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PROCESS OF REFINING GLYCERIDE OILS

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This invention relates to a process of refining glyceride oils, and more particularly to a process of refining so-called "degummed" oils.

An object of the present invention is to provide a process of refining degummed glyceride oils in which the soap stock is separated from the refined oil without stratification.

Another object of the invention is to provide a process of refining degummed oils in which a material is added to the process which will prevent the stratification of the soap stock during separation so that the soap stock can be cleanly separated from the refined oil.

A further object of the invention is to provide a process of refining degummed oils in which continuous centrifugal separation of soap stock is substantially completely effected.

A still further object of the invention is to provide a continuous process of refining degummed oils in which a material which will prevent stratification of the soap stock is present during continuous centrifugal separation of the soap stock from the oil so that substantially all of the soap stock and nigre is discharged as the heavy effluent.

Glyceride oils are ordinarily refined either by a batch or continuous process in which an alkaline refining reagent of a strength of concentration determined by long experience in the art is added to the oil to react with the free fatty acids thereof and also precipitated gums contained therein, so that the resultant soap stock, including soap, gums, water and excess refining reagent, can be separated from the oil either by gravity separation or centrifugal separation. The gums from vegetable oils include valuable phosphatides and other minor constituents and can be recovered as a valuable byproduct prior to alkali refining, for example, by adding a small portion of a gum precipitating reagent such as water, boric acid solution, etc., and separating the precipitated gums from the oil. If the oils are refined with the gums contained therein, the gums are largely destroyed by the alkali and only small portions of valuable phosphatides and other minor constituents can be recovered from the soap stock. On the other hand, if the gums are removed prior to alkali refining, satisfactory batch refining cannot be accomplished and continuous refining becomes extremely difficult.

When a substantial part of the gums is removed prior to alkali refining, the soap stock tends to stratify into a soap layer and nigre layer. The soap layer is lighter than the liquid nigre layer, and in batch refining does not settle cleanly from the oil. Thus, in batch refining there is an upper layer of oil, an intermediate layer of soap stock admixed with oil, and a lower layer of nigre, which, for example, in refining cottonseed oil, is a red liquor consisting mostly of water, coloring matter and excess caustic. Large amounts of the oil are admixed with the soap and either the refined oil contains large amounts of soap or the refining losses are excessive.

In continuous refining of degummed oil, a similar stratification many times takes place in the centrifuge, and the nigre, along with a portion of the soap, is discharged as the heavy effluent, while a large portion of the soap discharges with the oil as the lighter effluent, such that the oil must be further processed to remove the soap.

The present invention is particularly adapted to the continuous process, but can, of course, be employed with some measure of success in batch refining. Continuous processes of refining, however, accomplish a considerable saving in refining losses over the batch process and in combination with the present invention are made applicable to processes in which the oil is first degummed, thus enabling even greater reduction in refining losses.

In such continuous refining processes, an alkaline refining reagent in a concentration and amount suitable for the particular oil being treated in accordance with the experience of the art is continuously admixed with a stream of the oil so that the refining reagent reacts with the free fatty acids to form soap, reduces the color of the oil, and precipitates any gums present to form a soap stock which is continuously centrifugally separated from the oil. The centrifugal separation is carried on at a temperature, usually between 120° and 160° F., which is suitable for the particular oil being treated. The oil may be heated, preferably during flow and in a closed heating device, before admixture with the refining agent, or the mixture of oil and resultant soap stock may be heated after mixing has been effected. Also, heating may be partly accomplished before admixing and partly after admixing. The time and extent of heating of the oil or mixture will depend also upon the nature of the oil being refined. Certain oils will more readily respond to preheating and other oils to heating after mixing. In any event, the emulsion which tends to form between the oil and soap stock must be broken before centrifugal separation can be satisfactorily accomplished. This emulsion forms readily at low temperatures but does not form or is broken at temperatures between approximately 120° F. and 160° F., depending upon the oil be-

ing refined. Also, the concentration of the refining reagent employed to some extent determines the temperature which must be employed to break the emulsion. In general, the more concentrated the refining solution, the lower the temperature at which the emulsion breaks, and in the case of certain oils with which extremely concentrated lye solutions can be employed without damaging the oil, centrifugal separation can be effected at temperatures lower than those indicated, even, in some cases, as low as room temperature, such that no heating is required.

