

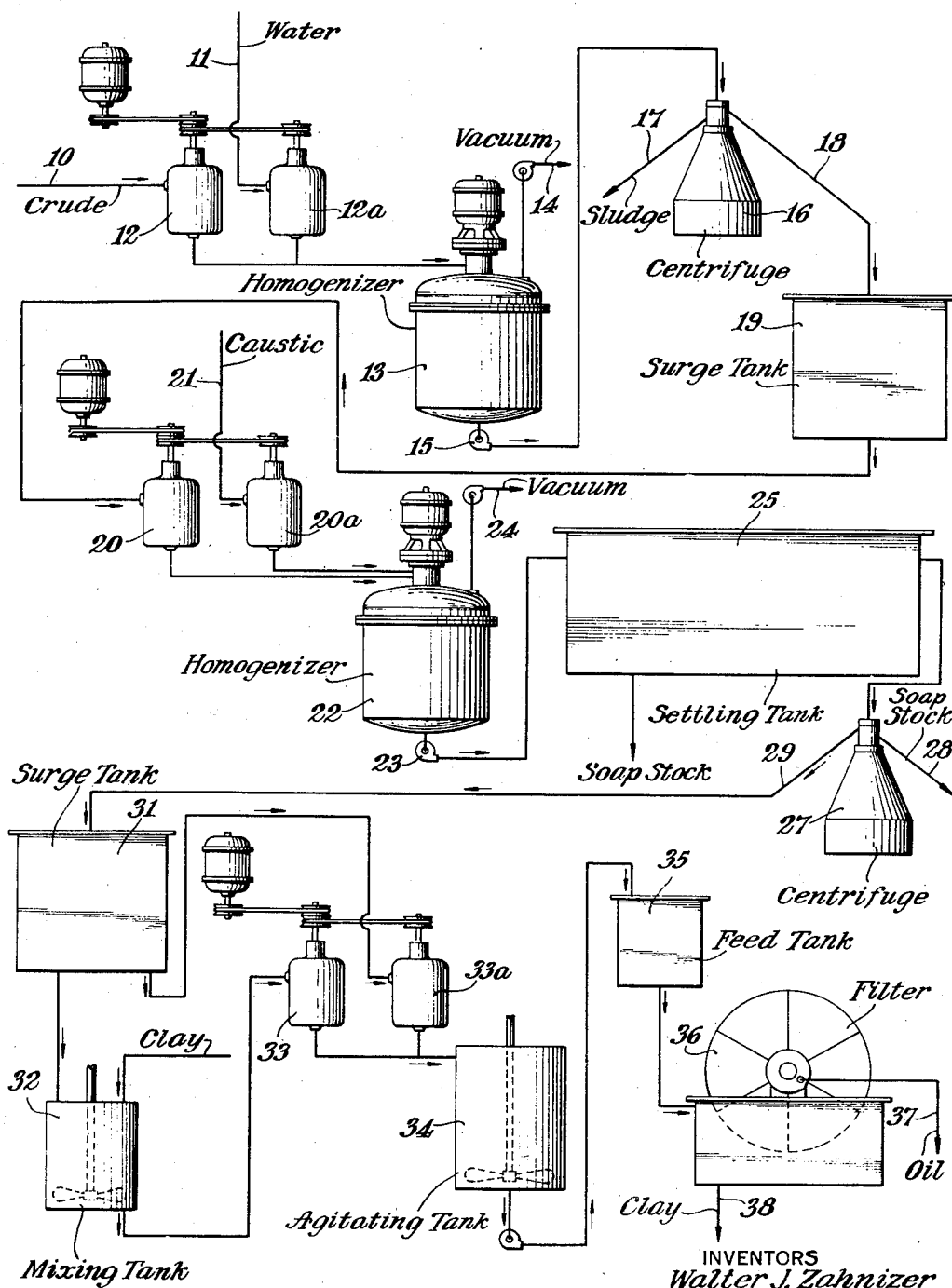
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W. J. BLOOMER ET AL

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REFINING OF COTTONSEED OIL

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INVENTORS
Walter J. Zahnizer
BY and Ward J. Bloomer
Nathaniel G. S.
ATTORNEY

UNITED STATES PATENT OFFICE

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REFINING OF COTTONSEED OIL

Ward J. Bloomer, Westfield, and Walter J. Zahner, Bayonne, N. J., assignors to The Lummus Company, New York, N. Y., a corporation of Delaware

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This invention relates to improvements in refining fatty oils such as animal and vegetable oils, and, more particularly, to the refining of fatty acid containing glyceride oils such as cottonseed oil.

