas.

Among the better known of these compounds that have been suggested to replace soaps, are the sulphonated alcohols and certain other sulphonated products derived from fatty acids in which the carboxylic acid group has been either neutralized with an amine or esterified with ethyl or a similar alcohol. Each of these types of products has been found to have rather desirable characteristics. Most of the compounds suggested are relatively stable to acids and alkalis, have a higher detergent power in hard water than soap and good emulsifying properties, and form no precipitates with calcium and other metals that are commonly encountered. However, the difficulty of their manufacture is such that they are necessarily quite expensive in comparison with ordinary soaps, and hence, while they have been found commercially feasible for some industrial uses, particularly in the treatment of textile materials preparatory to dyeing, where their stability and the solubility of their salts are of particular importance, they do not find a ready market in competition with ordinary soap for domestic use.

Accordingly, it is the purpose of this invention to provide at a cost comparable to the cost of ordinary soap, a material that is superior to soap in wetting, emulsifying and detergent characteristics especially in hard water, that is non alkaline, and the metal salts of which are practically all soluble. To accomplish this object the present invention provides a product that looks, and foams in water much as does ordinary soap of high quality. The new product, however, has a far greater detergent power than soap in hard water and leaves no soap curd on the walls of the vessel in which it is used. Furthermore, this new product may be formed directly from the fatty oils and some other relatively inexpensive chemicals without the necessity of first converting the fatty oils to fatty acids and thereafter large quantities of by-products to be removed. Thus the cost of production is sufficiently low to enable the new product to compete directly on a price basis with ordinary soaps even under the present conditions of keen competition.

In the practice of this invention, fatty oils, which may or may not contain free fatty acid are reacted with anhydrous or at least substantially anhydrous glycerine and fuming sulphuric...
acid. Preferably, the resulting product is there-
after neutralized. The manner in which the
components are reacted may be varied, although a
procedure which has been found particularly sat-
satisfactory will be hereinafter described in detail.

Other examples of satisfactory procedures for
accomplishing this reaction are given in a co-
pending application, Serial No. 28,711, filed June
27, 1935.

Preferably, about two molecular weights of
substantially anhydrous glycerine are reacted
with one molecular weight of fatty oil and three
molecular weights of fuming sulphuric acid. In
order to bring the reaction to completion it has
been found desirable to have a considerable ex-
cess of sulphuric acid present. From this excess
a quantity of an inorganic sulphate is formed by
the neutralization of the resulting product.

After neutralization, water is usually removed
to solidify the product. This may be done by
the use of drying oils or by spraying, in much
the same way that soap is dried. The inorganic
sulphate from the neutralization may either be
left as a part of the final product or removed,
according to the properties desired in the prod-
uct. The deterging effectiveness of the product
is not impaired by the presence of the inorganic
salt, and the mixture has been found satisfac-
tory for most purposes, especially when the said
salt is present, while the elimination of the removal step
results in a considerable saving. Of course, if
the inorganic salt is removed, the concentration
of the organic detergent material in the product
is increased and hence the deterging power of a
given quantity of the final product is raised ac-
cordingly.

In describing, by way of example, an illustrative
process by which the new products may be pro-
duced, reference will be had to the accompanying
drawing in which the process is shown diagram-
matically by a flow sheet. The method illus-
trated and hereafter described is, however, only
given by way of example, and other methods may
be used for the preparation of the new products
within the scope of this invention.

One method, then, by which a new product has
been formed in accordance with this invention,
and as diagrammatically shown in the drawing,
consisted in reacting a quantity of fatty oil and
glycerine and then acting on the resulting prod-
uct with sulphuric acid and finally neutralizing.
For example, to 260 grams of distilled
water were added 64 pounds of anhydrous glycerine
(99.5%) and 1.2 pounds of caustic soda. The
mixture was heated to 175 to 205° C. for ap-
proximately an hour at the end of which time the re-
sulting product was found to be miscible with
95% ethyl alcohol. The caustic soda apparently
acts to catalyze the reaction but its use may be
eliminated provided the temperature of the fatty
oil and glycerine mixture is raised to 280-300°
C. and held there until the product is homo-
geneous and soluble in 95% ethyl alcohol.

To 281 pounds of the product of this first re-
action there were next added 526 pounds of fum-
ing sulphuric acid (102.8%, which contains ap-
proximately 12 1/2% free SO3) and the mixture
stirred at a temperature of around 40° C. until
the resultant product was miscible with water.

