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(71) Applicant: **ARCHER DANIELS MIDLAND COMPANY** [US/US]; Corey M. Crafton, 4666 Faries Parkway, Decatur, Illinois 62526 (US).

(72) Inventor: **HOGAN, Phil**; 12 Stirling Road, Kings Hill, West Malling Kent Me194rd (GB).

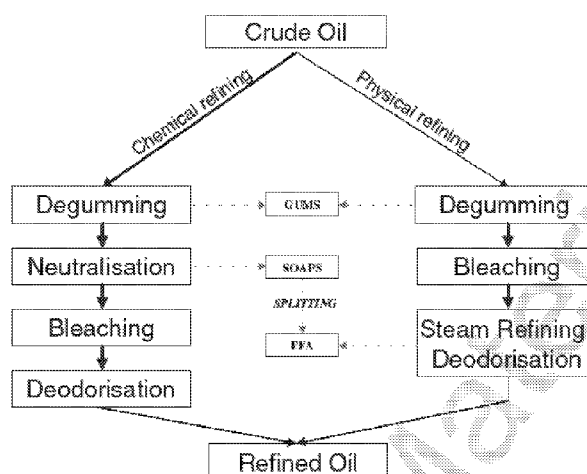
(74) Agent: **BLOOMER, Scott**; ARCHER DANIELS MIDLAND COMPANY, 4666 Faries Parkway, Decatur, Illinois 62526 (US).

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(54) Title: PROCESSED PALM OIL

Figure 1.



(57) Abstract: A central problem confronting the edible oil industry is that physical refining employed on a wide scale to process palm oil has been shown to generate the process contaminants glycidol, glycidol esters, 2-MCPD, 2-MCPD esters, 3-MCPD, and 3-MCPD esters. We disclose herein methods for reducing the process contaminants in physically refined oil by contacting the oil with steam in a packed stripping column.

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KM, ML, MR, NE, SN, TD, TG).

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PROCESSED PALM OIL

CROSS REFERENCE TO RELATED APPLICATION[S]

- 5 **[0001]** This application claims the benefit under 35 U.S.C. § 119(e) of U.S. Provisional Application Serial No. 62/345,940, filed June 6, 2016, the contents of the entirety of which is incorporated by this reference.

BACKGROUND OF THE INVENTION

- 10 **[0002]** The present invention relates to the field of edible fats and oils. More particularly, the invention relates to a method for reducing the amount of process contaminants in edible oils. Glycidol, glycidol esters (GE, also known as glycidyl esters) and monochloropropanediols, such as 3-monochloropropane-1, 2-diol (3-MCPD), 2-monochloropropane-1, 3-diol (2-MCPD), as well as esters thereof, are
15 removed from physically refined palm oil or fractions thereof by the methods of the invention.

- [0003]** The process contaminants MCPD, MCPD esters, and glycidol esters can be substantially absent through the earlier processing steps of refining crude oil into finished oil, but formed in the deodorizer or physical refining step of oil processing.
20 Matthaes and Pudel opined that the most promising way of mitigation would be to prevent the formation of the compounds or its precursors (Lipid Technology, Vol. 25, No 7, pg 151-155, 2013). The formation of MCPD esters in palm oil and rapeseed oil during oil processing steps was monitored by Franke et al. ("Influence of chemical refining process and oil type on bound 3-chloro-1, 2-propanediol contents in palm oil
25 and rapeseed oil," Food Sci. Technol. 42 (2009) 1751–1754). The Weishaar method was used to determine the content of MCPD esters in oil before refining and after laboratory hydration, degumming, neutralization, washing, drying, bleaching, and deodorizing steps. MCPD was below detection limits in rapeseed oil until after deodorization. Crude palm oil reportedly contained about 1 mg/kg liberated MCPD (although subsequent
30 reports have concluded that the analysis method used at the time was not fully correct) and remained at that level through refining until the deodorization step, where it increased to over 4 mg/kg. When physically refined palm oil was subjected to a second

bleaching and passage through a physical refining fatty acid stripper/deodorizer, the levels of 3-MCPD were lowered by bleaching but substantially increased by the second passage through the fatty acid stripper/deodorizer. This was illustrated in US patent Application Publication No. US2012238770: "Physical refining of palm oil in the control experiment caused an undesirable increase in the content of glycidyl esters in palm oil. Starting palm oil contained 0.8 ppm glycidyl esters, but when it was subjected to physical refining, the content of glycidyl esters in the palm oil increased from 0.8 ppm glycidyl esters to 15.6 ppm." The vacuum in this experiment was 3 mm Hg (4 mbar).

[0004] The German Federal Institute for Risk Assessment (BfR) reported that refined vegetable fats derived from palm oil, used for margarines and infant formula, may contain glycidol fatty acid esters (<http://www.foodnavigator.com/Product-Categories/Food-safety-and-labelling/First-hint-of-concerns-in-refined-vegetable-fats-from-palm-oil>, report dated April 24, 2009 accessed Oct. 1, 2009). Although glycidol is a known carcinogen, it is currently not known what amounts of glycidol could be released from glycidol esters during digestion. The European Food Safety Authority expert Panel on Contaminants in the Food Chain recently released a report on process contaminants. They set a tolerable daily intake (TDI) of 0.8 micrograms per kilogram of body weight per day for 3-MCPD and its fatty acid esters based on evidence linking this substance to organ damage in animal tests. However, the panel did not set a safe level for glycidol esters, concluding that sufficient evidence shows that glycidol (formed from glycidol esters) is genotoxic and carcinogenic.

