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(54) **Enzymatic degumming**

(57) The present invention relates to a method for treating vegetable oils and/or animal fats. The method comprises adjusting temperature, treatment with acid, adjusting pH, contacting the aqueous mixture with en-

zyme, crystallization of high melting glycerides and separation.

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Description

[0001] The present invention relates to a method for treating vegetable oils and/or animal fats.

5 Background

[0002] Most crude edible fatty oils - of vegetable or animal origin - contain impurities which must be removed before the oil is suitable for consumption. Also fatty oils for technical use often have to be purified to some extent to make them suitable for their purpose.

10 **[0003]** The removing of impurities could be carried out by a degumming and/or winterization process and may be combined into one process a so-called cold degumming process. However, the traditional cold degumming process is not always successful because:

- The separation efficiency is relatively low because of the increased gum viscosity at low temperatures;
- 15 - The wax crystallization and the crystal growth are, to some extent, inhibited by the presence of gums.

The invention

20 **[0004]** Accordingly, the present invention solves the above mentioned technical problems by the new inventive method. Thus, the present invention relates to a new method for treating vegetable oils and/or animal fats to reduce the content of impurities, such as various phospholipids i.e. gums, wax and/or high melting glycerides. One aspect of the invention to provide a method for efficiently removing both the phospholipids and the high melting glycerides by phospholipase at the same time. Another aspect of the invention to provide a method for utilizing the enzyme reaction feature such as the reacted gum has lower viscosity and less emulsification strength to achieve less oil loss.

25 **[0005]** The main purpose of a degumming process is to remove phospholipids from the oil. For some oil types such as sunflower seed oil, rice bran oil, corn oil, winterization process is needed to remove the high melting glycerides to avoid problems in the use of the oils at lower temperature or in later process.

30 **[0006]** The enzymatic degumming process has been proven effective in gum removal. In degumming processes, the phospholipids are converted to lyso-phospholipids and free fatty acids i.e. FFA. The lyso-phospholipids have much less emulsion capacity and lower viscosity. So, it is expected that the separation at lower temperature in enzymatic degumming process is much better than in a conventional process.

[0007] On the other hand, since the lyso-phospholipids are water-soluble, it is expected that most lyso-phospholipids will stay in the water phase during wax crystallation and crystal growth, so that the inhibition due to the presence of gums is eliminated.

35 **[0008]** In short, the cold enzymatic degumming process will provide the possibility of making degumming and dewaxing simultaneously, and with significant low loss of neutral oil

[0009] The new method for treating vegetable oils and/or animal fats according to the invention, comprises the following steps:

- 40 (i) adjusting the vegetable oils and/or animal fats to a temperature within the range from about 20 to about 90°C, preferably within the range from about 40 to about 90°C;
- (ii) pre-treating the vegetable oils and/or animal fats with acid for at least 1 minutes;
- (iii) adjusting the pH with lye to a pH within a range from about 4 to about 8 at a temperature of at least 20°C obtaining an aqueous mixture, preferably at a temperature of at least 40°C;
- 45 (iv) adding enzymes to the aqueous mixture;
- (v) reducing the temperature of the aqueous mixture to crystallization temperature of high melting glycerides;
- (vi) separating the aqueous mixture into an aqueous phase and a treated vegetable oils and/or treated animal fats phase; and
- 50 (vii) optionally treating the treated vegetable oils and/or treated animal fats phase with hot water or with silica adsorption.

[0010] In step (i) the temperature of the vegetable oils and/or animal fats may be adjusted within the range from about 60 to about 90°C.

55 **[0011]** In the pre-treating step (ii) the vegetable oils and/or animal fats may be treated with acid from about 1 to about 60 minutes, preferably from about 5 to about 60 minutes, most preferred from about 20 to about 40 minutes.

[0012] The pH in step (iii) may be adjusted with lye to a pH within a range from about 4 to about 8 at a temperature preferably from about 40 to about 60°C. The lye in step (iii) is selected from the group consisting of sodium hydroxide, potassium hydroxide, sodium silicate, sodium carbonate, calcium carbonate, and a combination thereof, preferably

sodium hydroxide or potassium hydroxide. According to the invention the mixing of the lye in step (iii) may be continued within the range from about 1 min to about 4 hours.

