Abstract:
The present invention relates to a process of manufacturing palm oil fractions containing virtually no 3-monochloropropanediol fatty acid esters. More particularly, the present invention provides a process of manufacturing a palm oil fraction that contains virtually no 3-MCPD-esters, said process comprising: fractionating a crude palm oil having a free fatty acid content of less than 1.5% and a diglyceride content of less than 5.5 wt.% to produce at least one crude palm oil fraction, said crude palm oil fraction being selected from crude palm olein having an iodine value of at least 55 and crude palm stearin having an iodine value of less than 48; and deodorising the crude palm oil fraction to produce a deodorised palm oil fraction having an 3-MCPD-ester content of less than 1 ppm. The invention further provides a method of preparing a food product, said method comprising incorporating into said food product 3-99 wt.% of a palm oil fraction obtained by the aforementioned process.
PROCESS FOR MANUFACTURING PALM OIL FRACTIONS CONTAINING VIRTUALLY NO 3-MONOCHLOROPROPANEDIOL FATTY ACID ESTERS

TECHNICAL FIELD OF THE INVENTION

The present invention relates to a process of manufacturing palm oil fractions containing virtually no 3-monochloropropanediol fatty acid esters (3-MCPD-esters).

BACKGROUND OF THE INVENTION

Palm oil fractions are produced from palm oil by means of fractionation. The term "fractionation" is used to describe fractional crystallisation processes of triglycerides in which a solid high melting fraction (stearin) is separated from a liquid low melting fraction (olein). The fractionation process has two main stages, the first one being the crystallisation stage and the second one being the separation stage. During the crystallisation stage fat crystals grow when the temperature of the molten fat or its solution is lowered, and their solubility at the final or separation temperature determines the triglyceride composition of the isolated fat crystals (stearin) and of their mother liquor (olein).

Dry fractionation is the oldest type of fractionation. An alternative fractionation technique is solvent fractionation. Solvent fractionation employs an organic solvent during crystallisation in order to enhance fractionation yields.

GB-B 1491 170 describes a process for preparing a soft palm oil which comprises degumming raw palm oil by contact with phosphoric acid, decolourising the product by contact with an adsorbent, removing free fatty acids and odour components from the resulting decoloured oil by subjecting it to steam distillation under reduced pressure, and subjecting the resulting deodorised oil to winterisation (fractionation), thereby removing higher melting materials and obtaining low melting materials.

Free 3-monochloropropane-1,2-diol (3-MCPD) has been identified as a contaminant for a long time in various foods like liquid seasoning (e.g. soy sauce) or bakery goods heated to high temperatures. This substance is formed when fat- and chloride salt-containing foods are processed at high temperatures. In animal experiments 3-MCPD has led to an increase in the cell count (hyperplasia) in renal tubules and, at higher levels, it triggered benign tumours.
No genotoxic effect was observed. It can, therefore, be assumed that the tumours observed in the long-term animal study (mainly benign) only occur above a threshold value. There are no findings from human studies.

Recent studies have identified 3-MCPD fatty acid esters in refined edible fats and in fat-containing foods. 3-MCPD esters are compounds of 3-MCPD and various fatty acids which are formed at high temperatures under hydration following a reaction between fats and chloride ions. Studies by the food control authorities in Baden-Württemberg showed that all refined vegetable oils and fats contain 3-MCPD fatty acid esters. Only oil that had not undergone any heat treatment (e.g. native olive oil) did not contain the substance.

3-MCPD fatty acid esters are formed at high temperatures, probably during the deodorisation of edible fats and oils, the last stage in refining, during which undesirable odorous and taste-bearing substances are removed. Besides 3-MCPD fatty acid esters also 2-MCPD fatty esters, 1,3-dichloropropane-2-ol (1,3-DCPO) and 1,2-dichloropropane-2-ol (1,2-DCPO) fatty acid esters have been identified in heat processed foods.

No toxicological data are available on 3-MCPD fatty acid esters, nor for that matter on any of the other above mentioned propanol fatty acid esters. Nonetheless, it would be desirable to have available oil processing techniques that minimize formation of these unwanted chloropropanol fatty acid esters.

