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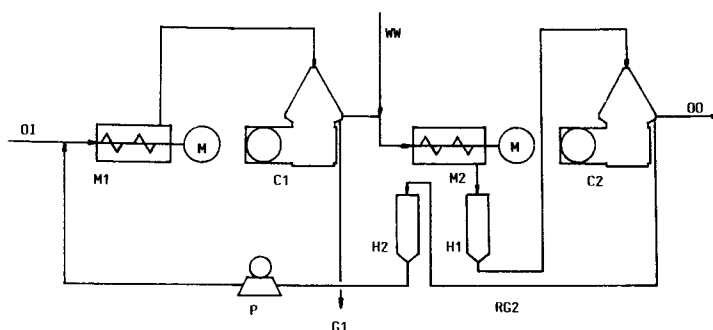
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(54) **Process for the continuous removal of a gum phase from triglyceride oil.**

(57) Process for the continuous removal of a gum phase from triglyceride oil comprising a) centrifugation of the oil containing a separate gum phase in a first centrifugal separator to yield a gum phase with minimal oil content and oil that still contains a fraction of the gums originally present in the feed; b) mixing into the oil obtained from the first centrifugal separator an amount of washing water and optionally passing said mixture to a holding vessel; c) centrifugation of said mixture in a second centrifugal separator to yield oil with a minimum residual gum content and a wet gum phase with a rather high oil content; and d) recycling said wet gum phase into the oil stream fed to the first centrifugal separator.

**FIGURE:**

OI: oil inlet  
M1: mixer 1  
C1: first centrifugal separator  
M2: mixer 2  
C2: second centrifugal separator  
WW: washing water  
H1: first holding tank  
H2: second holding tank  
G1: gum phase  
P: pump  
RG2: recirculation of second gum phase  
OO: oil outlet

**EP 0 507 363 A1**

**Background of the invention**

The present invention relates to a process for the continuous removal of a gum phase from triglyceride oil resulting in a gum phase with low oil content and a degummed oil which after bleaching may be physically refined. The present process comprises the following stages:

- a) in a first stage the oil containing a separate gum phase is subjected to centrifugal separation in a first centrifugal separator to yield a gum phase with minimal oil content and an oil that still contains a fraction of the gums originally present in the feed;
- b) in a second stage an amount of washing water is mixed into the oil obtained from the first centrifugal separator, which mixture is optionally passed to a holding vessel;
- c) in a third stage the mixture obtained at stage b) is subjected to centrifugal separation in a second centrifugal separator to yield oil with a minimum residual gum content and a wet gum phase with a much higher oil content than the gum phase obtained at stage a);
- d) in a fourth stage the wet gum phase obtained at stage c) is recycled into the oil stream fed to the first centrifugal separator.

Crude triglyceride oils as obtained by pressing and/or extracting oil seeds or animal matter contain several compounds other than triglycerides. Some of these compounds such as phosphatides, free fatty acids, odours, colouring matter, waxes and metal compounds must be removed because they adversely affect taste, smell, appearance and keepability of the refined oil.

Several processes for the removal of these unwanted compounds are known and frequently discussed in the literature.

A process for the continuous removal of a gum phase from triglyceride oil is described in EP 0 348 004. This process involves in a first stage degumming the oil by any conventional degumming method causing phosphatides to be hydrated, and, in a second stage, separating the undissolved and originally non-centrifugable particles from the degummed oil after optional agglomeration, by microfiltration, filtration, centrifugation, sedimentation or decantation. The main disadvantage of this process is the important loss of triglyceride oil entrained during removal of both gum phases.

Another process for the continuous removal of a separate gum phase from triglyceride oil is described in EP 349 718. In a first stage of this process, the oil containing a separate gum phase is subjected to centrifugal separation to yield gums with minimal oil content and an oil that still contains a fraction of the gums originally present in the feed. In a second stage of this process, the oil obtained from the first stage is subjected to a second centrifugal separation to yield oil with a minimum residual gum content and a gum phase with a much higher oil content than the gums obtained in the first stage, which gums are recycled as such. This recycling results in a decreased loss of triglyceride oil.

Finally, if sale of degummed oil and thus a low phosphorus content is aimed at, one or more washing stages are recommended, because the oil leaving the second centrifugal separator still has a phosphorus content that is considered to be unduly high.

