Nov. 3, 1942. B. CLAYTON 2,301,110

PROCESS OF REFINING OIL

Filed Feb. 27, 1942

Fig. 1.

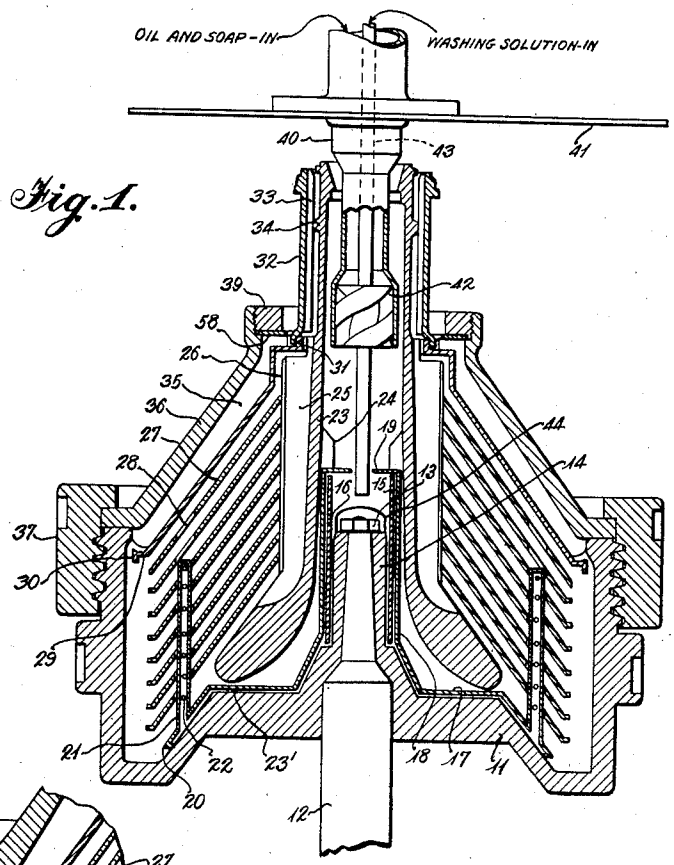


Fig. 2.

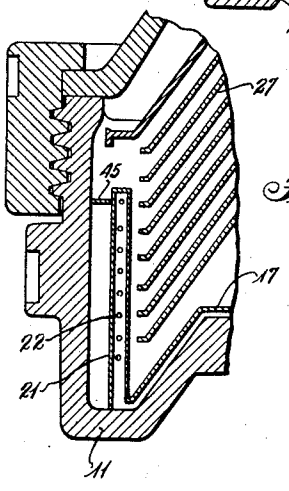
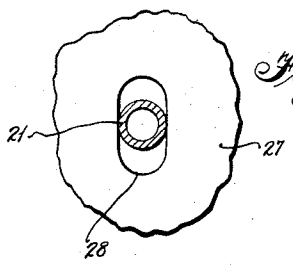


Fig. 3.



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2,301,110

PROCESS OF REFINING OIL

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8 Claims. (Cl. 260—428)

This invention relates to a process of refining animal and vegetable oils, and more particularly to a process of removing residual impurities from such oil after it has been treated with alkali and the resultant soapstock separated from the oil.

In processes of refining animal and vegetable oils by alkali treatment, the oil, after the resultant soapstock has been separated therefrom either by a settling or a centrifugal separation step, contains small amounts of residual alkali, soap and water. Even in oil separated from soapstock by means of a very efficient centrifugal separation such as that effected by means of a centrifuge having a large diameter bowl and inclined discs between which the light effluent is adapted to flow to render the separation more effective, it has been found that the oil thus separated may contain soap in the proportion of from 300 to 1500 parts per million. It is the practice now to wash this oil in separate equipment by the use of 10 to 20% of water and if the soap particles left in the oil after separating the soapstock are very difficult to remove, even a second washing operation may be employed. Present commercial practice favors a refined oil in which the soapstock has been reduced to from 10–12 parts per million. Following the washing step the oil is ordinarily subjected to a vacuum drying step to remove the last traces of water.

