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METHOD OF REFINING CRUDE WOOL GREASE

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Fig. 1.

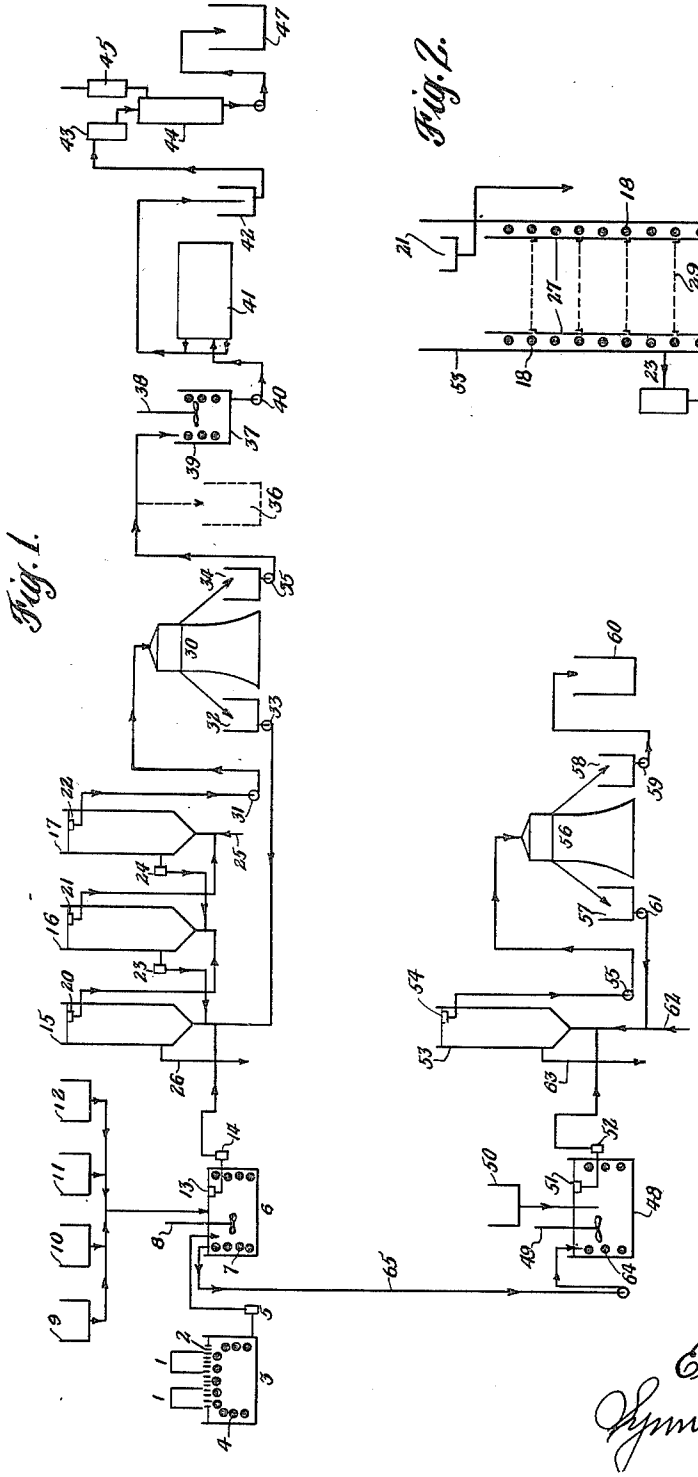


Fig. 2.

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METHOD OF REFINING CRUDE WOOL GREASE

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26 Claims. (Cl. 260—412.5)

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This invention relates to an improved method of refining crude wool grease and has for its primary object the provision of a simple and economical method for producing either hydrous or anhydrous lanolin of high grades, but before describing the invention in detail or reciting its objects more specifically it is desired to refer briefly to certain of the present prior art practices in the production of crude wool grease because it is believed that the nature of the invention will be more fully understood and appreciated with these facts in mind.

