PROCESS OF REMOVING MATERIALS CONTAINING PHOSPHATIDES FROM VEGETABLE OILS

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Fig. 1

Fig. 2

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My wife, Edith, and I, Benjamin H. Thurman
This invention relates to a process of removing materials containing phosphatides from vegetable oils and more particularly to a process of continuously removing such materials from vegetable oils to improve the quality of the oils and recover valuable products therefrom. Vegetable oils, for example, cottonseed, soybean, linseed, etc., as they come from the press or expeller contain various impurities commonly referred to as "foots." These foots are gummy viscous materials containing large amounts of phosphatides. Upon standing, a portion of the foots settle slowly in the cold to form a mass referred to as "foot mass." This foot mass is common practice to maintain the oil in storage for weeks or even months to allow the foots to settle and then decant the supernatant oil from the sludge. A clean separation cannot be effected in this manner and large amounts of valuable oil are retained in the sludge. Filtering of the oil to remove foots has also been suggested but the gummy and viscous nature thereof makes this operation slow and difficult.

Contrary to the view entertained by the experts of the art I have heretofore discovered that phosphatides, in utilisable form, were contained in the gums of expressed vegetable oils obtained, for example, from cottonseed, soybean, linseed, rape seed, etc. An application based upon this discovery has been filed by me Serial No. 6,446, filed February 14, 1935, which application is a continuation in part of Serial No. 644,637, filed November 22, 1933, and Serial No. 676,932, filed June 21, 1933. In those applications a precipitating reagent is employed and phosphatides are recovered without the theretofore existing practice of using a solvent for extracting the phosphatides from the beans.

The present invention constitutes a variation from the application aforesaid in that no precipitating media is employed while the tendency of the separated foots to stick to the bowl of a centrifugal separator is overcome in the manner hereinafter explained.

It is therefore one of the objects of the present invention to provide a process by which such gums or foots can be continuously centrifugally separated from the oil without the employment of a precipitating reagent and without injury to the oil or to the phosphatides contained in said gums.

It is another object of the invention to provide sufficient heat to cause the precipitation of the naturally occurring gums in the oil while limiting the duration of the time the oils remain in a heated condition to such a brief time as to prevent injury to the oils or to the phosphatides contained therein.

It is still further an object of the invention to maintain the oils in such a heated condition at the time of subjecting the same to centrifugal separation as to cause the precipitated materials to coalesce or agglomerate whereby to condition the same for separation from the purified oil irrespective of whether the heat is applied before or during centrifugation.

Desirably, the process contemplates separation of the foots from the vegetable oils promptly after they are expressed and before the same have a deteriorating effect upon the oil or upon the phosphatides contained therein although it is recognized that the invention has utility with respect to the treatment of oils which have been maintained in storage for a considerable period of time.

As indicated above, the separated foots or gums contain valuable constituents, such as phosphatides. The gums from edible oils, such as cottonseed or soybean, may be further treated to separate out and purify the phosphatides as disclosed in applicant's co-pending application above referred to. The purified phosphatides form valuable stabilizing materials for edible products, while the gums separated from drying oils, such as linseed, form valuable materials for making putty and other analogous substances because of their anti-oxidant properties. Also the removal of the gums from any of the vegetable oils improve their color and facilitate their further refining. The acid sludge from acid refining is reduced in amount and also valuable phosphatides are removed before being degraded by the acid. The soap stock separated from alkali treated oil, which has previously been centrifuged to remove the gums or foots, is also of higher quality as it is not contaminated with large amounts of gums, less refining reagent is necessary and the refining loss is reduced. Drying oils, such as linseed treated in accordance with the present process, become "non-break oils" if all traces of moisture are removed therefrom. That is to say the oils will not precipitate further foots or gums when heated to a temperature of 500° F. or higher.

It is, therefore, an object of the invention to provide a process by which vegetable oils may be conditioned for alkali refining operations to reduce the refining loss therein and provide a higher quality soap stock.
A further object of the invention is to provide a process of making "non-break" drying oils.

