

# UNITED STATES PATENT OFFICE

2,084,632

## HYDROGENATED NAPHTHA SOAP GELS AND METHOD OF MAKING SAME

Carleton Ellis, Montclair, N. J., assignor to Stand-  
ard-I. G. Company

No Drawing. Application April 25, 1933, Serial  
No. 667,830

4 Claims. (CL. 87-5)

This invention relates to soap and the process of making same and relates especially to soap gels, pastes, or emulsions made with the aid of petroleum derived products.

5 The invention is concerned in a specific way with detergents containing hydrogenated or hydrogen-treated petroleum solvents, particularly hydroformed products ranging from the hydro-naphthas through the hydro kerosenes and to  
10 oils of even higher boiling point.

In co-pending application 658,151, filed February 23, 1933, (Patent No. 2,058,781, granted October 27, 1936) hydrogenated naphtha soap is disclosed and claimed broadly and the present in-  
15 vention relates to specific improvements over the application referred to.

These hydroformed solvents, or hydrogenolates, are derived from petroleum or its various liquid distillation or extraction products. They  
20 may be produced from petroleum distillates, such as burning oil or gas oil, and in general from hydrocarbon oils of a boiling range including that of gasoline and also of ranges extending up to about 650° to 700° F. or somewhat higher. In  
25 producing such solvents a petroleum distillate oil may be passed in the vapor phase with free hydrogen over a suitable catalyst at a pressure in excess of 20 atmospheres (preferably 50 to 200 atmospheres or higher) desirably at a tempera-  
30 ture above 900° F. and preferably within the range of about 930° to 1050° F. with a suitable partial pressure of hydrogen and time of contact to secure reconstituting of the hydrocarbon oil molecules without necessarily forming apprecia-  
35 ble amounts of polymerized or coky material. The hydrogen supplied is preferably within the range of about 1000 to 4000 cubic feet per barrel of feed oil, the amount generally depending upon the gravity and boiling range of the stock. A  
40 greater proportion of hydrogen may be used with suitable variation in feed rate, temperature and pressure. The feed rate depends upon the reaction temperature, and other operating conditions such as the partial pressure of hydrogen.  
45 This rate may be suitably about 1.5 to 4 volumes of oil per volume of catalyst-filled reaction-space per hour. The catalysts preferably comprise the oxides or sulfides of the elements of the 6th group together with suitable promoters of the alkaline  
50 earth or earth oxides. Such catalysts are substantially insensitive to sulfur poisoning. In the presence of hydrogen they possess the power of hydrunsulfing the petroleum feed stock, thus removing any stench due to the presence of ob-  
55 jectionable sulfur compounds.

The hydroformed petroleum products obtained thereby are mostly characterized by highly desirable solvent powers through their boiling ranges. Ordinarily, however, I prefer to use dis-  
5 tillate fractions boiling in a range which lies within the points included between the tempera- 5 tures of 300° and 550° F., though fractions having an initial boiling point of less than 300° F., or a final boiling point higher than 550° F., are not excluded. Such fractions generally possess  
10 rather high flash points and therefore eliminate to a large degree any fire hazard which may be encountered either during the manufacture of such soap emulsions or gels or during such periods  
15 of time they may remain in storage. An example of such a hydroformed naphtha, which is admirably suited for my purpose, is that of the type designated "safety fuel", and which may be pre-  
20 pared by subjecting the residues obtained in the distillation of heavy naphtha to a reconstituting process as just described, redistilling the product over clay, and retaining the fraction boiling be-  
25 tween 313° and 453° F. In one case a product made in this manner was found to exhibit a flash point of 121° F.

One method of making soap gels with these hydronaphthas is to dissolve a soap in the hydro-  
25 naphtha at a temperature near its boiling point and then allow the solution to cool. On cooling, the solution sets to a more or less stiff mass, de-  
30 pending upon the quantity of soap employed.

Soaps which are made by any of the well-  
35 known processes and which contain a low content of water are suitable for my purposes. Such soaps are usually produced by the saponifica-  
40 tion of tallow or other vegetable or animal fats or oils by means of alkalies, as for example sodium or potassium hydroxide, or by the direct action of such alkalies on the higher fatty acids, as for example stearic acid or distilled cottonseed fatty  
45 acids, or on wax acids, or on mixtures of these acids.

Another procedure for making gels is to grind  
45 the soap to a fine powder, and then slowly add hydronaphtha while grinding. The mass slowly sets to a gel, and any desired stiffness or rigidity can be obtained by regulating the quantity of  
50 hydronaphtha used. Still another manner in which these gels may be produced is by dissolving the higher fatty acids, such as stearic, oleic, or  
55 distilled cottonseed acids, wax acids, or mixtures of these acids, in the hydronaphtha, then triturating the solution of acids in hydronaphtha with an equivalent quantity of alkali, such as sodium or potassium hydroxide, dissolved in

water. The stiffness of the gels made by this latter process may be regulated by the proportion of acids employed, and consequently the quantity of alkali needed to react with the acids, the proportion of hydronaphtha used, and the volume of water employed in dissolving the alkali before causing it to react with the acids. Many combinations of these methods are possible, and in some instances it may be desirable to use such combinations. An example of such combinations would be, incorporating the hydronaphtha with the soap at ordinary temperatures, warming the hydronaphtha soap gel so produced, and finally cooling to allow it to set to a more solid mass.

