Aug. 24, 1943. B. H. THURMAN 2,327,569

PROCESS OF REFINING

Filed March 29, 1941

Fig. 1.

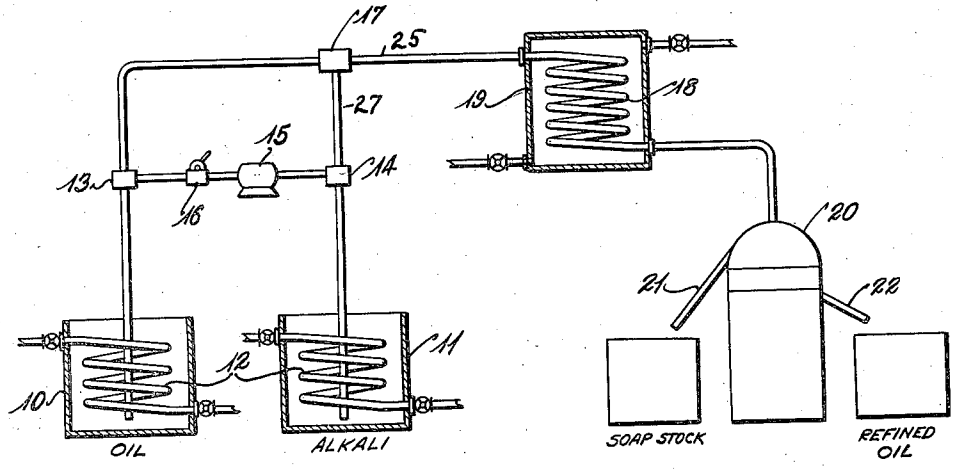
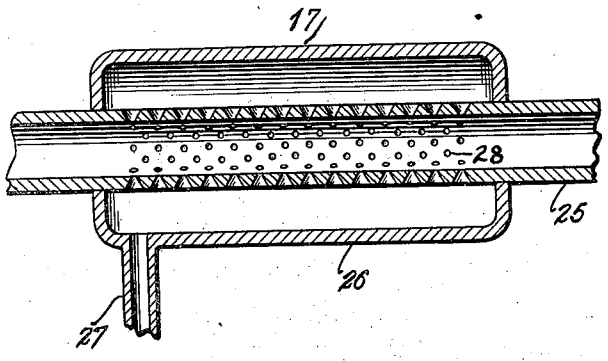


Fig. 2.



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PROCESS OF REFINING

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5 Claims. (Cl. 260—425)

The present invention relates to a method of refining crude fatty oils such as animal and vegetable oil.

It has been found that in conventional oil refining processes, whether batch or continuous, in which a refining agent such as caustic alkali is mixed with the oil to neutralize the free fatty acids and to remove color and gums, the mixing of the alkali and the oil, while relatively vigorous, is far from that necessary to insure substantially instantaneous uniform distibution of the alkali through the oil. Such conventional mixing procedures result in the distribution of relatively large globules of alkali through the oil and the distribution of relatively large globules of oil through the alkali, the former predominating. The alkali reacts with the fatty acids almost instantaneously, forming soap, which in turn emulsifies and envelops crude oil particles. Also, unreacted alkali is enveloped in crude oil. Thus, there are areas of untreated crude and of unreacted alkali, surrounded by soap layers. In due time and after prolonged stirring, some of these enveloped masses may be worked back into the bulk of the material, thereby permitting reaction. It will be apparent, therefore, that quantities of reactants are segregated from the reaction zone, and consequently there can be no uniformity of reaction. Furthermore, because of the different manner in which fatty acids, phosphatides, coloring matter, and neutral oil react, it is possible to start one reaction without allowing the other to progress. For instance, if the alkali is not uniformly distributed instantly throughout the oil, the immediate formation of soap not only shuts out crude oil but prevents the other necessary reactions from proceeding in areas where uncombined caustic is not available.

Refining operations employing the above type of mixing require a relatively extended period of contact to complete the various phases of the refining. It will be apparent, therefore, that the period of contact between the oil and the alkali must be sufficient to permit, either by diffusion or by continued agitation, contact between alkali and the impurities and to permit reaction between them. This extended period of contact, while considerably reduced in the continuous operation over that encountered in batch operation, is still sufficient to permit material losses by saponification and entrainment of neutral oil.

