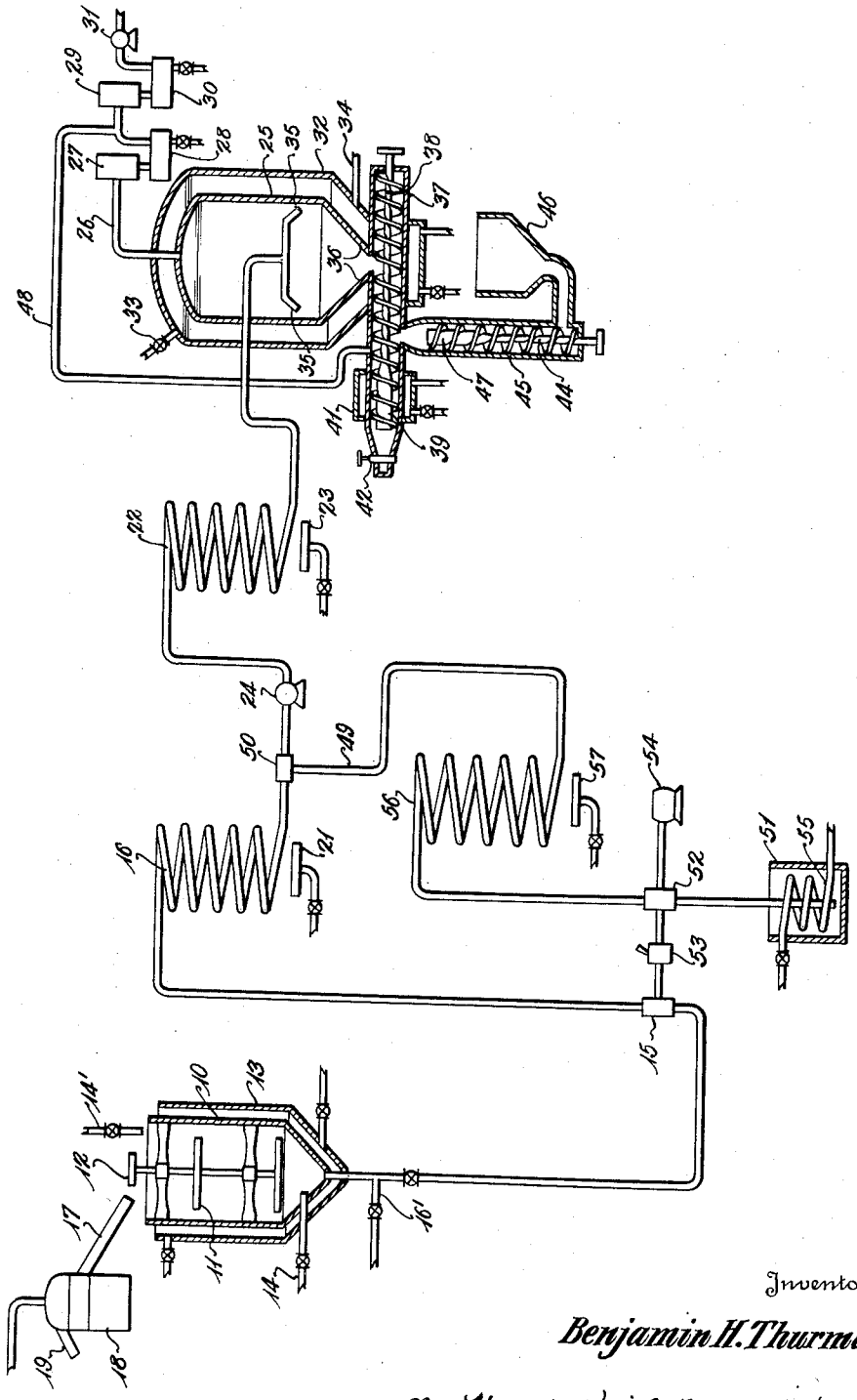
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SOAP STOCK PURIFICATION AND PRODUCT

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SOAP STOCK PURIFICATION AND PRODUCT

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5 Claims. (Cl. 252—117)

This invention relates to a soap product and more particularly to a product resulting from the treatment of soap stock from the alkali refining of animal and vegetable oils.