Other objects and advantages of the invention will appear in the following detailed description of preferred embodiments of my invention, described in connection with the attached drawings, of which there are three.

Figure 1 is a schematic drawing of an apparatus suitable for carrying out the present invention, and

Figure 2 is a diagrammatic drawing of a centrifugal separator particularly adapted for carrying out the present process.

The centrifugal separator 9 illustrated diagrammatically in Figure 2 includes a centrifugal bowl 10 capable of being rotated at high speed by any suitable drive means, for example, a pulley 11. A stationary inlet member 13 carried by a cover 14 may be employed to introduce materials to be separated into an inner member 15, which delivers the materials to the interior portion of the centrifugal bowl. The heavier effluent material flows upwardly along the walls of the bowl 10 and is discharged through a port 16 into a pan 17 which delivers the heavier effluent from the centrifuge. The lighter effluent passes upwardly adjacent the inner member 15 and is discharged through a port 18 into the cover 14 and thus from the centrifugal through a pipe 19.

The centrifugal machine is preferably provided with a heating jacket 20 completely surrounding the bowl 10 and provided with an inlet 21 and an outlet 22 for any desired gaseous heating medium, for example, steam. The oil containing foams may be delivered to the centrifugal through a pipe 23 which is connected to the stationary inlet member 13.

One of the salient features of the present invention is the introduction of water or other liquid material, such as a salt solution which is of greater specific gravity than the oil, immediately into the entrance of the centrifugal separator along with the oil to be centrifuged to carry the foams out of the centrifuge. The gums or foots constitute the heavier effluent and are thrown against the walls of the bowl 10 by centrifugal force. These materials are extremely viscous and sticky and will not by themselves flow upwardly along the walls of the bowl and discharge through the port 16. I have found that water or aqueous salt solution will cause the foams to be continuously discharged from the centrifuge. No intimate mixing of the oil and other liquid is necessary or desirable. The other liquid is, therefore, delivered into the oil as close to the entrance of the centrifugal as practicable and may even be carried well down into the bowl by a separate liquid pipe before contacting the same with the oil.

The application of heat to the outer surface of the bowl also reduces the viscosity and helps to prevent sticking of the gums or foots to the bowl walls. With certain types of oil the application of heat may be sufficient to cause the foots to flow or slide out of the centrifugal bowl in which case the introduction of other liquid through the pipe 24 may become unnecessary. With other oils better results may be obtained by employing the carrying liquid alone without the application of heat. In most instances the employment of both produces best results but it will be understood that certain oils may be best separated with the application of heat only while with certain other oils the introduction of the carrier liquid alone produces best results.

The temperature at which the oil is introduced into the centrifugal separator will depend upon the nature of the oil being treated and no exact temperature or temperature range suitable for all oils can be given. The nature and amount of gums or foots contained in the same varies. The oil will vary with the soil and climate at the point or origin of the oil as well as the process used in extracting the oil. In general more gums are removed at the lowest temperature at which the oil is sufficiently liquid to provide efficient separation. With soybean oil, for example, better results are usually obtained when the oil entering the centrifuge is at a relatively high temperature, for example, 140° to 160° F., while with other oils separation is more complete at a lower temperature, for example, 70° to 100° F. With some oils which remain liquid below 70° F. temperatures lower than 70° F. may be employed and still others may require temperatures between 100° and 140° F. Separation at temperatures materially above 160° F. is usually impractical as water vapors tend to form in the layer of foots in the centrifugal separators causing the foots lighter than the oil, thus causing "priming" or delivery of the foots with the oil.

Vegetable oils ordinarily come from the press or expeller at temperatures from approximately 140° to 160° F., and thus in many instances these heated oils may be delivered directly to the centrifuge. In other cases it may be desirable to reduce the temperature of the oils before centrifuging. This may be done either by allowing the oils to stand in storage or passing the same through a continuous cooling device. Also, oils which have been allowed to cool in storage may have their temperature raised by heating the same until the proper temperature for the particular oil is obtained before they are delivered to the centrifuge.

