(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 269 277**
A2

(12) EUROPEAN PATENT APPLICATION

(21) Application number: 87309597.0

(51) Int. Cl.4: C11B 3/04 , C11B 3/10

(22) Date of filing: 29.10.87

(30) Priority: 13.11.86 US 929889

(43) Date of publication of application:
01.06.88 Bulletin 88/22

(84) Designated Contracting States:
CH DE ES FR GB IT LI NL

(71) Applicant: THE CAMBRIAN ENGINEERING GROUP LIMITED
2200 Argentia Road
Mississauga Ontario L5N 2K7(CA)

(72) Inventor: Kaji, Takashi
33303 Mainsail Crescent
Missisauga Ontario, L5L 1H3(CA)

(74) Representative: Seaborn, George Stephen
c/o Edward Evans & Co. Chancery House
53-64 Chancery Lane
London WC2A 1SD(GB)

(54) Process for degumming triglyceride oils.

(57) A process is provided for removing phospholipids or gums from triglyceride oils. The process comprises (a) dispersing in the oil an organic acid or acid anhydride, at a temperature not greater than about 40°C; (b) subsequently dispersing water in the oil, while maintaining the temperature at not greater than about 40°C; and then (c) separating a sludge containing the gums from the oil to produce an oil product substantially reduced in phosphorus-containing compounds. The oil product of this degumming process, when subjected to the further step of bleaching, consistently results in a refined oil product containing less than 3 ppm of phosphorus, usually less than about 0.5 ppm phosphorus.

EP 0 269 277 A2

PROCESS FOR DEGUMMING TRIGLYCERIDE OILS

The present invention relates to a process for degumming triglyceride oils.

Edible oils are triglycerides which have been extracted from animal, marine or vegetable matter. Oils of marine and vegetable origin contain,in addition to the triglycerides of fatty acids, extraneous materials, for instance free fatty acids, colour bodies, sterol-type compounds and phosphorus-containing compounds. The phosphorus-containing compounds are known in the art as phosphatides, phospholipids or gums. It is necessary in refining triglyceride oils to remove one or more of these components. In particular, it is desirable to reduce the amount of the phospholipids in the oil from an initial concentration of about 500 to 3000 ppm to less than about 3 ppm (based on phosphorus). Oils which contain greater than 1 or 2 ppm phosphorus have an unstable flavour and colour. Also, the presence of phosphorus compounds above this level can interfere with subsequent processing of the oils, for instance in the hydrogenation of the oils.

The phosphatides present in triglycerides are generally thought to be of two types, hydratable phosphatides and hydrolyzable phosphatides. The hydratable phosphatides are easily removed from the oil by treatment with water to hydrate the compounds, followed by removal, for example by centrifugal separation. Extractors of triglycerides will often carry out this initial degumming process to produce what is termed in the art, partially or industrially degummed or deslimed oil. This industrial degumming process is capable of reducing the phosphorus content of the oil to about 200 ppm. However, this phosphorus level is still too high for any practical application, and the industrially degummed oil must be further refined to remove the remaining phospholipids, which are present in a hydrolyzable form.

The conventional commercial process for degumming triglycerides comprises treating the oil with phosphoric or citric acid, at a temperature of about 60 to 90°C for about 20 minutes, to hydrolyze and hydrate the phosphatides, and subsequently neutralizing the oil with caustic soda to remove the free fatty acids, phospholipids and soaps. The neutralization step produces what is called a soapstock, which is a combination of free fatty acids with caustic soda. The soapstock and phospholipids are removed by centrifugal separation. Following this step, the oil is bleached with a bleaching clay to remove colour bodies, and then deodorized by steaming to remove odours, flavours and the remaining free fatty acids.

This conventional process has one major drawback, and that is the production of the soapstock. This soapstock is an undesirable waste product which must be further processed prior to disposal. The soapstock also carries with it undesirable quantities of entrained oil.

In spite of this major disadvantage, this process remains one of the most widely used commercial processes to refine triglycerides because it is capable of reducing the phosphorus content to less than about 3 ppm. Most of the attempts to improve on the process have failed to attain an acceptable level of phosphorus reduction.

