

1

3,510,501

GLYCERIDE OILS

Paulus Johannes Seip, Zwijndrecht, Netherlands, assignor to Lever Brothers Company, New York, N.Y., a corporation of Maine

No Drawing. Filed Mar. 14, 1966, Ser. No. 541,437
Claims priority, application Luxembourg, Mar. 18, 1965, 48,219

Int. Cl. C11 3/06

U.S. Cl. 260—425

10 Claims

ABSTRACT OF THE DISCLOSURE

Alkali refining of glyceride oils is accomplished by separately heating the oil and an aqueous alkali solution, and by thereafter bringing the separately heated materials into contact with each other in a neutralization zone essentially free from obstruction at a temperature of from 105° C. to 170° C. The refined oil is subsequently separated from the soap-containing alkali solution formed by neutralization.

The invention relates to the alkali refining of glyceride oils containing free fatty acids and other impurities. The glyceride oils concerned include animal and vegetable glyceride oils which are normally solid, as well as those which are normally liquid.

Glyceride oils obtained by solvent extraction, pressing or other methods, from animal or vegetable raw material always contain a greater or smaller amount of free fatty acids. Before these oils can be used in the preparation of foodstuffs, like margarine, shortening, mayonnaise, and the like, the impurities must be removed. It has become standard practice to reduce or remove these impurities by a treatment wherein the oil is contacted with aqueous alkali at temperatures below the boiling point of water and generally at atmospheric pressure and the soap so formed, together with the other reaction products of impurities initially present, is removed in solution and subsequently split to recover fatty material therefrom. It is known that during neutralisation with an alkaline solution a combined effect of purifying, de-gumming and part-decolorising may occur because a number of undesirable substances present in the crude oil are absorbed in the soap. However, in this method still considerable losses of neutral oil are experienced. These losses are caused by undesirable side reactions occurring during neutralisation. Sometimes emulsions are formed of oil in water stabilised by the soap solution. These emulsions may be very stable, which has as a consequence that during the separation of the two phases oil may be entrained with the soap solution.

In the processes applied so far it has not been found possible to avoid all oil losses, because, for instance, by suppressing one of the causes of oil losses, other undesirable side reactions may be further or other undesirable process conditions may be necessary. Hence, when the soap solution is subsequently split by acid hydrolysis to recover fatty material a considerable amount of neutral oil may be found in the acid oil, leading to a reduction in the yield of neutral oil. In general acid oils containing 30 to 40 percent or more of neutral oil are still quite usual. It is possible to recover neutral oil of a fair quality from the soap solution, but this is rather expensive and therefore industrially unsatisfactory.

In the literature several methods have been proposed to reduce the losses inherent in alkali refining, e.g. the use of less alkaline compounds, such as soda ash and ammonia, and the use of centrifuges, giving very short contact times between the oil and the alkaline solution. Although these proposed methods give some improvement,

2

the oil losses are still considerable, especially when glyceride oils containing predominantly fatty acids with a chain length of 16 carbon atoms or more are refined.

In the course of extensive research carried out with the object of devising a satisfactory method of neutralising oil in which the oil losses are considerably reduced without the necessity of carrying out the process with undue complexity, it was found that the neutralisation process is almost completely controlled by what happens at the oil-lye interface. At this interface local differences in interfacial tension occur during the transfer of fatty acids from the oil to the aqueous phase. These differences in interfacial tension cause currents in the immediate vicinity of the interface, which may be so violent that the interface becomes unstable and oil droplets are struck off spontaneously, which are taken up by the aqueous phase; this means that spontaneous emulsification takes place to a greater or lesser extent. In investigating the phenomenon we have found that the emulsified oil droplets are extremely small so that they will not settle, even in a very strong gravity field. These oil droplets are lost, even if they are not saponified by the alkali.

It has been found that the currents in the immediate vicinity of the interface and the so-called spontaneous emulsification are dependent on the process conditions, as well as on the type of oil used and it is the principal object of the present invention to provide an improved refining process in which the usual refining losses are considerably reduced.

In British patent application No. 11861/64 corresponding to U.S. application Ser. No. 441,378, filed Mar. 19, 1965, now U.S. Pat. No. 3,423,442; a method of reducing refining losses is described in which the oil and alkaline solutions are contacted continuously in a bed of chemically inert packing material by causing each of said liquids to make random contacts with the other while flowing along tortuous paths through said bed and subsequently separating the oil and aqueous liquids by virtue of their different specific gravities.