The present invention contemplates the admixing of the oil and alkali substantially instantaneously. It has been found that by physically distributing the alkali through the oil at such a rate that distribution is accomplished before substantial chemical reaction has had time to take place with the fatty acids, that all the refining reactions can start simultaneously throughout all the oil and will be complete much sooner than in conventional refining processes. It has been found that the reactions of neutralization, hydration of the gums and phosphatides, absorption of color, and saponification of neutral oil occur at rates decreasing in the order named. Therefore, by securing complete distribution of the alkali through the oil before any substantial amount of chemical reaction takes place, the refining reactions of neutralization, hydration and absorption of color can be completed before any material saponification of neutral oil can occur. Also, degradation of phosphatides due to long contact with strong alkali is materially reduced. That is, the alkali is promptly used up in the desired reactions or is promptly removed from the oil and is not retained in the mixture so as to be available for the undesirable slower reactions for substantial periods of time.

It is, therefore, an object of the present invention to provide a refining process in which the oil and refining agent are thoroughly admixed substantially instantaneously.

It is another object of the present invention to provide a refining process which results in decreased neutral oil losses.

A further object of the present invention is to provide an improved refining process which permits the employment of a minimum quantity of refining agent.

A still further object of the present invention is to provide an improved process of removing and recovering phosphatides in an undecomposed condition.

Another object of the present invention is to provide an improved refining process in which the oil and the refining agent are substantially completely admixed physically before chemical reaction begins.

These and other objects of the present invention will be apparent to those skilled in the art from the following description of the invention:

Crude animal and vegetable oils are composed principally of glycerides of fatty acids, and in addition thereto contain small quantities of free fatty acids, gums, phosphatides, coloring matter and the like. It is the object of the refiner to remove these minor constituents and obtain, as far as possible, a substantially colorless oil composed solely of the glycerides. Conventional

refining includes the treatment of the crude oil with aqueous solutions of caustic alkalies such as sodium hydroxide. The sodium hydroxide reacts readily with the free fatty acids to form soaps thereof. The gums and phosphatides hydrate readily in the presence of water. In the presence of aqueous sodium hydroxide the phosphatides become hydrated and lose some of their oleophilic character and thus become less soluble in oil. They do, however, still retain powerful emulsifying properties. The sodium hydroxide, however, then proceeds to react with the phosphatide molecule to form what is believed to be an absorption complex. This complex is even less soluble in the oil and to a large extent has lost its emulsifying properties, and can be removed from the oil efficiently by centrifugal force. In the course of time this sodium hydroxide phosphatide complex decomposes into sodium glycerophospheric acid and other products. This decomposition is at a relatively low rate and probably occurs at a rate similar to that of saponification of the neutral oil.

The color constituents of the crude oil are removed by means of caustic alkali, probably by modification and adsorption by the soap. This adsorption is believed to occur at a rate less than the neutralization of the free fatty acids and the formation of the phosphatide complex but at a faster rate than the saponification of neutral oil. From the above description it will be apparent that by physically distributing the alkali substantially uniformly through the oil before any substantial amount of chemical reaction can occur, the above reactions will occur simultaneously and will be completed in the order of the reaction rates. It will thus be seen that the main steps of refining, namely the neutralization of free fatty acids, the hydration of the phosphatides and the absorption of color, can be completed before any large quantity of the neutral oil has been saponified and phosphatides decomposed. By rapidly separating the aqueous phase containing the impurities, from the oil phase after refining is complete, substantially all the neutral oil can be recovered in a relatively pure state. Similarly, by treating the separated aqueous phase containing the phosphatides in the alkali complex, with an acid, it is possible to stop the decomposition of the complex and to recover phosphatides in a substantially undecomposed form.