Probably the most common method of removing grease from raw wool is by washing the wool in solutions containing soap and soda ash, which solutions then contain emulsified wool grease, dirt, salts, soaps from the neutralization of free fatty acid in the wool, pollen, proteins from various sources, etc. The crude grease is then separated from such liquors by settling, centrifuging or both, and enters the market as crude wool grease. It is sometimes sold as "hydrous crude wool grease" which is a "cream" still containing approximately 15 to 40% of water as well as other impurities associated with the scouring solution emulsified therein. It may be recentrifuged to a water content of from 1 to 3% and sold as "dry crude wool grease." In either case, this crude material must be refined before it can be used for pharmaceutical or cosmetic purposes, and even for less critical uses it often needs to be at least partially purified before it is sufficiently free of objectionable odor, grit, soap, etc.

There are, however, two other rather common ways of recovering crude wool grease. One is to scour the wool with a solvent and to recover the grease by distilling off the solvent from the filtered solution. This yields a "dry" crude grease. The other method recovers not only wool grease but also all the fatty acid from the soap in the scouring liquor. It consists in breaking the grease emulsion in the scouring liquor by acidulating it—generally with sulphuric acid—whereupon there separates a foul-smelling upper layer sold in England as "Yorkshire grease." It has also been sold as degras, although the latter name is used to denote other products, notably a product which contains the free fatty acids recovered from the soaps associated with crude wool grease together with some of the unsaponified

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grease not recovered in the separation of the grease.

My improved process is most easily applied to a creamed emulsion of grease, containing water soap, dirt, coloring matter, proteins and other impurities known as crude hydrous wool grease although it may also be applied to other types of impure crude wool grease such as "dry" crude grease and even, indeed, to "acid cracked wool grease" or "Yorkshire grease," although, in this latter case, it lacks one of the main advantages of my invention as will more fully appear hereinafter.

With the foregoing in mind my invention has among others, the following objectives:

(1) To refine crude wool grease without using alcohol or any other organic solvent in the refining process which feature of the invention obviates the provision of stills or other equipment ordinarily employed in the recovery of the solvent used, and (2) to provide a method of refining crude wool grease by means of which it is possible to avoid subjecting the grease to be refined to acid or to acidified water, in fact, to avoid subjecting the grease to a pH below 7 at any stage of the refining operation. This is an important advantage of my improved process because, in my experience, I have found that the color and odor of the refined grease is adversely affected by acidulation at any stage of the refining operation even though the grease may later be treated so as to contain no free fatty or other acid.

In connection with the following detailed description of my improved invention, attention is directed to the accompanying drawing wherein Figure 1 is a diagrammatic representation of the steps pursued and the preferred apparatus employed in carrying out the process as applied to hydrous crude wool grease, and Figure 2 illustrates certain details of the construction of one of the washing towers employed in the process. I wish it to be understood, however, that the apparatus shown is merely illustrative because other types of equipment might well be employed in carrying out the steps of the process.

In carrying out my process I first emulsify the melted crude grease in water solution containing soap. "Hydrous" crudes may contain enough soap to produce this emulsion merely upon heating and stirring the melted crude, but with the

so-called "dry" crudes, in addition to the water needed, it may be necessary to add some soap in order to produce a good emulsion.

Next I add to the emulsion alkaline chemicals, preferably such as contain an alkali silicate. Insofar as certain basic advantages of my improved process are concerned, this addition may not always be necessary but it assures the removal of free fatty acid contained in the crude grease. Such free fatty acid may be present even in crude which is alkaline in reaction due to hydrolysis of soap. The alkali also aids in dissolving protein impurities and in suspending solid dirt in the aqueous phase which is separated in accordance with the step to be described below.

If it is desired to recover a very light-colored grease, hydrogen peroxide or a per-salt yielding hydrogen peroxide in aqueous solution, such as sodium perborate or sodium percarbonate, is added to the emulsion and if such bleaching agents are used it is well to keep the mixture very hot, preferably near the boiling point, until the peroxide in the water is decomposed as indicated by the cessation of gas evolution. Otherwise, gas bubbles adhering to solid impurities may prevent the latter from passing out of the grease layer into the water layer as in the next step.