Our invention is adapted to the refining of various types of fatty oils of the animal and vegetable type such as soy bean, castor, corn, maize, lard and fish oils, by the removal of the fatty acid content thereof. It is particularly suitable for refining a cottonseed oil which normally has a fatty acid content of around 1 to 2% and a gum and resin content of approximately the same amount. It is the principal purpose of the invention to treat such oils so that they may be fit for human consumption, viz., free of gums, resins, coloring matter, and substantially neutral.

It has heretofore been proposed that the refining of cottonseed oil be carried out under very careful temperature control to assure an effective neutralization and a rapid separation of the soap stock. It has been found by some that it was necessary to neutralize at an emulsion forming temperature to assure an effective neutralization and prevent the setting of the color bodies and to prevent the increase of the acid number, and then heating has been required to give an emulsion breaking temperature so that the soap stock could be effectively removed from the oil. Very elaborate heating controls have been suggested with very expensive apparatus as the result.

The best prior practice has found it extremely difficult, however, to obtain a suitable color oil without excessive caustic or bleaching treatments both of which are expensive and both of which materially add to the operating losses. Even with the best of care and the most expensive of equipment, it has been impossible to reduce the refining losses to substantially below 4% which is still excessive.

In the prior application of Giles et al., Ser. No. 200,923, filed April 8, 1938, entitled Refining of cottonseed oil, there has been disclosed a process for refining animal and vegetable oils which includes the steps of water washing, decolorizing and neutralizing of the oil usually followed by the subsequent steps of oil extraction of the clay and soap treatment. Such a process has been found to have superior results in comparison to heretofore well known processes, but we have since found that certain improvements in operating conditions as generally set out therein will assure a superior product and a higher yield.

The principal object of our invention is to pro-

vide an inexpensive, rapid, commercial method of treating cottonseed oil to remove the gums, resins, coloring matter and fatty acid content so that it may be rendered satisfactory for human consumption and so that losses are reduced to a minimum.

A more specific object of our invention is to provide an improved arrangement of the sequential steps of water washing and neutralizing a glyceride oil such as cottonseed oil which can be carried out under uniform temperature conditions and with a minimum of mechanical apparatus and with a comparatively short total time interval.

Another specific object of the invention is to provide improved neutralizing and soap stock separating steps in the refining of cottonseed oil whereby high thruput can be obtained without critical operating conditions and so that slight variations in operating conditions will not materially impair the results.

Further objects and advantages of the invention will appear from the following disclosure of a preferred form of embodiment thereof taken in connection with the attached drawing which is a flow diagram showing the manner in which the process is preferably carried out.

In accordance with the preferred form of embodiment of our invention, it is proposed to first remove the gums and resins from the crude oil. This is accomplished by intimately mixing or homogenizing as by the apparatus described in Cornell's U. S. Patent 2,042,880 of approximately one to two percent of water with the initial crude. It is preferable to use a means to accomplish a mixture which readily separates and the homogenizer referred to is particularly satisfactory for such purpose. Thereafter, the sludge which consists of mucilaginous matters, gums, resins and some color bodies is separated out either by a centrifuge or by settling, this latter being facilitated by using a coagulant such as of the metal salts type. This water washed oil has markedly superior qualities as respects original crude and greatly facilitates the neutralizing steps as not only does the oil have a lower acid number, but it is substantially free of emulsifying material.

The preferred second step in the operation is to neutralize this water washed oil with the theoretical quantity of alkali, a slight excess from 2-10% and preferably about 5% being used as a practical matter. The alkali is preferably introduced as an aqueous solution or an alcohol solution or a combination of the two. The concentration may range from 5 to 50% and for most

purposes, 10% to 30% is found most effective. A relatively small excess appears adequate probably because there is no extraneous matter for the alkali to react with. Furthermore, there is no saponification of the oil itself. The neutralization is also preferably carried out in an homogenizer or device which will permit intimate mixing in a short period of time, also preferably of the type described in Cornell U. S. Patent No. 2,042,880. The use of a device of this kind permits of the introduction of the alkali as a highly concentrated solution without damage to the product, if this is desirable.

Because of this unique apparatus, we obtain an admixture of the neutralizing agent and oil of a type which has been impossible by prior devices heretofore known. The alkali and oil are blended and compounded in an exceptionally intimate attenuated stream or film and under the desired vacuum. The total treating time is not substantially in excess of a second and ordinarily is but a fraction of a second the film is subjected to a substantial centrifugal force. This force acts to continuously carry the mixed oil and soap particles off the rotating disc. The film is extremely thin, in the neighborhood of .005" thick and is preferably not allowed to become subdivided or to substantially flash or vaporize. The soap thus remains soft and can be continuously treated. No emulsion results from this treatment at temperatures below 140° F. and even as low as 70° F.