The usual approach to overcoming the problems of the above-described commercial process is to avoid the neutralization step by practising a more intensive initial degumming step. To that end, the oil is treated with phosphoric or citric acid at a temperature of about 90 to 100°C for one hour or more. The gums are then removed by centrifugal separation. The oil is subsequently subjected to the bleaching and deodorizing steps.

This intensive degumming process has been unsatisfactory in that, with most triglyceride oils, it is not capable of reducing the phosphorus content to an acceptable level. After the intensive degumming process the oil has a phosphorus content of about 20 to 50 ppm. Following the subsequent bleaching and deodorization steps, the oil still typically contains about 15 to 20 ppm phosphorus. As previously stated, it is desirable to reduce the phosphorus content to less than 3 ppm and more preferably to less than 1 ppm.

In Canadian Patent No. 1,060,041, issued August 7, 1979 to Unilever Ltd., there is disclosed a process for degumming triglyceride oils. An acid or acid anhydride, preferably citric acid, is dispersed in the oil at a preferred temperature of 65 to 90°C, and thereafter water is dispersed in the oil and the oil, water, acid mixture is held at a temperature of less than 40°C for a short time. An aqueous sludge containing the gums is then removed from the oil. The oil may thereafter by subjected to neutralization, bleaching and/or deodorizing steps to further reduce the phosphorus, colour bodies and free fatty acids. The initial degumming step is exemplified only with an acid contacting step which is conducted at very high temperatures, typically at 70°C. The phosphorus removal, even after water washing, bleaching and deodorizing or after an undesirable neutralization step, was not consistently at an acceptable level of less than 3 ppm. Phosphorus reduction levels of less than 1 ppm are not achieved at any of the exemplified conditions.

In Canadian Patent No. 1,157,883, issued November 29, 1983 to United Oilseed Products Ltd., there is disclosed an improvement in a process similar to that of the Unilever patent. In the improved process, the

temperature of the process throughout the steps of acid contacting, water contacting and centrifugal separation, is greater than 40°C, and preferably is in the range of about 45-55°C. The process, as exemplified, reduces the phosphorus to a level which varies between about 6 and 100 ppm, but which is typically less than about 50 ppm.

According to the present invention, it has been discovered that a triglyceride oil can be efficiently and economically degummed by dispersing in the oil an organic acid or acid anhydride, at a temperature not greater than about 40°C, subsequently dispersing water in the oil, while maintaining this temperature, and then separating a sludge containing the gums from the oil. This process is effective in reducing the phosphorus content in the oil, at this stage in the refining to less than about 15 ppm.

The inventors have further discovered that, surprisingly, the oil product of the degumming process of the invention, when subjected to the further step of bleaching, consistently results in a refined oil product containing substantially less than 3 ppm phosphorus. The phosphorus reduction after bleaching is typically less than about 1 ppm and usually less than about 0.5 ppm.

While not being bound by the same, it is believed that the degumming process of this invention as a result of being conducted at a lower temperature than the prior art processes, results in a conditioning of the phosphorus-containing compounds in the oil, which makes them readily adsorbable on bleaching clay and therefore more completely removable in the subsequent bleaching operation. It is further believed that the phospholipids in the oil, once they are hydrolyzed and hydrated by this process are less oil-soluble at the lower temperature conditions of this process, and are therefore more completely removable than if the process were conducted at a higher temperature.

Thus, the process of this invention, when practiced in combination with a bleaching step, is capable of reducing the phosphorus content to at least the same level as that of the conventional alkali refined and bleached oil. However, the process of this invention achieves this result without the production of the undesirable soapstock and the loss of oil entrained in the soapstock. Furthermore, this result is achieved without the necessity of maintaining a high temperature during the acid contacting step. Furthermore, the oil product produced by the process of this invention leaves the free fatty acids in a relatively stable form such that they can be removed and recovered in a subsequent deodorization and fatty acid stripping step comprising steam distillation.

Accordingly, the present invention broadly provides a process for degumming a triglyceride oil which is substantially liquid at about 40°C, comprising (a) dispersing in the oil an organic acid or acid anhydride, at a temperature not greater than about 40°C; (b) subsequently dispersing water in the oil, while maintaining the temperature at not greater than about 40°C; and then (c) separating a sludge containing the gums from the oil to produce an oil product substantially reduced in phosphorus-containing compounds.