[0005] Since the first report of the process contaminants glycidol/GE and MCPD/MCPD esters in edible oils, many patent applications have been filed. Although many applications focus on earlier steps, the predominant focus of the patent applications is adjustments to deodorization (including PCT Patent Application Publication No. WO2011009841, PCT Patent Application Publication No. WO2011009843, Japanese Patent Application Publication No. JP2011074358, PCT Patent Application Publication No. WO2011090240, PCT Patent Application Publication No. WO2011122184, Czech Republic Patent Application Publication No. CZ2010303281, PCT Patent Application Publication No. WO2012130744, PCT Patent Application Publication No. WO2014012548, PCT Patent Application Publication No.

WO2014012759) or treatments after deodorization (including PCT Patent Application Publication No. WO2011005081, Japanese Patent Application Publication No. JP2011074358, PCT Patent Application Publication No. WO2011040539, PCT Patent Application Publication No. WO2011069028, PCT Patent Application Publication No. WO2011090239, PCT Patent Application Publication No. WO2011074574, PCT Patent Application Publication No. WO2011074575, PCT Patent Application Publication No. WO2012107230, PCT Patent Application Publication No. WO2013093093, PCT Patent Application Publication No. WO2013145808, PCT Patent Application Publication No. WO2015057139).

- 10 **[0006]** Packed stripping columns are used in combination with deodorizer trays in conventional physical refining of palm oil to remove free fatty acids and volatile malodorous compounds from fats or oils (J. Amer. Oil Chem. Soc. 65(3) 306-311 1988; “Deodorization”, Bernardini, M. Proceedings of the World Conference on Oilseed Technology and Utilization, Thomas H. Applewhite, Editor, AOCS Press, Champaign, IL p 186-195, 1993; Oils-Fats-Lipids 1995, Proc. 21st World Congr. Intl. Soc. Fat Res., vol 1, p. 149-154 1996; British Patent GB816522, United States Patent US2508919, United States Patent US4599143, United States Patent US6001220, United States Patent US7670634, United States Patent US8951592, United States Patent US9114329, United States Patent Publication No. US201622780, PCT Patent Application No. WO215073359, European Patent EP 1505145, and European Patent EP1905816B1).
- 15 However, these disclosures focus on either standalone packed deodorizers, or the use of a packed column as a separate step before conventional deodorizer trays. The packed column portions of the deodorizers operate within a range of vacuum of 1.1 to 7 mbar. In these systems, process contaminants such as MCPD, esters thereof, glycidol, and glycidol esters are known to form in the deodorizer trays after the oil contacts steam in the packed column under a vacuum of 1.1 to 7 mbar.
- 20
- 25

[0007] The number of patent applications filed underscores the strong demand for methods to eliminate these process contaminants.

- [0008]** We have developed an efficient method for process contaminant removal. The method can be adapted to existing oil production facilities with minimal effort, or established as stand-alone processes outside an existing oil production facility. The method of the present disclosure comprises contacting physically refined palm oil
- 30

with steam and vacuum in a packed stripping column apparatus to remove process contaminants which can be generated in conventional physical refining and deodorizing.

SUMMARY OF THE INVENTION

5 **[0009]** In one aspect, the present invention relates to a method for removing process contaminants from physically refined palm oil or a fraction obtained from physically refined palm oil, the method comprising contacting the oil or fraction with steam and vacuum in a packed stripping column, wherein the process contaminants comprise at least one of 3-monochloropropanediol, 2- monochloropropanediol, glycidol,
10 monochloropropanediol esters, glycidol esters, or the sum of monochloropropanediol esters and glycidol esters.

[0010] In an additional embodiment, the amounts of process contaminants are determined by AOCS Official Method Cd 29c-13.

[0011] In an additional embodiment, the oil or fraction is contacted with steam
15 and vacuum in a packed stripping column at about 220 - 260 degrees centigrade and at a pressure of about 0.05 - 1.0 mbar.

[0012] In another embodiment, the vacuum is within the range 0.64- 0.78 mbar.

[0013] In a further embodiment, after the physically refined oil is contacted
20 with steam and vacuum in the packed stripping column, the oil is not subjected to another deodorization step.

[0014] In a still further embodiment, the fraction obtained from physically refined palm oil is selected from the group consisting of palm olein, palm stearin, and palm olein, palm stearin, second olein, third olein, palm mid-fraction, double stearin,
25 and combinations of any thereof.

[0015] In another embodiment, a reduction in the sum of MCPD esters and glycidol esters greater than 50% is obtained. In yet another embodiment a reduction in the sum of MCPD esters and glycidol esters greater than 80% is obtained.

[0016] In another embodiment a reduction in the glycidol level of greater than
30 50% is obtained. In a still further embodiment, a reduction in the glycidol level of greater than 95% is obtained.

[0017] In yet another embodiment, the residence time in the packed stripping column is less than 10 minutes. In an embodiment, the residence time in the packed stripping column is less than 5 minutes.

[0018] In an embodiment, the steam is provided at 0.5- 1.2% of the oil. In certain embodiments, the oil is provided at 15-72 kg/hour and the steam is provided at 0.3 kg/hour.

[0019] In a further embodiment, the physically refined palm oil or fraction obtained from physically refined palm oil is passed into the top of a packed stripping column, steam is passed into the bottom of the stripping column, and vacuum is applied; the steam passes upward and contacts the falling oil while the oil falls over the packing.

[0020] In a still further embodiment, the oil is allowed to pass out of the bottom of the packed stripping column and the process contaminants pass out of the packed stripping column through the vacuum outlet with the steam.

[0021] In another embodiment composition comprising a processed oil obtained by any of the above embodiments is disclosed.

BRIEF DESCRIPTION OF THE DRAWINGS

[0022] FIG. 1 depicts processing steps commonly used to process edible oil according to the prior art (From De Greyt & Kellens, Chapter 8, "Deodorization," in Bailey's Industrial Oil and Fat Products, Sixth Edition, Volume 5, p 341- 382, 2005, F. Shahidi, editor).