In this way the velocity of the oil and alkaline streams is so reduced that emulsification is minimised and in consequence considerably reduced refining losses occur.

It has now been found that even when the oil and aqueous liquid are contacted in a space free from obstructions, such as would prevent straight line movement of either liquid, a considerable reduction in emulsification and in consequence a valuable decrease in refining losses can be effected by contacting the two liquids at a temperature which is not less than 105° C. but not high enough to lead to significant saponification of the oil, under a super-atmospheric pressure which is high enough to prevent boiling.

The present invention, therefore, provides a process for alkali refining a glyceride oil with an aqueous liquid by contacting said oil with aqueous alkali and removing in aqueous solution the soap formed, characterised in that the oil and aqueous liquid are contacted at a temperature not less than 105° C. and not high enough to lead to significant saponification of the oil, under a superatmospheric pressure which is sufficiently high to prevent boiling, in a space which is free from obstructions such as would prevent straight line movement of either oil or aqueous liquid.

The optimum temperature depends primarily on the nature of the crude oil treated, especially the chain length and proportion of the predominating free fatty acids and the strength of the alkaline solution used. In general with oils in which the predominating free fatty acids contain at least 16 carbon atoms, and oils in which the free fatty acid content is high, e.g. 8–10% or more, relatively high temperatures, e.g. 130° or 140° to 150° or 160° C., are most advantageous, although even at temperatures be-

3

tween 105° and 130° C., e.g. 110°–120° C., some advantage is achieved with such oils. On the other hand, with oils in which acids of lower chain length predominate in the free fatty acid, e.g. coconut, palmkernel and other oils of the lauric acid class, temperatures of 105° to 110° or 120° have given excellent results even when the initial free fatty acid content has been as high as 8%. The temperature, however, should not be higher than is necessary to obtain the desired reduction in refining losses and in general it has not been found desirable to work at temperatures higher than about 160° or 170° C.

The pressure should be so chosen with regard to the temperature that the formation of vapour bubbles is avoided completely. Pressures higher than are required to achieve this end are in general to be avoided especially when an inert gas is used in obtaining the desired total pressure. At unduly high pressures the consumption of such gas tends to be prohibitively costly.

The process of the invention may be carried out in a variety of ways. Thus, e.g. the oil may be neutralised in a stirred pressurised cylindrical kettle having a cone-shaped bottom, the alkali being sprayed onto the oil. Or, as in a more recently described method, the oil in the form of droplets may be caused to rise through a static body of aqueous alkali, in a vessel which, in accordance with the present invention, is suitably pressurised and heated.

Another advantageous method of carrying out the invention is by applying at suitable elevated temperatures and pressures the method of neutralisation described in British application No. 19008/63, corresponding to U.S. application Ser. No. 366,240, filed May 11, 1964, now U.S. Pat. No. 3,454,608; where the oil and the aqueous solution are caused to flow in the same or opposite directions through a succession of treating regions in each of which the oil flows smoothly, horizontally or upwards at a small angle, on the surface of the aqueous solution, and transfer between each such region and the next is effected in such a way that any layer of aqueous soap solution that has formed immediately below the oil layer is displaced to bring the oil into direct contact with aqueous alkaline solution containing less soap and more unreacted alkali than is present in such layer, substantial intermixture of the aqueous and oil phases being avoided throughout.

The process of the present invention is also applicable to the continuous alkali refining of glyceride oils with the aid of centrifuges. The process of the invention is also applicable to refining processes other than neutralisation which involve treatment with aqueous alkaline liquids and hence tend to lead to refining losses due to emulsification. Such processes include the desliming of these oils, which still contain some mucilage by treating them with suitable aqueous alkaline liquids, such as sodium silicate.

The effectiveness of the process of the invention is surprising, since at higher reaction temperatures the saponification reaction will be accelerated and one would expect the saponification losses, owing to saponification at the oil surface, to increase rapidly with the rise in temperature. It appears, therefore, that a direct saponification does not contribute appreciably to the refining losses. The greater part of the refining losses seems to be caused by emulsification and not by saponification. If saponification occurs, this will generally be in the form of an indirect saponification that is at the surface of the emulsified oil particles, which follows the spontaneous take-up of oil through the interface in the alkaline phase.

The process according to the invention is of particular importance for the application to types of oils that during neutralisation have a tendency to form emulsions, especially spontaneous emulsions.

The difference in behaviour during neutralisation of the oils is especially determined by the type of free fatty acid that is predominantly present in the oil.