In Fig. 1 of the drawing there is shown a diagrammatic illustration of one embodiment of the present invention. In this figure there is shown a supply of crude oil in tank **10** and a supply of refining agent in tank **11**. Each of these tanks may be supplied with heat exchange coils **12** by means of which the temperature of the oil and the refining agent may be suitably adjusted. Measured quantities of the proportioned streams of the oil and alkali may be withdrawn from tanks **10** and **11** by means of pumps **13** and **14** respectively. These pumps may be driven by a common variable speed motor **15**. The variable speed mechanism **16** permitting the desired proportioning of the streams of oil and alkali. The proportioned streams may be mixed in a mixer **17** in which the alkali is uniformly distributed throughout the oil in a substantially instantaneous manner, to be described hereinafter. The mixed stream of oil and alkali may then be passed through the coil **18** disposed within tank **19** which may contain a heat exchange medium for the purpose of conditioning the mixture of oil and impurities for the step of centrifugal operation. The mixture is then discharged into centrifuge **20** in which it is separated into soap stock and refined oil which are discharged through spouts **21** and **22**, respectively.

Fig. 2 illustrates a section through a mixer which has been found suitable for the present invention. This mixer is composed of a central pipe **25** surrounded by a jacket **26** having a suitable inlet **27**. The central pipe **25** is provided with a large number of minute orifices **28** which permit communication between the jacket **26** and the interior of the pipe **25**. These orifices are extremely minute and may, for example, be from .001 to .01 inch in diameter. The proportioned stream of crude oil from pump **13** is passed through the pipe **25**. The proportioned stream of alkali from pump **14** is introduced into the jacket from the inlet **27**. It is preferred to employ a large pressure differential for example from 100 to 1000 pounds per square inch across the orifices **28**. By means of this high pressure differential minute quantities of alkali in the form of mist or spray distribute themselves through the body of flowing oil. By means of this high pressure differential the mixing of the oil and alkali can be completed before any substantial amount of chemical reaction occurs. It is preferred to spray the reagent into a moving cylinder of crude oil without disruptinging the body of the cylinder to any substantial extent by mechanical means. The reagent is broken into extremely fine particles or droplets at the point where it is flashed or exploded into the flowing mass of oil. The particles of reagent have such a high velocity and possess so much kinetic energy that they penetrate the crude oil and are dispersed instantly. In this way the high velocity particles of refining agent break up the oil physically before substantial chemical action occurs.

In this manner the various refining reactions may be instituted simultaneously and the resultant mixture of oil and impurities is ready for separation when the absorption of color, apparently the slowest refining reaction, has been completed. The mixture may be prepared for centrifugal separation by heating to an emulsion breaking temperature or in any other manner.

Where it is desired to recover the phosphatides in undecomposed condition, the degradation of the phosphatides by the alkali can be stopped in several ways, for example, by rapidly drying the soap stock or by neutralizing the alkali present.

Thus, the soap stock immediately after it has been separated from the oil may be dried, preferably in a vacuum, following which phosphatides may be extracted with a solvent such as benzol, petroleum ether, or a mixture of benzol and alcohol. Alternatively the dried soap stock may be treated with a solution containing enough acid to neutralize the alkali, and then the phosphatides may be extracted with an oil solvent.

Another method is to extract the dried soap stock with acetone, which removes the oil, leaving behind the phosphatides and soap. The phosphatides may be dissolved in an oil solvent, leaving behind the soap. Or, the mixture of soap and phosphatides may be treated with enough acid to decompose the soap yielding a mixture of fatty acid and phosphatides. This mixture is capable of a number of uses without

separation of the phosphatides. These may, however, be isolated by extracting with an alcohol which removes the fatty acids.

Instead of drying the soap stock, the same may be treated directly with an acid, such as hydrochloric or sulphuric, in amounts sufficient to neutralize the excess sodium hydroxide. A further method is to treat the soap stock discharged from the centrifuge with a barium chloride solution which serves to neutralize excess alkali. The action of the alkali in the discharged soapstock may be stopped by means of adding alcohol or alcoholic solutions of magnesium chloride.

By means of any of the above procedures the decomposition of the sodium hydroxide phosphatide complex may be stopped. Furthermore, the phosphatide may be isolated from the soap stock mixture in any of the above stated ways.

While the present invention is particularly adapted to the alkali refining of vegetable oils in view of the nature of the multiplicity of reactions occurring therein, the present method of mixing may also advantageously be used in other refining operations such as degumming, employing either water oran electrolyte as the refining reagent, or in acid refining, since the present method of mixing obviates any disadvantage produced by too extended a contact of the refining agent with the neutral oil.