In Figure 1 a tank 25 indicates a source of supply for vegetable oils and may be either a large storage tank or a small tank for temporarily receiving the oils from the presses. If the oil is at the correct temperature for separation of the particular oil being treated, it may be delivered directly to the centrifuge 9 by pipes 26 and 27 by opening the valve 28 in the pipe 26 and closing the valve 29 in the pipe 30. If it is desirous to increase or lower the temperature of the oil it may be circulated with the heat exchanger 31 by a pump 32. The heat exchanger 31 may include a coil 33 enclosed in a casing 34 through which a heating or cooling medium, such as, for example, water, may be circulated. The oil is then delivered to the centrifuge through the pipe 23. A small amount of water or other carrying liquid is withdrawn from a tank 35 and delivered by a pipe 24 into the stream of oil entering the centrifuge 9. The proportions of carrying fluid and oil are not in general extremely critical so that accurately proportioning devices are not usually necessary, but such proportioning devices are well known in the art and can be employed if desired. Foots are discharged from the centrifuge from the pan 17 and delivered to a receiver 36 while the oil is discharged through the pipe 19 into a receiver 38. The oil from which the foots have been removed may be then carried to subsequent refining steps or may be sold as an article of commerce.

In the case of drying oils particularly, it is many times desirable to remove traces of water, present in the oil as it comes from the press or
introduced therein by admixture of small portions of the carrying fluid from the tank 33. This may be conveniently accomplished by vacuum drying the oil in a vacuum chamber 39 provided for with a condenser 40 for condensing water vapors withdrawn from the oil and a receiver 41 for receiving the condensed water. A vacuum may be maintained in the vacuum chamber 39 by means of a vacuum pump 42 connected to the receiver 41. In many cases the amount of water vapor withdrawn from the oil is sufficiently small that the condenser 40 and receiver 41 may be eliminated and the vacuum pump 42 connected directly to the vacuum chamber 39.

If the oil being dried is centrifuged at a relatively high temperature, for example, 140° to 160° F., the oil may be delivered directly to the vacuum chamber through the pipes 43 and 44 but if low temperatures are found desirable in the centrifuge 9 for a particular oil, the centrifuged oil may be pumped by a pump 45 through a heat exchanger 46 which may be entirely similar to the heat exchanger 31. The oil is heated in the heat exchanger to the temperature necessary to substantially complete all moisture in the evaporating chamber 38. Thus, for example, the oil may be heated to temperatures from 140° to 212° F. or even higher if necessary. The dried oil may be withdrawn from the evaporating chamber 38 through a pump 47 and delivered through a pipe 48 to a receiver 49. An extremely stable oil is thereby produced which in the case of drying oils is classed as a "non-break" oil, i.e., no further precipitation of gums or foots will take place even if the oil is treated to 600° F. Eisold oils may also be passed through the vacuum chamber 39 if desired. The drying of the oils renders them extremely stable against the formation of additional free fatty acids and the oils may be maintained in storage for extended lengths of time prior to refining without degrading.

Even if the oil is delivered to the centrifuge at temperatures as high as 160° F., the application of further heat by passing a heating medium through the heating jacket 20 surrounding the centrifugal bowl produces advantageous results. When the bowl is not thus heated, the rapidity of solidification of the oil in contact with air within the heating jacket 20 violently agitates the air and causes heat to be rapidly transferred from the bowl wall to the wall of the heating jacket 20 and thus to the atmosphere. This effect is even more pronounced if no jacket is provided around the centrifugal bowl as heat from the bowl is rapidly dissipated into the atmosphere. This lowers the temperature of the bowl walls and in many cases causes the foots or gums to tenaciously adhere thereto. The temperature of the foots may, by the present invention, be maintained when the oil is delivered in a heated condition to the centrifuge and when relatively cool oil is delivered to the centrifuge the temperature of the foots may be raised immediately adjacent the centrifugal bowl so that the foots will slide off the wall of the bowl or flow out of the centrifuge. As before indicated, it is impractical to heat the oil entering the centrifuge to a temperature sufficient to cause this sliding or flowing of the foots as such temperatures will cause water vapor to form in the foots so that they become lighter and portions of the foots are delivered with the oil. This condition is known as "priming" and results in imperfect separation. The heating of the bowl walls, however, allows the foots leaving the centrifuge to be raised or to be maintained at the highest temperature practical without priming and also allows the foots to be heated before distilling even though separation in the centrifugal bowl is more efficient at lower temperatures than the discharge temperature of the foots.