It has been found that relatively small quantities of water are ample to remove large quantities of soap if the water can be brought into sufficiently intimate contact with the oil in order for the water to reach a soap concentration which is in equilibrium with the concentration of soap remaining in the oil. It will be seen that if such a small quantity of water as 1% is added to an oil containing 1,000 parts per million of soap, there would be approximately ten times as much water as the soap in the oil. If all the soap were extracted in a single step the resulting soap solution would only have a soap concentration of about 10%. Therefore, by effecting intimate contact between the oil and the water it is possible to remove substantial quantites of the soap by means of a relatively small quantity of water.

The present invention contemplates the introduction of a small amount of washing solution into the centrifugal bowl which is used for separating the oil from the soapstock or into the centrifuge which is being used for the first washing operation. In either event the water is brought into contact with the oil just after the oil has been separated from either the soapstock or the wash water as the case may be. That is, the washing solution is introduced inside the neutral zone. The washing solution thus extracts a considerable quantity of soap from the separated oil and then flows outwardly in the centrifugal bowl to join the heavy effluent composed of either soapstock or wash water. In this manner the centrifugal separation and one washing step can be accomplished in a single centrifuge, or two washing steps may be conducted simultaneously within a single centrifuge.

Also with many oils an undesired amount of coloring matter persists in the oil after neutralization and separation and this coloring matter is extremely difficult to remove in subsequent operations. The color of the oil can be reduced if a large excess of caustic alkali is employed during naturalization but such operation results in increased losses due to saponification of neutral oil. Also neutralization with non-saponifying neutralizing agents such as alkali salts of alkali metals or alkali earth metals, ammonia or various amines, frequently does not remove the color to the desired extent. I have found that the introduction of a solution of a color reducing agent instead of water alone into the centrifugal separator as the washing liquid will markedly reduce the color of the oil without substantially increasing refining losses in the process. The soap or other solid impurities are removed from the oil to substantially the same extent as when water alone is employed and the amount of free alkali in the separated oil is not substantially increased even when a relatively concentrated solution of caustic soda is employed as the color reducing agent.

The color reducing step of the present invention is particularly useful in combination with the soapstock separation step in refining operations in which non-saponifying neutralizing agents such as soda ash are employed as a high quality oil of desired color can be produced with low refining losses. However, the color reducing step of the present invention may be employed as a second washing operation or as part of the separation of various impurities from oil. If desired, any separation in which the color reducing agent is employed as a washing solution may be followed by a washing operation with water alone. Since the washing action of the color reducing agent removes substantially all of the solid materials suspended in the oil and reduces the color of the oil, a final washing operation need only remove a small amount of residual color reducing agent which is usually a water soluble material and easily removed from the oil with a

relatively small amount of water. Thus an advantageous operation in accordance with the present invention is to neutralize the crude oil with a non-saponifying neutralizing agent such as soda ash, then centrifugally separate the resulting soapstock from the oil using a color reducing agent as a washing solution in the separator to reduce the color of the oil and remove solid impurities, and then subject the resultant oil to a water washing step utilizing another centrifugal, in which step a small amount of water may be mixed with the oil before entering the centrifugal or the washing may be performed within the centrifugal or both ways of washing can be used.

It is the principal object of the present invention to provide an improved method of washing refined oil in which small quantities of a washing liquid may be used to effect a substantial reduction in the impurities of the oil.

It is another object of the present invention to provide a method of introducing a small quantity of a color reducing agent as a washing liquid into the oil in a centrifuge just after the oil has been separated from impurities.

It is a further object of the invention to provide a method of introducing a color reducing agent into the oil in a centrifuge just after the oil has been separated from a washing liquid.

Other objects and advantages of the invention will appear in the following description of the process and the apparatus shown in the drawing, in which

Fig. 1 represents a vertical section through a centrifugal bowl which may be used to carry out the present invention.

Fig. 2 represents a fragmentary vertical section of a modified centrifugal bowl; and

Fig. 3 represents a fragmentary plan section of the washing liquid inlet pipe.