Such soap stock is produced by adding an alkali, such as caustic soda, to the animal and vegetable oils in sufficient amount to neutralize the free fatty acid therein and to provide an excess for reaction with coloring matter. The fatty acids react with the alkali to form soap and there is usually some reaction with neutral oil to form additional soap and liberate glycerine. Coloring matter and gums are precipitated along with the soap. These substances are separated from the refined oil as soap stock either by settling or centrifugal separation usually at a temperature between 100 and 160° F. The soap stock is ordinarily a viscous, slimy and dark colored material containing soap, uncombined oil, caustic soda solution, phosphatides, glycerine, albuminous or proteinaceous matter and other non-fatty matter. For example, the average composition of cottonseed oil soap stock according to Lewkowitsch is as follows:

	Per cent
Fatty anhydrides	48.50
Glycerine	3.98
Caustic soda (Na_2O)	3.20
Foreign organic matter	5.90
Water	36.00
Coloring matter	2.42
	100.00

Such a mixture is subject to fermentation and decomposition and cannot be kept in storage for any length of time without deterioration. This material has been employed in soap powder or cheap bar soap after being subjected to repeated boilings in soap kettles including the employment of salt or large amounts of a caustic soda to drain out the kettle and cause a partial separation between the soap and impurities therein. The soap lyes resulting from settling operations contain large quantities of organic impurities and considerable soap, as well as glycerine, but have no value and are sent into the sewer. This procedure is expensive and time consuming and according to Thomssen and Kemp's "Modern Soap Making" it requires a thousand pounds of soap stock having a 50% fatty acid content, 100 pounds of solid, 76% caustic soda and 100 pounds of salt to produce 750 pounds of settled soap. The resultant soap is soft bodied and has a characteristic and disagreeable odor. On account of these undesirable properties, the use of cottonseed soap stock is limited to soap powders and similar preparation which seldom contain more than 25% soap. Soda ash and other filling materials which disguise the odor, hide the color and also serve to preserve the soap are employed in large amounts. As further stated by Thomssen and Kemp, this soap was made up in cakes and was sold to some extent, but in recent years it has fallen in disrepute and little, if any, is made or used in bar form.

In accordance with the present invention, cottonseed soap stock, as well as soap stocks from other animal and vegetable oils, is purified by a simple process so as to produce a substantially pure soap stock product, non-odorous and non-decomposable, capable of shipment and of being incorporated into high quality soaps in proportions as high as 40 to 60%. The gums, resins, proteins and other foreign substances, including coloring matter, are largely removed from the soap stock to produce a relatively high quality soap and also valuable by-products.

An object of the invention is to provide an improved soap stock which is substantially dehydrated and which will not ferment or decompose during storage or shipment.

Another object of the invention is to provide an improved soap stock substantially free of odorous and putrifiable materials.

A further object of the invention is to provide an improved soap stock relatively free of impurities and capable of being employed as soap or being incorporated into high quality soap products in large proportions.

In accordance with the present invention, the soap stock may be subjected to high temperatures and pressures for sufficient length of time to break down impurities, such as proteins, etc., into compounds of less molecular weight so as to render them capable of vaporization or of being dissolved in water. The heated soap stock may then be introduced into a voporizing chamber at a lower pressure to vaporize the impurities as well as water from said soap stock. Sufficient alkali may be introduced into the soap stock to saponify all of the neutral oil or other fatty material in the soap stock during the process so that a completely saponified product results. The process is preferably carried out as a continuous process and will be described in connection with a suitable apparatus capable of carrying out such a continuous process. Such an apparatus is diagrammatically shown in the accompanying drawing.