As hereinbefore indicated, the introduction of a carrying fluid such as water or a salt solution into the centrifugal bowl materially assists in carrying the foots out of the bowl and with certain types of oil may be employed without the application of heat to the centrifugal bowl. Very small amounts of carrying liquid are ordinarily sufficient, for example, one-fourth to 1% of the weight of the oil, although larger proportions of water up to, for example, 10% are sometimes advantageous. The carrying liquid should be of greater specific gravity than the oil and substantially immiscible with the oil, and is desirable of even greater specific gravity than the foots so as to mix with and float the foots out of the centrifuge. Water is ordinarily employed for this purpose although salt solutions, such as solutions of sodium chloride, are many times advantageous because of their tendency to prevent the formation of emulsions between the oil and foots. Any water soluble salt which is substantially inert to the oil can thus be employed. The carrying liquid does not function as a reagent for precipitating and rendering gums insoluble and it is, therefore, desired to avoid as far as possible mixing of the carrying solution with the oil.

The temperature of the carrier fluid is preferably that of the oil entering the centrifuge but may be of a higher temperature in order to raise or maintain the temperature of the foots so as to reduce their viscosity and assist in carrying them out of the centrifuge. Thus carrier fluids at a temperature of 100° to 180° F. can be successfully employed.

In the process of the present invention the foots or gums which usually settle as a sludge in vegetable oils are quickly separated from the oil in a continuous manner. No precipitating agent need be employed. The oil may be thoroughly mixed with the oil to precipitate the gums. However, I have discovered that there is a certain amount of moisture contained in the oils as a result of the usual hot pressing operation which inherently functions, to some extent, as a precipitating reagent. The separated foots or emulsions discharge continuously from the centrifugal when the carrier liquid described or the application of heat to the bowl of the centrifugal or both are employed depending upon the nature of the oil being treated.

While I have disclosed the preferred embodiments of my invention it is understood that the details thereof may be varied within the scope of the following claims.

What I claim is:

1. A continuous process of separating gums containing phosphatides from fatty oils which comprises the steps of quickly precipitating said gums with the aid of a precipitating agent comprising moisture in such a small quantity as to produce gums incapable of being removed from the oil by centrifugal separation, promptly and continuously, before injury to said oil by said precipitated gums, centrifugally separating the precipitated gums from the oil, facilitating such centrifugal separation of the previously precipitated gums by introducing a carrier liquid heavier than said oil.
oils into the centrifugal separator whereby to condition the previously precipitated gums for discharge from the bowl without adherence thereto.

3. The process as defined in claim 1 in which the carrier liquid is no greater than 10% by weight of the oil.

4. The process as defined in claim 1 in which the carrier liquid is inert to said oil whereby to prevent injury thereto.

5. The process as defined in claim 1 in which the carrier liquid is of greater specific gravity than the foams.

6. The process as defined in claim 1 in which the carrier liquid comprises a salt solution.

7. A continuous process for separating gums containing phosphatic material from fatty oils which comprises the steps of: continuously subjecting said oils, in the presence of a precipitating reagent of a character and amount insufficient to substantially attack the oil or the free fatty acids contained therein and insufficient to form foams capable of said centrifugal separation, to a temperature sufficient to rapidly precipitate the gums in the form of sticky and difficultly separable foams; promptly, before injury to the oil by said foams, centrifugally separating said foams from the oil, facilitating such separation of the previously precipitated gums by applying heat to the zone of centrifugal separation whereby to cause said sticky foams to be discharged from the centrifugal without adherence thereto and further facilitating said separation by simultaneously introducing a heavier carrier liquid during the centrifugal separation.

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