[0023] FIG. 2 depicts an embodiment of a packed stripping column.

DETAILED DESCRIPTION OF THE INVENTION

[0024] As used herein, "process contaminant" means one or more of 3-monochloropropanediol, 2-monochloropropanediol, esters of 3-monochloropropanediol, esters of 2-monochloropropanediol, glycidol, or glycidol esters.

[0025] As used herein, "monochloropropanediol" means a mono-chloropropane-1,2-diol and includes 3- mono-chloropropane-1,2-diol (3-MCPD) and 2-mono-chloropropane-1,2-diol (2-MCPD). MCPD is a contaminant that occurs in food in its non-esterified (diol) form.

[0026] As used herein, “MCPD esters” means a monoester or diester (with fatty acids) of MCPD. MCPD esters are contaminants that occur in food in an esterified (with fatty acids) form.

[0027] As used herein, “glycidol ester” refers to a fatty acid ester of glycidol.
5 Glycidol esters are contaminants that occur in food in an esterified (with fatty acids) form.

[0028] As used herein, “edible oil” can be selected from a vegetable oil, an animal oil, a marine oil, a microbial oil, and combinations of any thereof.

[0029] As used herein, “RBD palm oil” refers to physically refined palm oil.

10 [0030] Crude (unprocessed) edible oils can contain phospholipids, free fatty acids, lipid oxidation products, and unsaponifiable matter (including chlorophyll and carotenoid pigments, tocopherols, sterols, and hydrocarbons). Oil processing removes undesirable components through several unit operations, generally by either chemical refining or physical refining. Chemical refining is often applied to seed oils. Chemical
15 refining steps often include: (1) degumming, (2) neutralization (alkali refining), (3) bleaching, and (4) deodorization to provide edible oil (Figure 1). In chemical refining, free fatty acids present in the oil are removed primarily in the “neutralization (alkali refining)” step by neutralizing the acid with a metal ion or group 1 alkali metal ion such as sodium.

20 [0031] Alternatively, physical refining is favored for palm oil and the so-called lauric oils. Physical refining steps include: (1) degumming, (2) bleaching, (3) and steam refining/deodorization. In physical refining, bleached oil passes through a packed stripping column to remove free fatty acids by evaporation, then the oil passes through a deodorizing step in which the oil is contacted with steam to remove other contaminants
25 (Figure 1).

[0032] As used herein, “crude oil” means unprocessed oil after it has been extracted from vegetable or animal raw material. Crude oils normally need refining to render them fit for human consumption. A crude (unprocessed) oil is one that has been removed from an oil source, such as an oilseed, by one or more processing steps such as
30 extraction, pressing, extrusion, or expeller pressing. A processed oil as an oil that has been subjected to one or more of degumming, crude degumming, superdegumming, neutralizing, alkali refining, caustic refining, once refining, bleaching, fatty acid

stripping, deodorizing, hydrogenation, hardening, winterizing, fractionation, interesterification, transesterification, ester exchange, hydrolysis, alcoholysis, ester synthesis or combinations of any thereof.

[0033] A broad array of edible oils are available for processing, including
5 animal fat, beef tallow, borneo tallow, butterfat, camelina oil, candlefish oil, canola oil, castor oil, cocoa butter, cocoa butter substitutes, coconut oil, cod-liver oil, coriander oil, corn oil, cottonseed oil, flax oil, hazelnut oil, hempseed oil, herring oil, illipe fat, kokum butter, lard, mango kernel oil, marine oils, meadowfoam oil, menhaden oil, milk fat, mowrah fat, mustard oil, mutton tallow, olive oil, orange roughy oil, palm oil, palm
10 kernel oil, palm kernel olein, palm kernel stearin, palm olein, palm stearin, peanut oil, phulwara butter, pile herd oil, pork lard, rapeseed oil, rice bran oil, safflower oil, sal fat, sardine oil, sasanqua oil, shea fat, shea butter, soybean oil, sunflower seed oil, tallow, tsubaki oil, triacylglycerols, used cooking oil, vegetable oil, whale oil, white grease, yellow grease, and derivatives, conjugated derivatives, genetically-modified derivatives,
15 and mixtures of any thereof. Edible oils are often triacylglycerols, but also comprise diacylglycerols, monoacylglycerols, and phospholipids (lecithin).

[0034] Palm oil and fractions thereof are prone to the development of glycidol, GE and MCPD/MCPD esters as process contaminants. The content of process
contaminants in crude palm oil before physical refining is typically low, and may be
20 below detection limits. The exposure of oil to the elevated temperatures and/or long treatment times used in the physical refining process problematically results in the formation of process contaminants.

[0035] As used herein, "palm fraction" means a component of palm oil obtained from fractionation of palm oil. Valuable fat fractions are obtained from refined
25 palm oil by the process of fractionation, which produces fractions enriched in harder, more solid fat and fractions enriched in softer, more liquid oil. Fractionation has been described in, for example, Deffense, E. "Fractionation of Palm Oil," Journal of the American Oil Chemists' Society 62(2), 376-385 (1985). Fractionation of physically refined palm oil problematically concentrates MCPD in the more liquid fraction, called
30 an "olein" fraction. Subsequent additional fractionation steps, such as second stage, third stage, and fourth stage fractionations can concentrate the MCPD in further olein

fractions, such as second olein and third olein fractions, palm mid-fraction, and double stearin. Palm oil fractions frequently require bleaching and deodorization.