Referring to the drawing, 10 indicates a mixing receptacle in which the soap stock is mixed with a suitable viscosity reducing material, such as steam, water or caustic soda solution with or without additional fat or fatty acids, so as to render the same capable of being pumped through the system for treating the soap stock. This mixer may be of any suitable construction such as a mechanical agitator or colloid mill for effectively mixing the water or caustic soda solution with the soap stock. By way of example, the mixer 10 is shown as a conventional mechanical agitator provided with paddles or impellers 11 driven from any suitable source of power by a pulley 12. The mixer may be surrounded by a heating jacket 13 so as to raise the temperature of the materials in the mixer 10 to reduce the viscosity of the same, or live steam or other heating medium may be introduced into the mixer 10 through a pipe 14 so as to directly contact the contents of the mixer to heat the same. The mixer 10 may be of large size and employed as a batch mixing device into which a quantity of soap stock is introduced and sufficient water or alkali solution added through the pipe 14' to give a proper consistency of soap stock and the resultant mixture continuously pumped from the mixer by the pump 15 and through a heating device shown as a coil 16. When employed as a batch mixing device, the agitators 11 of the mixer 10 are kept running during the withdrawal of material from the mixer so as to maintain a substantially uniform mixture leaving the mixer 10 and entering the coil 16. If desired, the soap stock in the mixer may be given one or more preliminary washes and impurities removed through pipe 16'.

Alternatively, soap stock from a refining process may be continuously introduced into the mixer 10 from the spout 17 of a continuous centrifugal separator 18 forming part of a refining process. In such a centrifugal separator the alkali treated oil can be continuously separated from the soap stock, the soap stock discharged as a heavy effluent from the spout 17 and the refined oil as a lighter effluent from the spout 19. Water or caustic soda solution can also be introduced in proper proportions through the pipe 14' into the mixer 10 so that the mixer functions as a continuous mixer, in which case it may be of relatively small size.

In either a batch or continuous mixing, it is preferred to introduce sufficient alkali into the mixer 10 to completely saponify during the process all of the neutral oil and other fatty materials contained in or added to the soap stock. Although there is usually some uncombined alkali in the soap stock, it is usually necessary to add additional alkali if complete saponification is to be effected. Also, sufficient water is added either as part of the alkali solution or water alone to produce a pumpable mixture. As will be hereinafter discussed, water alone may frequently be employed to produce a high quality soap stock even though all of the fatty materials in the soap stock have not been converted into soap.

The fluid mixture of soap stock and water or caustic solution is heated in the heating device 16 under an elevated pressure imposed by the pump 15. Heat can be supplied to the heating coil 16, for example, by a burner 21 for liquid or gaseous fuel. Since a substantial period of time of treatment under pressure and high temperatures is usually required for proper treatment of the soap stock, one or more additional flow heating devices, for example, heating coils 22 provided with burners 23, are usually desirable, and a second pump 24 may be provided for forcing the mixture through this second heating coil or coils 22. The heated mixture is then preferably discharged into an evaporating chamber 25 in which vaporizable materials are separated from the soap stock. The vaporizable materials are withdrawn through a pipe 26 to a condenser 27 provided with a receiver 28. If fractional condensation of the vaporizable materials is desired, one or more additional condensers 29 provided with receivers 30 may be employed and a vacuum pump 31 is preferably provided for maintaining a vacuum in the evaporating chamber 25, condensers 27 and 29 and receivers 28 and 30.

