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DETERGENT AND METHOD OF PREPARATION

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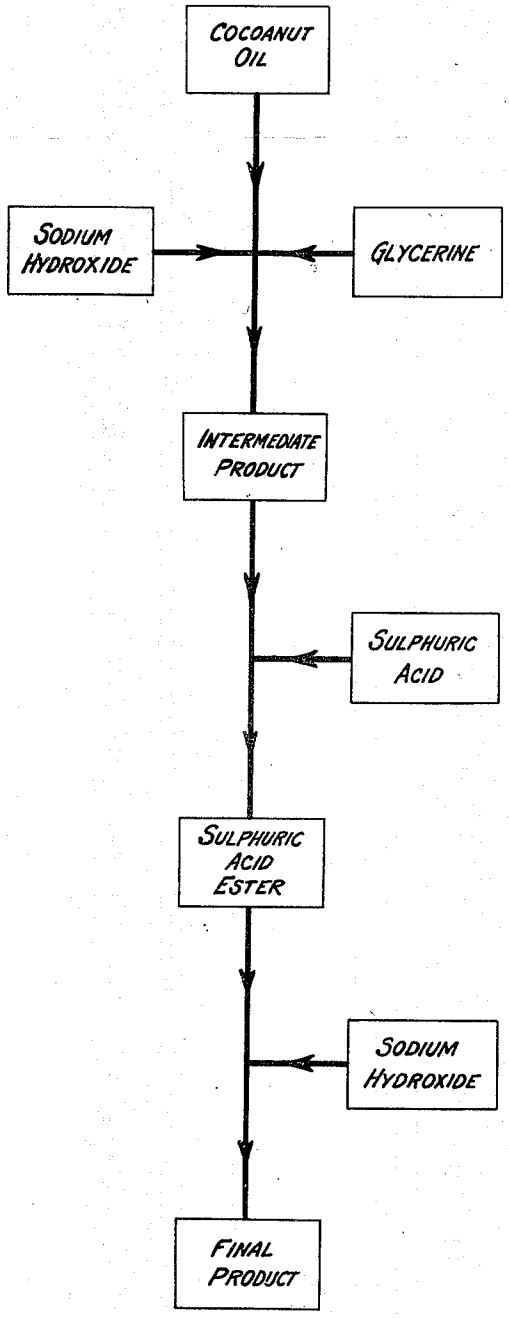

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DETERGENT AND METHOD OF PREPARATION

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22 Claims. (Cl. 260—400)

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 deterging 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 there are no 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 thereafter neutralized. The manner in which the compounds are reacted may be varied, although a procedure which has been found particularly satisfactory will be hereinafter described in detail.

Other examples of satisfactory procedures for accomplishing this reaction are given in a copending 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 excess 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 rolls 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 product. The deterging effectiveness of the product is not impaired by the presence of the inorganic salt, and the mixture has been found satisfactory for most purposes, even when the 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 accordingly.

In describing, by way of example, an illustrative process by which the new products may be produced, reference will be had to the accompanying drawing in which the process is shown diagrammatically by a flow sheet. The method illustrated 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 product with sulphuric acid and finally neutralizing. For example, to 216 pounds of cocoanut oil there 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 approximately an hour at the end of which time the resulting 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 280–290° C. and held there until the product is homogeneous and soluble in 95% ethyl alcohol.

To 281 pounds of the product of this first reaction there were next added 526 pounds of fuming sulphuric acid (102.8%, which contains approximately 12½% free SO_3) 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 sufficient 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 reacting 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 requirements given above, namely, three molecular weights for every two molecular weights of glycerine and one molecular weight of fatty oil, and in addition there should be an excess 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 concentration of the sulphuric acid, which it is desirable to use, was readily calculated. The product may be 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. Lamont No. 1,652,900 or by other well known soap drying methods.

The dry material contained around 60% of sodium sulphate and 40% of the organic material and formed a dry, granular, non-hygroscopic powder at room temperature. When warmed somewhat or compounded with an appropriate amount of water, glycerine or mineral oil, it could be milled, plodded or pressed into cakes by the use of the usual soap molding machinery.

The sodium sulphate or other sulphate formed by the neutralization can be removed, if desired, but this is not usually necessary, and, in fact, the presence of this inorganic salt in the final products, in many instances, appears to improve the desirability of the material, particularly for domestic use. The product made according to the first of the above examples will contain about 60% sodium sulphate. For some purposes, however, it may be desired to produce a detergent according to the present invention but free from the inorganic sulphates formed by the neutralization. To eliminate this inorganic sulphate it has been found easier to modify the original process by which the material is produced than to attempt to remove the inorganic sulphate, once it has been formed in the composition.

As an example of a process by which the new materials may be formed and freed of inorganic salts, the fatty oil, glycerine and sulphuric acid may be reacted as in the first example above. Then, instead of neutralizing with caustic soda, about 400 pounds of hydrated lime may be added to the mixture as a 10% slurry. Calcium sulphate 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 solution of a suitable calcium precipitant, as, for example, sodium carbonate or sodium phosphate or sodium oxalate in an amount slightly in excess of that required to replace all of the calcium in the detergent with sodium and precipitate the calcium as the carbonate, phosphate or oxalate. This precipitate may then be filtered off and the remaining solution is ready for concentration as desired

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

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 product made 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 new 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 dynes per cm. and a 0.125% solution registered 31.8 dynes. The du Nuoy tensionometer 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:

	Reagent	#1	#2
1	$CaCl_2.2H_2O$	No precipitate	No precipitate.
2	$BaCl_2$	White flocculent precipitate	Do.
3	$(NH_4)_2SO_4$	No precipitate	Do.
4	KCl	Slight turbidity	Slight turbidity.
5	$MgCl_2.6H_2O$	No precipitate	No precipitate.
6	$AlCl_3$	do	Do.
7	$Pb(C_2H_3O_2)_2$	White flocculent precipitate	Do.
8	$CuSO_4.5H_2O$	No precipitate	Do.
9	$FeCl_3$	do	Do.
10	$ZnCl_2$	do	Do.
11	$AgNO_3$	do	Do.
12	$HgCl_2$ (saturated)	do	Do.
13	Sea water	do	Do.
14	HCl	No turbidity	No turbidity.
15	Hard water (600 ppm.)	No turbidity cold or hot	No turbidity cold or hot.

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 Pulfrich photometer:

	Product tested	Relative detersive efficiency	
		Soft water 50 ppm.	Hard water 300 ppm.
1	Palm and olive oil soap	100.0	100.0
2	Sodium lauryl sulphate	106.9	123.6
3	Sodium oleyl methyl taurine	96.4	106.7
4	Present product	111.5	140.9

While cocoanut 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 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 for the purpose of 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, abietic 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 order to aid in understanding this invention and with the reservation that they are but tentative.

In the examples above given, cocoanut oil, glycerine and sulphuric acid were specified as the main raw constituents. Cocoanut 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 cocoanut oil are usually about as follows:

Lauric acid	45%	$CH_3(CH_2)_{10}COOH$
Myristic acid	20%	$CH_3(CH_2)_{12}COOH$
Capric acid	10%	$CH_3(CH_2)_8COOH$
Caprylic acid	9%	$CH_3(CH_2)_6COOH$
Oleic acid	6%	$CH_3(CH_2)_7CH{:}CH(CH_2)_7COOH$
Palmitic acid	5%	$CH_3(CH_2)_{14}COOH$
Stearic acid	3%	$CH_3(CH_2)_{16}COOH$
Caproic acid	2%	$CH_3(CH_2)_4COOH$

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

$$\begin{array}{l} CH_2OOCR \\ | \\ CHOOCR \\ | \\ CH_2OOCR \end{array}$$

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

$$\begin{array}{l} CH_2OOCR \\ | \\ CHOOCR \\ | \\ CH_2OOCR \end{array} + 2 \begin{array}{l} CH_2OH \\ | \\ CHOH \\ | \\ CH_2OH \end{array} \longrightarrow 3 \begin{array}{l} CH_2OOCR \\ | \\ CHOH \\ | \\ CH_2OH \end{array}$$

This reaction takes place upon the application of heat and is aided by the presence of caustic soda or other alkali, soaps, alcoholates, certain metals and other catalysts. The resultant products may be designated monoglycerides.

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

$$\begin{array}{l} CH_2OOCR \\ | \\ CHOH \\ | \\ CH_2OH \end{array} + H_2SO_4 \longrightarrow \begin{array}{l} CH_2OOCR \\ | \\ CHOH \\ | \\ CH_2OSO_3H \end{array} + H_2O$$

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

$$\begin{array}{l} CH_2OOCR \\ | \\ CHOH \\ | \\ CH_2OSO_3H \end{array} + NaOH \longrightarrow \begin{array}{l} CH_2OOCR \\ | \\ CHOH \\ | \\ CH_2OSO_3Na \end{array} + H_2O$$

At the same time any excess of sulphuric acid is converted into sodium sulphate.

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, talc, pumice, whiting, feldspar, naphtha, phenols, titanium dioxide, barium 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 dyestuff 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 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 steps that comprise 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, and thereafter neutralizing the mixture.

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, separating the product from the mixture by extracting with alcohol, and drying.

6. In a process of forming a composition of matter consisting principally of a substantially pure salt of a sulphuric acid ester of a cocoanut oil monoglyceride, the steps that comprise reacting a cocoanut oil monoglyceride with 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, separating the product from the mixture by extracting with alcohol, and drying.

7. 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.

8. In a process of forming a composition of matter including a substantial proportion of a sulphuric acid ester of a cocoanut oil monoglyceride, the step that comprises reacting a 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.

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 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.

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 an acid of the group consisting of fatty acids, naphthenic acids, abietic acid, and the carboxylic acids produced by the oxidation of petroleum hydrocarbons 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 cocoanut oil fatty acid, the steps that comprise reacting a polyhydric alcohol mono-ester of a cocoanut oil 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.

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 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 step that comprises reacting 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 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, the step that comprises 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.

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 cocoanut oil fatty acid, the step that comprises reacting a polyhydric alcohol mono-ester of a cocoanut oil 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 concentra-

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 comprising 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 mols 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 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 mols 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 alkaline earth metals, separating the inorganic sulphate from the mixture, reacting the mixture with an alkali metal salt of an acid capable of forming water insoluble salts with alkaline earth metals and removing the resultant alkaline earth metal salt.

19. 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 calcium hydroxide, separating calcium sulphate from the mixture, reacting the mixture with sodium carbonate, and removing the resultant calcium carbonate.

20. 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 alkaline earth metals, separating the inorganic sulphate from the mixture, reacting the mixture with a salt of an acid capable of forming water insoluble salts with alkaline earth metals, said salt having a positive ion selected from the group consisting of alkali metals, ammonium, and organic bases, 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 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.

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.

FRED WEAVER MUNCIE.