SUMMARY OF THE INVENTION

The inventors have developed a process that enables the manufacture of palm oil fractions that contain virtually no 3-MCPD-esters. Unexpectedly, the inventors have discovered that the formation of 3-MCPD-esters during the production of palm oil fractions can be minimized effectively by first fractionating a crude palm oil having a low free fatty acid content and a low diglyceride content, followed by deodorisation of the crude palm oil fractions so obtained. In contrast, in conventional processes for the production of palm oil fractions, crude palm oil is deodorised before fractionation.

More specifically, the present invention provides a process of manufacturing palm oil fractions comprising:

- fractionating a crude palm oil having a free fatty acid content of less than 1.5% and a diglyceride content of less than 5.5 wt.% to produce at least one crude palm oil fraction,
said crude palm oil fraction being selected from crude palm olein having an iodine value of at least 55 and crude palm stearin having an iodine value of less than 48; and

- deodorising the crude palm oil fraction to produce a deodorised palm oil fraction having a 3-MCPD-ester content of less than 1 ppm.

Although the inventors do not wish to be bound by theory, it is believed that the low 3-MCPD-ester levels in the palm oil fraction obtained by the present process can be realised by utilizing a crude palm oil of very low free fatty acid (FFA) content and by fractionating such crude palm oil prior to deodorisation.

The invention further provides a method of preparing a food product, said method comprising incorporating into said food product 3-99 wt.% of a palm oil fraction obtained by above described process.

DETAILED DESCRIPTION OF THE INVENTION

Accordingly, one aspect of the invention relates to a process of manufacturing a palm oil fraction comprising:

- fractionating a crude palm oil having a free fatty acid content of less than 1.5% and a diglyceride content of less than 5.5 wt.% to produce a crude palm oil fraction, said crude palm oil fraction being selected from crude palm olein having an iodine value of at least 55 and crude palm stearin having an iodine value of less than 48; and

- deodorising the crude palm oil fraction to produce a deodorised palm oil fraction having a 3-MCPD-ester content of less than 1 ppm.

The term "oil" whenever used herein encompasses both oils that are liquid at ambient temperature and oils that are solid at ambient temperature (fats).

The crude palm oil employed in the present process typically contains at least 90 wt.% of triglycerides. Other components that may be contained in the crude palm oil in significant quantities include diglycerides, monoglycerides, fatty acids and phospholipids.

The present process yields a palm oil fraction that not only has a very low 3-MCPD ester content, but wherein also the levels of glycidol fatty acid esters are much lower than in conventionally produced palm oil fractions. Glycidol fatty acid esters are believed to be a direct precursor of 3-MCPD (and 2-MCPD and 1,2-DCPO). Unfortunately, there is not yet available an analytical method that enables reliable quantification of glycidol fatty acid ester concentrations. What is believed to be the best currently available analytical technique for the determination of glycidol fatty acid esters is described by Masukawa et al. (A New Analytical Method for the Quantification of Glycidol Fatty Acid Esters in Edible Oils, J. Oleo Sci. (2010) 59(2), 81-88).

According to a particularly preferred embodiment of the present process, the deodorised palm oil fractions produced in the present process has a 3-MCPD-ester content of less than 0.8 ppm, even more preferably of less than 0.6 ppm and most preferably of less than 0.5 ppm. Preferably, the process yields a deodorised palm olein having a 3-MCPD ester content of less than 0.8 ppm and a palm stearin having a 3-MCPD ester content of less than 0.6 ppm. The very low 3-MCPD-ester contents in the deodorised palm oil fractions can be realised with the present process without using techniques for removing 3-MCPD-esters, e.g. by means of selective adsorption and/or by means of solvent extraction. In the present process the 3-MCPD ester content of the crude palm oil and the crude palm oil fraction is usually below detection limit, e.g. below 0.15 ppm. In an advantageous embodiment of the present process, the fractionation of the crude palm oil yields a crude palm olein having a 3-MCPD ester content of less than 0.15 ppm and a crude palm stearin having a 3-MCPD ester content of less than 0.15 ppm.

As explained herein before, it is believed that in order to achieve very low 3-MCPD levels in the deodorised palm oil fraction it is crucial to start from a crude palm oil having an exceptionally low FFA content. Advantageously, the FFA content of the crude palm oil does not exceed 1.3%, most preferably it does not exceed 1.2%.

