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(54) Title: A PROCESS FOR THE PRODUCTION OF A VEGETABLE-OIL PRODUCT

(57) Abstract

The invention relates to a process for the preparation of a vegetable-oil product from oilseed, such as rapeseed, and to the product obtained by the process. In the process the seed is comminuted, possibly heat-treated, and slurried in water. According to the invention, an enzyme is added to this slurry, the purpose of the enzyme being to retain in the aqueous phase the phosphatides present in the seed, while the oil separates to form a separate phase of its own, which is separated mechanically by centrifugation, for example. The obtained crude vegetable oil, which does not contain organic solvent residues and is substantially devoid of phosphatides, is refined physically to produce a final product, the refining comprising a treatment with an adsorption agent and/or a deodorization.

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A process for the production of a vegetable-oil product

The present invention relates to a process for the production of a vegetable-oil product from oilseed, in which process the seed is comminuted and slurried in water and the slurry is treated so that the oil is detached from the seed tissue, whereafter the oil is separated from the aqueous phase and is refined to produce the final product. In addition, the invention relates to a vegetable-oil product produced by the process.

Conventional methods for preparing vegetable oil are based on the separation, by extraction, of the oil from the other constituents of the seed. Before the extraction step, crushed seed is heated and pressed, and in certain cases these steps may substitute for the extraction. Organic solvents, such as hexane, have been used for the extraction.

The disadvantages of oil separation by extraction include the high capital costs of the necessary equipment, as well as high operating costs. In addition, solvent residues which may constitute a safety and health hazard and to which maximum values have therefore been set in legislation are left in the oil obtained and also in the seed material which is left over from the separation and is used for fodder.

In addition to the above-mentioned disadvantages, the pressing and extraction process involves a problem in that the phospholipids, i.e. phosphatides, present in the seed end up in the extract, together with the oil. In this case the phosphatides must be removed from the oil in the first step of the refining so that they will not disturb the subsequent steps of the re-

fining, in which the oil is purified of aldehydes and of free fatty acids, as well as of pigments.

The removal of phosphatides as part of oil refining has most commonly been carried out by using water or an acid. In each case, owing to their amphoteric character, the phosphatides hydrolyze and become insoluble in oil. Thereupon they can be separated from the oil by centrifugation, for example. Other methods for removing phosphatides include the use of membranes according to GB Patent 7 421 813 and the adsorption methods using bleaching clay or silica according to US Patents 635762 and 823217.

The object of the present invention is to provide a method simpler than previous ones for the preparation of a vegetable-oil product, eliminating the separate phosphatide removal step belonging to the refining. The process according to the invention is characterized in that an enzyme is added to an aqueous slurry of the seed so that the oil is separated under its action to form a separate phase, while the phosphatide present in the seed remains mainly in the aqueous phase, that the oil phase is separated and that the separated oil is transferred directly to physical refining, which consists of a treatment with an adsorbing agent and/or of deodorization.

What is accomplished with the enzyme addition according to the invention is that, as early as the primary oil separation step, the phosphatides remain in the aqueous phase left over from the separation, whereupon the obtained oil phase can, substantially without intermediate steps, be transferred to the subsequent physical refining. For the physical refining there suffices, according to need, an adsorption treatment mainly for the removal of pigments and/or a deodorization for the removal of the aldehydes or free fatty acids present. Since

the separation of the oil phase from the aqueous phase can be by mechanical means, for example, by centrifugation, the use of an organic solvent for the extraction of the oil is avoided in the invention, as is also the separation of the oil from the solvent, and an oil is obtained which is completely devoid of hazardous solvent residues.

The mechanism of the enzyme action, which in itself in no way restricts the invention, in the separation of oil and the other constituents of seed according to the invention is obviously as follows. In seed, such as rapeseed, oil is present in the cytoplasm of the seed tissue, in the form of small bodies separated from each other by monomolecular phosphatide layers. Under the action of the enzymes, the said walls between the bodies are hydrolyzed, breaking down so that the oil can separate from the cytoplasm. The hydrolyzing phosphatides at the same time become insoluble in the oil phase. The phosphatides thus remain, dissolved, in the aqueous phase and are separated from the oil definitively in the centrifugation or other such mechanical separation. According to the observations made, an oil phase which has been separated according to the invention by an enzyme treatment and centrifugation has an even lower phosphatide content than has an oil which has been separated from seed by conventional prior-art methods and has been treated separately for the removal of phosphatides.