Sulphuric acid of this proportion and strength
was found to bring the reaction to such a state that a solution of 1% of the product in
water is substantially clear and non-turbid by re-
acting with the glycerine-fatty oil compound and
absorbing the water of reaction. It was found
that, in general, enough sulphuric acid should be provided to fulfill the molecular weight require-
ments given above, namely, three molecular
weights for every two molecular weights of gly-
cerine and one molecular weight of fatty oil, and
in addition there should be an amount of sulphuric acid sufficient to absorb one molecular weight of
water for each molecular weight of sulphuric acid
that is to enter the reaction. Since this water
needs to be absorbed readily, it is preferable that
the amount and concentration of the excess of
sulphuric acid be such that after absorbing the
water the concentration of the excess sulphuric
acid will still be not less than about 99.3%.

From this information the amount and concen-
tration of the sulphuric acid, which it is desirable
to use, was readily calculated and was neutralized
to a pH of about 6.0 to 7.0 with caustic soda, (it took around 815 pounds of a 50°
Baumé solution), and thereafter dried. The
drying can be accomplished either by a spraying
method such as set forth in the United States
patent to Dallas R. Lyfmont No. 1,852,800 or by
other well known soap drying methods.

The dry material contained around 60% of so-
dium sulphate and 40% of the organic material
and formed a dry, granular, non-hygroscopic
powder at room temperature. When warmed
to somewhere when the said is present, while the elimination of the removal step
resulted in a considerable saving. Of course, if
the inorganic salt is removed, the concentration
of the organic detergent material in the product
is increased and hence the deterging power of a
given quantity of the final product is raised ac-
cordingly.

As an example of a process by which the new
materials may be formed and freed from
salts, the fatty oil, glycerine and sulphuric acid
may be reacted as in the first example above.

Then, instead of neutralizing with caustic soda,
around 400 pounds of hydrated lime may be added
to the mixture as a 10% slurry. Calcium sul-
phate will be formed from the excess sulphuric
acid and lime and will precipitate. The calcium
salt of the new detergent product being soluble,
the mixture may be filtered to remove the calcium
sulphate. Preferably, an amount of water equal
to the amount of the solution is used to wash the
calcium sulphate free of detergent product.

The solution containing the calcium salt of the
new material may then be treated with a suitable calcium precipitant, as, for example,
sodium carbonate or sodium phosphate or sodium
oxide in an amount slightly in excess of that re-
quired to replace all of the calcium in the deter-
genit with sodium and precipitate the carbonate,
the carbonate, phosphate or oxalate. This pre-
icipitate may then be filtered off and the remain-
ing solution is ready for concentration as desired.

In order to stabilize the new product, the solution may, if it is found necessary, be ac-

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justed to a pH of 6.0 to 7.0. Thereafter, it may be concentrated by boiling until it has the correct solids content for whatever use it is required. The solution may be evaporated to form a solid product from a concentration of around 25% or over, by spraying or by the use of drying rolls. The dried product when warm is soft and flexible, but becomes quite hard and brittle at room temperature. It can be prepared as a powder. A 20% aqueous solution of the salt-free product is liquid above 10°C.

In some instances, it may be desirable to use the calcium salt of the new material directly without converting it into the sodium salt, and in other cases it may be to advantage to form the corresponding ammonium, triethanolamine, magnesium, potassium or other salts. These may be formed in the same manner as the sodium salts, or ammonia or triethanolamine may be added together with carbon dioxide to precipitate the calcium, instead of adding their carbonates or oxalates.

The calcium salt itself may be dried over a heated roll. In this case, a low temperature of drying is preferable, since the product has a tendency to char. The use of vacuum with the roll is desirable, or the product may be mixed to a slurry with a filler, such as calcium sulphate, chalk, bentonite, pumice or clay and dried by means of a heated roll.

The salt-free product may, however, be formed from the new material according to the example, if so desired, by extracting the product with alcohol to remove the product and leave the inorganic salt. Alternatively, the product may be extracted before neutralization, but after sufficient dilution to prevent reaction, with butyl alcohol. The butyl alcohol solution is thereafter neutralized with caustic or the like to the proper pH of around 6.0 to 7.0. Other solvents can be used in place of the alcohol and butyl alcohol mentioned.

In order to aid in the identification of the products and to demonstrate their effectiveness as detergents certain tests have been made upon a product made according to the first example and containing 60% sodium sulphate, and upon the same product without the sodium sulphate. The sodium sulphate containing product gave 450 cubic centimeters of foam when 50 cubic centimeters of an equeous solution containing 1% of the material (total solids) were shaken thoroughly in a 500 cubic centimeter closed graduated cylinder. The surface tension of a 0.25% solution was measured as 31.5 dyne per cm. and a 0.125% solution registered 31.8 dynes. The Du Nuoy tensiometer was used according to the Du Nuoy procedure.