The evaporating chamber 25 is preferably provided with a heating jacket 32 through which any desired heating medium, such as steam or heated mineral oil, may be circulated by the pipes 33 and 34 so as to maintain the desired temperature in the evaporating chamber 25. In the preferred operation in which the temperatures imparted to the mixture in coil 22 and maintained in the evaporating chamber 25 are sufficient to cause the soap stock to be deposited in the evaporating chamber in substantially anhydrous molten form, the soap stock is discharged by nozzles 35 positioned to direct the soap stock against the walls of the chamber. This molten material flows down the walls of the chamber in thin films such that vapors are substantially completely separated therefrom and pass inwardly and upwardly through the center of the evaporating chamber in a substantially unimpeded path. This procedure provides for substantially no entrainment of liquids or solids in the vapors being removed from the evaporating chamber in the pipe 26. It is, of course, apparent that in certain operations where the temperatures are not sufficient to maintain the material discharged into the evaporating chamber in liquid form, a conventional spray nozzle may be employed to direct solid material toward the lower portion of the evaporating chamber.

The purified soap stock deposited in the evaporating chamber is directed by the sloping walls 36 of the lower portion of the evaporating chamber into the housing 37 of a screw conveyor 38. The housing 37 of the coveyor communicates with the interior of the evaporating chamber in fluid-tight relationship and in fact forms a part of the evaporating chamber 25. The conveyor 38 is preferably provided with an enlarged shaft portion 39 adjacent its discharge end so as to form a constricted passage 40 between the shaft portion 39 and the conveyor housing 37. A cooling jacket 41 is also preferably positioned around the discharge portion of the conveyor housing 37 so as to cool the materials being discharged and this cooling, in conjunction with the constricted passage 40, forms a vacuum seal for the evaporating chamber 47 while at the same time providing for the continuous discharge of soap stock therefrom. The discharge end of the conveyor housing 37 is preferably provided with a manually operable valve 42 which may be closed during starting and stopping of the apparatus so as to seal the vacuum when insufficient soap stock for that purpose is present in the constricted passageway 40. This valve is normally maintained open during the continuous operation and such soap stock is continuously discharged from the conveyor housing.

When it is desired to mix purified and substantially completely saponified soap stock with other

soap so as to provide a high grade soap mixture having desired physical characteristics, a second conveyor 44 provided with a housing 45 communicating with the housing 37 of the conveyor 38 may be provided to introduce a mixture of soap and water into the conveyor housing 37 intermediate the evaporating chamber 25 and the discharge end of the conveyor housing 37. A hopper 46 for such a mixture of soap and water may communicate with the housing 45 of the conveyor 44 so that, by adjusting the relative speeds of the conveyors 38 and 44, any desired proportion of soap and water mixture may be introduced into the conveyor housing 37. The conveyor 44 is also preferably provided with an enlarged shaft portion 47 adjacent its discharge end to form a vacuum seal similar to that for the conveyor 38. The soap and water mixture delivered into the conveyor housing 37 may contain sufficient water to hydrate the mixture of soap stock and additional soap to the desired degree such that the mixture may be extruded from the conveyor housing 37 in bar form suitable for pressing into cakes. Any vapors formed when the mixture of soap and water contacts the heated soap stock in the conveyor housing 37 may be withdrawn through a pipe 48 communicating with the condenser 29. The mixture of soap and water introduced into the conveyor housing 37 assists in cooling the purified soap stock therein so that the temperature of the soap being discharged to the atmosphere is below that at which contact with the air will damage the same.

Instead of supplying additional alkali to the soap stock mixture in the mixer 10, sufficient water or steam may be added in the mixer 10 to render the soap stock pumpable and additional alkali mixed with a heated stream of the soap stock by delivering an alkali solution through the pipe 49 to a flow mixer 50 positioned between the heating coil 16 and the pump 24. This can be accomplished by providing a caustic supply tank 51 and a proportioning pump 52 connected to the soap stock pump 15 through a variable speed device 53 and driving both the pumps 50 and 52 through a variable speed motor 54. Any other suitable means for accurately proportioning the amount of soap stock and alkali solution may be employed.