It has been found that in general the refining losses decrease with decreasing chain length of the predominant-

4

ing free fatty acid that is present in the oil. Also the presence of unsaturated bond in the free fatty acids is of influence. Oils that contain free fatty acids with one or two unsaturated bonds do show less emulsion formation than oils containing predominantly saturated free fatty acids. At a higher chain length of the free fatty acid, the losses due to spontaneous emulsification become appreciably greater. In particular oils containing free fatty acids with a large chain length, which oils have a strong tendency towards the formation of emulsion during refining, may be efficiently neutralised when the neutralisation temperature is chosen well over 100° C. Contrary to all expectation the loss during neutralisation with this type of oil is considerably reduced, and leads to a high quality end product, when neutralising at high temperatures.

Generally the various oils can be grouped together as follows, according to their behaviour during neutralisation:

Oils like coconut oil, palmkernel oil and Babassunut oil, which contain predominantly free fatty acids with a chain length below 16 carbon atoms, oils like palm oil, hardened fats and oils, cocoa butter, Borneo tallow, Illipé butter, lard and tallow, which contain predominantly free fatty acids with a chain length of 16 carbon atoms or more containing a substantial amount of saturated fatty acids, and vegetable oils like cottonseed oil, soybean oil, groundnut oil, olive oil, sunflower oil, safflower oil, sesame oil, linseed oil, rapeseed oil and mustardseed oil, which contain predominantly fatty acids with a chain length of 16 carbon atoms and more, containing unsaturated bonds.

Especially oils of the second as well as the third kinds give rise to many difficulties during refining, as indicated above, owing to spontaneous emulsification. It is understood that many oils take an intermediate position, especially between the last-mentioned two kinds of oils.

The superatmospheric pressure may be due simply to the vapour pressure of the aqueous liquid or may be imparted by an inert gas like nitrogen so that the total pressure is well above the saturated vapour pressure of the water at the treating temperature. The total pressure, being the sum of the vapour pressure and the pressure of the inert gas is generally in the range of 1–10 atmospheres especially 3–5 atmospheres, depending on the processing temperature.

The use of more concentrated alkaline solutions, for instance higher than 0.3 N, is of importance, especially in view of the smaller amount of heat necessary for the warming up of the aqueous alkaline solution. It has been observed that when increasing the concentration of the alkali from for instance 0.1 N to 0.4 N, a temperature increase of about 20° C. above the temperatures that are conventionally used in refining with a 0.1 N alkaline solution is required to reduce the refining losses to the level of more dilute caustic soda. Therefore, when working at relatively high temperature, a low refining loss is obtained as compared with more dilute alkali, and a markedly higher heat economy is reached.

In the process as envisaged aqueous sodium carbonate may be used. This kind of alkali reagent, however, generally gives rise to a strong spontaneous emulsification. This stronger emulsification of carbonate in proportion to sodium hydroxide can be overcome by increasing the temperature during refining as compared with the temperature applied when sodium hydroxide is used. Generally solutions of sodium carbonate of 0.05 to 2.0 molar are suitable.

If the reaction is carried out using a temperature that is about 25° C. higher than that conventional for the use of sodium hydroxide, the detrimental effect of the sodium carbonate can be completely overcome and the same low refining losses are obtained as when using sodium hydroxide. Generally, aqueous solutions of caustic soda of normality 0.05–2.0 are suitable.

It is known that the refining of glyceride oils which contain a relatively high amount of free fatty acids, say of 1 to 5% and higher often meets with many difficulties. Such types of oils give rise to a strong spontaneous emulsification causing relatively high oil losses. The process of the invention is effective for this kind of oil in that when carrying out the refining operation at temperatures well over 100° C. the refining losses are strongly reduced and comparable with those obtained when refining oils of the same kind having a much lower free fatty acid content. In general the temperature during the refining of oils having a free fatty acid content well over 1% has to be chosen about 25° C. higher to obtain low refining losses as compared with those obtained for glyceride oils having a free fatty acid content below 1%.

The kind of free fatty acid present in the crude oil strongly depends upon the glyceride structure of the oil because these fatty acids are to a great extent degradation products of the triglycerides present in the oil. As indicated above the fatty acids in the oil can in general be distinguished in acids containing a long carbon chain and acids containing a relatively short carbon chain.