These washing stages require additional centrifugal separators and cause an effluent stream, and accordingly increase investment and processing costs.

**Objects of the invention**

Therefore it is an object of the invention to provide an alternative process for the continuous removal of a gum phase from triglyceride oil resulting in gums with low oil content and degummed oil with low phosphorus content.

It is an additional object of the invention to provide a process for the continuous removal of a gum phase from triglyceride oil, which process does not entail high investment and processing costs.

It is also an object of the invention to provide a process for the continuous removal of a gum phase from triglyceride oil, avoiding polluting effluents.

These and other objects and advantages of the invention will become apparent as the description of the invention proceeds.

**Detailed description of the invention**

The invention relates to a process for the continuous removal of a gum phase from triglyceride oil resulting in a gum phase with low oil content and a degummed oil which after bleaching may be physically refined.

The process according to the invention is a process for the continuous removal of a gum phase from

triglyceride oil, comprising the following stages:

a) in a first stage the oil containing a separate gum phase is subjected to centrifugal separation in a first centrifugal separator to yield a gum phase with minimal oil content and an oil that still contains a fraction of the gums originally present in the feed;

b) in a second stage an amount of washing water is mixed into the oil obtained from the first centrifugal separator, which mixture is optionally passed to a holding vessel;

c) in a third stage the mixture obtained at stage b) is subjected to centrifugal separation in a second centrifugal separator to yield oil with a minimum residual gum content and a wet gum phase with a much higher oil content than the gum phase obtained at stage a), and

d) in a fourth stage the wet gum phase obtained at stage c) is recycled into the oil stream fed to the first centrifugal separator.

It has surprisingly been found, that mixing an amount of washing water into the oil obtained from the first centrifugal separator prior to being fed to the second centrifugal separator does not affect triglyceride oil content of the gum phase leaving the first centrifugal separator. It has also surprisingly been found that this additional amount of water does not affect the water content of the oil leaving the second centrifugal separator, in that the first centrifugal separator can effectively remove the additional amount of water that has been recycled together with the gum phase from the second centrifugal separator. This additional amount of water further causes the oil obtained from the second centrifugal separator to have a much lower phosphorus content.

It has also been found, that a usual amount of bleaching earth is required for bleaching the oil obtained according to the present invention, to provide bleached oils having a phosphorus level which is considered to be acceptable.

It is thus an advantage of the process according to the invention over the process disclosed in EP 0 349 718 that one or both washing stages can be omitted, as a result of which loss of triglyceride oil entrained during these washing stages, effluent problems and investments in washing centrifuges are avoided.

In order that the invention may be fully understood, the accompanying figure schematically represents a process line.

The process according to the invention can advantageously be used in the degumming process according to US 4,698,185. In this process water degummed vegetable oils are treated with finely dispersed aqueous acid whereupon this acid is partially neutralised so that a gum phase is formed. If such oils containing a gum phase are processed according to the present invention, the oils thus obtained may be physically refined.

Similarly the process according to the invention can advantageously be applied to degum oils treated by the process according to GB 1 565 569. This process entails adding an acid to crude oil, allowing a contact time of approximately 10 min., partially neutralising this acid with a base and then allowing an extended contact time for the development of a separate gum phase which is finally to be removed from the oil.

The process according to the invention can also advantageously be applied to degum oils treated by the process according to US 4,049,686. In US 4,049,686 a process is described in which oil is treated with an acid and cooled to below 40 °C whereupon the nonhydratable phospholipids form gums in a form that can be removed from the oil e.g. by centrifuge.

The type of oil to be degummed by the process according to the invention is not critical. Thus 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 provided the gum phase has been successfully developed before the oil is fed to the first centrifugal separator.

The washing water to be mixed into the oil obtained from the first centrifugal separator can be water, diluted non-toxic acid, e.g. citric acid, water containing salts or effluent water resulting from the optional washing stage. The amount of washing water to be mixed into the oil leaving the first centrifugal separator generally ranges from 0.05 to 10 wt.%, preferably between 0.5 and 5 wt.%.

The oil - washing water mixture is then optionally passed to a holding vessel. This holding time generally ranges from 10 seconds to 30 minutes, preferably from 0.5 to 15 minutes. The temperature in said holding vessel is preferably between 60 and 110 °C.