The triglyceride oils commonly used as feedstocks in this process include edible oils of vegetable or marine origin obtained by any of the known extraction techniques, including pressing and solvent extraction from an appropriately prepared oilseed or solvent extraction from a residue of a pressing operation. The oil may also be an oil prepared by rendering, pressing or solvent extraction from a whole marine species or a part thereof. Such feedstocks may be used in their crude form, but will usually be received from a commercial extractor of these oils in a partially degummed (industrially degummed) form, wherein the hydratable phospholipids have been substantially removed. The oil feedstock will be substantially liquid at the temperatures used in this process, which are less than about 40°C.

To achieve the above mentioned benefits of the process of this invention, the steps wherein the oil is contacted with an organic acid or acid anhydride are conducted at a temperature not greater than about 40°C. At temperatures greater than 40°C, the phosphorus removal achieved by the process drops off significantly. The lower temperature limit in these steps is generally set by the physical characteristics of the oil feedstock. The oil should be substantially liquid at the process temperature. For economic reasons, the preferred temperature of the process is in the range of about 20-25°C. In this ambient temperature range the oil does not require heating or cooling throughout the process.

Certain triglyceride oils, for example corn and sunflower oil, contain undesirable waxes when such oils are chilled. These waxes precipitate, giving the oil a cloudy appearance. Also, the waxes deteriorate at high temperature processing, deliteriously affecting the stability of the final product. Such oils are usually subjected to a procedure known as winterization, wherein the oil is cooled to crystallize the waxes and then cold filtered to remove the waxes.

In accordance with the process of the present invention, triglyceride oils containing waxes can be simultaneously dewaxed and degummed by maintaining the temperature throughout the degumming process in the range of about 0 to 10°C. Thus, the organic acid, and thereafter the water, are dispersed in the oil, and the sludge is separated from the oil at a temperature of about 0 to 10°C. The sludge which is removed from the oil contains both the phosphorus-containing gums and the majority of the waxes.

In the first step of the process, that is the acid contacting step, the acid-oil mixture is preferably maintained under agitation for a time sufficient to hydrolyze the majority of the hydrolyzable phospholipids. The time needed for this step will vary with the particular oil and the temperature being used, but will usually be less than about one hour, and will preferably be 10 to 20 minutes.

The organic acid or acid anhydride used in this process will most preferably be citric acid or maleic anhydride, and will preferably be of food grade quality. Other acids comparable to citric acid and maleic anhydride, and suitable for use in this process include maleic acid, lactic acid, oxalic acid and acetic anhydride. The acid is preferably added as an aqueous solution containing an amount of the acid up to the saturation point. A 50% by weight aqueous solution of the acid is preferred.

The amount of acid added to the oil will vary with the type of oil being processed, the amount and type of impurities in the oil and the other process conditions. Typically, an amount from about 1,000 to 10,000 ppm (0.1 to 1.0% by weight) of the oil, calculated on the basis of dry acid, is sufficient for the process.

Once the oil has been acid treated, and the phospholipids have thus been hydrolyzed, water is dispersed in the oil while maintaining the temperature at not greater than about 40°C. The amount of water needed in this step will vary with the quantity of phospholipids in the oil, but an amount in the approximate range of 0.1 to 3% by weight of the oil will usually be sufficient. An amount of water in the range of about 1.5 to 2.5% by weight of the oil is preferred.

In this second step of the process, the oil, acid and water mixture is preferably maintained under agitation for a time sufficient to hydrate the majority of the phospholipids. Again, the time will vary with the type of oil and temperature, but will usually be less than about one hour, and will preferably be about 20 to 30 minutes.

Once the phospholipids have been hydrated they become oil insoluble and precipitate out of the oil in the form of an aqueous sludge. This sludge is separated from the oil, preferably by centrifugation in a manner well known in the art. For practical reasons, the centrifugation is preferably carried out at a temperature not greater than about 35°C. After separation, the oil product is typically found to have a phosphorus content of less than about 15 ppm.

The process of this invention may be carried out either as a batch process or as a continuous process.

The oil product produced by this process is in a suitable form for further treatment by the known procedures of bleaching, deodorization and free fatty acid stripping. As previously disclosed, the oil product resulting from the process of this invention, after subsequent bleaching has a very low level of residual phosphorus, typically less than 1 ppm. If desired, the oil product may be neutralized in accordance with the known neutralization techniques, however, this step is preferably omitted in order to avoid the production of the undesirable soapstock.