At this point I add to the mixture a relatively small amount of a water-soluble sulphonated fixed oil such as sulphonated castor oil, sulphonated neat's-foot oil, sulphonated rape seed oil, sulphonated corn oil, sulphonated soya bean oil, or other sulphonated animal or vegetable oil commonly available commercially as such. Normally these are substantially neutralized materials and do not lower the pH of the mixture below 7.0 which is important in attaining the objectives of my invention as described above. This addition causes a rapid separation of clear grease on top of the mass while the water solution settles simultaneously and rapidly to the bottom carrying with it the soap, the solid dirt and the water-soluble sulphonated oil. The phenomenon following the addition of the sulphonated oil is extremely striking and, insofar as I am aware, entirely novel in the refining of grease and much of the success of my improved process depends to a large extent on this rapid clean separation of grease on the one hand from a water solution or suspension of the impurities on the other hand. The separation is made so thorough, in fact, as to make possible countercurrent water washing of the grease in accordance with the following step. If the separation is not thorough losses of grease in the waste wash water may become serious so that the process is uneconomical.

The grease layer is now separated from the water layer and is washed in hot water in one or more washing tanks in which, preferably, the wash water flows in counter-current to the grease. In this way the last traces of water-soluble impurities such as soap, alkali, etc. are removed and the grease separated from the mass at the top of the last washing tank where it appears as a clear neutral, light-colored material which, except for a little extra moisture, is of U. S. P. lanolin grade.

The grease may now be separated from any remaining wash water and dehydrated if a dry product is required. This is best accomplished by passing the grease, together with some water, if desired, through a centrifugal separator by means of which the grease can readily be brought to a water content of 0.2% or less.

With the foregoing procedure the grease is now generally marketable as U. S. P. lanolin. However, for some purposes, especially for the cosmetic industry, a further improvement in the quality of the refined grease may be desirable in which event either or both of two further steps may be included as follows. Add to the grease a small amount of bleaching earth or decolorizing carbon or both, stir hot for a short time, and pass through a filter press. Or pass the hot grease, either filtered or unfiltered, through a chamber in which a partial vacuum is maintained, which treatment serves to remove traces of volatile odoriferous impurities along with traces of moisture. This step may be assisted by the introduction of live steam, air or inert gas beneath the surface of the grease while it is under vacuum. The apparatus for use in this step need not be elaborate. If no steam or gas is bubbled through it the grease should be spread out into thin layers, as by allowing it to trickle over a series of inclined trays while it is under vacuum, so that the time of retention in the "deaerating" chamber may be short. Another method of assuring quick removal of volatile impurities from the grease is to introduce it into the vacuum chamber as a spray. After removal from the "de-aerating" chamber the refining operation is concluded and the grease may be run directly into drums, barrels, or other tight containers for shipment.

With the foregoing outline of the essential features of my improved process in mind, I will now describe in detail the accompanying drawing in order to more fully illustrate the steps of the process. The crude grease is received in steel drums 1 which are inverted over the steam-pipe grille 2 at the top of the melting tank 3 in which the crude grease is kept molten and hot by steam coils 4.

After melting, the hot crude grease, containing from 20 to 40% moisture, is transferred to the processing tank 6 by means of a blow case 5, tank 6 being equipped with steam heating coils 7 and a good mechanical stirrer 8. From tanks 9 or 10, or both, there is then added an appropriate material or mixture which contains, in soluble form,

	Pounds
Silica (SiO_2)	0.77
Sodium oxide (Na_2O)	0.40

for every 100 lbs. of anhydrous crude grease present.

The exact quantity of the above alkaline mixture used as well as its SiO_2:Na_2O ratio are not critical and may be varied considerably without greatly affecting the behavior of the process. According to the grade of crude grease in process the sodium oxide added may be varied from 0.3 to 1.0 lb. and the silica from 0.5 to 2.0 lbs. per 100 lbs. of dry crude.