The centrifugal separators to be used in the process according to the invention can be disc centrifuges, decanters or other equipment capable of continuously separating a gum phase from an oil phase. The performance of such equipment can commonly be adjusted to yield either a gum stream with low oil content or an oil stream with low gum content as set out in EP 349 718.

The oil phase obtained by the process according to the invention may further be dried and bleached with commonly known bleaching means prior to being physically refined or refined by alkali neutralisation.

The invention is now illustrated by the following examples.

### Example 1

In this example the continuous removal of a gum phase from triglyceride oil according to the invention is illustrated.

The feed consisted of water-degummed soybean oil having a temperature of approximately 90 °C, a residual phosphorus content of approximately 120 ppm and an iron content of approximately 1.15 ppm. The separate gum phase was formed according to US 4,698,185 applying 0.20 vol.% phosphoric acid of 80 % strength, a contact time equal to 2.5 min. (at a throughput of 9.3 tons/h.) and a 50 % neutralization of the phosphoric acid with 12° Bé caustic soda.

The soybean oil comprising a separate gum phase was, in the first stage of the process according to the invention, fed to a centrifugal separator, resulting into a gum phase having a triglyceride oil content of approximately 19 wt.% (calculated on dry matter) and triglyceride oil which still contained a fraction of the phosphatides originally present in the feed.

Into the oil resulting from the first centrifugal separator, 4.10 % of washing water was mixed, which mixture was passed to a holding vessel and allowed to stand for approximately 2 minutes prior to being fed to the second centrifuge.

During the second centrifugal stage, a wet gum phase was removed from the triglyceride oil, which gum phase was fully recycled into the oil fed to the first centrifugal separator over a second holding vessel, which allowed a holding time of approximately 1 hour. The flow rate of this recycled stream was estimated at 2,3 tons/h.

In a comparative example, the same soybean oil, in which the separate gum phase was formed as mentioned above, was treated by the process according to EP 0 349 718, including two washing stages.

The quality of the oils at different stages of the processes is summarized in table 1. The oils were analysed for phosphorus and iron by plasma emission spectroscopy (A.J. Dijkstra and D. Meert, J.A.O.C.S. 59 (1982), 199).

Table 1

	example 1	comparative example 1
before first centrifugal separator moisture %	6.86	2.93
after first centrifugal separator moisture %	0.54	0.60
phosphorus ppm	26.8	64.8
iron ppm	0.15	0.17
before second centrifugal separator moisture %	4.68	0.59
after second centrifugal separator moisture %	0.42	0.42
phosphorus ppm	10.3	37.5
iron ppm	0.15	0.12
after two washing stages moisture %	-	0.53
phosphorus ppm	-	13.3
iron ppm	-	0.10
after drying phosphorus ppm	9.6	10.0
iron ppm	0.11	0.08

The above table clearly illustrates that the residual phosphorus content of the oil resulting from the second centrifugal separator is much lower when operating in accordance to the invention rather than according to EP 0 349 718. Besides, the phosphorus content of the oil obtained according to the invention is about the same as the phosphorus content of the oil obtained according to EP 0 349 718, which process included two washing stages. This example thus illustrates that both washing stages can be omitted when processing in accordance to the invention.

The above table also shows that the moisture content of oil resulting from the second centrifugal separator does not increase when operating in accordance with the invention. It also demonstrates, that the first centrifugal separator is able to remove the extra water introduced into the oil at the stage in between

the centrifugal separators.

## Example 2

In this example the continuous removal of a gum phase from triglyceride oil according to the invention is illustrated.

The feed consisted of partially water-degummed rapeseed oil having a temperature of approximately 90 °C, a residual phosphorus content of approximately 158 ppm and an iron content of approximately 1.58 ppm. The separate gum phase was formed according to US 4,698,185 applying 0.16 vol.% phosphoric acid of 80 % strength, a contact time equal to 2.5 min. (at a throughput of 9.0 tons/h.) and a 50 % neutralization of the phosphoric acid with 12 ° Bé caustic soda.

The rapeseed oil comprising a separate gum phase was, in the first stage of the process according to the invention, fed to a centrifugal separator, resulting into a gum phase having a triglyceride oil content of approximately 14 wt.% (calculated on dry matter) and triglyceride oil which still contained a fraction of the phosphatides originally present in the feed.

Into the oil resulting from the first centrifugal separator, 2 % of washing water was mixed, which mixture was passed to a holding vessel and allowed to stand for approximately 2 minutes prior to being fed to the second centrifuge.