The process is further illustrated by the following examples which demonstrate the operability and preferred conditions of the process, but which in no way limit the scope of the invention.

Example 1

This example shows the effect of temperature on the process of this invention.

An industrially degummed soyabean oil having the following initial characteristics, colour (LOVIBOND 25mm cell) 25Y + 3.5R, free fatty acids 0.27% by weight as oleic acid, and phosphorus content 129 ppm, was contacted, at the temperatures indicated in Table I, with a 50% by weight aqueous solution of citric acid to give 5000 ppm, based on dry acid.(LOVIBOND is a trade mark of The Tintometer Limited of Salisbury, England, for visual colorimeters with a generally recognized colorimetry scale.) The oil and acid were vigorously agitated for 15 minutes. Thereafter 1.0% by weight water was added to the oil, while maintaining the same temperature as in the acid contacting step. The oil mixture was agitated for an additional 30 minutes. The sludge containing the gums was removed by filtration. The analytical results of the degummed oil are shown in Table I.

The degummed oil was thereafter decolorized with bleaching clay and acid and then deodorized, in accordance with the following procedures:

Decolourization (Chemicals were added at room temperature) Addition of phosphoric acid 300 ppm (as dry matter) added as an 85% aqueous solution
Addition of citric acid 300 ppm (as dry matter) added as a 50% aqueous solution
Addition of bleaching clay 2.0 wt.%
Process conditions 110°C, 10 minutes, atmospheric pressure
Clay removal filtration

DeodorizationTemperature 250°C
Pressure 0.6 mm Hg
Time 60 minutes
Steam Sparge 2.4 wt %/hr

The analytical results of decolourization and deodorization are shown in Table I.

From the results it will be noted that the degumming process of this invention is capable of significantly reducing the phosphorus content of the oil. While the phosphorus content is still higher than desirable after the initial removal of the sludge, following the decolourization of the degummed oil, an oil product very low in phosphorus is produced, provided the temperature of the initial degumming process was not greater than about 40°C. The best results with respect to phosphorus removal were achieved when the degumming process was conducted at a temperature not greater than 35°C.

TABLE I

Exp't. No.	Degumming Temperature (°C)	Degummed Oil P (ppm)	Degummed Oil FFA (%)	Degummed Oil Colour (LOVIBOND 25mm cell)	Decolourized Oil P (ppm)	Decolourized Oil FFA (%)	Decolourized Oil Colour (LOVIBOND 25mm Cell)	Deodorized Oil Colour (LOVIBOND 133mm Cell)	Deodorized Oil FFA (%)	Deodorized Oil Flavour Score (/100)
1	25	6.8	0.25	20Y + 3.3R	0.3	0.30	2Y + 0.1R	4Y + 0.4R	0.02	90
2	30	4.3	0.25	20Y + 3.3R	0.3	0.30	2Y + 0.1R	4Y + 0.4R	0.02	90
3	35	6.9	0.25	20Y + 3.3R	0.3	0.30	2Y + 0.1R	4Y + 0.4R	0.02	90
4	40	9.3	0.25	20Y + 3.3R	0.5	0.30	2Y + 0.1R	4Y + 0.4R	0.02	90
5	45	12.0	0.25	20Y + 3.3R	1.0	0.30	2Y + 0.1R	5Y + 0.4R	0.02	85
6	50	16.7	0.25	20Y + 3.3R	1.7	0.30	2Y + 0.1R	5Y + 0.4R	0.02	85
7	55	23.5	0.25	20Y + 3.3R	2.6	0.31	3Y + 0.1R	5Y + 0.5R	0.02	80
8	60	37.5	0.25	20Y + 3.4R	4.0	0.31	3Y + 0.1R	5Y + 0.5R	0.02	75
9	80	83.9	0.26	25Y + 3.5R	12.1	0.33	3Y + 0.2R	7Y + 0.7R	0.02	55