If desired, a concentrated emulsion can be formed by adding a small amount of water to the gel composition made as described above. The exact amount of water is not important except in that if too much is used the product will be too thin to set to a gel. In such a case it forms an emulsion paste and if still more water is used a fluid emulsion results. The advantage in mixing a small amount of water with the hydronaphtha soap gel composition is that it facilitates subsequent mixing with water.

Hydronaphtha soap gels made by any of the processes just mentioned may be used as detergents, particularly for the removal of greases or oils. Such soap compositions are especially applicable as a mechanics' soap, or as a soap for the washing of automobiles or other vehicles whose continual use generally results in the accumulation of grease or oil on certain portions of the body.

A hydronaphtha of the safety fuel type may be converted to a clear jelly-like substance by heating it with as little as 2 to 3 percent by weight of soap. The clear water-white gelatinized material thus obtained may be employed for cleansing purposes in various ways. Ordinarily, however, I prefer to use a much larger proportion of soap, generally the weight of the soap employed being about 50 percent or more of the weight of the hydronaphtha.

Soaps which are made by any of the well-known processes and particularly those which have a low content of water are suitable for my purpose. Such soaps are usually produced by the saponification of tallow or other vegetable or animal fats or oils by means of alkalies, as for example sodium or potassium hydroxide, or by the direct action of such alkalies on the higher fatty acids, as for example stearic acid or distilled cottonseed acids.

I prefer, however, to use those soaps made by saponifying the wax acids with alkalies, particularly potassium hydroxide, and then drying. Such soaps form jellies which are somewhat darker in color but which are remarkably free from syneresis and therefore better suited for employment in cleansing compounds of this general character than products in which the gel in the course of time shrinks with the exudation of naphtha.

By wax acids is meant the acids derived by the oxidation of paraffin wax, or other mineral waxes such as those from petrolatum, ceresin, ozokerite, Palembang wax, slop wax, wax-tailings, and in some instances Montan wax, by means of strong nitric acid, air, oxygen, or oxidizing gases. Such oxidation is generally carried out at temperatures corresponding to the melting point of the wax or higher (e. g. 300° to 400° F.); and preferably in the presence of catalysts such as previously oxidized wax, manganese or cobalt resins,

barium cinnamate and the like, mixtures of these acids, or other appropriate catalysts. Also the acids obtained by the oxidation of heavy petroleum oils and similar substances may be used.

According to the present invention it becomes possible to use a very substantial proportion of hydrofined petroleum oil since the odor of such hydrogen treated material is relatively mild and sweet. At the same time, however, it becomes possible to use, if desired, perfuming agents in very moderate quantities to yield a perfumed hydronaphtha soap gel. In such instances the perfume may be dissolved in the hydronaphtha before it is incorporated with the soap to produce the hydronaphtha soap gel.

Fillers and/or abrasives may be used also. Examples of such substances are pumice powder or silix; also softer materials of the polishing type such as tripoli or infusorial earths. Another ingredient which could be used is vegetable ivory dust, of which large quantities are available from the button industry. One method of incorporating these fillers and/or abrasives in the hydronaphtha soap gels is to grind the filler and/or abrasive with the soap to a fine powder, and then with constant agitation to heat the powder soap and inert material with sufficient naphtha so that the soap dissolves but leaves the filler and/or abrasive dispersed throughout the liquid. On cooling, a hydronaphtha soap gel is obtained in which the filler and/or abrasive is suspended throughout. Another method is to incorporate the filler and/or abrasive with powdered soap, and then while grinding to add slowly the desired proportion of hydronaphtha.

As has been indicated, by the term "hydronaphtha" I mean any hydrogenated naphtha, kerosene, and the like, within the scope of the present invention, including hydrofined petroleum solvents of various boiling ranges extending through light naphtha, heavy naphtha, light kerosene, heavy kerosene, and even higher. I prefer, however, hydrofined products substantially or wholly free from stench forming hydrogenated naphthalenes, such as Tetralin. The term "hydronaphtha" as employed herein, therefore, should be understood to embrace petroleum fractions of a boiling range higher than that ordinarily comprehended by the term "naphtha", such fractions, however, having a bland sweet odor and being substantially free from naphthalene derivatives such as Tetralin; the boiling range of the hydronaphthas preferably being in the zone embracing so-called heavy naphtha or light kerosene.

The following examples will serve to illustrate the various phases of my invention. The proportions given are parts by weight.