During the second centrifugal stage, a wet gum phase was removed from the triglyceride oil, which gum phase was fully recycled into the oil fed to the first centrifugal separator over a second holding vessel, which allowed a holding time of approximately 30 minutes. The flow rate of this recycled stream was estimated at 2,4 tons/h.

In a comparative example, the same rapeseed oil, in which the separate gum phase was formed as mentioned above, was treated by the process according to EP 0 349 718, including two washing stages.

The oil content of the gum phase obtained by the process according to EP 349 718 was approximately 12.6 % (calculated on dry matter).

The oils thus obtained were bleached with 0.5 wt.% bleaching earth (Tonsil ACCFF, Süd Chemie, Munich, Germany) at 100 °C under vacuum for 30 minutes whereupon the oil was allowed to cool to below 90 °C before the bleaching earth was filtered off. Subsequently, the bleached oils were physically refined at 240 °C for 2 hours at a vacuum below 400 Pa (30 mmHg). Both oils had a bland neutral taste and showed excellent keepability.

The quality of the oils is summarized in table 2. Again, the oils were analysed for phosphorus and iron by plasma emission spectroscopy (A.J. Dijkstra and D. Meert, J.A.O.C.S. 59 (1982), 199).

Table 2

	example 2	comparative example 2
after second centrifugal separator phosphorus ppm	13.7	22.4
iron ppm	0.08	0.07
after drying phosphorus ppm	9.2	4.6
iron ppm	0.10	0.10
after bleaching with equal amounts of bleaching earth phosphorus ppm	2.9	1.0
iron ppm	0.01	0.01

The above table 2 clearly illustrates that the residual phosphorus content of the oil resulting from the second centrifugal separator is much lower when operating in accordance to the invention rather than according to EP 0 349 718.

Furthermore, the above table shows that the oil according to the invention can, after drying, be bleached with the usual amount of bleaching earth in order to obtain a high quality refined oil.

## Claims

1. Process for the continuous removal of a gum phase from triglyceride oil comprising the following stages:

a) in a first stage the oil containing a separate gum phase is subjected to centrifugal separation in a

first centrifugal separator to yield a gum phase with minimal oil content and an oil that still contains a fraction of the gums originally present in the feed;

b) in a second stage an amount of washing water is mixed into the oil obtained from the first centrifugal separator, which mixture is optionally passed to a holding vessel;

c) in a third stage the mixture obtained at stage b) is subjected to centrifugal separation in a second centrifugal separator to yield oil with a minimum residual gum content and a wet gum phase with a much higher oil content than the gum phase obtained at stage a), and

d) in a fourth stage the wet gum phase obtained at stage c) is recycled into the oil stream fed to the first centrifugal separator.

2. Process according to claim 1, characterized in that the amount of washing water added in stage b) ranges from 0.05 to 10 wt.%, preferably from 1 to 5 wt.%.

3. Process according to claims 1 or 2, characterized in that the washing water is selected from the group consisting of water, diluted non-toxic acid and water containing salts

4. Process according to any of the preceding claims, characterized in that the washing water is demineralized water.

5. Process according to any of claims 1 to 3, characterized in that the washing water is diluted non-toxic acid, preferably citric acid.