Example II

An industrially prepared solvent extracted rapeseed oil (300g), containing 1,078 ppm phosphorus and 1.35% by weight of free fatty acids, was heated to 35°C with gentle agitation. To this was added either 50% aqueous solution of citric acid, to give a concentration of 2,500 ppm citric acid based on the dry acid in the oil, or powdered maleic anhydride at the level of 1,500 ppm. The mixture of oil and acid was vigorously agitated for 10 minutes while maintining the temperature of 35°C. An amount of water, 3.0% by weight, was then added and the mixture was agitated, at the same temperature, for an additional 20 minutes. The mixture was then filtered to remove the sludge containing the gums. The oil product was analyzed for phosphorus and free fatty acid content. For comparison purposes, the same oil was degummed with water only (3.0% by weight) with agitation for 30 minutes at 60°C. The analyses of the three filtered products are shown in Table II

TABLE II

Expt No.	Acid Contacting Acid (ppm)	T (°C)	Time (min)	Water Addition Water (%)	T (°C)	Time (min)	Residual Phosphorus(P) (ppm)	Residual Free Fatty Acid(FFA) (%)
10		-		3.0	60	30	26.5	1.06
11	Citric (2,500ppm)	35	10	3.0	35	20	11.5	0.90
12	Maleic Anhydride (1,500ppm)	35	10	3.0	35	20	14.6	0.92

Example III

To examine the effect of scaling up the process of this invention the procedures of experiments 11 and 12 as above-described, were repeated with 2,700g and 3,000g of the same oil. The process conditions and results are shown in Table III. After degumming, the oil product was decolourized with bleaching clay with acid addition, and then steam refined. The conditions of these two steps were as follows:

Decolourization Addition of phosphoric acid 500 ppm as 85% aqueous solution, added to oil at 30°C.
Addition of citric acid 500 ppm as 50% aqueous solution, added to oil at 30°C.
Addition of bleaching clay 3.0% by weight added at 90°C.
Process conditions 110°C, 15 minutes, atmospheric pressure with agitation.

Steam Refining Temperature 225°C
Time 60 minutes

TABLE III

Exp't No.	Acid Contacting			Water Addition			Degummed Oil		Decolourized Oil		Deodorized Oil		
	Acid (ppm)	T (°C)	Time (min)	Water (%)	T (°C)	Time (min)	P (ppm)	FFA (%)	P (ppm)	FFA (%)	Flavour	FFA (%)	Colour (LOVIBOND 133mm Cell)
13	Citric (2,500)	35	10	3	35	60	2.9	0.96	0.31	0.95	Very Mild (95/100)	0.01	3Y+0.3R
14	Maleic Anhydride (1,500)	35	10	3	35	60	12.9	0.94	0.22	0.96	Very Mild (95/100)	0.01	3Y+0.2R

Exp't No.	Decolourized Oil Colour (LOVIBOND 25mm Cell)
13	1.5Y+0.2R +0.1N
14	1.3Y+0.2R +0.1N

Example IV

To examine the reproducibility of the process of this invention, a blended feedstock of pressed and extracted oils which had previously been industrially degummed by a conventional process was treated. In each run, 3000g of oil having an initail colour of 40Y +3.0R + 0.5B (LOVIBOND 25mm Cell) an initial phosphorus content of 230 ppm and an initial free fatty acid content of 0.88% was contacted with an aqueous solution of citric acid to add 4,000 ppm citric acid, based on dry acid. The oil and acid were vigorously agitated for 30 minutes. Because the oil had been partially degummed, it was more difficult to effect the degumming. For this reason the citric acid addition level and agitation time were increased over the amounts and times used in the previous examples. Thereafter, 2.0% by weight water was added and the mixture was agitated for an additional 60 minutes. The sludge containing the gums was removed by filtration. The analytical results of the degummed oil are shown in Table IV.

The degummed oil was thereafter decolourized with bleaching clay and acid addition and then deodourized by the following procedures:

Decolourization (2,500g of degummed oil) Addition of phosphoric acid 500 ppm as an 85% aqueous solution added to the oil at 30°C.
Addition of citric acid 500 ppm as a 50% aqueous solution added to the oil at 30°C.
Addition of bleaching clay 3.0% by weight added at 105°C.
Process conditions 110-112°C, 15 minutes, atmospheric pressure with fast agitation.
Clay removal vacuum filtration.

Deodorization (1,200g of decolourized oil) Equipment CAMPRO stainless steel deodorizer. (CAMPRO is a trade mark of Cambrian Processes Limited of Mississauga, Ontario, Canada.)
Temperature 255°C
Pressure 0.5 to 1.0 mm Hg
Time 60 minutes
Steam Sparge 3% by weight of the oil
Cooling under vacuum

The analytical results are shown in Table IV.