[0036] As used herein, “olein” and “stearin” refer to the two fractions obtained in a fractionation step. “Olein” refers to the fraction having a higher iodine value and
5 “stearin” refers to the fraction having a lower iodine value. Olein fractions are enriched in oil components having a lower melting point than either the unfractionated oil or the stearin fraction and are thus softer than the corresponding stearin fraction. Olein is predominantly liquid oil at a fractionation temperature, and stearin is predominantly solid oil at fractionation temperatures. Generally the iodine value of an olein fraction is
10 higher than the iodine value of the oil or fraction from which it was derived. As used herein, “palm stearin” means a palm fraction enriched in palm oil components having a higher melting point than the unfractionated palm oil from which it was obtained, or is predominantly solid oil at the filtration temperature. The iodine value of a stearin fraction is lower than the iodine value of the oil or fraction from which it was derived.
15 The yields of olein and stearin from a given filtration step can be varied within relatively broad ranges. For example, in a fractionation the yield of stearin can comprise 10- 90 weight percent of the starting material; the corresponding yield of olein will comprise 90- 10 weight percent of the starting material, so that the weights of the stearin and olein will sum to approximately the weight of the starting material. Each olein or stearin
20 fraction can be designated with a number indicating the iodine value of the fraction. Palm fractionators often develop their own unique terminology for each fraction. However, regardless of any other names ascribed to a fraction, the question of which fraction from a fractionation step is the stearin or the olein can be answered by comparing their iodine values. Palm oil fractions, such as palm olein, have been
25 categorized as food oils having high levels (greater than 4 mg/kg) of MCPD (Larsen, J. C., “3-MCPD Esters in Food Products,” Summary Report of a Workshop held in February 2009 in Brussels, Belgium, International Life Sciences Institute). Process contaminants are known to be concentrated in the valuable olein fraction.

[0037] Deodorization is often the last step in edible oil processing. In
30 deodorization, oil is heated under vacuum and steam is sparged into the hot oil while the oil is held in trays. In the deodorization process a several compounds are removed to form deodorizer distillate: odor-causing compounds, residual free fatty acids,

compounds causing off-flavors and color reversion, pesticide residues, oxidation residues, low molecular weight contaminants, hydrocarbons, some polar compounds, unsaponifiable matter, phytosterols, sterols, and some natural antioxidants (such as tocopherols). Processed oil at this stage is clear and brilliant finished oil ready for consumption, and is known as refined, bleached, deodorized (RBD) oil, physically refined (RBD) oil or fully refined oil.

[0038] As used herein, “alkali refining,” “neutralizing,” or “chemical refining” means removing free fatty acids from oil by contacting with a solution of alkali and removal of resulting fatty acid soaps that form from the bulk of triacylglycerols. Alkali refined oil may be, but not always, subsequently bleached and deodorized before being considered to be edible grade (Figure 1).

[0039] Physical refining comprises two steps, a free fatty acid stripping step and a deodorization step, which may be carried out in a single piece of equipment in which the steps are separated by the direction of oil flow; that is, oil first passed through the fatty acid stripper section, then through the deodorizer section of a single piece of equipment. Physical refining is typically carried out at temperatures of 240- 270 °C. Steam usage in deodorizing can vary depending on the type and age of the unit in use. De Greyt & Kellens (Chapter 8, “Deodorization,” in Bailey’s Industrial Oil and Fat Products, Sixth Edition, Volume 5, p 341- 382, 2005, F. Shahidi, editor) indicated that “for chemically refined oils, for example, a stripping steam consumption of 0.5–0.7% is reported as being sufficient for packed columns, compared with 1.0–1.2% for tray deodorizers. However, modern tray deodorizers today operate with even less steam, as low as 0.7–0.9%” (p.369). Physical refining is widely known to cause an increase in process contaminants.

[0040] The methods used to measure process contaminants include AOCS Official Method Cd 29c-13, the entirety of which is incorporated herein by reference. AOCS methods Cd 29a-13 and Cd 29b-13 are commonly regarded as methods giving equivalent results as are several DGF methods (DGF C VI 18 (10) part A, DGF C VI 18 (10) part B, DGF C VI 18 (10) part B modified, and DGF C VI 18 (10) part A+B), the entirety of each of which is incorporated herein by reference.

[0041] In the methods presented herein, physically refined palm oil or a fraction thereof, containing a first level of process contaminants, is admitted to the top

part of a column and steam is admitted to the bottom. The column contains inert structured packing material to increase surface area of contact between oil, vacuum, and a “carrier agent” (e.g. steam, vapors, gases). Purified oil, containing significantly reduced process contaminants leaves the column at the bottom. The oil feed is heated (1) before passing the structured package material in the vacuum column (2) and is cooled (3) down to temperatures at which no additional process contaminants are formed. Vacuum is applied to the column. Vapors and aerosols leave the top of the column and may pass into a cooling unit where stripping steam and removed process contaminants are condensed. Contaminants may react with the steam and be converted to molecules which are non-toxic at the levels remaining in the purified oil. The incoming RBD oil stream can be heated by the outlet streams of the column, simultaneously cooling the outlet stream to save energy consumption.

[0042] Physically refined oil may be piped directly from a deodorizer to the packed stripping column after the physical refining step or deodorization process step of an existing physical refining process line. Cooling or heating may be needed before oil enters the packed stripping column. Horizontal versions of the packed stripping column can be used. Alternatively, a packed stripping column can be integrated into existing oil refining deodorization units that have different size, shape or are partitioned in manifold contaminant reactive distillation units.

[0043] The present invention teaches a post- refining technology running at low operations costs for retrofitting to existing refineries, enabling oil refiners to produce oils and fats having low levels of glycidol esters or MCPD esters without decreasing the existing refining production capacities.