The centrifuge in Fig. 1 is composed of a bowl 11 mounted on a shaft 12 and secured thereto by means of nut 13. The bottom of the bowl has an upwardly extending cylindrical portion 14 through which the shaft 12 passes. A sleeve 15 having perforations 16 therein is disposed around the cylindrical portion 14. The bottom of this sleeve rests upon the bottom of the bowl and the top of the sleeve is adapted to support a dished member 17 having an upstanding cylindrical portion 18 surrounding the sleeve 15. An inwardly extending flange 19 fastened to the top of the cylindrical portion 18 bears upon the top of the cylinder 15 and thus supports the dished member 17. The dished member follows the general contour of the bottom of the bowl and is supported in spaced relation thereto by means of inwardly extending flange 20. Thus, there is a passageway extending from the inside of the cylindrical member 15, through the perforations 16, between the cylindrical members 15 and 18 and between the dished member 17 in the bottom of the bowl. Spaced near the periphery of the dished member 17 are pipes 21 which extend upwardly from the dished member and are fastened thereto to provide a flow communication with the space between the bottom of the bowl and the dished member. This pipe 21 is provided with suitable perforations 22 through which the washing liquid may be discharged into the main portion of the bowl. The pipe 21 is preferably closed at the end so that the washing liquid will have to pass through holes 22 rather than directly out the end of the pipe. These pipes are preferably disposed inside the neutral zone of the bowl, that is, inside the interface between the heavy and light effluents.

Surrounding the cylindrical portion 18 of the dished member is a tubular member 23 which is flared out near the bottom of the bowl to provide a passageway between the dished member and the tubular member. The bottom of the tubular member 23 is disposed above the dished member to provide a passage 23' beneath the tubular member. This is accomplished by supporting the tubular member by means of ribs 24 which rest upon the flange 19 of the dished member. The tubular member is provided with a plurality of outwardly extending fins 25 which in turn are provided with shoulders 26. A plurality of discs 27 are adapted to be slid over the tubular member and are provided with slots adapted to engage the shoulders 26 of the ribs 25, so that the discs will rotate with the tubular member. These discs may be of the conventional type employed in this type of centrifuge, in which case they would be provided wtih perforations 28. By suitably constructing the dished member 17 and the water pipe 21 the perforations in the disc may pass over and surround the water pipes. In this case it is preferred to have the perforations 28 of sufficient size so that after the discs are disposed over the water pipe there will still be sufficient room between the outside of the pipe and the adjacent disc to allow the passage of heavy effluent along the outside of the water pipe. In this manner the advantages obtained by means of the perforated discs are not lost by the use of these perforations to provide a space for the water pipe. Where the perforations in the disc are not necessary for efficient separation it is apparent that the water pipe may be of sufficient size to occupy the entire space made by the perforations. The discs 27 are provided with the usual means of spacing so that the discs will be supported in spaced relation with each other. Above the last discs is disposed dam member 29 which has a downwardly extending flange 30 which is disposed to permit only heavy effluent to pass beyond it. At its upper end the dam member 29 has an upwardly extending flange 31 to which is attached the light effluent discharge tube 32. This tube has inwardly extending ribs 33 which engage the annular rib 34 of the tubular member 23 to hold the tube 32 in spaced relation with the tubular member 23, thus providing a passage for the light effluent. The discs and the dam member 29 are held together by means of the spider member 35 which in turn is held in position by the closure member 36. The lock ring 37 serves to hold the entire assembly together. A ring dam 38 supported by a shoulder on the inner surface of the closure 36 is held in position by the nut 39. This ring dam serves to determine the neutral zone in the centrifuge and may be replaced by another of different size when it is desired to shift the neutral zone.

The feed mechanism is composed of a central feed tube 40 supported by cover 41. The feed tube is provided with a spiral 42 which serves to feed the mixture to be centrifuged against the inside wall of the tubular member 23 and from there it may pass through the space between the tubular member 23 and the tube 18 and out through passage 23'. The washing liquid inlet 43 is disposed within the feed tube and passes through the center of the feed spiral. It extends down a considerable distance into the centrifuge and passes through a hole in the

flange 19. The cap 44 may be provided to cover the nut 13 on the end of the shaft 12.

In Fig. 2 I have illustrated a modification of the centrifuge in which the pipe for washing liquid is disposed beyond the discs and inside the neutral zone. In this modification a support 45 between the inside of the bowl and the top of the washing liquid pipe may be provided since in this case the discs do not surround the pipe and accordingly the pipe cannot be supported by them.

