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METHOD FOR THE CONTINUOUS ALKALI REFINING OF VEGETABLE OILS

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Fig. 1

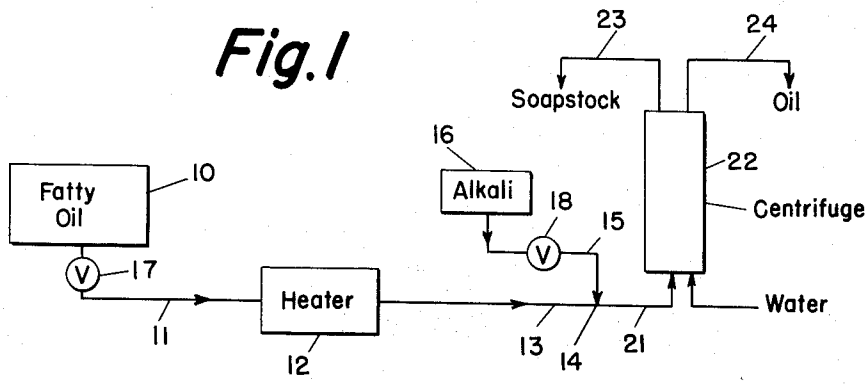
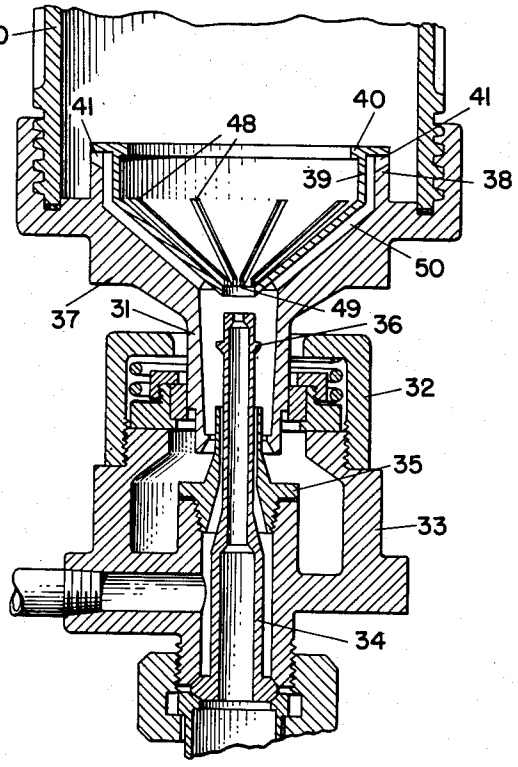


Fig. 2



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METHOD FOR THE CONTINUOUS ALKALI REFINING OF VEGETABLE OILS

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8 Claims. (Cl. 260—425)

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This invention pertains to the refining of fatty oils, (e. g. glyceride oils), and more particularly to the refining of vegetable and animal oils, such as cottonseed oil, soya bean oil, peanut oil, corn oil, sunflower seed oil, sesame oil, rape seed oil, cocoanut oil, babasu oil, palm oil, palm kernel oil, linseed oil, tallow, lard, grease, fish oil, whale oil, etc.

Fatty oils are ordinarily refined by adding to the oil an aqueous caustic soda solution, followed by conditioning, including at least some agglomeration of soap stock, and then separating from the oil such soap stock together with other impurities. The purpose of such refining is to neutralize and remove free fatty acid, to remove gums, and to improve oil color. In the conventional processes of the prior art, a considerable excess of alkali is employed above that needed to neutralize the free fatty acid present in the oil, not only to insure complete neutralization of free fatty acid but also to provide an excess for treatment and removal of gums and of color bodies present.

In the kettle refining of vegetable oil, which was the earliest method used, the caustic soda solution in excess was introduced into a kettle of oil and agitated until the caustic soda reacted with the free fatty acid, gums, etc., forming soap stock particles. The body of oil was then allowed to be quiescent so that the soap stock formed could settle to the bottom, permitting the refined oil to be drawn off from the top of the kettle. In this kettle refining, the excess of caustic soda had an opportunity to attack the gums and color bodies so as to improve the quality and color of the oil.

An improvement over this method of refining is the continuous method in which the caustic soda solution, in considerable stoichiometric excess, is added to a flowing stream of vegetable oil, and the mixture agitated and conditioned to cause the caustic soda to react with the free fatty acids, gums, and color bodies. This results in agglomeration of soap stock. The composite stream is then continuously separated into oil and soap stock by the use of a centrifugal separator. In this continuous process improved yields are effected by the fact that centrifugal force, being very much more powerful than gravity, is able to squeeze a portion of the entrained oil out of the body of soap stock. Then too, the time of contact between caustic and oil is materially reduced over the time required for gravity subsidence in a kettle, and further improved yields are made possible by the fact that in this time of shorter treatment there is less opportunity for the excess caustic soda to attack and saponify neutral oil.