As no emulsion is formed, the soap particles readily separate from the oil and we find that in one hour, approximately 90% of the sludge or soap stock will separate out. In some cases, a coagulant such as sodium silicate, and/or aluminum hydroxide may be added in very small amounts to facilitate this action. The settling action can take place in a single tank or series of tanks of the intermittent type or may be accomplished in a continuous settling device as disclosed in the Dorr Company patents. It is preferable to centrifuge this settled oil to remove the residual oil from the soap stock, so that a completely neutral and soap free oil can be obtained promptly and in large quantities and with a minimum of centrifugal apparatus. Thereafter, the oil can be further treated with clay if a premium color is desired and the soap stock will be salted out or acidified as desired. By the sequence of steps outlined, only a small quantity of clay is necessary as compared with preliminary decolorizing procedure as the alkali neutralizing agent has already acted on the color bodies to a limited extent.

In accordance with the attached drawing, the preferred process includes the mixing of the crude oil at 10 with approximately 1% of water at 11 controlled by suitable proportionating devices 12 and 12a, for example, as described in Cornell U. S. Patent No. 2,109,331. The properly proportioned oil-water mixture is then introduced into the mixing device 13. The mixing device which is preferably of the type described in Cornell Patent No. 2,042,880, and is preferably operated under a vacuum obtained by vacuum line 14, intimately mixes the oil and water. The mixture is then removed by the pump 15 and discharged into the centrifuge 16. This may be of either the batch or continuous type, but is preferably of the continuous type with the sludge removed at 17 and the gum-free oil removed at 18. If desired, a settling tank may be used to remove part of the sludge prior to centrifuging

to reduce the number of centrifuges otherwise necessary.

This gum-free oil removed at 18 is particularly characterized by its freedom from gummy, resinous and mucilaginous material and its freedom from a tendency to emulsify. It is temporarily collected in surge tank 19 from which it is pumped through proportionating device 20 into mixing device 22. The theoretical amount of caustic alkali from line 21 is established by proportionating device 20a and is separately introduced into the mixing device 22. This is preferably of the homogenizer type disclosed in the Cornell Patent 2,042,880 which is the only apparatus now known which will accomplish the neutralization without forming a permanent emulsion. It is, of course, understood that the theoretical amount of caustic may include quantities of 105% of that theoretically necessary to neutralize the fatty acids but due to the very intimate attenuation within the homogenizer 22 and the complete contact in the short time, the theoretical amount of caustic is adequate to neutralize the fatty acids and there is no saponification of the oil.

The homogenizer 22 is also preferably operated under a vacuum induced at 24. The vacuum prevents oxidation and prevents entrainment of air and assists in maintaining the thin film which is so essential. Having eliminated the emulsifying material and having operated under the vacuum, and with an attenuated and continuous stream in the homogenizer, the soap and oil mixture does not emulsify. It will readily separate and is preferably discharged by pump 23 into a settling tank 25. Such mixture has no free caustic therein and the oil phase has a very low acid content in the range of .03% or less.

Settling of this oil for an hour period and with a coagulant, if desired, will permit approximately 90% of the oil to become substantially free of soap particles. To make the process continuous and to materially reduce the number of centrifuges required, the tank 25 is conveniently of two to three hours' capacity which, at 1500 gallons per hour capacity of the homogenizer, is from 3000 to 5000 gallons. Such oil is then drawn off at the top and centrifuged or mechanically separated at 27 to complete the freeing of the oil from the soap stock which discharges at 28. The oil is discharged at 29 to surge tank 31, and in this case is bright in color, free of gums and resins and is neutral. From time to time or continuously with a continuous type of settling tank, soap stock is removed from the bottom of settling tank 25.

If desired, the oil collected in surge tank 31 may be decolorized further by mixing it with clay or other decolorizing agent. Preferably a part of the oil enters mixing tank 32 for mixing with the clay to form a slurry and then the clay slurry and oil are passed through proportionating pumps 33 and 33a into an agitating tank 34 and thence into the filter feed tank 35. The oil is filtered from the clay in filter 36 and the bleached oil discharges at 37. The clay is removed at 38.

The temperatures of operation may be varied within considerable limits. If the entire process, including water washing, neutralizing, etc., is operated at 90° F., the loss of material as gums, resins and soap stock can be kept down to 3.9% of the charge with a free fatty acid content not to exceed 2%. When the process is continuously operated at 140° F., the refining loss

in the water washing and neutralizing is reduced to around 2.9%, which is probably due to the reduced viscosity of the oil at the higher temperature. The thruput is also more rapid than at 90° F. Operating much above 140° F. does not appear to be helpful. The range of 125-145° F. appears to be the beneficial limits of the operation.