The alkali solution in the tank 51 may be preheated to any desired temperature below the boiling point of the solution in the tank by means of a heating coil 55 through which any suitable heating medium, such as steam, may be circulated. Also, it is preferable to preheat the alkali solution in a flow heating device under pressure such as a coil 56 provided with a burner 57 such that the temperature of the alkali solution mixed with the soap stock in mixer 50 is approximately that of the soap stock from the coil 16. The mixer 50 in conjunction with the pump 24 provides an efficient mixing between the soap stock and caustic and in many cases this variation of the process is preferable to mixing the alkali with the soap stock in the mixer 10 as the additional alkali and soap stock are brought together under heated conditions during flow and a more uniform admixture may sometimes be obtained.

In the simplest and preferred process of the present invention, a quantity of soap stock, such as cottonseed soap stock, is placed in the mixer 10. This material is extremely viscous and slimy and as indicated above, contains unsaponified oil, phosphatides, proteins, coloring matter, etc. Sufficient alkali to completely saponify the saponifiable materials contained in the soap stock is preferably introduced along with sufficient water to render the soap stock flowable. The mixture is then vigorously agitated during heating by means of the heating jacket 13 or the introduction of steam through the pipe 14 until a substantial uniform mixture is produced. It is preferred to heat the mixture before it is withdrawn from the mixer to a temperature approaching that of the boiling point thereof in order to reduce its viscosity and the amount of heat it is necessary to impart to the mixture in the heating coils, but a lower temperature can be employed as long as it is sufficient to produce a flowable mixture. The preferred alkali is caustic soda, although in the present process other alkalies, such as sodium carbonate or sodium peroxide, may be employed. In this connection the liberation of oxygen from sodium peroxide in some cases has a valuable bleaching effect upon the soap stock. Other bleaching agents, such as sodium hypochlorite or other chlorine containing compounds, may similarly be employed to bleach the soap stock. It is unnecessary to accomplish complete saponification in the mixer 10, although when employing caustic soda substantially complete saponification may be attained therein. By maintaining the agitators 11 running during withdrawal of the soap stock mixture from the mixer 10, a substantial uniform mixture may be delivered to the coil 16 by the pump 15.

This mixture is subjected to high temperatures and pressure in the coil 16 and then pumped through one or more coils 22 where it is additionally subjected to high temperature and pressure conditions. For example, the pressure in coils 16 and 22 may range between 250 to 1000 pounds per square inch and the temperature reached in coil 22 may be as high as 450 to 700° F. A preferred temperature and pressure in coil 22 is approximately 550° F. and 400 pounds per square inch. By subjecting the soap stock mixture to these high temperatures under conditions of flow in the coils 16 and 22, the saponifiable materials in the soap stock mixture are substantially completely saponified and the proteins and other undesirable impurities are broken down into simpler compounds, for example, ammonia and amines, peptides, amino acids or amides. Such materials as the ammonia, amines, etc., are vaporizable in the evaporating chamber 25 and other material, such as certain amino acids or amides, will combine with the alkali to produce materials having excellent detergent properties or are water soluble such that the treated soap stock may be given a subsequent washing with water to remove such water soluble impurities. Sufficient time is provided for the breaking down of such compounds which can usually be accomplished in periods of time ranging from 2½ to 10 minutes. This will vary with different soap stocks and the time necessary for a particular material can be determined by experiment and one or more coils 22 employed of sufficient length to provide the necessary time of treatment.

