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(54) **Continuous refining process with reduced waste streams.**

(57) The present invention relates to a process for refining glyceride oils which process results in refined oils and waste streams, the disposal of which latter does not entail high processing costs and investments. The process according to the present invention is a process for refining glyceride oil comprising addition of alkali, a separation step and a water washing treatment, in which said alkali is concentrated and at least part of the aqueous water washing effluent is recycled to control the alkali strength.

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Background of the invention

The present invention relates to a process for refining glyceride oils which process results in refined oils and waste streams, the disposal of which latter does not entail high processing costs and investments. The process according to the present invention is a process for refining glyceride oil comprising addition of alkali, a separation step and a water washing treatment, in which said alkali is concentrated and at least part of the aqueous water washing effluent is recycled to control the alkali strength.

The term 'glyceride oils' as used herein is intended to encompass both vegetable and animal oils. The term is primarily intended to describe the so-called edible oils, i.e. oils derived from fruits or seeds of plants and used chiefly in foodstuffs, but it is understood that oils of which the end use is non-edible are to be included as well.

Crude glyceride oils of in particular vegetable origin, such as soybean oil, rapeseed oil, sunflower oil, cottonseed oil and the like contain a significant amount of non-triglyceride compounds including phosphatides, free fatty acids, odours, colouring matter, waxes and metals. Many of these impurities adversely affect taste, smell, appearance and keepability of the oil. It is therefore necessary to refine, i.e. to remove the gums and other impurities from the crude glyceride oils as much as possible.

In general the first step in the refining of glyceride oil is the so-called degumming step, i.e. the removal of phosphatides (gums). In conventional degumming processes water is added to the crude glyceride oil to hydrate the phosphatides, which are subsequently removed by centrifugation. The resulting water-degummed oil often still contain unacceptably high levels of 'non-hydratable' phosphatides and in general residual phosphorus levels are achieved in the order of 100-250 ppm. This water-degummed oil is then normally treated with acid (GB 2,038,863), optionally followed by cooling and holding (DE 26 09 705) or a treatment with alkali (EP 0 195 991), to remove the residual phospholipids and associated trace-metals such as iron and copper. Subsequent refining includes either neutralisation of free fatty acids and separation of the soapstock thus formed (alkali refining), or physical refining, i.e. removal of free fatty acids by distillation. The resulting oil is then further refined by bleaching and deodorization.

Despite the fact that physical refining has a number of advantages over alkali neutralisation, such as the avoidance of soapstock formation and its potential lower refining loss, the vast majority of refiners are currently still using the conventional caustic soda refining method, followed by at least one washing stage, the main reason being the expensive conversion to physical refining.

Apart from the saponification of neutral oil through contact with the alkali solution and the entrainment of neutral oil upon separation of the soapstock and the subsequent washing stages, alkali refining further has the disadvantage over physical refining to yield vast effluent streams of high biological oxygen demand. As a matter of fact, disposal of wastewater from the conventional washing of alkali refined glyceride oil presents a problem to refiners because of increasingly stringent law. An efficient removal of remaining impurities by water washing is however essential for the oil quality and for subsequent bleaching, hydrogenation, winterizing, deodorization, etc. to be successful, and cannot simply be omitted.

The washing water is used more efficiently if the alkali refined oil is washed counter currently as suggested by G. Haraldsson in 'Degumming, Dewaxing and Refining', J.A.O.C.S., Vol. 60, Febr. 1983, Pages 205A. Counter current water washing results in a reduction of the wash water consumption by almost fifty percent but its impurity content and, as a result, its biological oxygen demand is increased in accordance.

A method to overcome the disposal of wastewater from the washing stages is suggested by R.E. Beal, L.T. Black, E.L. Griffin and J.C. Meng in "Water-Recycle Washing of Refined Soybean Oil: Plant Scale Evaluation", J.A.O.C.S., Vol. 50, July 1973, Pages 260 to 263. This method entails treatment of the wash water, upon decantation of the organic matter, over cation exchange resins to regenerate it for subsequent alkali neutralised oil washings. However, because of high investments and operational costs involved, this method is hardly used industrially if at all.

