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SOAP AND PROCESS FOR MAKING SAME

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This invention relates to compositions containing fats, oils or fatty acids, which are brought into contact with the human skin during their use and to processes for producing such compositions free from skin irritating characteristics. Such compositions would include soaps of all types, such as bar, powdered, cream and liquid soaps, shampoos, dentifrices, and cosmetic preparations, such as cold cream, vanishing cream, and other such substances containing fatty acids, saponified and unsaponified oils. The invention has been found to apply with particular advantage to the obtainment of soaps having superior mildness and improved sudsing and lathering properties and for this reason will be described with relation to soap. It will be appreciated however, that this invention is readily applicable to all substances of the general type indicated.

A common type of soap is a blended mixture of animal and vegetable oils. However, other compositions or blends may be used and soaps have been produced without blending. Cosmetic preparations may contain varying amounts or blends of animal, vegetable and mineral oils, fats or fatty acids, saponified or unsaponified. This invention is not limited to the use of any specific composition but is applicable to all types of oil, fats or fatty acid of vegetable, mineral or synthetic origin containing relatively low molecular weight components.

The higher grade tallows suitable for producing soaps of good keeping qualities have a high titre and a smaller percentage of oleic acid than low titre tallows. Palmitic and stearic fatty acids are also present in predominant proportions. This type of animal fat has only minimum proportions of low molecular weight components. Various other types of animal fats, however, have small but significant proportions of low molecular weight fats and fatty acids. Synthetic fats and fatty acids now being produced also contain in certain cases proportions of low molecular weight components. Vegetable oils such as palm kernel, babassu, tucuum, cohune and coconaut, are rich in low molecular weight glycerides and free fatty acids, or a similar type such as, for example, caproic, caprylic, capric, myristic and lauric acids.

It is known that soaps of good quality can be produced from high titre tallow, and that while mild in their action, such soaps generally have a relatively low solubility in water and are slow to lather.

It is also known that soaps made from oils of the type of cocoanut oil are readily soluble and

lather quickly. In the manufacture of soap it is consequently an almost universal practice to blend a proportion of cocoanut or similar oil containing lower fatty acids with the harder fats such as a high titre tallow to improve the solubility and lathering characteristics of the finished soap.

There are, however, certain disadvantages in the use for soap making of vegetable oils of the type, for example, of cocoanut oil. One of the major disadvantages is that a soap containing a fat or oil of the type of cocoanut or palm kernel oil, for example, is usually less mild than one made entirely from tallow, for example. A cocoanut oil soap, or one containing a substantial proportion of such soap, is known to be more irritating to the skin than a tallow soap. In the case of certain more sensitive skins, the effect may be so great as to make it necessary to avoid entirely the use of such soap.

We have discovered as the result of many tests that not all of the lower molecular weight components are irritating to the skin, but rather that only components of a molecular weight less than lauric are irritating. Such fatty acids would be, for example, capric, caprylic and caproic, each of which has a molecular weight less than 200, which is approximately the molecular weight of lauric acid.

We have found that not all the soaps of the lower molecular weight fatty acids which are derived from oils or fats of the type of cocoanut oil have desirable lathering properties. Of the soaps of the fatty acid of a molecular weight lower than palmitic, only those of myristic and lauric acids possess desirable lathering properties. The soaps of the acids lower than lauric acid, such as capric, caprylic and caproic have very inferior lathering or sudsing properties.

We have also found that a soap with desirable lathering or sudsing properties, improved mildness and freedom from irritating properties can be made from oils or fats of the type of cocoanut or palm kernel oil. This may be done if such oils or fats, their fatty acids or resulting salts are treated to remove the components of lower molecular weight than lauric acid, while retaining the lauric acid and other components of higher molecular weight.

An object of the invention is to produce soaps and cosmetic preparations free from skin irritating properties.

An object of the invention is to produce a soap having improved lathering properties and mildness.

A further object of the invention is to produce soaps having desirable lathering properties as a result of the use of low molecular weight fatty acids but which soaps are mild in their action on the skin. Other objects and advantages will become apparent to those skilled in this art in view of the following disclosure.