From the results, it will be noted that the degumming process was capable of reducing the phosphorus level to about 10 to 24 ppm. This level was further reduced, after decolourizing, to less than 0.20 ppm. The reproducibility of the phosphorus removal, colour, free fatty acid removal and flavour was excellent.

TABLE IV

Exp't No.	Acid Contacting			Water Addition			Degummed Oil			Decolourized Oil			Deodourized Oil		
	Acid (ppm)	T (°C)	Time Min.	Water (%)	T (°C)	Time Min.	P (ppm)	FFA (%)	Colour (LOVIBOND 25mm Cell)	P (ppm)	FFA (%)	Colour (LOVIBOND 25mm Cell)	Flavour score/100	FFA (%)	Colour (LOVIBOND 133mm Cell)
15	Citric (4000)	35	30	2.0	35	60	13.2	0.73	35Y+3.0R	0.16	0.75	2Y+0.1R	Mild, 90	0.01	4Y+0.4R
16							10.2	0.82	35Y+3.0R	0.13	0.80	2Y+0.1R	Mild, 95	0.01	4Y+0.3R
17							14.8	0.83	35Y+3.0R	0.14	0.86	2Y+0.1R	Mild, 95	0.01	4Y+0.4R
18							12.1	0.86	35Y+3.0R	0.16	0.90	2Y+0.1R	Mild, 95	0.01	4Y+0.4R
19	(as Exp't 15)			(as Exp't 15)			11.9	0.94	30Y+3.0R	0.13	0.91	2Y+0.2R	Mild, 95	0.01	4Y+0.4R
20							23.6	0.91	30Y+3.0R	0.29	0.87	2Y+0.2R	Mild, 95	0.01	5Y+0.4R
21							11.8	0.88	35Y+3.0R	0.22	0.87	2Y+0.1R	Mild, 95	0.01	4Y+0.4R
22							23.0	0.81	30Y+3.0R	0.28	0.87	2Y+0.2R	Mild, 95	0.01	4Y+0.4R
23							11.3	0.80	30Y+3.0R	0.29	0.84	2Y+0.2R	Mild, 95	0.01	4Y+0.4R

Example V

To demonstrate the process with industrial equipment, 4501 of an industrially prepared non-degummed canola oil (a blend of prepressed and extracted oil) was charged to a 7001 baffled reactor equipped with a turbine agitator. The initial characteristincs of the oil were colour 5Y + 3.3R (LOVIBOND 25mm Cell) free fatty acid content (as oleic acid) 0.81% and phosphorus content 541 ppm. The oil was contacted with a 50% aqueous solution of citric acid to add 2,500 ppm citric acid (based on dry acid). The temperature of the process was 35°C. The mixture was agitated at 280 rpm for 10 minutes. Thereafter, 3% by weight of water was added to the mixture and the mixture was agitated at 280 rpm for an additional 20 minutes. The aqueous sludge which formed was removed by centrifugation, under the following conditions:

Centrifugal SeparationEquipment WESTFALIA SA-7-01-576, automatic discharging centrifuge, equipped with 69 discs (6.4 cm top diameter, 16.2 cm bottom diameter), 65 of which were liquid-liquid-solid separator discs and 4 of which were clarifier discs located at 1, 23, 46 and 69 locations. (WESTFALIA is a trade mark of Centrico, Inc. of Northvale, New Jersey, U.S.A.)

Angle Conical half angle 35°
Bowl Diameter 24.9 cm
Bowl Speed 8,400 rpm

The analytical results of the degummed oil are set forth in Table V.

The degummed oil was then dried at 60-70°C under -27" Hg for one hour in the reactor. The degummed oil (1,5001 collected) was pumped into a 26001 jacketted reactor and from this 3001 of degummed oil was mixed with PEMBINA VEGA clay bleaching earth to prepare a 3% clay slurry based on the total oil. (PEMBINA VEGA is a trade mark of Pembina Mountain Clays Incorporated of Winnepeg, Manitoba, Canada.) To the remainder of the oil was added, with mixing, 500 ppm each of phosphoric and citric acid (as solutions). The oil-acid mixture was heated under vacuum to 100°C with agitation. The clay slurry was then added to this mixture, also at a tempterature of 100°C The total mixture was heated to 110 to 115°C and held for 15 minutes at -27"Hg. The bleached oil was cooled with a heat exchanger to 60°C and then filtered in a filter press. The analytical results of the decolorization are shown in Table V.