This material may be added in the form of an admixture of low-alkali silicate and high-alkali silicate, as an admixture of low-alkali silicate with caustic soda, or in any other convenient form, as long as sufficient of the equivalent Na_2O or equivalent alkali oxide and SiO_2 are present.

Other alkali may be added instead of or in partial replacement of the sodium silicate just referred to. Mixtures of more or less equivalent alkalinity compounded with soda ash, trisodium phosphate, borax, etc. can be used. However,

the emulsion formed with the grease is most easily and quickly broken by addition of the sulphonated oil which follows when the alkali used is a silicate, as described. Moreover, the silicate alkali causes better deflocculation of solid impurities such as dirt, lime soap, etc., which are present in the crude grease than do other alkaline mixtures. Thus these impurities pass more quickly and easily into dispersion in the lower aqueous layer on the breaking of the emulsion described later.

Due to the action of soap present in the crude grease or formed by neutralization of the free fatty acids in the crude grease by the added alkali, the mass, on stirring, forms a uniform white emulsion. To bleach the grease, to kill bacteria, and to destroy odors, there is now added, from tank 11, for each 100 lbs. of dry crude grease, from 1 to 3 lbs. of 30% hydrogen peroxide, for example, or an equivalent quantity of a hydrogen peroxide solution of different strengh, or of sodium perborate, sodium percarbonate, etc.

The mass is now stirred while being kept hot, preferably at 190° F. to 210° F., until all the peroxide has been destroyed as indicated by the cessation of gas evolution.

From 1 to 6 lbs., for example, of sulphonated castor oil for each 100 lbs. of dry crude grease, are now added from tank 12. When sufficient sulphonated oil has been stirred in, there occurs, on stopping the stirrer, a rapid separation of clear grease on top of the mass. Simultaneously, a dark colored aqueous solution of soap, alkali, excess sulphonated oil and other water-soluble matter in which is suspended dirt and other insoluble matter, rapidly accumulates at the bottom of the mass. If sufficient sulphonated oil has been added, a quite sharp separation between clear grease and "dirty water" occurs and is finished within from 1 to 5 hours.

The separated grease layer is removed through skimmer 13, and passed by pump 14 into the bottom of washing tower 15. From the top of 15, it passes via skimmer 20, to the bottom of the second washing tower 16; and sequentially, via skimmer 21, to the bottom of the third washing tower 17, as shown.

Fresh hot water enters the bottom of the third washing tower 17 at 25, leaves 17 at the lower side via pump 24, passes into the bottom of tower 16, leaves 16 via pump 23, enters 15 at the bottom, and flows to waste from the lower side of 15 at 26.

Details of the construction of one of the identical towers 15 are shown in Figure 2. It is equipped with heating coils 18 to maintain the grease and water at from 170–200° F. In addition, the tower contains a series of nesting screens 27 fitting into each other and into the rings 28, welded to the tower, as shown.

Water and grease enter at the bottom at 30A as shown and rise inside the cylinder formed by the nesting screens 29. The grease is broken into droplets by passing through the screens, and is thus brought into intimate contact with the water in the tower. The water passes upward with the grease, through 27, and then flows over the top of the upper screen ring of 27 and down between the side of the tower and the outside of screens 27 to the water outlet to pump 23.

The grease floats upward and collects at the top of the tower whence it is withdrawn by skimmer 21.

By passage through the series of wash towers the grease is thus repeatedly washed while broken up into droplets. Thus the grease collected by skimmer 22 of the third wash tower in which i has been brought into contact with fresh clea water is practically free of all impurities excep entrained water.

The water is completely separated by centrifug 30 into which the wet grease is passed at a regu lated rate by pump 31. The water discharged fron the centrifuge enters receiver 32 from which i is returned to the bottom of the first washin tower 15.

The dehydrated pure grease enters receiver 34 from whence pump 35 carries it either to drun 36 for storage and shipment, or to filter tank 37 if further purification is to be undertaken.