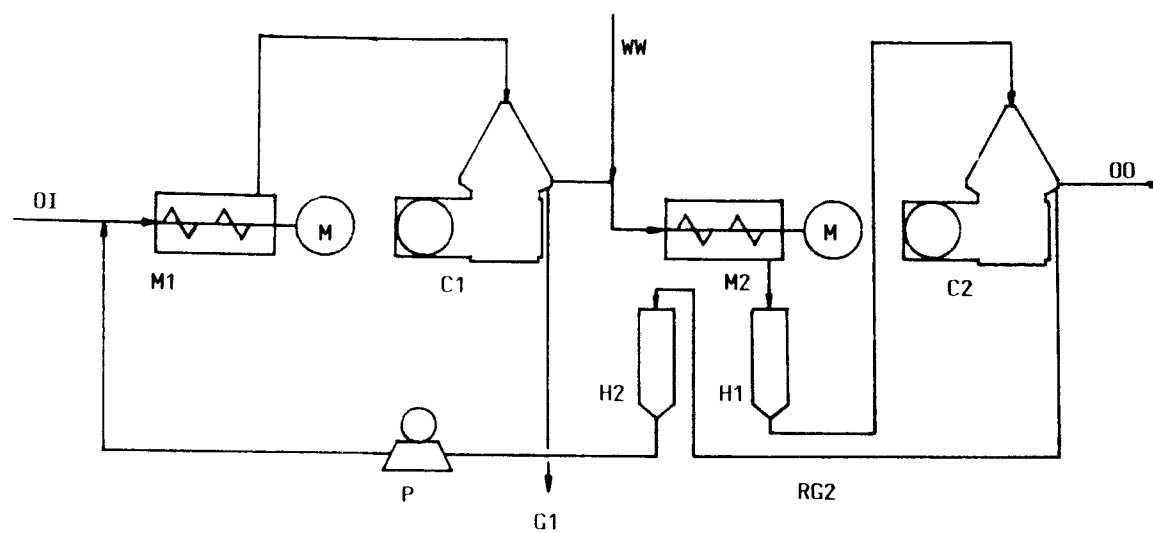
6. Process according to claims 1 or 2, characterized in that the washing water results from the optional washing stage.

7. Process according to any of the preceding claims, characterized in that the mixture in stage b) is passed through a holding vessel and allowed to stand for 10 seconds to 30 minutes, preferably for 0.5 to 15 minutes.

8. Process according to any of the preceding claims, characterized in that the temperature during the processing ranges between 60 and 110 °C, preferably between 80 and 100 °C.

9. Degummed oil as obtained according to any of the preceding claims.

FIGURE:



OI: oil inlet  
 M1: mixer 1  
 C1: first centrifugal separator  
 M2: mixer 2  
 C2: second centrifugal separator  
 WW: washing water  
 H1: first holding tank  
 H2: second holding tank  
 G1: gum phase  
 P : pump  
 RG2: recirculation of second gum phase  
 OO: oil outlet



DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,Y	EP-A-0 349 718 (NV. VANDEMOORTELE INTERNATIONAL) * page 3, line 15 - line 25; claim 1 * ---	1	C11B3/16
Y	BE-A-507 650 (S.A. DES APPAREILS CENTRIFUGES) * page 3, line 13 - line 22; claims 2,3 * * page 1, line 22 - page 2, line 5 * ---	1,3,6	
Y	THE JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY vol. 24, 1947, pages 261 - 264; H.J. DUTTON ET AL.: 'THE FLAVOR PROBLEM OF SOYBEAN OIL. I. A TEST OF THE WATER WASHING-CITRIC ACID REFINING TECHNIQUE' * page 261, right column * ---	1,3,6	
Y	FR-A-1 108 998 (S.A. FRANCAISE POUR LA SEPARATION, L'EMULSION ET LE MELANGE) * page 1, right column; figure * ---	1,3,6	
A	REVUE FRANCAISE DES CORPS GRAS vol. 27, no. 7, 1980, PARIS pages 333 - 339; G. DEVINAT ET AL.: 'SUR LA RAFFINABILITE DES HUILES. I. CONSIDERATIONS GENERALES ET ASPECTS TECHNOLOGIQUE ET ANALYTIQUE' * page 335, left column * ---	1-4,8,9	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	GB-A-481 580 (THE SHARPLES SPECIALTY COMPANY) * page 4, line 43 - line 82; claims 1-3,7,11; figures 1,2 * ---	1-3,7-9	C11B
A	EP-A-0 077 528 (CPC INTERNATIONAL INC.) * page 19, line 4 - page 20, line 6; claim 1 * --- -/--	1-3,5	
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 07 JULY 1992	Examiner KANBIER D.T.
<b>CATEGORY OF CITED DOCUMENTS</b> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ..... & : member of the same patent family, corresponding document			





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## EUROPEAN SEARCH REPORT

Application Number

EP 92 20 0543

Page 2

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	THE JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY vol. 61, no. 8, 1984, pages 1380 - 1382; E.G. LATONDRESS: 'ENERGY SAVING TECHNIQUES IN CONTINUOUS DEGUMMING AND REFINING' * page 1380, right column *  -----	2, 3, 7, 8	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 07 JULY 1992	Examiner KANBIER D. T.
<b>CATEGORY OF CITED DOCUMENTS</b> X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document  T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ..... & : member of the same patent family, corresponding document			