While various embodiments of the present invention have been described in detail, it is to be understood that the invention is not limited thereto but may be varied within the scope of the following claims:

I claim:

1. The method of refining glyceride oils, which comprises, mixing proportioned streams of oil and a refining agent by flowing a substantially uniformly advancing stream of said oil through a passageway having a small substantially uniform cross-section in sufficient amount to fill said passageway, injecting into said stream of oil through the walls of said passageway a plurality of minute high velocity jets of said refining agent, the cross-sections of said jets and passageway being sufficiently small and the velocity of said jets being sufficiently great to cause said jets to penetrate substantially instantaneously into all portions of said stream to substantially uniformly distribute minute droplets of said refining agent throughout said stream, and promptly thereafter separating from said oil impurities precipitated in said oil by said refining agent.

2. The method of refining glyceride oils, which comprises, mixing proportioned streams of oil and a refining agent by flowing a substantially uniformly advancing stream of said oil through a passageway having a small substantially uniform circular cross-section in sufficient amount to fill said passageway, injecting radially inwardly into said stream of oil in a plurality of radial directions through the walls of said passageway a plurality of minute high velocity jets of said refining agent, the cross-sections of said jets and passageway being sufficiently small and the velocity of said jets being sufficiently great to cause said jets to penetrate substantially instantaneously into all portions of said stream to substantially uniformly distribute minute droplets of said refining agent throughout said stream, and promptly thereafter separating from said oil impurities precipitated in said oil by said refining agent.

3. The method of refining glyceride oils and recovering phosphatides therefrom, which comprises, mixing proportioned streams of oil and an excess of a caustic alkali refining agent by flowing a substantially uniformly advancing stream of said oil through a passageway having a small substantially uniform cross-section in sufficient amount to fill said passageway, injecting into said stream of oil through the walls of said passageway a plurality of minute high velocity jets of said refining agent, the cross-sections of said jets and passageway being sufficiently small and the velocity of said jets being sufficiently great to cause said jets to penetrate substantially instantaneously into all portions of said stream to substantially uniformly distribute minute droplets of said refining agent throughout said stream, promptly thereafter separating from said oil soap stock including phosphatides precipitated in said oil by said refining agent, promptly thereafter treating said soap stock to prevent attack on said phosphatides by said excess caustic alkali, and recovering phosphatides from said soap stock.

4. The method of refining glyceride oils and recovering phosphatides therefrom, which comprises, mixing proportioned streams of oil and an excess of a caustic alkali refining agent by flowing a substantially uniformly advancing stream of said oil through a passageway having a small substantially uniform cross-section in sufficient amount to fill said passageway, injecting into said stream of oil through the walls of said passageway a plurality of minute high velocity jets of said refining agent, the cross-sections of said jets and passageway being sufficiently small and the velocity of said jets being sufficiently great to cause said jets to penetrate substantially instantaneously into all portions of said stream to substantially uniformly distribute minute droplets of said refining agent throughout said stream, promptly thereafter separating from said oil soap stock including phosphatides precipitated in said oil by said refining agent, promptly thereafter dehydrating said soap stock to prevent attack on said phosphatides by said excess caustic alkali, and recovering phosphatides from said soap stock.

5. The method of refining glyceride oils and recovering phosphatides therefrom, which comprises, mixing proportioned streams of oil and an excess of a caustic alkali refining agent by flowing a substantially uniformly advancing stream of said oil through a passageway having a small substantially uniform cross-section in sufficient amount to fill said passageway, injecting into said stream of oil through the walls of said passageway a plurality of minute high velocity jets of said refining agent, the cross-sections of said jets and passageway being sufficiently small and the velocity of said jets being sufficiently great to cause said jets to penetrate substantially instantaneously into all portions of said stream to substantially uniformly distribute minute droplets of said refining agent throughout said stream, promptly thereafter separating from said oil soap stock including phosphatides precipitated in said oil by said refining agent, promptly thereafter neutralizing said excess caustic alkali in said soap stock with an acid to prevent attack on said phosphatides by said excess caustic alkali, and recovering phosphatides from said soap stock.

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