With respect to the state of the art it should be noted that the use of enzymes in the treatment of oilseed is not in itself novel but a procedure known per se. For example, GB Application 2 127 425 discloses a method in which an enzyme is used to promote the extraction of oil by means of hexane. Furthermore, enzymes have been used in the separation of coconut oil (Mc Glone et al. J. Food Sci. 51, 1986, pp. 695-697) and avocado oil (Buenrostro et al., Biotechnol. Lett. 8, 1986, pp. 505-

506). However, the literature contains no mention of the action of enzymes on phosphatides, and there is no information given on the phosphatide contents in oils separated from seed by using enzymes. The separation of oil would thus be followed by a conventional refining treatment with the conventional steps for the removal of phosphatides, the adsorption of pigments, and the deodorization of the oil. By contrast, in the present invention, which is substantially based on the action of enzymes on the phosphatides present in the seed, it is essential that the obtained oil phase is transferred, without any separate steps for the removal of phosphatides, directly to the physical step of the refining, in which the oil is given an adsorption treatment and/or is deodorized.

The first step of the process for the preparation of a vegetable-oil product according to the invention is the comminution of the seed, for example by flaking or by coarse milling. Thereafter the seed is preferably heated so that its inherent enzymes are destroyed. Thus it is ensured that the action of the subsequently added enzyme acting on phosphatides will not be disturbed and that the enzymatic reactions will occur in the desired manner, with control. After the heating, the comminution of the preliminarily comminuted seed is continued using a disc attrition mill, a pin mill or some other fine-milling equipment. The finely milled seed is slurried in water, and the slurry is cooked for 10-60 min, preferably 30-40 min. If the cooking is continuous, the solids content of the slurry may be within the range 20-70 %, preferably 40-50%. On the other hand, if batch cookers are used for the cooking, the solids content of the slurry may be within the range 10-60%, preferably 15-40 %. After the cooking,the slurry can be wet milled by using, for example a colloid mill. After possible dilution and cooling, the slurry is ready for the addition of the enzyme having action on phosphatides.

The enzyme used has typically some optimal pH value to which the slurry is adjusted by adding an acid or a base. Thereafter the enzyme is added in a dose which may be 0.1-5.0 % by weight, most commonly 0.5-3.0 % by weight of the solids of the slurry, depending on the type of the seed and the enzyme used. After the adding of the enzyme, the slurry is incubated at the operational optimum temperature of the enzyme for 0.5-6 hours, preferably 3-4 hours. In this context it should be pointed out that the optimum conditions for the enzyme action in the process according to the invention are not necessarily those reported by the enzyme manufacturer on the basis of his characterizational studies; they have to be determined separately by experimentation. The enzymes used may vary according to the complexity of the cell walls of the seed; multi-activity enzymes have proven to be suitable, although carbohydralases have also yielded good results.

By the end of the incubation step, the desired division of the material into an oil phase, an aqueous phase and the remaining solids of the seed has taken place. By the use of the enzyme it has, according to the invention, been accomplished that the phosphatide present in the seed has in the main hydrolyzed and passed into the aqueous phase. The incubated slurry is heated to the temperature range 50-95 °C, preferably 70-95 °C, and is centrifuged in a decanting vessel in order to remove the solid seed material and any oil drops possibly adhering to it. The remaining liquid phase is maintained at a temperature of 70-95 °C and is clarified in a clarifier centrifuge, in which solid material will still separate out from the liquid. The solids obtained from the decanter are reslurried in water at a temperature of 60-80 °C, and the seed hulls are separated using a vibratory screen. The separated hulls are drained, pressed to dewater, and dried. The solids separated in the clarifier centrifuge are slurried in the washing water which

has passed through the vibratory screen. The obtained slurry is heated to the temperature range 60-80 °C and is divided in the clarifier centrifuge into a solid phase and a liquid phase. The liquid phases obtained form the clarifier in the said two steps are pooled together, maintained at a temperature of 50-95 °C, preferably 70-90 °C, and divided in a purifier centrifuge into an oil phase and a liquid phase. The solids obtained from the clarifier are homogenized and dried using, for example, a spray drier. To the obtained oil phase there is added, at a temperature of 30-70 °C, preferably 40-60 °C, approx. 0.1-3 % filter aid mixed with 0.1-0.5% sodium sulfate, whereafter mixing is carried out in a mixing tank. The oil is filtered using a conventional filter, such as a plate filter, and, when so desired, it can be further dried in a vacuum dryer. The phosphatide content in the oil obtained is so low that, after this, a mere physical refining treatment will suffice, without the chemical step normally carried out for the removal of phosphatides.