*Example 1.*—2 parts of a commercial white chip soap having a low content of water were ground to a fine powder and this powder heated with 100 parts of hydronaphtha, with constant agitation, to approximately 300° F.; when solution had apparently taken place heating was discontinued and the liquid allowed to cool. When cold the liquid had set to a clear water-white semi-solid mass.

*Example 2.*—The potassium soaps of wax acids were prepared by mixing a weighed quantity of the acids with an equivalent quantity of potassium hydroxide, dissolved in water, and removing the water by evaporation. A hydronaphtha soap gel was made, as described in Example 1, by using 2 parts of the dried potassium soaps of wax acids and 10 parts of hydronaphtha. This

gel was somewhat darker in color than that produced in Example 1, and also possessed more of a granular structure.

5 *Example 3.*—5 parts of the potassium soaps of wax acids were thoroughly incorporated with 10 parts of hydronaphtha to yield a relatively solid hydronaphtha soap gel.

10 *Example 4.*—5 parts of the dried potassium soap of wax acids were incorporated with 5 parts of hydronaphtha to secure a hydronaphtha soap gel. This gel was heated to a temperature somewhat below 300° F. and allowed to cool. The granular hydronaphtha soap gel so produced lathered with water and could be used for cleansing  
15 purposes.

*Example 5.*—A somewhat softer hydronaphtha soap gel was made from 5 parts of the dried potassium soaps of wax acids and 15 parts of hydronaphtha using the procedure described in Ex-  
20 ample 4.

*Example 6.*—The addition of tripoli earth as a filler was made in the following manner. 6 parts of the dried sodium soaps of wax acids were ground with 12 parts of tripoli earth to a  
25 fine powder, and 4 parts of hydronaphtha were thoroughly incorporated with the mixture of soap and tripoli earth. The hydronaphtha soap gel thus obtained lathered well with water and could be used as a detergent.

30 *Example 7.*—1 part of the dried potassium soaps of wax acids was thoroughly ground with 1 part of pumice, as a filler, and 5 parts of hydronaphtha thoroughly incorporated with the soap mixture. Additional filler, in the ratio of 4  
35 parts of pumice to 7 parts of hydronaphtha soap gel, was incorporated with the hydronaphtha soap gel. This product was used as a cleansing agent, the relatively large proportion of pumice furnishing a mild abrasive action.

40 *Example 8.*—A white cream-like hydronaphtha soap gel was obtained in the following manner. 5 parts of the sodium soaps of wax acids were thoroughly incorporated with 15 parts of hydronaphtha. 10 parts of water were then incorpo-  
45 rated with the hydronaphtha soap gel.

*Example 9.*—5 parts of the potassium soaps of wax acids were incorporated with 5 parts of hydronaphtha, and then 10 parts of water thoroughly incorporated with this hydronaphtha soap  
50 gel. The resulting gel was somewhat darker in color and less fluid than that obtained in Example 8.

*Example 10.*—10 parts of wax acids were mixed with 3 parts of hydronaphtha. A quantity of

potassium hydroxide sufficient to react with the wax acids was dissolved in a small quantity of water and the alkaline solution incorporated with the hydronaphtha solution of the acids. The hydronaphtha soap gel lathered well with  
5 water and possessed good detergent qualities.

*Example 11.*—A filler, such as pumice, was added to the hydronaphtha soap gel as follows: 10 parts of the wax acids were dissolved in 3 parts of the hydronaphtha and 10 parts of "safety  
10 fuel" and this solution mixed with 10 parts of pumice by grinding. Sufficient alkali to react with the wax acids was dissolved in a small quantity of water and the alkaline solution thoroughly incorporated with the hydronaphtha, wax  
15 acids, and pumice mixture. The hydronaphtha soap gel so produced possessed good detergent qualities.

It is not intended that the invention be limited by any of the examples which have been given  
20 only for illustrative purposes but it is intended to claim all inherent novelty in the invention as broadly as the prior art permits.

I claim:

1. A semi-fluid detergent composition in gel  
25 form consisting essentially of a water-soluble soap, incorporated in from about 1 to 50 parts by weight of a solvent consisting of hydrogenated petroleum naphtha boiling within the range from  
30 about 300 to about 550° F.

2. A semi-fluid detergent composition consisting of a water-soluble soap incorporated with  
35 from about 1 to about 3 parts of solvent consisting of hydrogenated petroleum naphtha boiling within the range from about 300° F. to about 550° F.

3. A method of making semi-fluid detergent compositions which comprises admixing a water-soluble soap with from about 1 to about 3 parts  
40 of a solvent consisting of hydrogenated petroleum naphtha boiling within the range from about 300° F. to about 550° F., heating the mixture until the soap is dissolved and thereafter cooling the solution.

4. A method of making a semi-fluid detergent  
45 composition which comprises admixing a water-soluble soap with at least an equal weight of a solvent consisting of hydrogenated petroleum naphtha boiling within the range from about  
50 300° F. to about 550° F., heating the mixture until the soap is dissolved in the solvent and thereafter cooling the solution.

CARLETON ELLIS.