Objects of the invention

It is now the object of the present invention to provide an alternative and simplified method to overcome the disposal of at least part of the wastewater resulting from the washing stages subsequent to alkali treatment.

This and other objects and advantages of the present invention will be apparent as the description of the invention proceeds.

Detailed description of the invention

The present invention relates to a process for refining glyceride oil comprising addition of alkali, a separation step and a water washing treatment, in which said alkali is concentrated and at least part of the aqueous water washing effluent is recycled to control the alkali strength.

In alkali refining, the control of the amount and strength of lye is particularly important. Optimal levels are commonly established by laboratory tests and are also based upon experience gained with similar oils and equipment. In general, it can be said that a minimum amount of water is needed to hydrate the phosphatides and the soaps. A shortage of water would lead to incomplete removal of these impurities by lack of hydration. If, on the other hand, the amount of water is too large, centrifugal separation may run into problems in that a three-phase system (hydrolysed impurities, aqueous phase and oil) may result. Consequently, the amount of water calculated as a percentage on oil has to be carefully balanced.

With respect to the use of alkali, too little lye, calculated as dry matter, will lead to incomplete neutralisation and too much lye is also to be avoided because of the cost involved and the undue saponification of glyceride oils. On the other hand, a certain excess over stoichiometric is required because lye may be needed to decompose non-hydratable phosphatides and the free fatty acid content of the oil shear may fluctuate.

In addition, lye strength is also a factor to be taken into account because this determines its density and thereby affects the separation characteristics during the centrifugal treatment or gravity settlement. In practice, amount of water and amount of lye are chosen within specific ranges determined as indicated above to arrive at a lye strength that allows separation and if this strength happens to be on the low side, salt is added to facilitate separation.

Caustic soda is usually received in a concentrated form and diluted with water to the desired concentration prior to mixing it, in a determined amount, into the glyceride oil. The soapstock thus formed is removed from the neutralized oil by centrifugal separation or sedimentation and the neutral oil is, in a subsequent step, washed at least once to remove residual soaps and/or phospholipids. The diluted soapstock removed from the neutralized oil and the effluent water resulting from the water washing treatments are usually combined and treated with mineral acid to recover and concentrate the fatty matter on the one hand and to discard the aqueous effluent on the other.

We have now surprisingly found that at least part of the effluent water resulting from the subsequent water washing treatments can be recirculated into the oil stream upon or prior to the addition of alkali to control the alkali strength, without affecting the refining process.

Furthermore, we have found that at least part of the effluent water resulting from water washing treatments subsequent to glyceride oil degumming processes involving an alkali treatment, may be recirculated into the oil stream upon or prior to the addition of alkali to control the alkali strength as well.

For instance, in European patent EP 0 195 991 a degumming process is disclosed to which the present invention may successfully be applied. This process comprises dispersing a non-toxic aqueous acid into the oil, mixing a base into the acid-in-oil dispersion in such a quantity that the pH of the aqueous phase is increased to above 2.5 but no substantial amount of soap is produced, separating the dispersion into an aqueous phase containing the gums and an oil phase comprising free fatty acids and finally washing the degummed oil.

European patent application EP 0 348 004 discloses a refining process in which the degumming step is followed by a separation step in which undissolved particles are removed from the degummed glyceride oil upon holding the oil for a period of time and under temperature conditions as to cause agglomeration of said particles in the presence of an agent which promotes the agglomeration and which, according to the description, may be lye or caustic soda. Because this process necessitates subsequent washing stages to be operated, the present invention may also successfully be applied.

Yet another such process is disclosed in European patent application 0 473 985. This process aims at reducing non-hydratable phospholipid content and, at the same time, eliminating natural waxes, by addition to the oil, at a temperature ranging from 20 to 70 °C and under gentle mixing, of a diluted non-toxic acid followed by addition of a diluted alkali in an amount of 40 to 150 percent of the stoichiometrical requirement to neutralize the added acid, heating the oil prior to centrifugal separation and finally washing the degummed and dewaxed oil with water.