The relatively high refining losses in refining glyceride oils containing long chain free fatty acids can be effectively reduced to the level of say coconut oil by choosing a temperature in the refining process which is about 40° C. higher as compared with that normally used. The long chain free fatty acids give rise to higher refining losses when using higher concentrations of alkali than the short chain free fatty acids so that the use of stronger alkaline solutions involves a further increase in temperature to counteract the detrimental effects. In these cases it will be necessary to choose the maximum temperature allowable in the range of 160 to 170° C., at which last temperature the rate of the hydrolysis reaction becomes rather strong and competes with the reduced loss due to avoidance of the spontaneous emulsification. When tallow, containing 5% free fatty acids which are composed of about 50% C₁₆ to C₁₈ saturated fatty acids and 50% C₁₈ unsaturated fatty acids, is refined in a kettle while stirring at 90° C., the oil loss amounts to 1 kg. oil per kg. fatty acid removed. At a temperature of 140° C., however, under the same reaction conditions the oil loss amounts to 0.4 kg. oil per kg. fatty acid removed.

In neutralisation neutral sodium salts, especially sodium chloride may be applied for instance in an amount of 0.3 to 3% and especially 0.5 to 2%, dissolved in the alkaline solution, to promote the separation of oil phase and soap solution. However, it was observed that salt also promoted spontaneous emulsification and the thus emulsified oil could no more be separated by the salt, for the influence of the salt does not extend to spontaneously emulsified oil, which is lost. The detrimental effect of salt in this respect can be reduced by choosing a higher neutralisation temperature to reduce the spontaneous emulsification. In this way the advantage of salt in the separation of oil and aqueous phase is still maintained without having the disturbing effect of increased spontaneous emulsification. If for instance safflower oil containing 3% free fatty acids is treated with 0.1 N alkali solution at 60° C., the refining factor is 1.15. If under the same conditions safflower oil is treated with 0.1 N lye containing 2% salt at a temperature of above 100° C., the refining loss is at the same level.

The process of the invention is specially applicable to oils such as soybean oil, rapeseed oil, cottonseed oil, linseed oil and groundnut oil, which usually contain a relatively high proportion of mucilage. This mucilage is normally removed by a desliming operation, e.g. by treating the crude oil with hot water to hydrate the phosphatides and precipitate them. However, this treatment does not remove all phosphatides, and for instance in the case of soybean oil about 0.3% by weight of phosphatide is commonly still present. During neutralisation the greater part of the remaining mucilage is removed, but in general so

much mucilage remains in the oil that a post-desliming is necessary if very pure oils are desired.

It is an advantage of the present invention that a post-desliming step can be avoided if the mucilage-containing oil is mixed with an aqueous acid, preferably 1 to 5 N hydrochloric acid in amount ranging from 1 to 5% of the weight of the oil, and this mixture, without removal of the acid, is subjected to neutralisation by contacting the mixture with an alkaline agent under the conditions characterising the process of the present invention. When such an acid pretreatment is applied, it is particularly desirable to effect the subsequent neutralisation according to the present invention in a totally unobstructed space.

The invention will now be illustrated by the following examples.

EXAMPLE 1

To illustrate the reduction in refining losses during refining obtained by maintaining the refining temperatures well above 100° C., a tallow having a free fatty acid content of 4.9% was treated at a temperature of 90° C. and thereupon another batch of the same oil was treated at 130° C.

At a temperature of 90° C. crude tallow (free fatty acid content 4.9%) was deacidified in a stirred cylindrical kettle having a cone-shaped bottom, with an alkaline solution of 0.8 N in an excess of 10% over the theoretical amount required. Then the oil was post-treated with 15 volume percent of dilute lye solution of 0.2 N followed by 3 washing treatments with water in an amount of 10% based on the amount of oil.

The results were: ultimate free fatty acid content 0.1%, ultimate soap content 0.15%, and neutralising factor 1.8 which corresponds to a neutral oil loss of 0.8 kg. per kg. fatty acid removed.

A similar quantity of tallow was thereupon treated in the same apparatus at a temperature of 130° C. and a pressure of 4 atmosphere gauge (nitrogen together with water vapour).

After neutralisation an ultimate free fatty acid content of 0.07%, a neutralising factor of 1.4 corresponding to an oil loss of 0.4 kg. per kg. fatty acid removed, and a soap content of 0.1% was obtained, so that no washing treatment was required any more. From the decrease in the neutralising factor it may be seen that the neutral oil loss was decreased with 50%.

EXAMPLE 2

A batch of 16 kg. of crude safflower oil was neutralised in the same apparatus as used in Example 1 at a temperature of 90° C. with an 0.8 N alkaline solution. Thereupon a post-treatment with a 0.2 N lye solution followed by water 3 washings were required to remove the soap which was formed by neutralisation.