The crude palm oil that is used as a starting material in the fractionation, besides having a very low FFA content, preferably also has a very low diglyceride content. Typically, the diglyceride content of the crude palm oil does not exceed 5.5 wt.%. Even more preferably, the diglyceride content does not exceed 5 wt.%. Most preferably, the diglyceride content does not exceed 4.5 wt.%.

The inventors have discovered that a low FFA crude palm oil can suitably be produced by:

- harvesting palm fruit;
• subjecting the harvested fruit to a heat treatment (e.g. steaming) to inactivate enzymes contained therein, said heating taking place within 24 hours, preferably 12 hours after the harvesting of the fruit;
• pressing said heat treated palm fruit to extract an oil-containing slurry; and
• removing water and non-oleaginous solids from said oil-containing slurry to produce a crude palm oil having a free fatty acid content of less than 1.5%.

By ensuring that the harvested palm fruit is heat treated very quickly after harvesting, formation of free fatty acids can be minimised effectively.

In the present process the time span from harvesting of the palm fruit till the deodorisation of the crude palm oil fractions typically does not exceed 4 days. Even more preferably, the latter time span does not exceed 84 hours, most preferably it does not exceed 72 hours.

Unlike conventional processes for the manufacture of palm oil fractions, in the present process degumming is suitably performed after fractionation. Thus, in accordance with a preferred embodiment, the crude palm oil fraction is subjected to degumming prior to the deodorisation. In another particularly preferred embodiment, the degummed crude palm oil fraction is subjected to bleaching prior to the deodorisation. Typically, the crude palm oil fraction is bleached to produce a bleached palm oil fraction having a Lovibond red number of less 1.5 and a Lovibond yellow number of less than 15.

The fractionation that is comprised in the present process may be carried out a dry or a wet fractionation. Preferably, the fractionation employed in the present process is a dry fractionation.

The fractionation employed in the present process advantageously uses a crystallisation temperature of less than 30°C, more preferably a crystallisation temperature in the range of 20-28°C. Typically, the olein yield of the fractionation process exceeds 50 wt.%. Most preferably, the olein yield is in the range of 60-85 wt.%.

The peroxide value of the crude palm olein obtained from the fractionation in the present process is very low, e.g. below 2, more preferably below 1.5 and most preferably below 1.

In the present process the crude palm oil is suitably deodorised at a temperature of 230-280 °C and a pressure of 0.1-4 mbar. Even more preferably, the crude palm oil is deodorised at a temperature of 250-270 °C, most preferably at a temperature of 260-265 °C. The pressure employed during deodorisation advantageously lies within the range of 0.3-2 mbar, most preferably of 0.5-1 mbar.
As explained herein before, the present process differs from conventional processes for the production of palm oil fractions in that fractionation precedes deodorisation. According to a particularly preferred embodiment, the crude palm oil is not deodorised prior to the fractionation, in other words, deodorisation is only performed after fractionation.

In a preferred embodiment of the present invention water is removed from the oil-containing slurry that is obtained after pressing the heat treated palm fruit in a clarifier that is preferably operated at 90-95 °C. The oil obtained from the clarifier is preferably dried in a vacuum drier to a moisture content of less than 0.2%.

Another aspect of the invention relates to a method of preparing a food product, said method comprising incorporating into said food product 3-99 wt.%, more preferably 4-60 wt.% and most preferably 5-50 wt.% of a palm oil fraction obtained by the process defined herein before.

Examples of food products in which the present palm oil fraction may suitably be incorporated include cooking oils, margarines, infant formulas, clinical food, peanut butter, cookies, chocolate, chocolate spread and nutritional formulations.

The advantages of the present invention are particularly appreciated when the deodorised palm oil fraction obtained by the present process is employed in products that are regularly consumed by children, especially infants. Hence, the present method is preferably used in the preparation of food product selected from margarines (including, bakery margarines), bakery fats, infant formulas and chocolate spread.

According to a particularly preferred embodiment, the present method is used to prepare an infant formula. Preferably, said infant formula is prepared by incorporating therein 0.5-10 wt.% of a deodorized palm olein that is obtained by the process described herein before.