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The purification of fatty oils, and particularly of vegetable oils, is conducted industrially on an extensive and rapidly expanding scale. The products produced enter a highly competitive market, and for this reason economies in production and improved yields, even though not glaringly large percentagewise, are of outstanding importance in the advancement of the art. The present invention is addressed to both of these considerations.

In the practice of this invention increased economies and yields are obtained through process simplifications and through reduction of losses encountered by virtue of (1) entrainment of neutral oil in the soap stock phase, (2) saponification of neutral oil, and (3) emulsification of neutral oil.

Entrainment of oil in the soap stock is primarily the result of agglomeration of the soap particles formed upon reaction of the caustic soda with the free fatty acid present in the oil undergoing refinement, and is aggravated by the presence of emulsion. Conditioning of the oil, involving large scale agglomeration of the soap particles, was originally thought necessary to the successful separation of the soap stock from the oil by centrifugal force, and although it was subsequently discovered that the degree of agglomeration might be materially reduced, without leaving in the separated oil an unduly large percentage of soap stock particles, yet the times and conditions suggested and thought necessary for contact between caustic soda and oil prior to separation by centrifuging have been such as to permit the originally-formed extremely small soap particles to build up into larger particles prior to centrifugal separation. This, among other things, was because it was considered necessary to have a significant increase in the mass of the individual particles present in order to successfully separate the soap stock particles by centrifugal force. Thus, while mixing times of less than one minute have been suggested, none have been for substantially less than about 20 to 30 seconds, and even during these shorter times agglomeration proceeds to such an extent as to entrain in the soap stock particles, although still small, a significant amount of neutral oil, especially under the conditions of mixing employed.

In all prior art continuous refining processes of which I am aware, a considerable stoichiometric excess of caustic over free fatty acid present in the oil is employed, whether the aqueous solution of the caustic is of ordinary or of relatively

high concentration. Thus, while the so-called short contact times of the prior art reduce the degree of saponification of neutral oil, conditions favorable to saponification are nevertheless present, and loss of some neutral oil, even though considered small, is the result. This is particularly true when the caustic is present in the aqueous solution in relatively high concentration. Furthermore, the conditioning of the mixture of caustic and oil prior to its separation, which heretofore has been considered essential, is obtained primarily by agitation, so that proportional increase in agitation was considered a necessary concomitant to any decrease in contact time.

All crude vegetable oils contain a varying percentage of gums in solution. These gums have an affinity for water. When a crude vegetable oil is treated with an aqueous solution of caustic, the water present tends to hydrate the gums, whereupon the gums absorb neutral oil in an emulsified state up to a very considerable percentage of their weight, such as up to one-third. The gums upon hydration separate from the neutral oil, and upon centrifuging are discharged with the soap stock with the result that their emulsified oil content is lost as far as neutral oil yield is concerned.

In the practice of the present invention a stream of aqueous caustic soda of relatively high caustic concentration, that is above 20° Bé., and preferably between 22° and 30° Bé., is continuously added, in substantially stoichiometric proportion, to a continuously flowing stream of oil to be refined. It is preferred that contact between caustic and oil be effected by virtue of turbulent flow, as distinguished from the extensive mechanical mixing heretofore considered essential, and which I have discovered is not essential even though extremely short contact times are used, and the combined stream of caustic and oil is immediately subjected to centrifugal separation, that is, within a period of not more than 5 seconds, (but not less than ½ second), between the time that any particular portion of caustic first contacts any particular portion of vegetable oil, and the time that said combined portions are subjected to centrifugal force for purposes of their separation.

By virtue of my process and the manner in which it is preferably practiced, agglomeration of soap stock and emulsion formation is held to a substantially irreducible minimum, and contra to the teachings of the prior art. I have discovered that the initially-formed soap stock may be efficiently separated from the neutral oil by centrifugal force in the ordinary centrifuges customarily employed for the purpose, such centrifuges being rated as developing centrifugal forces, for example, from 4000 to 20,000 times the force of gravity, a centrifuge being rated as developing a centrifugal force of 13,500 times the force of gravity being typical.