Using a 1% solution of the sulphate containing material (60% sodium sulphate) as solution #1 and a 1% solution of the sodium sulphate-free material as solution #2 and adding thereto equal volumes of a number of reagents in 10% solutions and at room temperature, the following results were noted:

<table>
<thead>
<tr>
<th>Reagent</th>
<th>$\text{f}_1$</th>
<th>$\text{f}_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaCl$_2$·2H$_2$O</td>
<td>No precipitate</td>
<td>No precipitate</td>
</tr>
<tr>
<td>Na$_2$CO$_3$</td>
<td>Slight turbidity</td>
<td>No precipitate</td>
</tr>
<tr>
<td>MgCl$_2$·6H$_2$O</td>
<td>No precipitate</td>
<td>No precipitate</td>
</tr>
<tr>
<td>AlCl$_3$</td>
<td>No precipitate</td>
<td>No precipitate</td>
</tr>
<tr>
<td>Pb(NO$_3$)$_2$</td>
<td>White flocculent precipitate</td>
<td>White flocculent precipitate</td>
</tr>
<tr>
<td>CuSO$_4$·5H$_2$O</td>
<td>No precipitate</td>
<td>No precipitate</td>
</tr>
<tr>
<td>FeCl$_3$</td>
<td>No precipitate</td>
<td>No precipitate</td>
</tr>
<tr>
<td>ZnCl$_2$</td>
<td>No precipitate</td>
<td>No precipitate</td>
</tr>
<tr>
<td>AgNO$_3$</td>
<td>No turbidity</td>
<td>No turbidity</td>
</tr>
<tr>
<td>NaOH (saturated)</td>
<td>No turbidity</td>
<td>No turbidity</td>
</tr>
<tr>
<td>HCl</td>
<td>No turbidity</td>
<td>No turbidity</td>
</tr>
<tr>
<td>Hard water (600 ppm)</td>
<td>No turbidity cold or hot</td>
<td>No turbidity cold or hot</td>
</tr>
</tbody>
</table>

A test was also made of the detergent power of several well known detergents and of the sodium sulphate containing product made according to the first example above. The standard soil test was used and the following results obtained with a PufFrich photometer:

<table>
<thead>
<tr>
<th>Product tested</th>
<th>Soft water 20 ppm</th>
<th>Hard water 300 ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Palm and olive oil soap</td>
<td>100.9</td>
<td>100.5</td>
</tr>
<tr>
<td>2 Sodium laurel sulphate</td>
<td>100.5</td>
<td>100.5</td>
</tr>
<tr>
<td>3 Sodium oleyl methyl taurine</td>
<td>98.3</td>
<td>98.3</td>
</tr>
<tr>
<td>4 Present product</td>
<td>111.1</td>
<td>114.9</td>
</tr>
</tbody>
</table>

While coconut oil has been named in the above examples, it is to be understood that the use of other fatty oils will also result in satisfactory products, varying proportions being used to compensate for the difference in the molecular weights. Among the oils which have been satisfactorily used are tallow, soy bean oil, and palm oil. In fact, it has been found, that oils ordinarily considered to be inferior in making soap, may be used according to this invention to produce products of a very good grade and the use of even such materials as fish oils and garbage grease is within the scope of this invention. Also, glycol or other polyhydric alcohols may be substituted for glycerine, and other sulphating or even phosphating compositions such as pyrophosphoric acid or phosphoric acid containing phosphoric anhydride may be substituted for sulphuric acid. Monoglycerides may also be formed by the reaction with glycerine of fatty acids, naphthenic acids, abletic acid or the carboxylic acids produced by the air oxidation of paraffin hydrocarbons and thereafter reacted with sulphuric acid substantially as indicated above.

Although the process of forming the new product has been described in the examples as though it were a batch process, it is contemplated that by suitable arrangement of equipment the reacting compounds can be caused to flow continuously through a system of apparatus, thus permitting raw materials to be reacted in a continuous process to produce the new products. It is not desired in the present application to be in any way bound by the structural formula which the products herein described are believed to have, or by the chemical reactions which are
thought to take place during the formation of the products. The oils from which the products are made are known to be of a complex nature and the literature and researches upon the subject fail to give any absolute assurance as to the reactions that take place or the structural formulas of the compounds formed. Furthermore, since the oils are complicated mixtures of organic compounds it is possible that the different compounds react differently with the glycerine and sulphuric acid and it may be that this fact accounts for much of the value of the final product.