In tank 37 which is equipped with mechanica stirrer 38, the grease is admixed with bleachin solids such as decolorizing carbon, bleaching clay or both, as an example, approximately 2.5% eacl of clay and carbon. The mixture is stirred whil being kept hot by steam coils 39 for a short tim and is then passed via pump 40 through filte press 41.

The effluent from 41 enters receiver 42 fron whence it is drawn, by the vacuum produced b vacuum pump 45, into de-aerator 44. On its wa to 44, it is brought to at least 200° F. by passag through steam heater 43.

The filtered, de-aerated grease collects in th bottom of de-aerator 44, whence it is drawn b pump 46 into shipping drums 47.

If it be desired to recover the fatty acids fron the soaps associated with the crude grease, th aqueous layer remaining in processing tank 6 after withdrawal of the grease layer, is withdraw via pipe 65 to tank 48 where it is treated witl sufficient dilute sulphuric acid from tank 50 t liberate the free fatty acid from the soap, whil stirring with mechanical stirrer 49. The quantit of sulphuric acid used is not critical and depend upon the amount of soap and alkali present. I any case, enough acid should be added to rende the liquid in 48 acid—say, to a pH of 4 or less The liquid is kept hot by steam coils 64.

The free fatty acid rapidly rises to the top o the tank on stopping the stirrer 49. If desired the crude fatty acid ("degras") which contain traces of unrecovered wool grease, etc., may b purified by passage through an abbreviated wash ing system similar to that used for purifying th separated wool grease.

This is shown in Figure 1 where 51 is a skimmer 52 a degras pump, 53 a washing tower similar t 15, 16 and 17, 54 another skimmer, 55 anothe degras pump, 56 a centrifuge, 58 a degras receiv er, 59 a final degras pump and 60 a shipping o storage drum.

Water separated in the centrifuge 56 enters re ceiver 57. Pump 61 forces this water into the bot tom of washing tower 53 at which point fres water also enters through 62. The waste wate from the tower flows to waste at 63.

Degras being a relatively cheap material, i does not generally pay to refine it further.

Although I have described my invention as car ried out in a particular kind and design of equip ment, it will be understood that this apparatu has been described in detail only for purposes c illustration and to make the description of my im proved process more intelligible. Many varia tions in equipment and technique will sugges themselves to those skilled in the art and I do nc desire to be limited in process details or appa ratus except as indicated in the appended claim

I claim:

1. In the refining of crude wool grease th

process which includes emulsifying the grease by agitating and heating it with water and soap to a temperature above its melting point, adding to the emulsion a relatively small amount of a water-soluble, substantially neutral, sulphonated fixed oil, allowing the mass to subside to form a water layer and a supernatant grease layer, and then separating the grease layer from the water layer.

2. The process of claim 1 in which the separated grease is washed with hot water.

3. The process of claim 1 in which the separated grease is washed with hot water and then subjected to the effects of a vacuum while still hot.

4. The process of claim 1 which includes also the addition to the emulsion of a relatively small quantity of a silicate alkali prior to the addition of the sulphonated oil.

5. The process of claim 1 which includes also the addition to the emulsion of a relatively small quantity of a silicate alkali prior to the addition of the sulphonated oil and wherein the separated grease is washed with hot water.

6. The process of claim 1 which includes also the addition to the emulsion of a relatively small quantity of a silicate alkali prior to the addition of the sulphonated oil and wherein the separated grease is washed with hot water and finally subjected to the effects of a vacuum while still hot.

7. In the refining of crude wool grease the process which includes emulsifying the grease by agitating and heating it with water and soap to a temperature above its melting point, admixing with the emulsion a relatively small quantity of a silicate alkali, admixing also a relatively small quantity of a substance chosen from the class consisting of hydrogen peroxide and per-salts which liberate hydrogen peroxide when dissolved in water, thereafter adding a relatively small quantity of a water-soluble, substantially neutral, sulphonated fixed oil, allowing the mass to subside to form a water layer and a supernatant grease layer, and then separating the grease layer from the water layer.

8. The process of claim 7 in which the separated grease is washed with hot water.

9. The process of claim 7 in which the separated grease is washed with hot water and then subjected to the effects of a vacuum while still hot.