Under normal conditions, no heat need be added at any stage although it will probably be found advisable to insulate the apparatus to conserve an initial temperature so that the conditions are as nearly constant as possible.

While we have shown and described a preferred form of embodiment of the invention, we are aware that modifications may be made thereto and we therefore, desire a broad interpretation of the invention within the scope and spirit of the invention as described herein and as claimed hereinafter.

We claim:

1. The method of refining cottonseed oil which comprises the steps of mixing the cottonseed oil with a proportionate amount of water such that the gums, resins and mucilaginous compounds are converted to oil insoluble material, thereafter continuously removing said insoluble material, blending said oil, free of said oil insoluble material, with substantially the theoretical amount of caustic alkali necessary to neutralize the free fatty acids in the oil, maintaining said oil and caustic alkali mixture in a thin film, under a sub-atmospheric pressure, and under a substantial centrifugal force for a period not substantially in excess of a second, thereafter settling said oil and soap stock to partially remove the soap stock, and subsequently centrifugally separating the partially settled oil to completely remove the soap stock.

2. An improved method for continuously removing the gums and resins in a crude glyceride oil which comprises the steps of mixing the oil with approximately 1% of water, passing said mixed materials to a rotary disk mixing device, rotating said disk at a relatively high peripheral speed to cause said materials to flow across the disk in a continuous thin film under substantial centrifugal force, removing the thin film continuously and passing it into a liquid receiving zone without forming an emulsion of the oil, and continuously separating the oil and hydrated matter by centrifugal force, the movement of the oil being continuous and confined from the air.

3. An improved method for refining a glyceride oil as claimed in claim 2, in which the oil is subsequently combined with a caustic neutralizing agent on a rotary disk mixing device which is operated at a relatively high peripheral speed to cause said materials to flow across the disk in a thin film under substantial centrifugal force for the immediate neutralization of the fatty acids therein, and the oil is removed in a continuous film into a liquid receiving zone without forming an emulsion of the oil.

4. In the refining of cottonseed oil the improvement which comprises the steps of conducting separate streams of oil and a caustic neutralizing agent to a rotary disk mixing device, combining

said materials on said disk and rotating said disk at a relatively high peripheral speed to cause said materials to flow across said disk in a thin film under substantial centrifugal force whereby immediate neutralization of the fatty acids is accomplished, removing the mixture of oil and neutralized acid in a continuous film into a liquid receiving zone without forming an emulsion of the oil, continuously passing said oil and neutralized acid through said zone, said zone being of such size as compared to the flow of liquid therethrough that there is a partial separation of oil from neutralized acid, and thereafter continuously centrifugally separating the partially separated oil.

5. An improved method for neutralizing the free fatty acids in cottonseed oil as claimed in claim 4, in which the charge oil is first passed into a mixing zone with a proportion of water, said oil and water being subjected to a relatively high velocity force on a disk which attenuates the mixture to a thin film, and immediately hydrates the gums and resins in the oil, said mixing zone being maintained under subatmospheric pressure and at a temperature similar to the temperature in the neutralization step, said film being maintained until said liquids reach a liquid receiving zone whereby an emulsion is avoided, and thereafter said water washed oil is centrifugally separated to remove the sludge, the passage of the oil through the system being continuous and in a confined stream.

6. A continuous method for refining crude cottonseed oil having a relatively free fatty acid content and entrained resinous matter which comprises passing the oil with approximately 1% of water across a relatively high-speed rotating disk in a confined zone maintained under sub-atmospheric pressure, in which zone the oil and water are restrained to a thin film moving under high velocity, during which movement the resinous matter is hydrated, continuously removing the film of oil and hydrated matter into a liquid receiving zone without the formation of an emulsion, centrifugally separating the resinous matter from the oil, conducting the oil to a second rotating disk, separately conducting a neutralizing agent in substantially the theoretical amount required to neutralize the free fatty acids to said second disk, rotating said second disk to establish a thin film of oil and neutralizing agent under high velocity centrifugal movement for a short period, during which the relative thinness of the film, the small amount of neutralizing agent and the uniform dispersion of the neutralizing agent permit a preferential neutralization of the acids without saponification of the oil or the formation of an emulsion, continuously removing the oil and neutralized acids into a liquid receiving zone without the formation of an emulsion, passing said oil continuously through said zone at a rate at which some of the neutralized acids settle out, and thereafter centrifugally separating the balance of the neutralized acids from the oil.

WARD J. BLOOMER.
WALTER J. ZAHNISER.