The following tentative explanation of the reactions and formulas are therefore given merely in aid of an understanding of this invention and with the reservation that they are but tentative.

In the examples above given, coconut oil, glycerine and sulphuric acid were specified as the main raw constituents. Coconut oil is comprised mainly of triglycerides, that is glycerine esterified with three molecules of fatty acid per molecule of glycerine. The fatty acids present in these esters in coconut oil are usually as follows:

- Lauric acid 45% C12H23COOH
- Myristic acid 25% C14H27COOH
- Capric acid 10% C10H20COOH
- Capryl acid 5% C10H18COOH
- Oleic acid 30% C17H33COOH
- Palmitic acid 5% C15H31COOH
- Stearic acid 5% C17H35COOH
- Caproic acid 5% C6H13COOH

For every three molecules of fatty acid present there is, of course, approximately one molecule of glycerine and when these fatty acids and the glycerine are combined, three molecules of water are dropped from the composition. The fatty acids in the oil may vary and, of course, will differ when the kind of oil is changed, but by using R as the fatty acid radical, the formula for the triglyceride may be considered to be

\[ \text{R} \text{OH} \text{OH} \text{OH} \]

When one molecule of triglyceride of this formula is reacted with two molecules of glycerine it appears that the following reaction may take place:

\[ \text{R} \text{HOH} + 2 \text{HOH} \rightarrow \text{R} \text{HOH} + \text{HOH} \]

\[ \text{R} \text{OH} + \text{HOH} \rightarrow \text{R} \text{OH} + \text{HO} \]

The addition of fuming sulphuric acid to the monoglyceride effects the formation of a new product, apparently according to the following reaction:

\[ \text{R} \text{OH} \rightarrow \text{R} \text{O} \text{SOH} \]

This product when neutralized with caustic soda results in the final product apparently of the formula:

\[ \text{R} \text{O} \text{SOH} \rightarrow \text{R} \text{O} \text{SOH} \text{Na} \]

According to the reactions and the formula given, the final product is the monoglyceride sulphate of sodium either with or without a quantity of sodium sulphate formed in the neutralization.

It is intended that the present products shall be used directly in place of the ordinary soaps, particularly for laundry, toilet uses and general domestic purposes, but it is possible to use the new products in combination either with the ordinary type of soap or with other types of sulphonated detergents or wetting agents. The new material may be compounded with sodium silicate, tallow, saponin, whale, tallow, raphana, phenols, titanium dioxide, boric acid sulphate or other materials commonly incorporated in soap.

The final products may be obtained by suitable manipulation in the form of cakes, powder, flakes or solutions and are adaptable for use as ingredients in shaving, facial or dental creams, liquid shampoos, mouth washes or cleansing solutions, shampoo or dental powders, as an addition to drug stuff baths and for general detergent purposes either alone or in admixture with soap. Since the products are not precipitated by mercury or silver they may also be used to advantage in compounding antiseptic or germicidal detergents with mercury or silver salts. Furthermore, the product has a very much milder taste and odor than ordinary soaps prepared from the same oils and is therefore of particular value in dentrifices or mouth washes. Also, the material is not precipitated by sea water or hard water and hence is particularly valuable where such waters must be used.

For practical use as a detergent it is important that the material produce a clear solution with water. The substantially pure material produced according to this invention does this as is shown in the data given above.

What I claim is:

1. In a process of forming a composition of matter including a substantial proportion of a salt of a sulphuric acid ester of a monoglyceride, the steps that comprise reacting a monoglyceride with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid, and thereafter neutralizing the mixture.

2. In a process of forming a composition of matter including a substantial proportion of a salt of a sulphuric acid ester of a cocoanut oil monoglyceride, the steps that comprise reacting cocoanut oil monoglyceride with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid, and thereafter neutralizing the mixture.

3. In a process of forming a composition of matter including a substantial proportion of a salt of a sulphuric acid ester of a fatty acid monoglyceride, the steps that comprise reacting a fatty acid monoglyceride substantially free of glycerine and free fatty acid with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid, and thereafter neutralizing the mixture.
4. In a process of forming a composition of matter including a substantial proportion of a salt of a sulphuric acid ester of a monoglyceride, the step that comprises reacting a monoglyceride of an acid of the said group with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

5. In a process of forming a composition of matter consisting principally of a substantially pure salt of a sulphuric acid ester of a monoglyceride, the steps that comprise reacting a monoglyceride with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid, thereafter neutralizing the mixture.

6. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a monoglyceride, the step that comprises reacting a monoglyceride with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

7. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a monoester of a fatty acid, the step that comprises reacting a monoglyceride with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

8. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a fatty acid monoester, the step that comprises reacting a monoglyceride with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

9. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a fatty acid monoglyceride, the step that comprises reacting a monoglyceride substantially free of glycerine and free fatty acid with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

10. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a monoglyceride of an acid of the group consisting of fatty acids, naphthenic acids, abietic acid, and the carboxylic acids produced by the oxidation of paraffin hydrocarbons, the step that comprises reacting a monoglyceride of an acid of the said group with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

11. In a process of forming a composition of matter including a substantial proportion of a salt of a sulphuric acid ester of a polyhydric alcohol mono-ester of an acid of the group consisting of fatty acids, naphthenic acids, abietic acid, and the carboxylic acids produced by the oxidation of petroleum hydrocarbons, the steps that comprise reacting a polyhydric alcohol mono-ester of a fatty acid with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid, and thereafter neutralizing the mixture.

12. In a process of forming a composition of matter including a substantial proportion of a salt of a sulphuric acid ester of a polyhydric alcohol mono-ester of a fatty acid with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

13. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a polyhydric alcohol mono-ester of a fatty acid with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

14. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a polyhydric alcohol mono-ester of a fatty acid with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.

15. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a polyhydric alcohol mono-ester of a fatty acid with fuming sulphuric acid of such strength and quantity as will react therewith and leave an excess of sulphuric acid in the mixture, which excess will, after the completion of the reaction, have a concentration of not less than about 99.3% sulphuric acid.
tion of not less than about 99.3% sulphuric acid. 16. The process of forming a composition of
matter including a substantial proportion of a salt
of a sulphuric acid ester of a monoglyceride com-
prising reacting anhydrous glycerine, fatty oil, and
fuming sulphuric acid, the oil and glycerine being
present in substantially the proportions of 1 mol.
of fatty oil to 2 mos of glycerine and the
sulphuric acid being of such strength and quanti-
ty as will react and leave an excess of sulphuric
acid in the mixture, which excess will, after the
completion of the reaction, have a concentration
of not less than about 99.3% sulphuric acid and
thereafter neutralizing the mixture.

17. The process of forming a composition of
matter including a substantial proportion of a
sulphuric acid ester of a monoglyceride comprising
reacting anhydrous glycerine, fatty oils, and
fuming sulphuric acid, the oil and glycerine being
present in substantially the proportions of 1 mol.
of fatty oil to 2 mos of glycerine and the
sulphuric acid being of such strength and quantity
as will react and leave an excess of sulphuric
acid in the mixture, which excess will, after the
completion of the reaction, have a concentration
of not less than about 99.3% sulphuric acid.

18. In a process of forming a composition of
matter including a substantial proportion of a
salt of a sulphuric acid ester of a monoglyceride,
the steps that comprise reacting a monoglyceride
with fuming sulphuric acid of such strength and
quantity as will react therewith and leave an
excess of sulphuric acid in the mixture, which
excess will, after the completion of the reaction,
have a concentration of not less than about
99.3% sulphuric acid, thereafter neutralizing the
mixture with an agent of the group consisting of
hydroxides, oxides, and carbonates of the alka-
line earth metals, separating the inorganic sul-
phate from the mixture, reacting the mixture
with a salt of an acid capable of forming water
insoluble salts with alkaline earth metals, and
removing the resultant alkaline earth metal salt.

21. In a process of forming a composition of
matter including a salt of a sulphuric acid ester
of a monoglyceride and an inorganic salt, the
steps that comprise reacting a monoglyceride with
fuming sulphuric acid of such strength and quanti-
ty as will react therewith and leave an excess
of sulphuric acid in the mixture, which excess
will, after the completion of the reaction, have
a concentration of not less than about 99.3% sul-
phuric acid, and thereafter neutralizing the mix-
ture.

22. In a process of forming a composition of
matter including a salt of a sulphuric acid ester
of a monoglyceride and a by-product salt, the
steps that comprise reacting a monoglyceride
with fuming sulphuric acid of such strength and
quantity as will react therewith and leave an
excess of sulphuric acid in the mixture, which
excess will, after the completion of the reaction,
have a concentration of not less than about
99.3% sulphuric acid, thereafter neutralizing the
mixture, and spray drying the mixture.

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