The invention is described with respect to soaps for the purpose of illustration and as it is particularly applicable thereto in view of the control of the lathering properties as well as the skin irritating characteristics. This description is not to be considered, however, as imposing limitations upon the broader aspects of the invention.

The lathering properties of the soaps of caproic, caprylic, capric, lauric and myristic acids have been determined by tests. The following comparative data arranged in Table I has been compiled as a result of these tests and indicates the thickness of a layer of suds produced by uniform agitation of solutions of the several soaps.

Table I

Concentration of soap in grams/300 cc.	Sodium caproate C ₆	Sodium caprylate C ₈	Sodium caprate C ₁₀	Sodium laurate C ₁₂	Sodium myristate C ₁₄
3	Nil	1/2 inch not stable.	Inches Nil	Inches 1/2	Inches 1 3/4
5	Nil	do.	1/2	1	2 1/4
7	Nil	do.	3/4	1 1/2	2 3/4

These tests show that soaps made from fatty acids having molecular weights lower than lauric acid are in general substantially devoid of lathering qualities. When present with other soaps they add nothing to the sudsing qualities and may, in fact, even be detrimental to any lathering properties of the soaps of high fatty acids.

The lower molecular weight vegetable oils or their fatty acids may be separated by distillation. Distillation for this purpose may be carried out on the oils with or without the use of an inert carrier. For instance, steam may be used as a vehicle to assist in the selective fraction of the lighter molecular weight fatty acids.

We have found that fractionation may be advantageously accomplished by a continuous operation in which the fats, oils or fatty acids are fed into a fractionating column still and the selective distillation carried on preferably under a vacuum. The separated fractions having different boiling points may be drawn off continuously from the fractionating plates through outlets located at suitable points along the column. The lightest fractions or "tops" which contain the fatty acids of lowest molecular weights may be drawn off at the top of the column. The next heavier fractions or "middles" may be drawn off from intermediate plates or trays in the column. The heaviest fatty acids or "bottoms" may be removed from the lowermost zones of the column or even from the still itself.

Cocoonut oil fatty acids fractionated in accordance with the above method have been found to produce a "tops" fraction having an average molecular weight of between 165-175 which consists principally of caproic, caprylic and capric acids. The "middles" portion was found to have an average molecular weight of from 200-206 and consisted principally of lauric acid. The "bottoms" fraction removed from the lower portion of the column or from the still consisted prin-

cipally of fatty acids of higher molecular weight than lauric acid. When taken from the still this "bottoms" fraction may be subjected to a refining distillation for the purpose of removing polymerized and unsaponifiable material and improving its color. Since we have found that in vegetable oils, for example, it is only the fractions of molecular weights lighter than lauric acid which are detrimental to mildness and lathering, it is feasible to combine the "middles" and "bottoms" fractions to form what we will hereafter term the "heavy fraction." It is, of course, to be understood that the "middles" and "bottoms" fractions could be employed separately if desired.

The separated light fractions are generally light yellow liquids at room temperature and when brought into contact with the skin cause irritation and an unpleasant sensation that the skin is drawn and chapped. The light fractions also have to an intensified degree the unpleasant "soapy" taste characterizing cocoonut oil fatty acids, or palm kernel fatty acids.

The heavy fractions at room temperatures are brittle, water white solids. Their characteristics, in so far as taste and irritating properties are concerned, are found to be mild relative to the light fractions and, as tests have shown, these advantageous characteristics are carried through to the soap.

In order to further develop the advantages of the separation and removal of the light fractions, extensive tests were carried out to determine the irritating effect on the skin, if any, of comparative soaps made of (a) light fractions of palm kernel oil, (b) palm kernel oil, and (c) heavy fractions of palm kernel oil. 5% solutions of these soaps were skin tested on a large number of subjects. The comparative results of these tests are shown in the following chart:

	Soap from light fraction	Pure P. K. O. soap	Soap from heavy fraction
	Percent	Percent	Percent
No detectable reddening of the skin	29.8	55.3	74.5
Very slight trace of reddening	34.0	42.6	23.4
Slight reddening	29.8	2.1	2.1
Definite wide-spread reddening—patchy	6.4		
Complaint of itching	100.0 29.8	100.0 12.8	100.0 None

These tests show that soaps made from the light fractions are far more irritating to the skin than either the palm kernel oil soap or the soap made from the heavy fraction and that the heavy fraction soap is the least irritating to the skin of any of the soaps.