The physical refining of the oil may comprise, as the first step, an adsorption treatment in which, by using siliceous earth or other similar adsorption agent, mainly pigments and possibly remaining phosphatides are removed from the oil. The duration of the treatment may be, for example, 50 min. Thereafter the oil can be subjected to deodorization, which may be carried out, for example, by blowing steam through the oil for approx. an hour at a temperature of approx. 250 °C. The deodorization removes from the oil free fatty acids, aldehydes and other oxidation products, and the oil obtained is ready for use, for example, as a foodstuff.

Example 1

Low-glucosinolate rapeseed (Westar) were flaked and steam cooked at 95 °C for 30 min in order to inactivate the myrosinase enzyme inherently present in the seed. The cooked, flaked seed was thereafter comminuted in a disc attrition mill fitted with serrated discs. A slurry with a solids content of 40 % was prepared from the comminuted seed, and this slurry was cooked for 30 min. The slurry was then diluted with cold water to a 20 % solids content, whereby the temperature of the slurry was also lowered. The pH of the slurry was adjusted to 4.0 by using acetic acid. Thereafter, an enzyme mixture consisting of the enzymes Novo SP-249 and Pectinex 3XL was added to the slurry at 2 % of the dry weight of the slurry, and the slurry was incubated at 45 °C for 24 hours. After the incubation the slurry was heated to 80 °C and was centrifuged in a laboratory centrifuge so that three separate phases were obtained, i.e. the solid sediment, the aqueous phase, and the oil phase. The aqueous phase was centrifuged in a bucket centrifuge, whereby a solid sediment not containing hull material and an aqueous phase containing practically no oil were obtained. The solid sediments obtained in the centrifugation operations were resuspended in water at 70 °C and were centrifuged once more into three separate phases. The two oil phases obtained in the centrifugation operations were pooled together, and a mixture containing filter clay 1 % by weight of the oil amount and sodium sulfate 0.2 % by weight of the oil amount were added to them. The oil was mixed at 50 °C, whereafter it was filtered. The obtained crude oil was finally dried under vacuum. The measurement results characterizing the quality of the oil are given in the following table.

Table

Process	Peroxide value (meq/kg)	Iodine value (cg/100g)	Free fatty acids (%)	Chlorophyll (ppm)	Phosphate (ppm)
Solvent extraction	3.1	117.5	0.80	21.5	121.5
Process of Example 1	7.8	116.1	0.77	18.4	2.9

The reference process in the table is a solvent extraction in which the oil was produced in a laboratory, by using Soxhlet apparatus, from the same heat treated and flaked seed as in the enzyme process, by using hexane as the solvent.

Example 2

Rapeseed (Westar) was treated for the adding of the enzyme as in Example 1. After the cooking and dilution, the pH was adjusted as in Example 1 and the temperature was maintained at 50 °C. Enzyme Pectinex 3XL (Novo) was added to the slurry, and the slurry was incubated for 4 hours. After the incubation the oil was separated as described in Example 1. The measurement results characterizing the quality of the oil are presented in the following table.

Table

Process	Peroxide value (meq/kg)	Iodine value (cg/100g)	Free fatty acids (%)	Chlorophyll (ppm)	Phosphate (ppm)
Process of Example 2 (Pectinex 3XL)	10.2	116.0	0.55	19.5	1.8

Example 3

Low-glucosinolate rapeseed (Tobin), which differed from the seed used in Example 1, was treated for the adding of enzyme as described in Example 1. After the cooking, the slurry was wet milled in a Siego mill. The milled slurry was diluted to a solids content of 20 %, the pH was adjusted to 4.5 and the temperature to 50 °C. Enzymes Olease (Biocon) and Pectinex 3XL (Novo) at 2 % by weight of the solids of the slurry were added to the slurry. The slurry was incubated for 4 hours, and the oil was separated as described in Example 1. The measurement results characterizing the quality of the oil obtained are presented in the following table:

Table

Process	Peroxide value (meq/kg)	Iodine value (cg/100g)	Free fatty acids (%)	Chlorophyll (ppm)	Phosphate (ppm)
Process of Example 3 (Olease + Pectinex 3XL)	4.6	115.2	1.0	19.1	1.2