In the practice of the invention, there is no material excess of caustic present, (a substantially stoichiometric quantity being considered to vary approximately from 0.9 to 1.1 times that quantity which is theoretically required to neutralize the free fatty acid present). It follows that there is no opportunity for significant saponification of neutral oil by virtue of the presence of any significant quantity of excess caustic. On the other hand, I have discovered that contra to prior art teachings, an excess of caustic is not required, when used in high concentration, to effectively neutralize the free fatty acids in continuous refining. This is true even though the contact time is reduced to below 5 seconds, and even though mixing is mild, e. g., by turbulent flow.

In view of the extremely short time that the high concentration aqueous caustic is in contact with the oil, and in view of its lower water content, the hydration and separation of gums is brought to a substantially irreducible minimum. The result is that no significant total quantity of neutral oil is lost by absorption in separated gums.

While my process results in very little, if any, removal of gums and of color, it is found that this may be done more expeditiously and with less loss of neutral oil if performed in a separate step. Gums may be removed, for example, prior to my process, such as, by water hydration followed by centrifugal separation, if desired, by processes well known in the prior art, or otherwise. It will be appreciated that in the case of certain oils it is unnecessary to remove color depending upon the orginal color of the oil prior to treatment and/or the use to which the oil is to be put. Light-colored finished oils are preferred for some purposes, but for other purposes a dark oil may be preferred, or may be acceptable. Removal of color, if desired, may also be performed in a separate step by any means known in the art, or otherwise, for example, after refining by the use of this invention.

Further features of this invention will become apparent to persons skilled in the art from the following particular description taken in connection with the drawings, in which;

Figure 1 is a flow sheet illustrating an embodiment of the invention; and

Figure 2 is a sectional elevation, shown broken, of a form of centrifuge bowl useful in the practice of the invention.

Referring now to Figure 1 of the drawings, at 10 is shown a source of fatty oil to be refined. This fatty oil flows through line 11 to heater 12 in which it may be raised to any desired temperature, such as between 140° F. and 160° F., although higher or lower temperatures may be employed, if desired, as will be understood by persons skilled in the art. The oil flows from heater 12 through line 13, and is contacted at 14 with a solution of NaOH in water which is fed into line 13 through line 15 from a source 16. This aqueous caustic soda solution is of a concentration in excess of 20° Bé., such as between 22° and 30° Bé.

A valve for the control of the flow of fatty oil is conventionally illustrated at 17, and a valve for the control of the flow of caustic soda solution is conventionally illustrated at 18. However, it is to be understood that in commercial operation these flow controls represented by valves 17 and 18 usually will take the form of proportioning devices, such as, proportioning pumps.

As previously pointed out, the rate of flow of caustic soda solution with respect to the rate of flow of fatty oil to be refined, is proportioned, and is such as to provide a substantially stoichiometric quantity of caustic soda required to neutralize the free fatty acid present. In the refining of fatty oils generally, and particularly of vegetable oils, it is more or less conventional to determine by titration the free fatty acid content of an oil to be refined, and to calculate the percentage of free fatty acids present in terms of oleic acid. For the purposes of the practice of this invention, a substantially stoichiometric quantity is considered to be approximately from 0.9 to 1.1 times that quantity which is theoreti-

cally required to neutralize the free fatty acids present as indicated by such titration.

From point 14 the composite stream of fatty oil and caustic soda solution flows through line 21 to centrifuge 22 in which the soap stock, which has been formed by reaction of the caustic soda with free fatty acid, is separated by centrifugal force from the oil, the soap stock being removed through line 23, and the oil through line 24.

As previously pointed out, it is preferred that the flow through line 21 be a turbulent flow as distinguished from laminar flow, and that the turbulence be produced without the aid of a large degree of, that is, intensive mechanical mixing of the character heretofore employed in the art. This is to avoid conditions heretofore thought necessary for efficient separation of soap stock from oil, which resulted in losses of neutral oil, and to avoid or reduce the formation of emulsion. While mild mechanical mixing might be employed while retaining some of the advantages of this invention, for best operation it is preferred that this turbulence be substantially entirely produced by the flow of the combined stream of oil and caustic through line 21. As is well understood, for a given viscosity, turbulence is a function of the rate and cross-sectional area of the flow, so that the selection of a proper size for line 21 is a mere matter of design, after the desired rates of flow and the temperatures to be employed have been determined, as is well within the knowledge of persons skilled in the art.

It will be noted that the present invention makes possible outstanding simplifications in apparatus, for the manner in which the stream of aqueous caustic soda solution is fed into the stream of oil at point 14 is made not critical, and may be accomplished, for example, by the use of the well known T pipe connection.