The neutralising factor was 1.6, so that 0.6 kg. neutral oil was lost for each kg. of fatty acid removed.

When in the same equipment another 16 kg. batch was treated with the same alkaline solution at a temperature of 125° C. and a pressure of 4 atmosphere gauge pressure, no washing treatment with water was necessary to remove the soap.

The ultimate free fatty acid content in this case was then 0.05%, the soap content 0.05% and the neutralising factor 1.2 corresponding to an oil loss of 0.2 kg. per kg. fatty acid removed, that is to say that the neutral oil loss was diminished from 0.6 kg. per kg. of fatty acids removed, to 0.2 kg.

EXAMPLE 3

When comparing the neutralisation losses when using a soda ash and a lye solution respectively, it can be shown that at the same temperature and same conditions the treatment with lye leads to considerably lower refining losses than the soda ash treatment. By choosing a higher temperature in the soda ash treatment quite comparable results can be obtained. A tallow having a free

fatty acid content of 4.5% was neutralised in a tower provided with guide plates in zig-zag arrangement as described in our British application No. 19,008/63.

The apparatus consisted of 36 zig-zag mounted guide plates, having a length of 18 cm. and a width of 5 cm. placed in a slope of 5° in a housing having a length of 18 cm., a width of 5 cm. and a height of 100 cm. In each plate a pipe was mounted in a hole of 0.9 cm. diameter in the middle of the plate at a distance of 1 cm. from the end of the plate. The pipe has to carry the alkaline solution upwards through the oil layer onto the plate below. In this apparatus a batch of tallow having a free fatty acid content of 4.5% was neutralised at 110° C. with a 0.1 N alkaline solution, in an excess of 25% of the theoretical amount required, and washing treatments were not required at all. The results were: soap content 0.02%, ultimate free fatty acid content 0.3%, neutralizing factor 1.05. With a 0.1 molar soda ash solution a soap content of 0.1%, ultimate free fatty acid content of 0.6% and a neutralising factor of 1.3 were obtained under identical conditions.

However, when the soda ash treatment was carried out at a temperature of 140° C. an ultimate free fatty acid content of 0.4% and a neutralising factor of 1.05 were obtained.

EXAMPLE 4

The detrimental influence of a high caustic soda concentration on the neutralisation can be compensated for by increasing the temperature during neutralisation. This can be demonstrated by neutralisation of tallow at 95° C., having a free fatty acid content 4.3% with a 0.1 N caustic soda solution (25% excess of the theoretical amount required) in the same apparatus as described in Example 3, and by comparison of this result with a similar treatment but using a stronger caustic soda solution.

The neutralising factor was 1.10 and when neutralised under the same conditions with a 0.4 N caustic soda solution, a neutralising factor of 1.45 was obtained. A temperature of 140° C. was required in the neutralisation of tallow with 0.4 N caustic soda solution to obtain a neutralisation factor equal to that when using a 0.1 N caustic soda solution.

EXAMPLE 5

Three batches of tallow were neutralised using a 0.1 N caustic soda solution containing 1% sodium chloride in the apparatus according to Example 3, the first having a free fatty acid content of 1%, the last two having a free fatty acid content of 5.0%. Trials with the tallow with a low and a high free fatty acid content showed that the neutralisation factor in the latter case was considerably higher. When neutralising the tallow with a high free fatty acid content under the same conditions, except that the temperature during neutralisation was chosen 20° C. higher, a neutralising factor was obtained equal to that when neutralising a tallow with low free fatty acid content.

The results were as follows:

Temperature, degrees	Free fatty acid content percent	Neutralising factor
95.....	1.0	1.3
95.....	5.0	1.6
115.....	5.0	1.35

EXAMPLE 6

The influence of the presence of salt in the neutralisation of tallow (free fatty acid content 5%) could be demonstrated by 3 trials carried out in the apparatus as described in Example 3.

From the tabulated results it could be concluded that the presence of salt considerably increased the neutralisation loss when tallow was treated at a temperature of 95° C. When the treatment was carried out at a temperature of 130° C., the unfavourable effect of the salt addition was removed.

The results were as follows:

Temperature, degrees	Alkali	Neutralising factor
95.....	0.1 N.....	1.2
95.....	0.1 N with 1% salt.....	1.6
130.....	do.....	1.2

EXAMPLE 7

To obtain the same results for the neutralisation of safflower and tallow as were obtained under similar conditions in neutralising coconut oil, the temperature of the neutralising treatment had to be chosen substantially higher. The apparatus used was the same as described in Example 3.