The bleached oil was thereafter deodorized by steam refining with a CAMPRO pilot deodorizer under the following conditions:

Oil flow rate 700-750 lbs/hr
Steam sparge 21 lbs/hr
Vacuum 3 mm Hg
Temperature 257-260°C

The results are shown in Table V.

TABLE V

Expt No.	Degummed Oil P (ppm)	Degummed Oil FFA (%)	Degummed Oil Colour (LOVIBOND 25mm Cell)	Decolourized Oil (runs 15-18 Combined)	Deodorized Oil (runs 15-18 combined)
				Color	Flavour
24	21.0	0.52	35Y+3.1R	(LOVIBOND 25mm Cell) 2Y + 0.2R	Mild, 85/100
25	13.8	0.49	40Y+3.2R	(LOVIBOND 133mm Cell) 10Y + 1.0R	FFA 0.01%
26	21.1	0.54	40Y+3.1R	FFA 0.60% P 0.17 ppm	Peroxide value 1.8me/kg
27	24.3	0.57	40Y+3.0R		

Example VI

The following example illustrates the simultaneous degumming and dewaxing of a triglyceride oil contining waxes.

An industrially prepared degummed sunflower oil having the following initial characteristics, colour 30Y + 1.5R (LOVIBOND 25mm cell), free fatty acids (as oleic acid) 0.98%, and phosphorus content 114 ppm, was degummed at two temperatures, 23°C and 8°C. For the lower temperature degumming, the oil was held, prior to processing, at 8°C for 42 hours to accelerate the crystallization of the wax. The degumming process at both temperatures included contacting the oil with a 50% aqueous solution of citric acid to add 2,000 ppm citric acid, based on dry acid, followed by agitation for 15 minutes. Water (1%) was then added to the oil and the oil was mixed for a further 30 minutes, then filtered. The water addition step and filtration steps were also conducted at the temperatures of 23°C and 8°C. The results are shown in Table VI.

The degummed oils were then decolourized by adding phosphoric and citric acid (500 ppm each) at room temperature, followed by the addition of 3% by weight bleaching clay at 90°C. The bleaching was conducted at 110-115°C for 15 minutes under reduced pressure. The results are shown in Table VI.

The decolourized oil was thereafter deodorized at 225°C for 60 minutes. The steam sparge in this step was approximately 3%/hr. A cold test was conducted on the deodorized oil to examine for wax removal. The results are shown in Table VI.

As will be noted from the results, the process of the present invention, when conducted at 8°C, produced an oil product significantly reduced in wax and phosphorus content.

TABLE VI

Exp't No.	Degummed Oil P (ppm)	Degummed Oil FFA (%)	Degummed Oil Colour (LOVIBOND 25mm Cell)	Decolourized Oil P (ppm)	Decolourized Oil FFA (%)	Decolourized Oil Colour (LOVIBOND 25mm Cell)	Deodorized Oil Flavour Score/100	Deodorized Oil Colour (LOVIBOND 133mm Cell)	Deodorized Oil FFA (%)	Deodorized Oil Cold Test 24 hrs	Deodorized Oil Cold Test 48 hrs
28 ($23^{o}C$)	15	1.22	35Y + 1.8R	0.3	0.62	0.5Y + 0R	Very Mild 100	2Y + 0.2R	0.01	Deposit of fine waxes	Deposit of large waxes
29 ($8^{o}C$)	12	1.17	30Y + 1.7R	0.3	0.60	0.5Y + 0R	Very Mild 100	2Y + 0.2R	0.01	Clear	Slight Deposit of fine waxes

0 269 277

Claims

1. A process for degumming a triglyceride oil which is substantially liquid at about 40°C, comprising:
(a) dispersing in the oil an organic acid or acid anhydride, at a temperature less than 40°C;
(b) subsequently dispersing water in the oil, while maintaining the temperature less than 40°C; and then
(c) separating a sludge containing the gums from the oil to produce an oil product substantially reduced in phosphorus-containing compounds.

2. The process as set forth in claim 1, wherein the steps (a) and (b) are conducted at a temperature at which the oil is liquid.