Example 4

Rapeseed (Tobin), which was the same as that used in Example 3, was treated for the adding of enzyme as described in Example 1. After the cooking and dilution, the pH was adjusted to 5 and the temperature to 50 °C. Olease (Biocon) at 2 % by weight of the solids of the slurry was added to the slurry, and the slurry was incubated for 4 hours. After the incubation the oil was separated as described in Example 1. The measure-

ment results characterizing the quality of the oil obtained are presented in the following table:

Table

Process	Peroxide value (meq/kg)	Iodine value (cg/100g)	Free fatty acids (%)	Chlorophyll (ppm)	Phosphate (ppm)
Process of Example 4 (Olease)	3.9	115.9	0.82	14.0	1.4

Example 5

Rapeseed (Westar) was treated for the adding of enzyme as described in Example 1. After the cooking and dilution, the pH of the slurry was adjusted to 4.5 and the temperature to 50 °C. The enzyme mixture according to Example 1 at 2 % by weight of the solids of the slurry was added to the slurry, and the slurry was incubated for 4 hours. The oil was separated as described in Example 1, and its quality characteristics were determined. The measurement results obtained are presented in the following table.

Thereafter the oil was subjected to a physical refining, at the beginning of which the oil was treated with 100-400 ppm citric acid at 60 °C for 20 min. Adsorption clay was added to the oil, and the oil was maintained at 100 °C under a pressure of 310 kPa generated by using an inert gas. After the adsorption step the oil phase was deodorized under a pressure of 0.4 kPa at 245 °C by blowing steam through it for one hour. Finally the oil was cooled, clarified by filtering it with clay, and analyzed by measuring the parameters characterizing the

quality of the oil. The measurement results are presented in the following table.

Table

Process	Peroxide value (meq/kg)	Iodine value (cg/100g)	Free fatty acids (%)	Chlorophyll (ppm)	Phosphate (ppm)
Process of Example 5 Crude oil	0.44	119	0.74	17.1	3.0
Physically refined oil	0.00	117	0.04	0.0	<0.2

The examples show that the phosphorus content of the crude, unrefined oil was in all cases at maximum 3 ppm. In conventional rapeseed oil production processes, the upper limits of phosphorus for degummed and superdegummed oils are 200 ppm and 50 ppm (Canadian General Standard Board). The phosphorus content of the crude rapeseed oil obtained according to the invention is thus only a fraction of the said values, and also considerably lower than the upper limit, 15 ppm, set for refined rapeseed oil (Canadian General Standard Board). It can be seen from the results of Example 5 that in the physical refining step of the process according to the invention the phosphorus content of the oil further considerably decreases.

It is evident for an expert in the art that the different embodiments of the invention are not restricted to the examples presented above but can vary within the accompanying claims. Thus it is possible to use, instead of the rapeseed cultivars used in the examples, other types of oilseed, such as sunflower seed or soybean or cotton seed, or even corn.

Claims

1. A process for the production of a vegetable-oil product from oilseed, in which process the seed is comminuted and slurried in water and the slurry is treated so that the oil is detached from the seed tissue, whereafter the oil is separated from the aqueous phase and is refined into the final product, characterized in that an enzyme is added to the slurry so that the oil separates under its action to form a separate phase, while the phosphatide present in the seed remains in the main in the aqueous phase, that the oil phase is separated, and that the separated oil is transferred directly to physical refining, which consists of a treatment with an adsorption agent and/or of a deodorization.

2. A process according to Claim 1, characterized in that the partly or completely comminuted seed is heated before the slurrying step in order to destroy the enzymes inherently present in the seed.

3. A process according to Claim 1 or 2, characterized in that the seed slurried in water is heated for 10-60 min, preferably 30-40 min, before the adding of the enzyme.

4. A process according to any of the above claims, characterized in that the enzyme is added at 0.1-5.0 % by weight, preferably 0.5-3.0 % by weight, calculated from the solids in the slurry.

5. A process according to any of the above claims, characterized in that, after the adding of the enzyme, the slurry is incubated for 0.5-6 h, preferably 3-4 h.

6. A process according to Claim 5, characterized in that after the incubation, before the separation of the oil phase, the temperature of the slurry is raised to the range 50-95 °C, preferably 70-95 °C.

7. A process according to any of the above claims, characterized in that the oil phase is separated from the aqueous phase by centrifugation.

8. A process according to Claim 7, characterized in that the oil separated by centrifugation is filtered and dried before refining.

9. A process according to any of the above claims, characterized in that the refining comprises an adsorption treatment and a deodorization using blown steam at an elevated temperature.