In the manufacture of soaps from fats, such as tallow and the vegetable oils, it is common to use between 65% and 85% tallow, for example, and about 35% to 15% of a vegetable oil suitable to produce a soap having desired lathering qualities. Such a soap has ordinarily been considered a mild soap. A comparison of this blended type of soap with a soap made from solely the heavy fraction of a vegetable oil discloses that the soap from the heavy fraction with no animal fat soap present has a superior mildness. Similarly, a blended soap of tallow and the so-called heavy fraction of a vegetable oil is very superior in mildness and other desirable properties to the usual blended type already referred to.

Many skin tests were made, using a large group of individuals, to determine and determine

strate these properties and the results have been compiled in the table below. In this series of tests, coconut oil soaps were used. Each skin test was made with a 5% solution of the soap.

As a further basis of comparison, there is included in the table of data below the results of tests with a soap made solely of the fatty acids of coconut oil. The first column relates to soaps made from the heavy fraction of coconut oil. The second column relates to a solely coconut oil soap. The third column relates to a soap made from a mixture of 80% tallow and 20% of the heavy fraction of coconut oil. The fourth column relates to a soap containing 80% tallow and 20% ordinary coconut oil.

	Soap from heavy fraction CNO	CNO soap	20% heavy fraction CNO soap—80% tallow soap	20% CNO soap—80% tallow soap
	Percent	Percent	Percent	Percent
No detectable reddening.	50.0	22.0	66.0	43.0
Very slight trace of reddening.	44.0	38.0	32.0	38.0
Slight (patchy).	6.0	36.0	2.0	8.0
Definite wide-spread reddening.	0.0	6.0	0.0	6.0
Complaint of itching.	100.0 0.0	100.0 30.0	100.0 0.0	100.0 10.0

The results tabulated above show that coconut oil soap alone, or mixed with tallow soap, is definitely more irritating than the respective soaps made from distilled or rectified coconut oil minus the light fractions containing the fatty acids having molecular weights lower than the molecular weight of lauric acid.

A typical example of a soap made in accordance with this invention having excellent lathering properties and superior mildness may consist of a mixture of about 80% by weight of tallow soap, and about 20% of the soaps of coconut and/or palm kernel oils which have been rectified or distilled to remove the free fatty acids having lower molecular weights than lauric acid. However, the proportions of animal fats and vegetable oils may be varied, as desired. Soaps having excellent lathering properties may be made from mixtures of between 65% and 85% tallow, or other similar fats, and 35% to 15% of the heavy fractions of the vegetable oils. In fact, desirable mild soaps can be made with increased amounts of the heavy fractions of the vegetable oils up to 100% of such fractions and no blended fat. Other vegetable oils may be used in obtaining a blended soap. If desired, only the separated lauric acid or the myristic acid fractions may be used, or they may be blended, to make a soap. These fractions, in turn, may be blended with other oils or animal fats within the entire range to give a soap of desired characteristics.

The use in soap making of these vegetable oils free from fractions of a molecular weight less than lauric acid eliminates the tendency to irritate the skin which characterizes soaps made from oil of the type of palm kernel and coconut oil, for example. At the same time the solubility in hard water and the lathering characteristics are improved, particularly when blends of soaps containing high molecular weight components, such as fats of the tallow type, are used.

The soap may be made in the usual manner, for example, by saponifying, salting out, crutching, framing or milling and drying. Any of the

known methods for treating vegetable oil containing soaps may be used and deflocculating agents, perfumes and coloring matter may be added as desired. Also the soap may be blended with additional agents, such as other detergent agents, sulphonated compounds, abrasives, or chemicals for modifying the detergent agent of the soap. These additional agents may be added by suitable mixing devices available to the trade. The finished soap may be in any desired form, for example, bars, chipped, sprayed, liquid or creamed. By the term soap we mean to include all types of soap, soap blends and soap containing compositions utilizing fats, oils and fatty acids.