Fig. 3 shows a modification of the present invention in which the perforations in the discs are of an elongated shape. The short dimension of the elongated perforation is disposed along a radius. The pipe for washing liquid is brought in contact with the sides of the elongated perforation and in this manner the discs serve to support the pipe. The space provided by the elongated perforation permits the passage of the heavy effluent alongside of the pipe.

In operation the oil containing the soapstock to be separated therefrom is conducted into the feed pipe 40, is forced to the periphery of the feed pipe by means of the spiral 42 at the end of which it is discharged against the inner wall of the tubular member 23 and flows downwardly through the space between this tubular member and the tube 18 and is distributed toward the periphery of the centrifuge bowl by means of the outwardly flared portion of the tube 23. The soapstock is thrown against the inside of the bowl while the oil is forced inwardly to pass between the discs 27. The washing liquid is introduced through the pipe 43, passes through the perforations 16, the space between the tubes 15 and 18 and into the pipe 21 from which it is distributed through the holes 22. This liquid flows countercurrent to the inward flow of the oil and effects intimate contact with the oil, thus removing a substantial portion of the soapstock and other impurities from the oil. Some of the washing liquid containing the soapstock and the impurities may discharge through the perforations 26 in the discs and thus take a short path to the heavy effluent discharge. Other portions of the washing liquid pass outwardly between the discs and join the main portion of the soapstock which is withdrawn at the heavy effluent discharge. The oil after passing the stream of washing liquid flows inwardly along the surface of the discs and is discharged into the space between the ribs 25 and from there it flows upwardly through the space between the tubes 32 and 23 and is discharged through the light effluent discharge.

The heavy effluent along the inner wall of the bowl passes around the flange 30 of dam member 29, up through the space between members 29 and 36, over the ring dam 33 and out the heavy effluent discharge.

It will be apparent that by means of the present process a single centrifuge may be used to effect the primary separation of impurities from the oil as well as to effect a substantial washing of the separated oil.

The washing accomplished by this small amount of washing liquid either makes a separate washing unnecessary or, in the case of oil from the primary separation containing an excessive amount of light floating soap, only one separate operation may be required.

The present process may also be employed to effect a double washing within a single centrifuge. Thus, a refined oil from which the majority of the soapstock has been separated but which contains from 300 to 1500 parts per million of soap can be admixed with a considerable quantity of water or of color reducing agent, which mixture can be separated in the present centrifuge. This washing will remove up to 95% of the residual soapstock and a large amount of color if a color reducing agent is employed and by the use of the additional washing liquid discharged into the separated washed oil it is possible to reduce the content of the residual soapstock and color reducing agent, if employed to a very small amount.

The quantity of washing liquid employed may vary with the different oils as well as with different batches of the same type of oil. In some cases washing liquid in the amount of 1% of the oil treated has been found effective. In others 2 or 3% may be more effective. Likewise less than 1% may be found satisfactory in particular instances.

In the treatment of certain oils it may be desirable to heat the material in the centrifuge. This may be done in the manner disclosed in my Patent No. 2,100,277. Likewise, heat may be added by the use of hot water, even above its atmospheric boiling point if pressure is employed.

The present process is not limited to the removal of residual soapstock from a refined oil but may also be employed to wash any impurities from the oil after it has been subjected to any kind of refining treatment. For example, where the oil is subjected to a degumming treatment the present process may be employed to remove traces of gums and other impurities which have precipitated but which have remained with the oil and have not been separated in the primary separation. Likewise, it can be employed to remove residual traces of impurities as well as acid from an oil which has been subjected to acid refining.

Similarly, the present process while preferably employed in conjunction with the continuous refining of oil, such as according to U. S. Patent No. 2,100,274, is not limited to continuous refining but may also be employed to remove residual impurities from oil refined according to the batch process.

This case is a continuation in part of my copending application Serial No. 369,510 filed December 10, 1940.

The various modifications of the process of the present invention are applicable to vegetable and animal oils in general. Such vegetable oils as cottonseed oil, corn oil, soy bean oil, linseed oil, sesame oil, etc., are within the contemplation of the invention. The term "animal oils" is also intended to include fish oils, such as sardine, menhaden, or herring oils. By the employment of the term "oils" or "oil" in the claims, I intend to embrace glyceride fats or oils, irrespective of the viscosity thereof.

While the description of the present invention has been particularly directed to the use of the type of centrifuge illustrated, it will be apparent that other types of centrifuge may be modified to perform the present invention.