EXAMPLES

EXAMPLE 1

[0044] A pilot scale packed stripping column having an inner diameter of 82.5 mm was packed with Rombopak™ 6M (Kühni, from Sulzer, Winterthur, Switzerland) packing to a height of 2 meters. A stylized rendering of a similar column is shown in FIG. 2. A fraction from physically refined palm oil, Palm Olein S (ADM Hamburg, Germany, Sample #VO04/16, received March 24, 2016) was continuously fed to the top of the packed column at 12.5 – 13.5 kg/h; the liquid loading was 2560 kg/m²/hr. The oil

was heated in a heat exchanger before the packed column with a contact time of 1 minute, and the residence time in the packed stripping column was 4- 5 minutes.

Vacuum was applied at a point in the column above the packing (Table 1, Pressure column). Steam at 180 degrees C was supplied to the bottom of the column at 0.5- 1.2% of the oil for some of the tests (Table 1, Steam column). Treated oil was cooled in a heat exchanger to 115 degrees C after leaving the bottom of the column. Samples of treated oil were taken at intervals (Table 1). Deuterated internal standards were used to quantify the 3-MCPD esters and glycidol esters using the AOCS Official Method Cd 29c-13, the entirety of which is incorporated herein by reference.

10

Table 1. Packed stripping column parameters.

	Column Temp.	Oil Feed	Pressure (Top)	Pressure (bottom)	Sample time	Steam	Flow rate	Steam dose
	°C	°C	(mbar)	(mbar)	(min)		(kg/h)	(kg/h)
1	219	88	1.08	1.2	10	No	14.7	--
2	219	87.9	1.07	1.2	20	No	14.7	--
3	219	87	1.08	1.2	30	No	14.7	--
4	219	85.2	1.06	1.2	16	No	14.7	--
5	219	87.4	1.06	1.2	26	No	14.7	--
6	219	88	1.25	1.77	10	Yes	14.7	0.13
7	219	88.3	1.26	2.27	20	Yes	14.7	0.13
8	219	88	1.25	1.87	30	Yes	14.7	0.13
9	220	90	1.35	1.77	10	Yes	15.1	0.12
10	220	90.2	1.32	1.87	20	Yes	15.1	0.12
11	220	90.4	1.29	1.87	30	Yes	15.1	0.12
12	263	89.5	1.17	1.97	8	No	15.1	--
13	263	--	1.25	2.17	18	No	15.1	--
14	262	87.9	1.3	1.97	20	No	15.1	--
15	263	89	1.08	1.2	8	Yes	15.1	0.12
16	263	89.3	1.09	1.2	18	Yes	15.1	0.12
17	263	89	1.09	1.2	28	Yes	15.1	0.12

[0045] The results of the treatments outlined in Table 1 on the levels of process contaminants are shown in Table 2.

- [0046] Table 2. Removal of process contaminants under conditions outlined in Table 1 as determined according to AOCS Official Method Cd 29c-13. "S" indicates the feed palm olein S. All values are given in mg/kg.

	Steam	Sum of MCPD esters and glycidol esters	3-MCPD	Glycidol
		mg/kg (calculated as free 3-MCPD)	mg/kg (calc. as free 3-MCPD)	mg/kg (calc. as free Glycidol)
S	--	20.93	3.82	11.46
1	No	18.9	3.59	10.26
2	No	18.7	3.43	10.23
3	No	18.4	3.49	9.99
4	No	18.1	3.39	9.86
5	No	18.4	3.54	9.96
6	Yes	7.41	3.40	2.69
7	Yes	8.08	3.40	3.14
8	Yes	7.77	3.24	3.04
9	Yes	7.76	3.36	2.95
10	Yes	7.98	3.31	3.13
11	Yes	7.57	3.30	2.86
12	No	23.38	3.78	13.14
13	No	22.91	3.60	12.93
14	No	23.74	3.71	13.42
15	Yes	3.78	3.51	0.18
16	Yes	3.7	2.97	0.49
17	Yes	3.56	2.93	0.42

[0047] When physically refined (RBD) palm olein S was rapidly heated and passed quickly through the packed stripping column without steam (# 1-5 and 12-14),

the levels of process contaminants were barely reduced at 219 degrees C (tests 1-5); some levels increased at 260 °C (tests 12-14).

[0048] However, passing the palm olein through the column with steam applied above the packing caused substantial decreases in process contaminants in a temperature-related manner. These tests were carried out under bottom-column vacuum levels of 1.2-2.27 mbar. Tests run with bottom-column vacuum levels of 1.77-2.27 mbar and steam temperatures of 219-220 degrees C (tests 6-11) caused the level of process contaminants in RBD palm olein to decrease substantially. The sum of MCPD esters and glycidol esters (calculated as free 3-MCPD) decreased from 20.93 mg/kg to 7.41-8.08 mg/kg, and free glycidol (calculated as free glycidol) decreased from 11.94 mg/kg to 2.69- 3.14 mg/kg. Although this is a substantial decrease, treatments with a bottom-column vacuum level of 1.2 mbar and steam at 260 degrees C produced oil with even lower levels of process contaminants: the sum of MCPD esters and glycidol esters (calculated as free 3-MCPD) in the palm olein decreased from 20.93 mg/kg to 3.56-3.78 mg/kg, and the level of glycidol (calculated as glycidol) decreased from 11.94 mg/kg to very low levels (0.18- 0.49 mg/kg). These levels correspond to a reduction in the sum of MCPD esters and glycidol esters of greater than 80% and a reduction in the glycidol level of 95-98%.

EXAMPLE 2

[0049] A stainless steel column having an internal diameter of 200 millimeters was constructed and filled with Sulzer Mellapakplus™ to a height of 1 meter. A stylized rendering of a similar column is shown in FIG. 2. Six lots of refined, bleached deodorized palm oil (Archer Daniels Midland Co. Hamburg, Germany) were obtained and tested for the process contaminants by AOCS Official Method Cd 29c-13 and reported as in Table 3. The levels of process contaminants in the six lots before treatment are given in Table 4.