10. A process according to any of the above claims, characterized in that the process is used for producing refined rapeseed cultivar oil.

11. A vegetable oil product, characterized in that the product is separated from an aqueous slurry of a comminuted seed material, which slurry has been treated with an enzyme, and that the product is devoid of residues of organic solvents such as hexane.

12. A vegetable-oil product according to Claim 11, characterized in that the phosphorus content of the product is 3 ppm or lower.

13. A vegetable-oil product according to Claim 12, characterized in that the phosphorus content of the product is approx. 0.2 ppm or lower.

14. A vegetable-oil product which has been produced by any one of the processes according to Claims 1-10.

AMENDED CLAIMS

[received by the International Bureau on 4 July 1991 (04.07.91), original claim 1 amended; other claims unchanged (1 page)]

1. A process for the production of a vegetable-oil product from oilseed, in which process the seed is comminuted and slurried in water and an enzyme is added to the slurry so that with the aid of the enzyme the oil is detached from the seed tissue and forms a separate phase, whereafter the oil is separated from the aqueous phase and is refined into the final product, **characterized** in that the oil, which has been separated from the aqueous phase and thereby from the phosphatides which are derived from the seed and remain in the aqueous phase, is transferred directly, i.e. without any further step for the removal of the phosphatides, to physical refining, which consists of a treatment with an adsorption agent and/or of a deodorization.

2. A process according to Claim 1, **characterized** in that the partly or completely comminuted seed is heated before the slurrying step in order to destroy the enzymes inherently present in the seed.

3. A process according to Claim 1 or 2, **characterized** in that the seed slurried in water is heated for 10-60 minutes, preferably 30-40 minutes, before the adding of the enzyme.

4. A process according to any of the above claims, **characterized** in that the enzyme is added at 0.1-5.0% by weight, preferably 0.5-3.0% by weight, calculated from the solids in the slurry.

5. A process according to any of the above claims, **characterized** in that, after the adding of the enzyme, the slurry is incubated for 0.5-6 h, preferably 3-4 h.

INTERNATIONAL SEARCH REPORT

International Application No PCT/FI 91/00062

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC5: C 11 B 1/04

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
IPC5	C 11 B

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched[8]

SE,DK,FI,NO classes as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category *	Citation of Document,[11] with indication, where appropriate, of the relevant passages[12]	Relevant to Claim No.[13]
X	EP, A1, 0113165 (IMPERIAL BIOTECHNOLOGY LIMITED) 11 July 1984, see claim 1-4 and page 12 line 29-30 "by aqueous flotation" --	1-12
X	WO, A1, 8909255 (BUCHER-GUYER AG) 5 October 1989, see the claims --	1-12
X	FR, A, 2196648 (CPC INTERNATIONAL INC.) 15 March 1974, see the claims --	1-12
X	FR, A, 2078467 (VEB SCHWERMASCHINENBAU-KOMBINAT ERNST THALMANN) 5 November 1971, see page 1, line 30 - line 32; claims 1,5 --	1-12

* Special categories of cited documents: [10]

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"Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
6th June 1991	1990 -06- 11
International Searching Authority	**Signature of Authorized Officer**
SWEDISH PATENT OFFICE	Kerstin Boije Jansson

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
X	DE, A, 1767027 (DREVICI, NOEET AL) 2 September 1971, see claims 1,3,4 --	1-12
X	GB, A, 1402769 (CPC INTERNATIONAL INC) 13 August 1975, see the whole document --	1-12
X	US, A, 4904483 (CHRISTENSEN ET AL) 27 February 1990, see the whole document --	1-12
X	DK, A, 64815 (A/S NIRO ATOMIZER) 9 September 1946, see the whole document --	1-12

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL PATENT APPLICATION NO.PCT/FI 91/00062

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the Swedish Patent Office EDP file on 91-04-30
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A1- 0113165	84-07-11	GB-A- 2127425	84-04-11
WO-A1- 8909255	89-10-05	CH-A- 675730	90-10-31
		EP-A- 0364545	90-04-25
FR-A- 2196648	74-03-15	NONE	
FR-A- 2078467	71-11-05	DE-A-B-C 2056896	71-08-26
DE-A- 1767027	71-09-02	NONE	
GB-A- 1402769	75-08-13	NONE	
US-A- 4904483	90-02-27	GB-A- 2215980	89-10-04
DK-A- 64815	46-09-09	NONE	