When the heated mixture is discharged into the evaporating chamber 25 under reduced pressure, the vaporizable material, such as ammonia and amines as well as water, is vaporized and withdrawn from the evaporating chamber through the pipe 26. Any glycerine present in the mixture is also vaporized and withdrawn. It is preferred to maintain the evaporating chamber under vacuum conditions by employing condensers such as 27 and 29 and the vacuum pump

31. Best results are obtained by employing a vacuum as high as commercially practicable and vacuums ranging from approximately 27 to 29 inches of mercury are suitable. The temperature of the material as discharged into the evaporating chamber is preferably high enough to maintain the resultant purified soap stock in liquid condition even though substantially all of the water and other vaporizable materials have been withdrawn therefrom. The liquid purified soap stock flows down the heated walls of the evaporating chamber and the temperature in the evaporating chamber is maintained sufficiently high to retain the purified soap stock in molten condition. Thus, temperatures ranging from 420 to 600° F. can be employed, depending upon the nature of the soap stock being treated and usually this temperature may be as low as 475° F. without causing solidification of the purified soap stock upon the walls of the evaporating chamber. To insure that all of the vaporizable materials are liberated in the evaporating chamber 25, the pressure in the last coil 22 may in some cases be sufficiently low that, at the temperature maintained therein, some vapors are formed in the coil so that the heat necessary for this vaporization is supplied in the coil rather than in the vaporizing chamber 25.

The condensible vaporizable materials, such as certain amines, as well as a small proportion of glycerine present in the soap stock, may be condensed in the condensers 27 or 29 and if sufficient cooling is employed any ammonia may be retained in water condensed in these condensers. The condensed materials are collected in any desired fractions in the receivers 28 and 30 and may be withdrawn therefrom through pipes 29' and 30' by any suitable means such as pumps (not shown) for subsequent separation or purification.

The purified soap stock, which consists essentially of soap along with other detergent materials, is cooled and discharged by the conveyor 38 without breaking the vacuum in the evaporating chamber 25. This material is substantially anhydrous and free of vaporizable materials and is also substantially odorless and free from materials which will cause subsequent deterioration of soap made therefrom. The material may be shipped or stored for indefinite lengths of time without deterioration.

If desired, the soap stock mixture in the mixer 10 may be washed and treated with salt or sufficient caustic soda to cause a separation of soap and impurities in a manner similar to that employed in soap making processes before the soap stock mixture is introduced into the heating coil 16. Thus one or more separations may be accomplished in the mixer 10 by washing and salting out impurities and allowing the same to settle as soap lyes which may be withdrawn through the pipe 16' and discarded. Such operation is ordinarily not necessary with most soap stocks and entails a loss of soap, reagents and valuable by-products.

As before stated, the additional caustic soda or other alkali may be supplied continuously through the pipe 49, in which case substantially instantaneous saponification of the saponifiable materials in the soap stock is effected because of the high temperatures in the mixer 50 and the rapid and efficient mixing accomplished at these high temperatures. Thus the soap stock may be heated to temperatures between approximately 300 and 420° F. in the coil 16 without causing decomposition of unsaponified glycerides or fatty acids therein. Also as indicated above, a mixture of high quality soap and water may be introduced into the conveyor housing 37 to assist in cooling the purified soap stock from the evaporating chamber 25, and to produce a hydrated soap containing sufficient moisture for the production of bar soap. It is apparent that if the particular soap stock being treated and the proportions of soap stock in the resultant mixture require more moisture than that which can be contained in a mixture of soap and water capable of being handled by the conveyor 47, a pump may be employed in lieu of the conveyor 47 for delivering a liquid mixture of soap and water into the conveyor housing 37. By balancing the amount of soap introduced and its moisture content against the amount of substantially anhydrous purified soap stock delivered by a conveyor 38, a properly hydrated soap product can be extruded from the conveyor housing 37. It is found that high grade soap products which will not deteriorate with age can be produced containing as high as 40 to 60% purified soap stock.

It is also possible to form the additional soap mixed with the purified soap stock as part of the process. Thus, desired amounts of saponifiable materials such as fatty acids or glycerides along with proper proportions of alkali can be introduced into the mixer 10 or during flow of the soap stock so that the additional saponified materials react to form soap which is thoroughly mixed with the purified soap stock. Fatty acids are more suitable for this purpose than glycerides, as valuable glycerine liberated from the glycerides is recovered in admixture with other materials and must be separated therefrom.