Table 3. Units reported using AOCS Official Method Cd 29c-13.

Analyte	Units
Sum of MCPD esters and glycidol esters	mg/kg (calculated as free 3-MCPD)
3-MCPD	mg/kg (calc. as free 3-MCPD)
2-MCPD	mg/kg (calc. as free 2-MCPD)

Glycidol	mg/kg (calc. as free Glycidol)
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Table 4. Process contaminants in six lots of RBD palm oil measured by AOCS Official Method Cd 29c-13. Units are mg/kg as indicated in Table 3.

Oil No.	Sum of MCPD esters and glycidol esters	3-MCPD	2-MCPD	Glycidol
1	7.4	0.7	0.4	4.5
2	43.3	4.7	1.8	25.9
3	12.7	4.4	3.0	5.6
4	23.4	3.0	1.5	13.6
5	7.6	2.2	1.1	3.6
6	22.2	3.5	2.1	12.5

- 5 **[0050]** The six lots of RBD palm oil contained unacceptable levels of process contaminants. The palm oil was subjected to steam stripping in the packed stripping column packed with Sulzer Mellapakplus™ by falling over the packing. Vacuum was pulled above the packing of the packed stripping column using a rotary vane pump and maintained within the range 0.64- 0.78 mbar. Steam (180 degrees C. 0.3 kg/hour) was
- 10 fed into the bottom of the reactor and passed out through the vacuum outlet. The column temperature was maintained within the range 254.6 – 285.3 degrees C. Oil was fed into the top of the packed stripping column at rates given in Table 5 and removed from the bottom. The contact time in the packed stripping column varied between 1 and 5 minutes depending on the flow rate of the oil.

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Table 5. Operating parameters tested in removing process contaminants from RBD palm oil in a packed stripping column. Steam (180 degrees C.) was supplied at 0.3 kg/hour.

Oil No. and Trial No.	Column	Pressure	Duration	Oil flow rate
	(°C)	(mbar)	(min)	(kg/h)
RBD PALM OIL 1				
TRIAL 1	267.7	0.67	75	no data

TRIAL 2	273.3	0.71	105	no data
TRIAL 3	280.2	0.74	135	no data
TRIAL 4	272.1	0.75	165	no data
RBD PALM OIL 2				
TRIAL 5	261.8	0.64	20	58
TRIAL 6	265.6	0.68	50	59
TRIAL 7	260.0	0.71	80	58
TRIAL 8	263.5	0.72	95	60
RBD PALM OIL 3				
TRIAL 9	254.6	0.70	60	60
RBD PALM OIL 4				
TRIAL 10	267.0	0.68	40	66
TRIAL 11	279.0	0.72	40	72
TRIAL 12	276.0	0.72	34	60
TRIAL 13	275.5	0.75	64	55
TRIAL 14	283.5	0.78	124	70
RBD PALM OIL 5				
TRIAL 15	269.9	0.72	30	15
TRIAL 16	275.0	0.75	60	15
TRIAL 17	269.9	0.74	90	16
TRIAL 18	270.2	0.70	120	15
TRIAL 19	272.9	0.69	44	15
TRIAL 20	275.0	0.68	54	15
TRIAL 21	271.8	0.66	84	15
TRIAL 22	273.4	0.69	129	15
TRIAL 23	274.0	0.64	30	15
TRIAL 24	276.0	0.75	60	15
TRIAL 25	278.7	0.78	90	15
RBD PALM OIL 6				
TRIAL 26	270.4	0.65	60	15
TRIAL 27	275.8	0.66	90	15
TRIAL 28	267.7	0.68	120	15

[0051] After falling over the packing in packed stripping column, the treated palm oil was passed through an economizer and heat exchanger that cooled the oil to less than 85 degrees C. in less than 2 minutes. The levels of process contaminants in the

5 feed oil and the treated oil are given in Table 6.

Table 6. Process contaminants in treated oil measured according to the methods in Table 3. Units are mg/kg as indicated in Table 3.

Oil No. and Trial No.	Sum of MCPD esters and glycidol esters	3-MCPD	2-MCPD	Glycidol
RBD PALM OIL 1	7.4	0.7	0.4	4.5
TRIAL 1	1.9	0.9	0.4	0.7
TRIAL 2	1.7	0.8	0.4	0.6
TRIAL 3	1.6	0.7	0.4	0.6
TRIAL 4	2.3	0.8	0.4	1.0
RBD PALM OIL 2	43.3	4.7	1.8	25.9
TRIAL 5	5.8	2.7	1.5	2.1
TRIAL 6	6.3	3.0	1.8	2.2
TRIAL 7	6.7	2.9	1.7	2.6
TRIAL 8	8.6	2.9	1.7	3.8
RBD PALM OIL 3	12.7	4.4	3.0	5.6
TRIAL 9	5.7	4.1	1.9	1.1
RBD PALM OIL 4	23.4	3.0	1.5	13.6
TRIAL 10	4.3	2.6	1.4	1.1
TRIAL 11	4.6	2.8	1.5	1.2
TRIAL 12	4.3	3.0	1.7	0.8
TRIAL 13	3.7	2.8	1.3	0.6
TRIAL 14	4.5	3.1	1.5	0.9
RBD PALM OIL 5	7.6	2.2	1.1	3.6
TRIAL 15	3.7	2.3	0.9	1.0
TRIAL 16	2.8	1.7	1.1	0.7
TRIAL 17	2.7	1.7	1.0	0.7
TRIAL 18	3.6	2.3	1.4	0.8
TRIAL 19	3.2	2.1	1.0	0.8
TRIAL 20	3.0	2.2	1.0	0.5
TRIAL 21	3.3	2.1	1.1	0.8
TRIAL 22	3.2	2.1	1.1	0.7
TRIAL 23	3.0	2.2	1.0	0.6
TRIAL 24	3.4	2.2	1.0	0.8
TRIAL 25	2.8	2.0	1.2	0.5
RBD PALM OIL 6	22.2	3.5	2.1	12.5
TRIAL 26	4.5	2.9	1.5	1.0
TRIAL 27	4.6	3.4	1.3	0.8
TRIAL 28	4.7	3.0	1.5	1.1

[0052] The packed stripping column was very effective at reducing the sum of MCPD esters and glycidol esters, and at reducing glycidol, as measured according to AOCS Official Method Cd 29c-13. The average reduction in process contaminants over the 28 trials, expressed in percent, is given in Table 7.