In the practice of the invention, the proportioned rate of flow of fatty oil and caustic is such that the time period over which the caustic and oil are in contact in line 21 does not exceed approximately 5 seconds, but is not less than ½ second, and preferably not less than one second. This time factor may be readily calculated from the volume rates of flow of oil and caustic soda solution, the length of line 21, and its cross-sectional area or areas, if line 21, for any reason, is not employed with uniform cross-sectional area. Ordinarily the time of residence of the combined stream of oil and caustic in centrifuge 22 before being subjected to centrifugal force for separation purposes may be neglected, but should there be a centrifugal construction such as to make such time of residence appreciable, such as ½ second or more, then such time of residence should enter into the calculations for time of contact.

It is found in the practice of the invention that the oil retained in the soap stock, separated in centrifuge 22, is so low as to seriously reduce the fluidity of the soap stock, making it tend to pack in the bowl of the centrifuge. This difficulty, when present, may be overcome by any suitable means, such as, by the continuous feed of water into the centrifuge bowl for purposes of rendering the soap stock sufficiently fluid to facilitate discharge. Any suitable means may be employed for this purpose, such as that illustrated, for example, in Figure 2, which in part shows structure similar to that shown in Figure 2 of U. S. Patent 2,138,468.

Referring now to Figure 2, the centrifugal

rotor 30 is provided with an inlet feed boss 31 which is surrounded by a guide bushing assembly 32. The guide bushing assembly 32 is secured in position on a base 33 which also carries a feed nozzle 34 through which the combined stream of oil and caustic is fed into the centrifugal rotor 30. An auxiliary feed nozzle 35 is also secured to the base 33, and surrounds the feed nozzle 34. The feed nozzle 34 is provided, at a point above the upper end of the nozzle 35, with a bead 36, which is adapted to be impinged by liquid fed through the nozzle 35, and to deflect that liquid against the inner wall of the boss 31.

The lower end 37 of the rotor 30 is provided with a cylindrical flange 38 extending longitudinally interiorly of the rotor. The upper end of the flange 38 forms a seat for a flange 40 of a feed liquid directing member 39 having a conical base portion. Member 39 may be secured in position by any suitable means, not shown. The flange 38 is cut away at spaced portions, as illustrated at 41, in order to afford egress from the space between the member 39 and the bottom portion of the bowl for liquid passing upwardly through that space.

The feed-liquid-directing member 39, as shown, is provided on the upper side of its conical surface with a plurality of radially extending blades or fins 48, and on the lower side of its conical surface with a plurality of radially extending blades or fins 50. The fins 48 extend radially inwardly toward the zone of the feed opening 49 in the lowermost part of the member 39 and are of relatively narrow width. The spaced fins 50 extend radially from the outer surface of the member 39 to the inner surface of the lower end 37 of the rotor, and thus serve rapidly to bring liquid entering this space up to the rotational speed of the rotor.

In the operation of the construction particularly described, the stream of soap-stock-containing oil is fed through the nozzle 34 into the centrifugal rotor 30 through feed opening 49, and is brought up to speed within the assistance of blades or fins 48. By virtue of centrifugal force, soap stock, being the heavier phase, is deposited on the inner wall of rotor 30.

To assist in the discharge of soap stock from the rotor 30 a suitable liquid, such as water, is passed through the annular space afforded between the nozzle 35 and the nozzle 34. This washing liquid impinges against the bead 36 formed upon the nozzle 34 and is deflected into contact with the inner wall of the boss 31 of the rotor 30. The centrifugal force generated by the rotation of the rotor 30 causes the washing liquid, e. g. water, to cling to this surface, and to be projected upwardly into the space occupied by the radially extending blades 50. These blades bring the washing liquid up to the speed of the rotor, and this liquid is impelled outwardly through the openings 41 between the flange 40 and the flange 38 under the influence of centrifugal force. Washing liquid impelled through the openings 41 flows upwardly along the interior wall of the rotor 30, thus coming into contact with the soap stock as separated, and is discharged with the soap stock from the rotor.

It will be understood that any other suitable means may be employed to effect, or assist in, the discharge of soap stock from the centrifugal rotor.

The following examples are by way of illustration, and not of limitation, since the procedure

may be applied to any of the oils to which this invention relates.