The process is also adaptable for treating soap stock without the addition of additional alkali. By merely adding water through the pipe 20 to the mixer 10 and passing the resultant mixture through the heating devices 16 and 22 under high pressure and temperatures, the water will react with neutral oil to cause the same to split into fatty acids and glycerine and the proteins and other impurities will be broken down into simpler compounds as is the case when additional caustic is employed. The fatty acids and glycerine can be vaporized in the evaporating chamber 25 and distilled over into the condensers 27 and 29 and recovered with other vaporizable materials. Also, by subjecting the mixture to somewhat less drastic temperatures in the coils 16 and 22, substantially all of the water may be removed from the soap stock as well as other vaporizable materials without to any great extent breaking down the glycerides, proteins, etc., so that a substantially dehydrated and cooled soap stock is extruded by the conveyor 38. Such soap stock, being substantially anhydrous, can also be stored for extended periods of time or shipped without deterioration, but is in general not suitable for incorporation into high quality soaps unless further treated. In this case it is apparent that a vacuum need not always be maintained in the evaporating chamber 25 and that a simple spray tower through which air is circulated may be in some cases sufficient to produce a stable soap stock capable of being stored and shipped.

Thus the present invention provides a process by which soap stock from vegetable and animal refining operations, which soap stock is at the present time of very little value, can be converted into a valuable material capable of being incorporated into high quality soaps and at the same

time valuable by-products can be recovered therefrom. Also, the invention may be employed merely to dehydrate the soap stock and stabilize the same so that it can be stored and shipped to points further distant than heretofore without fermentation or other deterioration. In this connection it is noted that the dehydrated soap stock is of much less weight and can be handled in ordinary containers instead of sealed drums as was necessary with untreated soap stock. When the soap stock is subjected to the preferred process of the present invention, it results in a purified soap stock product which is substantially free of disagreeable odor and impurities such as proteins, phosphatides, and other deleterious materials, as well as having a much lighter color than the original soap stock.

This application is a division of my application Serial No. 180,621, filed December 18, 1937, now Patent No. 2,239,131, granted April 22, 1941.

While I have described the preferred embodiments of my invention, it is understood that I am not to be limited to the details thereof but that the invention may be varied within the scope of the following claims.

I claim:

1. A purified soap stock prepared from soap stock from the alkali refining of vegetable oils, which comprises, a substantially completely saponified mixture consisting essentially of soap and substantial amounts of other detergent compounds resulting from the thermal decomposition of proteinaceous materials originally present in the soap stock, said purified soap stock being substantially free of decomposable proteinaceous material.

2. A purified soap stock prepared from soap stock from the alkali refining of vegetable and animal oils, which comprises, a soap material substantially free of water, decomposable proteins, and odorous materials and containing alkali salts of amino acids.

3. A high quality soap product containing substantially in excess of 25% of a purified soap stock prepared from soap stock obtained from the alkali refining of vegetable oils, said purified soap stock comprising a substantially completely saponified mixture consisting essentially of soap and substantial amounts of other detergent compounds resulting from the thermal decomposition of materials including proteinaceous material originally present in the soap stock and being substantially free of decomposable proteinaceous material.

4. A high quality soap product containing at least 40% of purified soap stock prepared from soap stock obtained from the alkali refining of vegetable oil, said purified soap stock consisting essentially of soap and substantial amounts of other detergent compounds resulting from the thermal decomposition of materials including proteinaceous material originally present in the soap stock and being substantially free of odorous and decomposable proteinaceous material.

5. A high quality soap product containing between 40 and 60% of purified soap stock prepared from soap stock resulting from the alkali refining of vegetable oils, said purified soap stock being a substantially completely saponified mixture consisting essentially of soap and substantial amounts of other detergent compounds resulting from the thermal decomposition of materials including proteinaceous material originally present in the soap stock and being substantially free of decomposable proteinaceous material.

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