Table 7. Average reduction in process contaminants.

Reduction in sum of MCPD esters and glycidol esters	Reduction in 3-MCPD	Reduction in 2-MCPD	Reduction in Glycidol
70.9 %	8.8 %	4.9 %	85.7 %

[0053] The steam removed from the packed stripping column was condensed and subjected to the analysis methods given in Table 3 to confirm the removal of process contaminants (Table 8).

Table 8. Process contaminant levels in condensed stripping steam measured according to the methods in Table 3. Units are mg/kg as indicated in Table 3.

Oil No. and Trial No.	Sum of MCPD esters and Glycidol esters	3-MCPD	2-MCPD	Glycidol
Condensate from Trial 1	4107	116	30.3	2674
Condensate from Trial 17	2704	99	10.5	1745

[0054] The high levels of process contaminants in the condensate confirmed that the process contaminants were actually removed in the process and not just chemically altered or just present in a different form in the oil obtained by contacting the oil or fraction with steam and vacuum in a packed stripping column.

CLAIMS

What is claimed is:

1. A method for removing process contaminants from physically refined palm oil
5 or a fraction obtained from physically refined palm oil, the method comprising contacting the oil or fraction with steam and vacuum in a packed stripping column, wherein the process contaminants comprise at least one of 3-monochloropropanediol, 2-monochloropropanediol, glycidol, monochloropropanediol esters, glycidol esters, or the sum of monochloropropanediol esters and glycidol esters.
- 10 2. The method of claim 1, wherein the amounts of process contaminants are determined by AOCS Official Methods Cd 29a-13.
3. The method of claim 1, wherein the oil or fraction is contacted with steam and vacuum in a packed stripping column at about 220 - 260 degrees centigrade and at a pressure of about 0.05 - 1.0 mbar.
- 15 4. The method of claim 3, wherein the vacuum is within the range 0.64- 0.78 mbar.
5. The method of claim 1, wherein, after the physically refined oil is contacted with steam and vacuum in the packed stripping column, the oil is not subjected to another deodorization step.
6. The method of claim 1, wherein the fraction obtained from physically refined
20 palm oil is selected from the group consisting of palm olein, palm stearin, second olein, third olein, palm mid-fraction, double stearin, and combinations of any thereof.
7. The method of claim 1, wherein a reduction in the sum of MCPD esters and glycidol esters greater than 50% is obtained.
8. The method of claim 1, wherein a reduction in the sum of MCPD esters and
25 glycidol esters greater than 80% is obtained.
9. The method of claim 1, wherein a reduction in the glycidol level of greater than 50% is obtained.
10. The method of claim 1, wherein a reduction in the glycidol level of greater than 95% is obtained.
- 30 11. The method of claim 1, wherein the residence time in the packed stripping column is less than 10 minutes.

12. The method of claim 1, wherein the residence time in the packed stripping column is less than 5 minutes.
13. The method of claim 1, wherein the steam is provided at 0.5- 1.2% of the oil.
14. The method of claim 1, wherein the oil is provided at 15-72 kg/hour and the steam is provided at 0.3 kg/hour.
15. The method of claim 1, wherein the physically refined palm oil or fraction obtained from physically refined palm oil is passed into the top of a packed stripping column, steam is passed into the bottom of the stripping column, and vacuum is applied, wherein the steam passes upward and contacts the falling oil while the oil falls over the packing.
16. The method of claim 15, wherein the oil is allowed to pass out of the bottom of the packed stripping column and the process contaminants pass out of the packed stripping column through the vacuum outlet with the steam.
17. A composition comprising a processed oil obtained by any of claims 1-16.

Figure 1.