3. The process as set forth in claim 2, wherein steps (a) and (b) are conducted at a temperature greater than 0°C and less than 40°C.

4. The process as set forth in claim 2, wherein the oil to be treated contains a negligible amount of waxes, and wherein the temperature of steps (a) and (b) is less than about 35°C.

5. The process as set forth in claim 1, wherein the oil to be treated contains waxes, and wherein the temperature of steps (a) and (b) is in the range of about 0 to 10°C, whereby in the separation step (c), the waxes are removed with the sludge.

6. The process as set forth in claim 1, wherein the organic acid or acid anhydride is citric acid or maleic anhydride.

7. The process as set forth in claim 1, wherein the acid is food grade citric acid.

8. The process as set forth in claim 7, wherein the citric acid is added to the oil as an aqueous solution and is included in an amount in the range of about 1,000 to 10,000 ppm, calculated as dry acid.

9. The process as set forth in claim 8, wherein the citric acid is added to the oil as an aqueous solution, the citric acid being included in the aqueous solution at a concentration up to saturation.

10. The process as set forth in claim 8, wherein the citric acid is added to the oil as a 50% aqueous solution.

11. The process as set forth in claim 1, wherein the amount of water included in step (b) is in the range of about 0.1 to 3% by weight of the oil.

12. The process as set forth in claim 9, wherein the amount of water included in step (b) is in the range of about 0.1 to 3% by weight of the oil.

13. The process as set forth in claim 9, wherein the amount of water included in step (b) is in the range of about 1.5 to 2.5% by weight of the oil.

14. The process as set forth in claim 1, wherein, in step (a), the mixture of oil and citric acid is maintained under agitation for a time sufficient to hydrolyze the majority of the hydrolyzable phosphorus-containing compounds.

15. The process as set forth in claim 1, wherein, in step (a), the mixture of oil and citric acid is maintained under agitation for up to about one hour.

16. The process as set forth in claim 13, wherein, in step (a), the mixture of oil and citric acid is maintained under agitation for a time in the range of about 10 to 20 minutes.

17. The process as set forth in claim 1, wherein, in step (b), the mixture of oil, citric acid and water is maintained under agitation for a time sufficient to hydrate the majority of the phosphorus-containing compounds.

18. The process as set forth in claim 1, wherein, in step (b), the mixture of oil, citric acid and water is maintained under agitation for up to about one hour.

19. The process as set forth in claim 16, wherein, in step (b), the mixture of oil, citric acid and water is maintained under agitation for a time in the range of about 20 to 30 minutes.

20. The process as set forth in claim 1, wherein the separation step (c) is conducted by centrifugation to remove an aqueous sludge containing the gums.

21. The process as set forth in claim 20, wherein the centrifugation is conducted at a temperature not greater than about 35°C.

22. The process as set forth in claim 1 wherein the triglyceride oil feedstock is of vegetable or marine origin and wherein the triglyceride oil feedstock has been previously industrially degummed.

23. The process as set forth in claim 21 wherein the triglyceride oil feedstock is of vegetable or marine origin and wherein the triglyceride oil feedstock has been previously industrially degummed.

24. A process for degumming a triglyceride oil, which is substantially liquid at about 40°C, wherein, after degumming, the oil is subjected to decolourizing with a bleaching clay, said process comprising of:

(a) dispersing in the oil an organic acid or acid anhydride, at a temperature less than 40°C;

(b) subsequently dispersing water in the oil, while maintaining the temperature less than 40°C; and then

(c) separating a sludge containing the gums from the oil to produce an oil product substantially reduced in phosphorus-containing compounds,

whereby the oil product of this process when subjected to a subsequent bleaching process results in a bleached oil product having a phosphorus content of less than about 1 ppm.

25. A process for degumming a triglyceride oil which is substantially liquid at about 40°C, comprising:

(a) dispersing in the oil an organic acid or acid anhydride, at a temperature less than 40°C;

(b) subsequently dispersing water in the oil, while maintaining the temperature less than 40°C;

(c) separating a sludge containing the gums from the oil to produce an oil product substantially reduced in phosphorus-containing compounds, and;

(d) decolourizing the oil product from step (c) by contacting the oil product with a bleaching clay, and then separating the clay from the oil to produce a bleached oil product having a phosphorus content of less than about 1 ppm.