Example 1

61,480 pounds of soya bean oil after being degummed were fed at the rate of 6500 pounds per hour, and at a temperature between approximately 158° and 160° F., through a pipe having an inner diameter of 1¼" to a centrifuge, being mixed with an aqueous caustic soda solution of 21° Bé., flowing at the rate of 25 pounds per hour, at a point in said pipe such that the length of flow of the combined stream of oil and caustic to the centrifuge was approximately 18". The time of contact between oil and caustic was approximately 3 seconds before the mixed stream entered the centrifuge. The oil recovered had a free fatty acid content of less than .04%. Its residual soap content was no more than an ordinary amount, and was readily removable by conventional water washing. The final soap-free moisture-free oil weighed 61,234 pounds, showing a net loss of only 0.40%. The original oil had a free fatty acid content of 0.35%.

Example 2

18,550 pounds of corn oil after being degummed were fed at the rate of 5000 pounds per hour, and at a temperature between approximately 158° and 160° F., through a pipe having an inner diameter of 1¼" to a centrifuge, being mixed with an aqueous caustic soda solution of 24.2° Bé. flowing at the rate of 40 pounds per hour. The respective flow rates of oil and caustic, and the length of the combined flow, were such that the time of contact between oil and caustic was approximately 4 seconds before the mixed stream entered the centrifuge. The oil recovered had a free fatty acid content of less than .045%. Its residual soap content was no more than an ordinary amount, and was readily removable by conventional water washing. The final soap-free moisture-free oil weighed 18,307 pounds, showing a net loss of only 1.2%. The original oil had a free fatty acid content of 1.15%.

Example 3

Cottonseed oil was fed at the rate of 4000 pounds per hour, and at a temperature of approximately 158° to 160° F. through a pipe having an inner diameter of 1" to a centrifuge while being mixed with a stream of aqueous caustic soda solution of 29° Bé. at the rate of 32 pounds per hour of solution. The rates of flow and the length of the combined flow were such that the time of contact prior to centrifuging was approximately two seconds. The soap stock obtained analyzed on a dry basis at 39.7% neutral oil.

A comparative run on the same crude oil was made under conventional commercial continuous refining conditions with a contact time of 1.5 minutes, and the soap stock obtained analyzed on a dry basis at 54% neutral oil.

Example 4

Peanut oil was fed at the rate of 4000 pounds per hour, and at a temperature of approximately 158° to 160° F. through a pipe having an inner diameter of 1" to a centrifuge while being mixed with a stream of aqueous caustic soda solution of 24° Bé. at the rate of 18 pounds per hour of solution. The rates of flow and the length of the combined flow were such that the time of contact prior to centrifuging was approximately two seconds. The soap stock obtained analyzed on a dry basis at 18% neutral oil.

A comparative run on the same crude oil was made under conventional commercial continuous refining conditions with a contact time of 1.5 minutes, and the soap stock obtained analyzed on a dry basis at 29% neutral oil.

From the foregoing it will be seen that I have provided a novel and highly useful process for the refining of fatty oils generally, and particularly of vegetable and animal oils.

Having particularly described my invention, it is to be understood that this is by way of illustration, and that modifications may be made without departing from the spirit of the invention. Accordingly, it is intended that the patent shall cover, by suitable expression in the claims, whatever features of patentable novelty reside in the invention.

I claim:

1. A process for the refining of a fatty oil containing free fatty acid which comprises combining a stream of said oil and a stream of aqueous caustic soda solution of at least 20° Bé. in measured quantity such that the proportion of caustic soda added to said oil is substantially stoichiometrically equivalent to the free fatty acid originally present in said oil, maintaining said combined streams of oil and caustic in mixed state under temperature conditions between approximately 140° F. and 160° F. and also under conditions such that the contact time does not substantially exceed five seconds to form soap stock in non-agglomerated condition, and then immediately separating said oil from the soap stock formed as a result of said treatment.

2. A process for the refining of a fatty oil containing free fatty acid which comprises flowing proportioned quantities of said oil and of an aqueous caustic soda solution of at least 20° Bé. into confluence and then concurrently under conditions of turbulent flow and of temperature between approximately 140° F. and 160° F., and after a contact time not substantially exceeding five seconds and while said soap stock is still in non-agglomerated condition immediately separating said oil from soap stock and spent caustic resulting from said treatment, said proportioned quantities of oil and aqueous caustic soda solution being such as to provide a substantially stoichiometric proportion of caustic soda to the free fatty acid originally present in said oil.

3. The process of claim 2 in which the oil is a vegetable oil.

4. The process of claim 2 in which the oil is cottonseed oil.

5. The process of claim 2 in which the oil is corn oil.

6. The process of claim 2 in which the oil is peanut oil.

7. The process of claim 2 in which the oil is soya bean oil.

8. The process of claim 2 in which the aqueous caustic soda solution is of between 22° and 30° Bé.

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