The results of the trials were as follows:

Oil	Temperature, ° C.	Free fatty acid content in g. equiv./kg.	Alkali	Neutralising factor
Coconut....	95	0.17	0.1 N with 1% salt..	1.1
Tallow.....	95	0.17	do.....	1.6
Safflower....	95	0.17	do.....	1.25
Tallow.....	140	0.17	do.....	1.1
Safflower....	120	0.17	do.....	1.1

EXAMPLE 8

This example illustrates effecting neutralisation according to the invention in a totally unobstructed space. In a column of stainless steel with a height of 160 cm. and a diameter of 10 cm., and having a conical bottom, a tallow was neutralised. The column was closed to the atmosphere and the neutralisation effected under a pressure of about 5 atm., the atmosphere being composed of nitrogen saturated with water vapour. The oil and alkaline solution flowed counter-currently, the oil being introduced at the bottom and flowing upwards from numerous small apertures to form the dispersed phase and the alkaline solution flowing downwards. The soap solution formed was continuously withdrawn from the bottom below the oil inlet. The alkaline solution had a concentration of 0.3 N and was present in an excess of 25%. The temperature during the neutralisation was kept at 140° C. The oil throughput amounted to 4 ton/m.²/h. The initial free fatty acid content was 5.62%. The neutralised oil had a free fatty acid content of 0.15%, and a soap content of 0.08%. The neutralising factor was estimated at 1.16.

In a method which is particularly advantageous when the oil treated is of relatively high free fatty acid content part of the refined oil is continuously fed back into the crude oil stream entering the space in which the refining is effected, the proportion fed back being such as to reduce the free fatty acid content of the oil coming into contact with the aqueous solution to such an extent that the soap content of the refined oil is not more than 0.1% by weight.

What is claimed is:

1. A process for neutralizing the free fatty acids contained as impurities in glyceride oil comprising contacting said oil with aqueous alkali and removing in aqueous solution the soap formed, characterised in that the oil and aqueous liquid are contacted at a temperature not less than 105° C. and not high enough to lead to significant saponification of the oil, under a superatmospheric pressure which is sufficiently high to prevent boiling, in a space which is free from obstructions such as would prevent straight line movement of either oil or aqueous liquid.

2. Process according to claim 1, in which the oil and aqueous liquid are continuously contacted in a completely unobstructed space.

3. Process according to claim 2, in which the oil flows upwards and the aqueous liquid flows downwards through said space.

4. Process according to claim 1, in which the oil and the aqueous solution are caused to flow in the same or opposite directions through a succession of treating regions in each of which the oil flows smoothly, horizontally or upwards at a small angle, on the surface of the aqueous solution, and transfer between each such region and the next is effected in such a way that any layer of aqueous soap solution that has formed immediately below the oil layer is displaced to bring the oil into direct contact with aqueous alkaline solution containing less soap and more unreacted alkali than is present in such layer, substantial intermixture of the aqueous and oil phases being avoided throughout.

5. Process according to claim 4, in which the oil and aqueous streams flow in the same direction.

6. Process according to claim 1, in which part of the refined oil is continuously fed back into the crude oil stream entering the space in which the refining is effected, the proportion fed back being such as to reduce the free fatty acid content of the oil coming into contact with the aqueous solution to such an extent that the soap content of the refined oil is not more than 0.1% by weight.

7. Process according to claim 1, wherein the oil is neutralised with the aqueous alkaline liquid by bringing

the two into contact while stirring and removing the soap stock by decantation or centrifuging.

8. Process according to claim 1, in which the aqueous liquid is an aqueous solution of caustic soda of normality 0.05-2.0.

9. Process according to claim 1, in which the aqueous liquid is a 0.05-2 molar sodium carbonate solution.

10. Process according to claim 1, wherein the alkaline solution contains 0.3-3% of its weight of sodium chloride.

References Cited

UNITED STATES PATENTS

439,515	10/90	Hunt	260-425
722,832	3/03	Fresenius	260-425
1,968,252	7/34	Gensecke	260-425
2,183,486	12/39	Colbeth	260-425
2,678,936	5/54	Afzelius et al.	260-425
2,714,114	7/55	Scott	260-425
2,838,553	6/58	Ayres et al.	260-425

FOREIGN PATENTS

1,471,772	1/67	France.
952,754	3/64	Great Britain.
1,025,965	4/66	Great Britain.

BERNARD HELFIN, Primary Examiner

H. T. MARS, Assistant Examiner

U.S. Cl. X.R.

260-424