It will be understood that the described soaps, cosmetic preparations and process may be modified in many ways without departing from the invention. For example, as to the cosmetic preparations, the blending of the treated fatty acids or oils with or without saponification, in desired amounts with other ingredients such as lanolin, mineral oil, glycerine, water or waxes may be carried out in any suitable apparatus to obtain a uniform distribution within the composition. Various types of blending devices are available on the market for this purpose. Therefore, the above described examples of the improved soap product and process should be considered as illustrative only and not as limiting the scope of the claims.

We claim:

1. A non-irritative soap product containing the soaps of fatty acids having at least 12 carbon atoms and substantially free of the soaps of fatty acids having less than 12 carbon atoms, at least a portion of said soap product consisting of soaps of the fatty material forming the residue of fatty oils of the class consisting of coconut, palm kernel, babassu, tucuum and cohune oils upon removal therefrom of fatty acids having less than 12 carbon atoms, including the fatty acids derived from the glycerides in said oil having fatty acid radicals of less than 12 carbon atoms, said soaps having at least as good lathering properties as those which characterize the soaps of the fatty acids of the fatty oils of said class and being free from the skin irritating properties normally characterizing said soaps of said fatty oils.

2. A non-irritative soap product containing the soaps of fatty acids having at least 12 carbon atoms and substantially free of the soaps of fatty acids having less than 12 carbon atoms, at least a portion of said soap product consisting of soap of the fatty material forming the residue of coconut oil upon removal therefrom of fatty acids, including the fatty acids derived from the glycerides in said oil having fatty acid radicals of less than 12 carbon atoms, said soap having at least as good lathering properties as those which characterize the soaps of the fatty acids of coconut oil and being free of the skin-irritating properties which characterize soaps of coconut oil.

3. A non-irritative soap product containing the soaps of fatty acids having at least 12 carbon atoms and substantially free of the soaps of fatty acids having less than 12 carbon atoms, at least a portion of said soap product consisting of soaps of the fatty material forming the residue of palm kernel oil upon removal therefrom of fatty acids, including the fatty acids derived from the glycerides in said oil having fatty acid radicals of less than 12 carbon atoms, said soap having at least as good lathering properties as those which characterize the soaps of the fatty acids of

palm kernel oil and being free of the skin-irritating properties which characterize the soaps of palm kernel oil.

4. In the process for making mild soap free from skin irritating properties from fatty oils containing low molecular weight components having skin irritating properties and higher molecular weight components free from skin irritating properties, the steps of fractionating the fatty acids of said fatty oils to remove a fraction containing fatty acids having less than 12 carbon atoms while retaining a fraction containing the fatty acids having at least 12 carbon atoms and making soap from the latter fatty acid fraction.

5. In the process for making a mild soap comprising the soaps of cocoanut oil fatty acids, said soap having good sudsing characteristics and freedom from skin irritating properties, the steps of fractionating cocoanut oil acids to remove a frac-

tion containing fatty acids of a molecular weight less than the molecular weight of lauric acid while retaining a fraction comprising the fatty acids of a molecular weight of lauric acid or greater, and reacting the latter fraction with an alkali to produce the soaps of the fatty acids thereof.

6. In the process for making a mild soap comprising the soaps of palm kernel oil fatty acids, said soap having good sudsing characteristics and freedom from skin irritating properties, the steps of fractionating palm kernel oil acids to remove a fraction having a molecular weight of less than the molecular weight of lauric acid while retaining a fraction comprising the fatty acids of a molecular weight of lauric acid or greater, and reacting the latter fraction with an alkali to produce the soaps of the fatty acids thereof.

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Disclaimer

2,300,416.—*Bror J. Grondal*, Watertown, and *Harry Morley Wylde*, Swampscott, Mass. SOAP AND PROCESS FOR MAKING SAME. Patent dated Nov. 3, 1942. Disclaimer filed Mar. 9, 1948, by the assignee, *Lever Brothers Company*.

Hereby enters this disclaimer of claims 2, 3, 4, 5, and 6 of said specification and that part of claim 1 thereof directed to cocoanut oil and palm kernel oil.

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