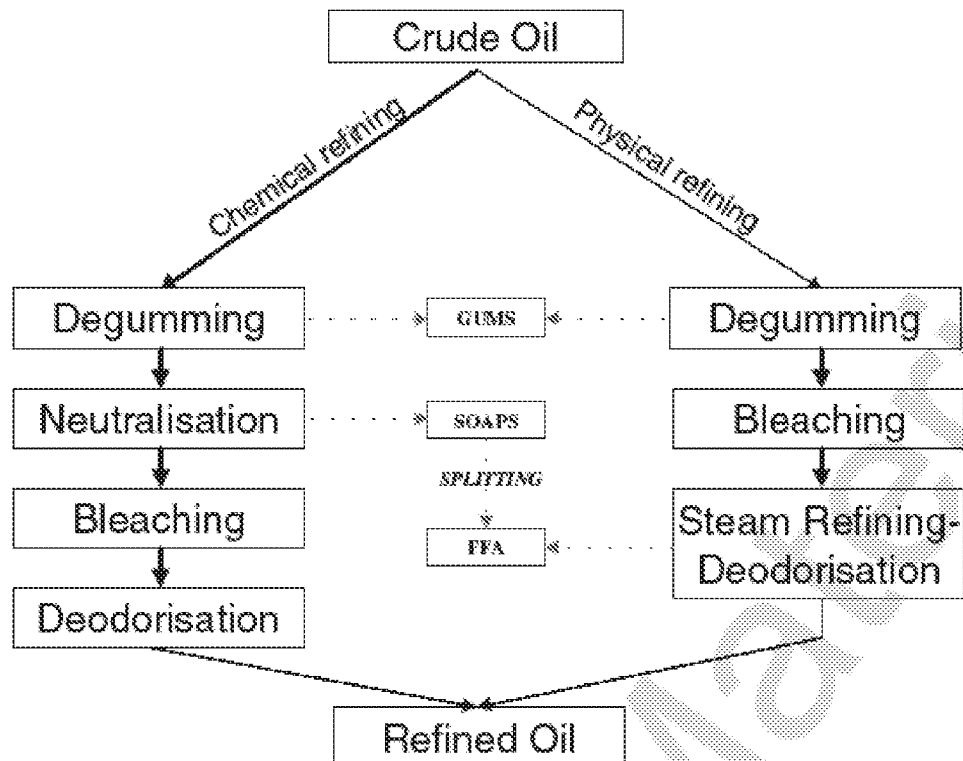
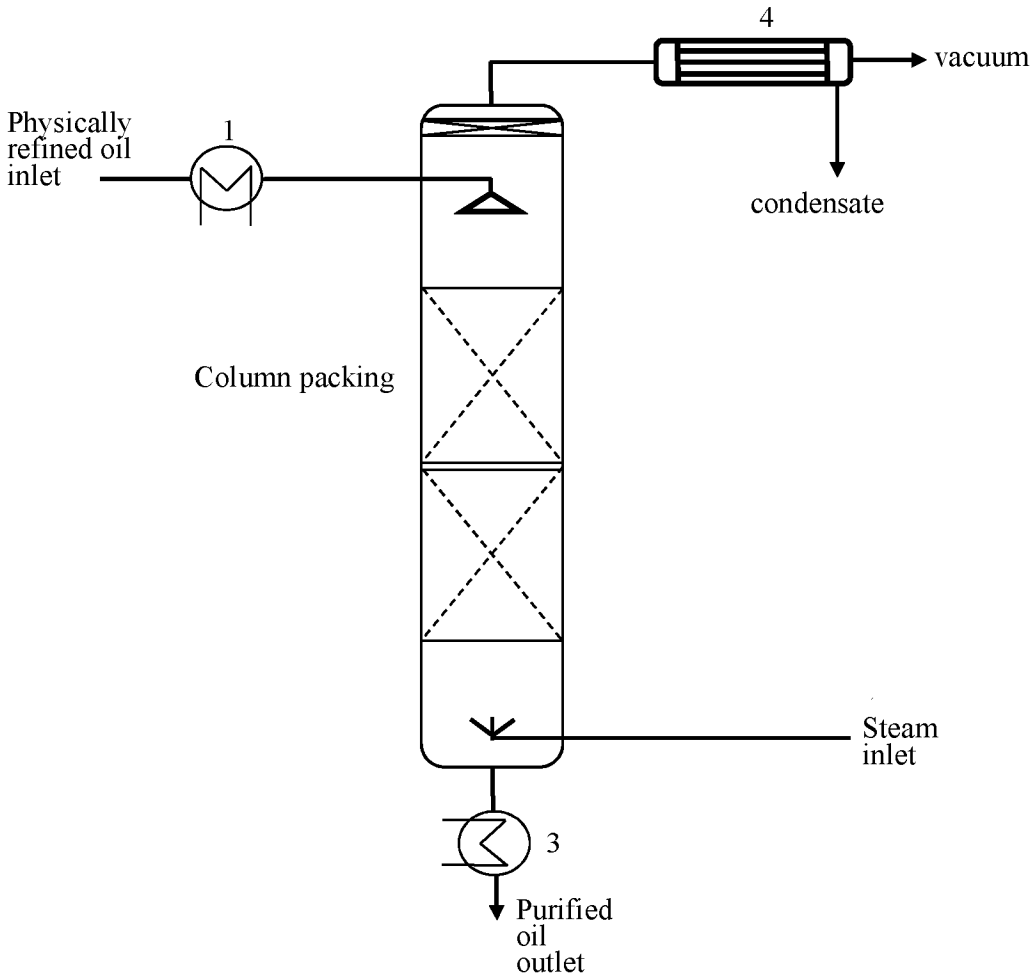


Figure 2



INTERNATIONAL SEARCH REPORT

International application No.

PCT/US2017/036052

A. CLASSIFICATION OF SUBJECT MATTER
 IPC(8) - C11B 3/10; C11B 3/00; C11B 3/08 (2017.01)
 CPC - A23L 5/20; C11B 3/00; C11B 3/14 (2017.05)

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)

See Search History document

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
 USPC - 554/175; 554/181; 554/183 (keyword delimited)

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

See Search History document

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2014/0135514 A1 (ARCHER DANIELS MIDLAND COMPANY) 15 May 2014 (15.05.2014) entire document	1-17
Y	US 2008/0081097 A1 (KELLENS et al) 03 April 2008 (03.04.2008) entire document	1-17
Y	WO 2015/174822 A1 (SIME DARBY MALAYSIA BERHAD) 19 November 2015 (19.11.2015) entire document	2
Y	US 4,601,790 A (STAGE) 22 July 1986 (22.07.1986) entire document	11, 12

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:

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Date of the actual completion of the international search

27 July 2017

Date of mailing of the international search report

16 AUG 2017

Name and mailing address of the ISA/US

Mail Stop PCT, Attn: ISA/US, Commissioner for Patents
 P.O. Box 1450, Alexandria, VA 22313-1450
 Facsimile No. 571-273-8300

Authorized officer

Blaine R. Copenheaver

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