As stated above, when degummed oils are attempted to be refined by such continuous processes, the soap stock tends to stratify in the centrifugal separator, and a clean separation is not effected. I have found that certain materials may be added in small amounts to the degummed oil or refining reagent, or even later in the process, so long as they are present during centrifugal separation so as to prevent the aforesaid stratification. These materials fall generally into two classes: (1) an emulsifying agent which will retain the soap emulsified with the water or nigre, and (2) a solvent for the soap which is miscible with water.

Apparently the excess refining reagent conventionally used in refining processes to reduce the color of the oil causes the soap to be salted out of the nigre in the absence of the gums which are present when undegummed oil is refined. It is, therefore, believed that the gums which separate with the soap stock, when undegummed oil is refined, act as an emulsifying agent between the soap and the nigre to retain the soap in admixture with the nigre and prevent it from being separated with the refined oil.

Thus, in accordance with the present invention, a small amount of gums separated from the same or different oil prior to alkali refining may be again admixed with the oil or again introduced into the process, preferably by admixing with the oil prior to adding the refining reagent thereto so as to act as an emulsifying agent to retain the soap dispersed in the nigre. Other gums, for example, Karaya or gum arabic, will also accomplish the same result. Other emulsifying agents can also be employed, for example, hydrolyzed gelatins such as peptones. I have also found that certain soaps which are extremely soluble in water, such as sodium resinate or sodium oleate, are effective to act as emulsifying agents between the soap and nigre to prevent stratification. Most potassium soaps of the usual fatty acids found in vegetable oils are also sufficiently water soluble to prevent stratification of the soap stock, if they are present in appreciable amounts during centrifugal separation. Such soap as sodium oleate or potassium soaps can be formed in situ during the refining process. For example, oleic acid may be added to the oil prior to refining and sufficient additional caustic soda employed during refining to produce the sodium oleate. In a similar manner, a portion of the caustic soda ordinarily employed as the refining reagent may be substituted by a potassium compound such as caustic potash or potassium carbonate so as to react with the free fatty acids present in the oil to form potassium soaps. Various other emulsifying agents may be employed for the same purpose, for example, powdered hydrophilic colloids such as colloidal clay, an example of which is bentonite, can be employed.

The various types of emulsifying agents referred to may be added to either the oil or refining reagent, depending upon which material they are most readily dispersed in, or may be added at any time prior to centrifugal separation. Preferably they are added prior to neutralization so as to be present in the mixture during the total time between neutralization and centrifugal separation, so as to retain the soap emulsified with the nigre.

The emulsifying agents referred to should be employed in substantially the minimum amount which will effectively prevent stratification, as such emulsifying agents will also tend to cause oil to be emulsified with the soap stock. Any oil emulsified with the soap stock will be separated with the soap stock and constitute a refining loss in the process. By employing the minimum amount which will just effectively prevent stratification, effective separation can be accomplished and refining losses markedly reduced over those occasioned in the alkali refining of crude or undegummed oil. Since the refining loss in a properly conducted degumming operation can be made extremely small, the over-all refining losses produced by first degumming and then alkali refining in accordance with the present invention are materially lower than for straight alkali refining of undegummed oil, even by a continuous process. This is true since substantially all undegummed oils contain more gums than necessary for the prevention of stratification, and therefore cause an unnecessary amount of refined oil to be emulsified and separated with the soap stock.

The amount of emulsifying reagent which must be employed in order to prevent stratification cannot be definitely given, as it will vary with the nature and quality of the degummed oils being refined. Different oils of even the same type vary widely in free fatty acid content, depending upon the origin of the seeds from which the oil was produced; the manner of removing the oil from the seeds, and the treatment of the oil between recovery from the seeds and refining. The amount of soap produced in refining will increase with the increase of free fatty acid content of the oil. In general, the greater the free fatty acid content of the oil, the greater the amount of emulsifying agent necessary to prevent stratification. Also, the greater the concentration and excess of the refining reagent found necessary for effective neutralization and color reduction, the greater the amount of emulsifying agent which must be employed to overcome the salting out action of the excess reagent. It is, therefore, desirable to employ the weakest lye and the smallest excess which is compatible with neutralization and color reduction when alkali refining degummed oil. This means that relatively high temperatures, i. e., in the neighborhood of 160° F., must usually be employed. Also, the amount of emulsifying agent will vary with the type of emulsifying reagent employed and the nature of the free fatty acids present. In general, the amount of emulsifying reagent is small, for example, less than 1% for low free fatty acid oils, but for extremely high free fatty acid oils producing soaps which are easily salted out of the oil, the amount of emulsifying agent necessary for effective separation may be considerably greater and may run as high as 5% to 10% for extreme oils.