[0013] The temperature of the aqueous mixture in step (v) may be adjusted by a cooling rate and by a residence time to optimize crystallisation, preferably by a cooling rate within the range of from about 0.5 degrees per hour to about 5 degrees per hour, and a residence time within the range of from about 4 to 24 hours, preferably from 6 to 12 hours.

[0014] The temperature of the aqueous mixture in separation step (vi) may be adjusted to facilitate separation, preferably the temperature is within the range of from about 15 to about 50°C.

[0015] The enzyme in treatment step (iv) may be a phospholipase enzyme, preferably one or more phospholipase A enzymes, or one or more phospholipase C enzymes, or a combination thereof.

[0016] The acid used in step (ii) is selected from the group consisting of phosphoric acid, acetic acid, citric acid, tartaric acid, succinic acid, and a mixture thereof, preferably phosphoric acid or citric acid.

[0017] Further aspects and embodiments of the invention are defined by the subclaims. The invention will be further illustrated in the Examples, which are for the purpose to clarifying the invention and not to limit its scope. If not otherwise stated in the examples and tables the percentage is given by percent by weight (wt%).

Examples

[0018] The equipment used in this experiment was an oil bath, Erlenmeyer flasks 500 ml, magnetic stirrer with heating and temperature control, an Ultra Turrax, a laboratory centrifuge. FFA is analyzed according to method according to American Oil Chemists' Society, AOCS, Ca 5a-40, moisture is analyzed according to method AOCS Ca 2b-38, and phosphorus is analyzed according to method DIN EN 14107.

[0019] Materials used were:

1. Citric acid, monohydrate
2. Sodium Hydroxide, dry
3. Enzyme , Lecitase Ultra®, i.e. a phospholipase A enzyme.
4. Water

[0020] The crude sunflower seed oil was heated in oven to 70°C to ensure all the wax crystals are melted and dissolved in the oil. Two 500 ml. Erlenmeyer flasks, A and B, were used, one for normal enzymatic deep degumming (A) and the other for cold enzymatic deep degumming (B). To each Erlenmeyer flasks were 250g of oil were added, and the flasks were placed in a 55°C oil bath. The oil was stirred with a magnetic rod during the whole reaction, i.e. approx. 350 rpm.

[0021] A citric acid solution, i.e. 5 ml, was prepared by dissolving 1,78 g citric acid monohydrate in distilled water. A sodium hydroxide solution was prepared, i.e. 5 ml, by dissolving 0,5075g sodium hydroxide pellets in distilled water.

[0022] To each flask were 0.5 ml of citric acid solution added, and the mixture were mixed by using an Ultra Turrax at high speed, approx.24000rpm, for 1½ min. After 1 hour 0.5 ml of NaOH solution were added and the mixtures were mixed with an Ultra Turrax for 1½ min. To each flask were 0.012 ml of enzyme added together with water summing up to a total of 6 ml for each sample, and the mixing continued for additional 1½ min.

[0023] After 3 hours of enzyme treatment, the oil bath for flask A is heated to 80°C to inactivate the enzyme; while flask B was moved together with the magnetic agitator to fridge (7 - 8°C), and the agitation was kept at ca. 40 rpm for an overnight.

[0024] After ½ hour heating at 80°C, the oil from flask A for 5 min was centrifuged at 2000X g. The moisture, FFA and phosphorus content in the light phase (oil phase) were analysed.

[0025] After an overnight agitation in the fridge, flask B and the magnetic agitator were removed from the fridge, and the agitation was kept at room temperature (about 22°C) for ca. 15 min. The oil from flask B was centrifuged for 5 min. at 2000X g and the moisture, FFA and phosphorus content in the light phase were analysed.

[0026] The residual phosphorus content in the degummed oil is about 1 ppm only, which implies the degumming in both samples is complete.