Palm olein is advantageously employed in infant formulas in combination with other oils to achieved an oil blend having a fatty acid composition that is similar to the fatty acid composition of human milk fat. EP-A 0 488 800 describes fat compositions for infant formulas that contain substantial quantities of palm olein. According to a particularly preferred embodiment of the present method, the deodorized palm olein is incorporated in an infant formula in combination with one or more non-hydrogenated vegetable oils, preferably including a highly unsaturated oil selected from rapeseed oil, safflower oil, soybean oil, sunflower oil, fish oil and combinations thereof.

Advantageously, the deodorized palm olein is incorporated in the infant formula as part of an oil blend, said oil blend comprising, by weight of the total amount of oil:
• 5-50%, preferably 8-40% of the palm olein obtained by the present process; and
• 15-60%, preferably 20-55% of a highly unsaturated oil selected from rapeseed oil, safflower oil, soybean oil, sunflower oil, fish oil and combinations thereof. Even more preferably, the latter oil blend additionally comprises 8-40%, most preferably 10-35% by weight of the total amount of oil, of a lauric fat selected from coconut oil, palm kernel oil and combinations thereof.

Typically, the oil contained in the infant formula provides 30-55% of the caloric content of the product.

According to another preferred embodiment of the present method, the deodorized palm fraction is incorporated in a chocolate spread in an amount of 10-35% by weight of the chocolate spread.

In another advantageous embodiment, the present method of preparing food product comprises incorporating a palm stearin obtained by a fractionation/deodorization process as described herein before into a margarine or a bakery fat. Typically, said palm stearin is incorporated in the latter products in a concentration of 10-80% by weight of the final product.

In one embodiment, the palm stearin is incorporated into a bakery margarine or a bakery fat in combination with a highly unsaturated oil selected from rapeseed oil, safflower oil, soybean oil, sunflower oil, fish oil and combinations thereof. More preferably, the palm stearin is incorporated into these bakery products in a concentration of 20-80% and the highly unsaturated oil in a concentration of 20-80%, both percentage being calculated by weight of the total amount of oil and fat that is contained in the bakery product.

In accordance with another embodiment of the present method, the palm stearin obtained by the present fractionation/deodorization process is incorporated in a margarine as part of a so called hardstock component, said hardstock component comprising an interesterified blend of 30-80% of the palm stearin and 20-70% of a lauric fat (notably palm kernel oil or coconut oil), both percentages being calculated by weight of the hardstock component. Besides said hardstock component, the margarine typically contains 20-80 wt.% of a highly unsaturated oil selected from rapeseed oil, safflower oil, soybean oil, sunflower oil, fish oil and combinations thereof.

The invention is further illustrated by means of the following examples.
EXAMPLES

Example 1
Marginally ripe palm fruit bunches were harvested at the Carey Island West Estate (Selangor, Malaysia). Loose fruits were kept separate and were not used in the production of low 3-MCPD ester palm fractions.

Within hours after harvesting, the fruit bunches were transported by road truck to the oil mill. Time between harvesting and onset of processing was approximately twelve hours. The oil obtained from the fruit contained in these fruit bunches had a FFA content of 0.9%, a diglyceride content of 3.8%, and the yield of oil on the total weight of the bunches was 20%. Processing at the oil mill was carried using a state of the art procedure, i.e. heating the fruit bunches with steam to inactivate the enzymes contained in the fruit, threshing to remove the empty fruit bunches, digesting, pressing and screening to separate the crude palm oil from the fruit, followed by separation of the water/oil slurry by a clarifier. In this example, the recovery stream of the clarifier which still contains some oil was not recycled.

The clarified oil was then cleaned by mixing it with water, followed by separation in a centrifuge. Finally, the crude palm oil was vacuum dried to a moisture content of less than 0.2%.

Approximately 24 hrs after pressing at the oil mill the crude oil was transported by road tankers to Golden Jomalina Food Industries Sdn Bhd, Selangor, Malaysia. At Jomalina, the road tankers where discharged in a dedicated storage tank.

The crude palm oil, having a FFA content of 0.9% and a diglyceride content of 3.8%, was then fractionated for 14 hrs into crude olein and crude stearin using DeSmet™ dry fractionation equipment with 74 ton fractionators. The characteristics of the crude palm oil feed and of the crude olein and the crude stearin are summarized in Table 1.