As stated above, stratification may also be prevented by the presence of solvent for the soap which is miscible with water. By way of example, the lower aliphatic alcohols, for example, ethyl

or isopropyl alcohol, can be employed. Such soap solvents are, in general, more difficult to employ than emulsifying agents, as they usually are of less specific gravity than the soap stock and tend to reduce the average specific gravity thereof, such that effective centrifugal separation is difficult. The amount of solvent necessary to keep the soap dispersed in the nigre will also vary with the free fatty acids and the concentration and amount of refining reagent employed. In general, the amount of such solvents will fall within the general limits discussed with respect to emulsifying reagents.

The present process is particularly applicable to vegetable oils, for example, cottonseed, soya bean, corn, sesame, peanut and kindred oils, from which the gums have been removed, but is also applicable to the alkali refining of animal oils whenever trouble is experienced with soap stock stratification. Also, in certain cases crude or undegummed vegetable oils contain such a small amount of naturally occurring gums that soap stock stratification occurs. In such cases the present invention is also applicable.

While I have disclosed the preferred embodiments of my invention, it is understood that the details thereof may be varied with the scope of the following claims.

I claim as my invention:

1. In the process of alkali refining degummed oils in which the soap stock is separated from refined oil by difference in specific gravity, the improvement which comprises performing said separation in the presence of an added emulsifying agent in an amount which will prevent stratification of the soap stock into soap and nigre without substantially increasing the entrainment of oil in the soap stock.

2. In a continuous process of refining degummed oil in which an alkaline refining reagent is added to the oil to produce soap stock which is separated from the oil, the improvement which comprises continuously centrifugally separating said soap stock from the oil while said soap stock contains in admixture therewith an added emulsifying agent in an amount which will prevent stratification of said soap stock during centrifugal separation without substantially increasing the entrainment of oil in the soap stock.

3. A continuous process of refining degummed oil which comprises adding an amount of emulsifying agent to said oil which will prevent stratification of soap stock subsequently produced in said process without substantially increasing entrainment of oil in said soap stock, thereafter admixing an alkaline refining reagent with said oil so as to form soap stock in the presence of said emulsifying agent and thereafter continuously centrifugally separating said soap stock from said oil at a temperature which will prevent substantial emulsification of oil with said soap stock whereby said soap stock is substantially completely separated from said oil.

4. In the process of alkali refining glyceride oils deficient in gum content so as to produce a soap stock which stratifies into soap and nigre with the soap mixed with substantial quantities of oil when an alkaline reagent is mixed with the oil, the improvement, which comprises, separating said soap stock from said oil in admixture with an added emulsifying agent in an amount which will prevent substantial stratification of said soap stock without substantially increasing the entrainment of oil in the soap stock.

5. In the process of alkali refining glyceride oils deficient in gum content so as to produce a soap stock which stratifies into soap and nigre with the soap mixed with substantial quantities of oil when an alkaline reagent is mixed with the oil, the improvement, which comprises, separating said soap stock from said oil in admixture with sufficient added emulsifying agent to prevent substantial stratification of said soap stock but insufficient to cause excessive amounts of oil to be entrained in said soap stock.

6. In the process of alkali refining degummed oils in which the soap stock is separated from refined oil by difference in specific gravity, the improvement which comprises, performing said separation in the presence of an added emulsifying agent comprising gums recovered from crude glyceride oils in an amount which will prevent stratification of the soap stock during separation without substantially increasing the entrainment of oil in the soap stock.

7. In the process of alkali refining degummed oils in which the soap stock is separated from refined oil by difference in specific gravity, the improvement which comprises, performing said separation in the presence of an added emulsifying agent comprising sodium resinate in an amount wnich will prevent stratification of the soap stock during separation without substantially increasing the entrainment of oil in the soap stock.

8. In the process of alkali refining degummed oils in which the soap stock is separated from refined oil by difference in specific gravity, the improvement which comprises, performing said separation in the presence of an added emulsifying agent comprising colloidal clay in an amount which will prevent stratification of the soap stock during separation without substantially increasing the entrainment of oil in the soap stock.

9. In the process of alkali refining degummed oils in which the soap stock is separated from refined oil by difference in specific gravity, the improvement which comprises, performing said separation in the presence of an added emulsifying agent comprising bentonite in an amount which will prevent stratification of the soap stock during separation without substantially increasing the entrainment of oil in the soap stock.

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