It is apparent that other washing liquids besides water and color reducing agents may be used if desired. For example, various electrolytes may be employed particularly where the process is employed for the removal of gums and phosphatides. Likewise, in certain instances, soap solvents such as alcohols which are immiscible with the oil may be employed, either

alone or in admixture with other liquids such as water.

When a color reducing agent is employed, the preferred agent is a solution of caustic alkali because of its effective color reducing action and its cheapness and availability. Such solutions of caustic soda will usually range in concentration between approximately 8° Bé. and 20° Bé. depending upon the nature of the oil being treated and the amount of color reduction desired. The amount of solution employed will also vary with the nature of the oil and the amount of color reduction necessary but will usually range between approximately the limits above given for washing liquids in general. However other color reducing agents such as caustic potash or alkali metal peroxides or caustic alcohol solutions may be employed.

While various modifications have been disclosed the invention is not to be limited thereto but it is understood that it may be varied within the scope of the appended claims.

I claim:

1. The process of separating impurities from glyceride oil which comprises subjecting said oil containing impurities to centrifugal separation in a centrifuge to remove a portion of the impurities therefrom, introducing a relatively small quantity of a color reducing agent as a washing liquid into the resultant separated oil while said oil is still within said centrifuge, and removing the washing liquid along with the previously separated impurities.

2. The process of separating soapstock from glyceride oil which comprises subjecting said oil containing soapstock to centrifugal separation in a centrifuge to remove a portion of the soapstock therefrom, introducing into the resultant separated oil while in said centrifuge a relatively small quantity of a color reducing agent as a washing liquid, and separating the washing liquid from the oil.

3. The process of separating soapstock from glyceride oil which comprises subjecting said oil containing soapstock to centrifugal separation in a centrifuge to remove a portion of the soapstock therefrom, introducing into the resultant separated oil while in said centrifuge a relatively small quantity of a color reducing agent, and separating the color reducing agent and coloring matter from the oil.

4. The process of separating impurities from glyceride oil which comprises subjecting said oil containing impurities to centrifugal separation to remove a portion of the impurities therefrom, admixing the resultant separated oil with a quantity of washing liquid and subjecting the mixture to centrifugal separation, introducing into the resultant washed oil while in the centrifuge used for said last mentioned separation and immediately following separation from the washing liquid a relatively small quantity of a color reducing agent as an additional washing liquid, and separating the washing liquid from the oil.

5. The process of separating soapstock from glyceride oil which comprises separating the major portion of the soapstock from the oil, admixing the separated oil with a quantity of washing liquid, subjecting the mixture to centrifugal separation in a centrifuge to remove a portion of the washing liquid and residual soapstock therefrom, introducing into the resultant separated oil while in said centrifuge a relatively small quantity of a color reducing agent as a washing liquid, and separating the washing liquid from the oil.

6. The method of refining glyceride oil, which comprises, treating the oil with an alkaline refining agent, subjecting the resulting mixture of oil and soapstock to centrifugal force in a centrifuge to separate a portion of said soapstock from said oil, introducing a color reducing agent as a washing liquid into said oil while the same is still passing through said centrifuge and after a portion of said impurities has been separated from said oil, and separating washing liquid from said oil in said centrifuge to remove additional impurities therefrom.

7. The method of refining glyceride oil, which comprises, treating the oil with a non-saponifying neutralizing agent, subjecting the resulting mixture of oil and soapstock to centrifugal force in a centrifuge to separate a portion of said soapstock from said oil, introducing a color reducing agent as a washing liquid into said oil while the same is still passing through said centrifuge and after a portion of said impurities has been separated from said oil, and separating washing liquid from said oil in said centrifuge to remove additional impurities therefrom.

8. The method of refining glyceride oil, which comprises, treating the oil with a non-saponifying neutralizing agent, subjecting the resulting mixture of oil and soapstock to centrifugal force in a centrifuge to separate a portion of said soapstock from said oil, introducing a color reducing agent as a washing liquid into said oil while the same is still passing through said centrifuge and after a portion of said impurities has been separated from said oil, separating washing liquid from said oil in said centrifuge to remove additional impurities therefrom, and thereafter washing residual color reducing agent from said oil.

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