Table 1

<table>
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<tr>
<th></th>
<th>Crude palm oil</th>
<th>Crude palm olein</th>
<th>Crude palm stearin</th>
</tr>
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<tbody>
<tr>
<td><strong>Yield</strong></td>
<td></td>
<td>70%</td>
<td>30%</td>
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</table>
The crude palm olein was semi-continuously acid degummed with $\text{H}_3\text{PO}_4$ (0.08 wt%), followed by bleaching with 1.2% bleaching earth [Wac Supreme IB by Taiko]. Essentially the same procedure was followed for the stearin.

Both fractions were separately deodorized in a 260 ton/day semi-continuous deodorizer at 265°C and 3.5 mbar.

The refined palm olein had a 3-MCPD ester content of 0.65 ppm and the refined stearin of 0.4 ppm.

### Comparative Example A
Ripe palm fruit bunches were harvested at the Carey Island West Estate (Selangor, Malaysia). Loose fruits were included in the harvest.

Time between harvesting and onset of processing was approximately 24 hours.
The oil obtained from the fruit contained in these fruit bunches had a FFA content of 2.5%, a diglyceride content of 5.5%, and the yield of oil on the total weight of the bunches and loose fruit was 21%.

Processing at the oil mill was carried using a state of the art procedure, i.e. heating the fruit bunches with steam to inactivate the enzymes contained in the fruit, threshing to remove the empty fruit bunches, digesting, pressing and screening to separate the crude palm oil from the

<table>
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<th>3-MCPD ester content</th>
<th>&lt; detection limit</th>
<th>&lt; detection limit</th>
<th>&lt; detection limit</th>
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<tr>
<td>Iodine value</td>
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<td>1</td>
<td>1</td>
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<td>c18:1</td>
<td>38</td>
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<tr>
<td>c18:2</td>
<td>10</td>
<td>11</td>
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fruit. followed by separation of the water/oil slurry by a clarifier. In this example, the
recovery stream of the clarifier which still contains some oil was recycled
The clarified oil was then cleaned by mixing it with water, followed by separation in a
centrifuge. Finally, the crude palm oil was vacuum dried to a moisture content of less than
0.2%.

Approximately 24 hrs after pressing at the oil mill the crude oil was transported by road
tankers to Golden Jomalina Food Industries Sdn Bhd, Selangor, Malaysia.
At Jomalina, the road tankers where discharged in a dedicated storage tank.

The crude palm oil was continuously acid degummed with H₃PO₄ (0.06 wt%), followed by
bleaching with 0.8 wt% bleaching earth [Wac Supreme IB by Taiko]. The palm oil was
deodorized in a 750 ton/day semi-continuous deodorizer at 265°C and 3.5 mbar.

The refined palm oil had a 3-MCPD ester content of 3.8 ppm.

The refined palm oil, having a FFA content of less than 0.1 wt.%, was then fractionated
during 7 hrs into olein and stearin using DeSmet™/Oiltech dry fractionation equipment with
35 ton fractionators. The characteristics of the refined palm oil feed and of the refined olein
and stearin are summarized in Table 2.

Table 2

<table>
<thead>
<tr>
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<th>refined palm oil</th>
<th>refined palm olein</th>
<th>refined palm stearin</th>
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<tbody>
<tr>
<td>Yield</td>
<td></td>
<td>80</td>
<td>20</td>
</tr>
<tr>
<td>3-MCPD ester content</td>
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<td>2.2</td>
</tr>
<tr>
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</tr>
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<td>10</td>
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<td>6</td>
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</table>
CLAIMS

1. A process of manufacturing a palm oil fraction that contains virtually no 3-
monochloropropanediol fatty acid esters (3-MCPD-esters), said process comprising:
   • fractionating a crude palm oil having a free fatty acid content of less than 1.5% and a
diglyceride content of less than 5.5 wt.% to produce at least one crude palm oil
   fraction, said crude palm oil fraction being selected from crude palm olein having an
iodine value of at least 55 and crude palm stearin having an iodine value of less than
48; and
   • deodorising the crude palm oil fraction to produce a deodorised palm oil fraction
      having an 3-MCPD-ester content of less than 1 ppm.