The effluent water resulting from the subsequent water washing treatments is preferably added into the oil stream prior to the alkali treatment to dilute the concentrated alkali immediately upon its addition into the oil stream. Extended contact of the oil with concentrated alkali would lead to undue saponification of the neutral oil and result in a rather considerable refining loss. If on the other hand the effluent water is added to the oil subsequent to the addition of the concentrated alkali, the temperature of the alkali-oil mixture is

preferably kept as low as possible, and most preferably below 40 °C. Upon addition of the effluent water and the alkali, the oil stream is preferably mixed intimately.

The concentration of the alkali added upon the alkali treatment is not that critical, although the higher is the alkali concentration and the more effluent water may be added to the oil stream to dilute the alkali, the higher the net savings will be. As a general rule, alkali concentrations ranging from 30 to 50 ° Baumé are suitable and advantageous.

A particular embodiment of the present invention is schematically represented in figure 1. It is an alkali neutralisation process which comprises an acid treatment (a) in which an acid is dispersed into the oil, an alkali treatment (b) in which a concentrated alkali is dispersed into the acidified glyceride oil to neutralize the dispersed acid as well as free fatty acids present in said acidified oil, a first centrifugal separation (c1) to yield an effluent phase comprising both phosphatides and soaps and having a low triglyceride oil content and an oil phase with reduced phosphorus and soap content, into which latter an amount of water is mixed (w) prior to being fed to a second centrifugal separator and a second centrifugal separation (c2) to yield an oil phase with minimum residual phosphorus and soap content and an aqueous heavy phase comprising a significant amount of triglyceride oil which is recycled (r) into the acidified oil at a stage prior or subsequent to the alkali treatment (b). This embodiment differs from our co-pending patent application EP 92200665.5 in that the aqueous heavy phase is recirculated into the oil stream right prior or subsequent to the alkali treatment, in that the alkali is concentrated and in that its strength is controlled in situ by the aqueous heavy phase.

An additional advantage of this embodiment is that better use is made of the alkali, compared to traditional neutralisation processes. As a matter of fact, the alkali addition upon alkali treatment (b) may be reduced shortly after start-up of the process line, because the wet heavy phase which is recycled (r) from the second centrifuge (c2) into the acidified oil has a pH of above 7, and, as a result, adds to the neutralisation of the dispersed acid and free fatty acids present in the acidified oil.

Another particularly useful embodiment of the present invention is a degumming process which comprises an acid treatment (a) in which an acid is dispersed into the oil, an alkali treatment (b) in which a concentrated alkali is dispersed into the acidified glyceride oil to raise the pH to above 2.5 without substantial formation of soaps, a first centrifugal separation (c1) to yield a gum phase with low triglyceride oil content and an oil phase with reduced phosphorus content into which oil phase an amount of water is mixed (w) prior to being fed to a second centrifugal separator and a second centrifugal separation (c2) to yield an oil phase with minimum residual phosphorus content and a wet gum phase which is recycled (r) into the acidified oil at the stage prior or subsequent to the alkali treatment (b). This embodiment differs from our co-pending patent application EP 92200543.4 in that the wet gum phase is recirculated into the oil stream right prior or subsequent to the alkali treatment, in that the alkali is concentrated and in that its strength is controlled in situ by the aqueous heavy phase.

The type of oil to be treated by the process according to the present embodiment of the invention is not critical. Thus crude or water-degummed edible triglyceride oils like soybean oil, sunflowerseed oil, rapeseed oil, palm oil and other vegetable oils as well as animal oils and fats can all be successfully processed.

The present invention is now illustrated by the following example wherein phosphorus and iron content of the oil are determined by plasma emission spectroscopy (A.J. Dijkstra and D. Meert, J.A.O.C.S. 59 (1982), 199), the soap content of the oil is determined by A.O.C.S. method Cc 17-79 and the free fatty acid content is determined by A.O.C.S. method Ca 5a-40.