Table

Analysis	Crude oil	Sample A	Sample B
Acid value [mg KOH/g]	0.84	0.85	0.82
Moisture [mg/kg]	947	1342	669
Phosphorous [mg/kg]	265	0.9	1.1

[0027] On the other hand, it was found some wax was removed together with the gum from the oil in the cold enzymatic deep degumming (B) sample after centrifuge separation. However, the amount of wax was not analyzed in this experiment.

[0028] Conclusion: The sunflower seed oil is successfully degummed in the cold enzymatic degumming process. Even though the separation temperature is much lower than that in ordinary degumming process, the residual phosphorus content in cold enzymatic degummed oil is at the same level as in the ordinary degummed oil.

Claims

1. A method for treating vegetable oils and/or animal fats, comprising the following steps:

- (i) adjusting the vegetable oils and/or animal fats to a temperature within the range from about 20 to about 90 °C;
- (ii) pre-treating the vegetable oils and/or animal fats with acid for at least 1 minute;
- (iii) adjusting the pH with lye to a pH within a range from about 4 to about 8 at a temperature of at least 20°C obtaining an aqueous mixture;
- (iv) adding enzymes to the aqueous mixture;
- (v) reducing the temperature of the aqueous mixture to crystallization temperature of high melting glycerides;
- (vi) separating the aqueous mixture into an aqueous phase and a treated vegetable oils and/or treated animal fats phase; and
- (vii) optionally treating the treated vegetable oils and/or treated animal fats phase with hot water or with silica adsorption.

2. The method according to claim 1, wherein the temperature in step (i) is adjusted within a range from about 40 to about 90 °C.

3. The method according to claim 1 or 2, wherein the pre-treating step (ii) the vegetable oils and/or animal fats is treated with acid from about 1 to about 60 minutes, preferably from about 5 to about 60 minutes, most preferred from about 20 to about 40 minutes.

4. The method according to claim 1, 2 or 3, wherein the pH in step (iii) is adjusted with the lye to a pH within a range from about 4 to about 8 at a temperature from about 40 to about 60 °C.

5. The method according to any one of claims 1 to 4, wherein the temperature of the aqueous mixture in step (v) is adjusted by a cooling rate and by a residence time to optimize crystallisation, preferably by a cooling rate within the range of from about 0.5 degrees per hour to about 5 degrees per hour, and a residence time within the range of from about 4 to 24 hours, preferably from 6 to 12 hours.

6. The method according to any one of claims 1 to 5, wherein the temperature of the aqueous mixture in separation step (vi) is adjusted to facilitate separation.

7. The method according to any one of claims 1 to 6, wherein the temperature of the aqueous mixture in separation step (vi) is adjusted to facilitate separation, preferably the temperature is within the range of from about 15 to about 50°C.

8. The method according to any one of claims 1 to 7, wherein the enzyme in treatment step (iv) is a phospholipase enzyme, preferably phospholipase A enzymes, or phospholipase C enzymes, or a combination thereof.

9. The method according to any one of claims 1 to 8, wherein the acid in step (ii) is selected from the group consisting of phosphoric acid, acetic acid, citric acid, tartaric acid, succinic acid, and a combination thereof, preferably phosphoric acid or citric acid.

10. The method according to any one of claims 1 to 9, wherein the temperature in step (i) is adjusted to be within the range from about 60 to about 90°C.

11. The method according to any one of claims 1 to 10, wherein the mixing of enzyme in step (iv) is continued within the range from about 1 min to about 6 hours.

12. The method according to any one of claims 1 to 11, wherein the mixing of the lye in step (iii) is continued within the

range from about 1 min to about 4 hours.

13. The method according to any one of claims 1 to 12, wherein the lye in step (iii) is selected from the group consisting of sodium hydroxide, potassium hydroxide, sodium silicate, sodium carbonate, calcium carbonate, and a combination thereof, preferably sodium hydroxide or potassium hydroxide.

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Application Number
EP 11 18 8382

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The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 16 May 2012	Examiner Adechy, Miriam
CATEGORY OF CITED DOCUMENTS 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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**ANNEX TO THE EUROPEAN SEARCH REPORT
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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