2. Process according to claim 1, wherein the crude palm oil is produced by:
   • harvesting palm fruit;
   • subjecting the harvested fruit to a heat treatment to inactivate enzymes contained
     therein, said heat treatment taking place within 24 hours, preferably 12 hours after the
     harvesting of the fruit;
   • pressing said heat treated palm fruit to extract an oil-containing slurry; and
   • removing water and non-oleaginous solids from said oil-containing slurry to produce a
     crude palm oil having a free fatty acid content of less than 1.5%.

3. Process according to claim 1 or 2, wherein the time span from harvesting of the palm fruit
till the deodorisation of the crude palm oil fraction does not exceed 4 days.

4. Process according to any one of the preceding claims, wherein the crude palm oil fraction
is subjected to degumming prior to the deodorisation.

5. Process according to claim 4, wherein the degummed crude palm oil fraction is subjected
to bleaching prior to the deodorisation.

6. Process according to claim 5, wherein the crude palm oil fraction is bleached to produce a
bleached palm oil fraction having a Lovibond red number of less 1.5 and a Lovibond
yellow number of less than 15.
7. Process according to any one of the preceding claims, wherein the crude palm oil is deodorised at a temperature of 230-280 °C and a pressure of 0.1-4 mbar.

8. Process according to any one of the preceding claims, wherein the crude palm oil is not deodorised prior to the fractionation.

9. Process according to any one of claims 2-8, wherein water is removed from the oil-containing slurry in a clarifier.

10. Process according to claim 9, wherein the oil obtained from the decanter is dried in a vacuum drier to a moisture content of less than 0.2%.

11. A method of preparing a food product, said method comprising incorporating into said food product 3-99 wt.% of a palm oil fraction obtained by a process according to any one of the preceding claims.

12. Method according to claim 11, wherein the method comprises incorporating a palm olein obtained by a process according to any one of claims 1-10 into an infant formula as part of an oil blend, said oil blend comprising, by weight of the total amount of oil:
   • 5-50%, preferably 8-40% of the palm olein obtained by the present process; and
   • 15-60%, preferably 20-55% of a highly unsaturated oil selected from rapeseed oil, safflower oil, soybean oil, sunflower oil, fish oil and combinations thereof.

13. Method according to claim 12, wherein the method comprises incorporating a palm stearin obtained by a process according to any one of claims 1-10 into a margarine or a bakery fat.

14. Method according to claim 13, wherein the palm stearin is incorporated into a bakery margarine or a bakery fat in combination with a highly unsaturated oil selected from rapeseed oil, safflower oil, soybean oil, sunflower oil, fish oil and combinations thereof, the palm stearin being incorporated in a concentration of 20-80% and the highly unsaturated oil in a concentration of 20-80%, both percentage being calculated by weight of the total amount of oil and fat that is contained in the bakery product.
15. Method according to claim 13, wherein the palm stearin is incorporated in a margarine as part of a so called hardstock component, said hardstock component comprising an interesterified blend of 30-80% of the palm stearin and 20-70% of a lauric fat, both percentages being calculated by weight of the hardstock component.
INTERNATIONAL SEARCH REPORT

PCT/MY2010/000113

A. CLASSIFICATION OF SUBJECT MATTER

INV. C11B3/12 C11B7/00 A23L1/015

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C11B A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal , BIOSIS, COMPENDEX, FSTA, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Further documents are listed in the continuation of Box C

See patent family annex

Date of the actual completion of the international search

8 November 2010

Date of mailing of the international search report

19/11/2010

Name and mailing address of the ISA/ European Patent Office, P B 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel (+31-70) 340-2040; Fax (+31-70) 340-3016

Authorized officer

Rooney, Kevin

Form PCT/ISA/210 (second sheet) (April 2005)
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<td>MAN Y B C ET AL: &quot;Detection of lard adulteration in RBD palm olein using an electronic nose&quot;, 1 May 2005 (2005-05-01), FOOD CHEMISTRY, ELSEVIER LTD, NL LNKD-DI:10.1016/J.FOODCHEM.2004.05.062, PAGE(S) 829 - 835, XP004615599, ISSN: 0308-8146 page 830, column 2, paragraph 2 table 1</td>
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