10. In the refining of crude wool grease the process which includes emulsifying the grease by agitating and heating it with water and soap to a temperature above its melting point, admixing with the emulsion a relatively small quantity of a silicate alkali, adding a relatively small quantity of a sulphonated, substantially neutral, fixed oil, allowing the mass to subside to form a water layer and a supernatant grease layer, separating the grease layer from the water layer, passing the grease layer through a plurality of washing towers in which it is subjected to intimate contact with hot water, centrifuging the washed grease, and finally subjecting the centrifuged grease to the effects of a vacuum while still hot.

11. In a process of recovering wool grease and degras from crude wool grease the steps which consist in heating the grease above its melting point while it is in the form of an emulsion with water containing soap, adding to the emulsion a relatively small amount of a water-soluble sulphonated, substantially neutral, fixed oil, allowing the grease to separate from a water layer containing the bulk of the soluble and insoluble impurities and washing the separated grease with hot water; separately acidulating the water layer separated from the emulsion, and removing the degras which separates from the acidulated water layer.

12. The process of claim 1 in which the fixed oil is sulphonated castor oil.

13. The process of claim 1 in which the fixed oil is sulphonated castor oil and in which the separated grease is washed with hot water.

14. The process of claim 1 in which the fixed oil is sulphonated castor oil and which also includes the addition to the emulsion of a relatively small quantity of a silicate alkali prior to the addition of the sulphonated castor oil.

15. The process of claim 1 in which the fixed oil is sulphonated castor oil and which also includes the addition to the emulsion of a relatively small quantity of a silicate alkali prior to the addition of the sulphonated castor oil and wherein the separated grease is washed with hot water.

16. The process of claim 1 in which the fixed oil is sulphonated castor oil and which also includes the addition to the emulsion of a relatively small quantity of a silicate alkali prior to the addition of the sulphonated castor oil and wherein the separated grease is washed with hot water and finally subjected to the effects of a vacuum while still hot.

17. The process of claim 7 in which the fixed oil is sulphonated castor oil.

18. The process of claim 7 in which the fixed oil is sulphonated castor oil and in which the separated grease is washed with hot water.

19. The process of claim 7 in which the fixed oil is sulphonated castor oil and in which the separated grease is washed with hot water and then subjected to the effects of a vacuum while still hot.

20. The process of claim 10 in which the fixed oil is sulphonated castor oil.

21. The process of claim 11 in which the fixed oil is sulphonated castor oil.

22. In the refining of crude wool grease the process which includes emulsifying the grease by agitating and heating it with water and soap to a temperature above its melting point, admixing with the emulsion a relatively small quantity of a silicate alkali, adding a relatively small quantity of a sulfonated, substantially neutral, fixed oil, allowing the mass to subside to form a water layer and a supernatant grease layer, separating the grease layer from the water layer and washing the separated grease with hot water, the silicate alkali containing alkali oxide equivalent to 0.3 to 1.0 lb. of Na_2O and containing silica in an amount of from 0.5 to 2.0 lbs., each per 100 lbs. of dry crude grease in process.

23. The process of claim 22 wherein the washed grease is subjected to the effects of a vacuum while still hot.

24. The process of claim 7 in which the quantity of hydrogen peroxide or hydrogen-peroxide-liberating per-salt is equivalent to from 1 to 3 lbs. of 30% hydrogen peroxide per 100 lbs. of dry crude grease in process.

25. The process of claim 7 in which the separated grease is washed with hot water and in which the quantity of hydrogen peroxide or hydrogen-peroxide-liberating per-salt is equivalent to from 1 to 3 lbs. of 30% hydrogen peroxide per 100 lbs. of dry crude grease in process.

26. The process of claim 7 in which the separated grease is washed with hot water and then

subjected to the effects of a vacuum while still hot and in which the quantity of hydrogen peroxide or hydrogen-peroxide-liberating per-salt is equivalent to from 1 to 3 lbs. of 30% hydrogen peroxide per 100 lbs of dry crude grease in process.

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