Example

Partially water degummed rapeseed oil, having a phosphorus content of 179 ppm, and iron content of 2.1 ppm and a free fatty acid content of 0.83 %, was alkali neutralized in accordance with the present invention. Into rapeseed oil, at a throughput of 9 tons per hour and at a temperature ranging from 100 to 110 °C, were finely dispersed 15 liters of phosphoric acid 80% strength and 20 liters of water per hour, whereupon the phosphoric acid - glyceride oil mixture was neutralized with 256 liters of lye 20 ° Bé per hour. The oil stream was then fed to a first centrifugal separator (R.S.A., Westfalia Separators A.G., Ölde, Germany) operating at conditions as to yield an effluent phase comprising both phospholipids and soaps and having a low triglyceride oil content and an oil phase with reduced phosphorus and soap content. The oil phase resulting from the first centrifugal separator was then fed to a second centrifugal separator (C.S.A., Westfalia Separators A.G. Ölde, Germany) operating at conditions as to yield an oil stream with minimum residual phosphorus and soap content and a heavy phase comprising the residual phosphorus and soaps as well as a significant amount of triglyceride oil, which heavy phase was fully recycled into the oil stream right

after the acid treatment. The resulting neutralized oil was then washed once with about 300 liters water per hour and vacuum dried. The operation conditions for both centrifugal separators were established in accordance with EP 0 349 718.

Soon after start-up of the alkali neutralisation line, the 256 liters of lye 20 ° Bé were replaced by 199 liters of effluent water resulting from the water washing treatment and 57 liters of lye 50 ° Bé.

The quality of the resulting oil is illustrated in table 1.

Table 1

	P (ppm)	Fe (ppb)	ffa (%)	soap (ppm)
lye 20 ° Bé	2	17	0.023	12
Example 1	3	22	0.024	15

From the above example it is clear that the quality of the oil is not significantly reduced when recirculating part of the aqueous water washing effluent to lower and control the alkali strength and to reduce overall water consumption of the alkali refining process by more than fifty percent.

Claims

1. Process for refining glyceride oil comprising addition of alkali, a separation step and a water washing treatment, in which said alkali is concentrated and at least part of the aqueous water washing effluent is recycled to control the alkali strength.
2. Process according to claim 1, characterized in that the oil, after addition of alkali, is subjected to a first centrifugal separator to yield an effluent having low triglyceride oil content and an oil phase with reduced impurity content which is fed to a second centrifugal separator to yield a heavy phase which is recycled into the oil prior to the alkali treatment and a refined oil phase which is subjected to a subsequent washing treatment.
3. Process according to claim 1, characterized in that the oil, after addition of alkali, is subjected to a first centrifugal separator to yield an effluent having low triglyceride oil content and an oil phase with reduced impurity content which is fed, upon addition of washing water, to a second centrifugal separator to yield an aqueous heavy phase which is at least partially recycled into the oil prior to the alkali treatment and a refined oil phase.
4. Process according to any of claims 1 to 3, characterized in that the alkali treatment is preceded by an acid treatment.
5. Process according to claim 4, characterized in that alkali is added in an amount sufficient to raise the pH to above 2.5 without substantial formation of soaps.
6. Process according to any of claims 1 to 3, characterized in that alkali is added in an amount exceeding the stoichiometric requirement to neutralize the free fatty acids present in the oil.
7. Process according to claim 4, characterized in that alkali is added in an amount exceeding the stoichiometric requirement to neutralize the dispersed acid and free fatty acids present in the acidified oil.
8. Process according to claim 1, characterized in that the concentration of the alkali ranges from 30 to 50 ° Bé.
9. Process according to any of the above claims, characterized by an aqueous effluent reduction of at least 50%.
10. Refined oil as obtained according to any of the above claims.

Figure 1

