

§ 170.225 - Part 1- Signed statements and certification

(1) Applicability of 21 C.F.R. part 170, subpart E

We submit this GRAS notice in accordance with 21 C.F.R. part 170, subpart E.

(2) Name and address of the notifier

Company: CoPack Strategies, LLC
Name: Bruce Spurlock & Amy Larson
Address: 2815 100th St, Ste 316, Des Moines, Iowa 50322
Phone: (515) 490-0618 or (515) 991-5676

(3) Name of notified substance

Corn Oil Zero (COZ)

(4) Applicable conditions of use of the notified substance

(a) Foods in which the substance is to be used

As explained in this notice, the substance is to be used in (1) all food where standard vegetable oil would be used such as baked goods, confections, etc, etc, and (2) all processes where standard vegetable oil would be used such as frying, cooking, etc, etc.

(b) Levels of use in such foods

The substance may be used in all foods and processes where vegetable oil would be typically used at levels deemed appropriate based on common uses of foods.

(c) Description of market intention

CoPack Strategies, LLC intends to market (COZ Corn Oil), corn oil derived from distiller's corn oil, as an ingredient for use in conventional foodstuffs the same as all corn oils and/or other edible oils. The standard serving size or reference amount for an edible oil is approximately 15 ml or 1 Tablespoon.*

*RACC - Reference Amounts Customarily Consumed Per Eating Occasion (21 CFR § 101.12) (U.S. FDA, 2015a) When a range of values is reported for a proposed food-use, particular foods within that food may differ with respect to their RACC.

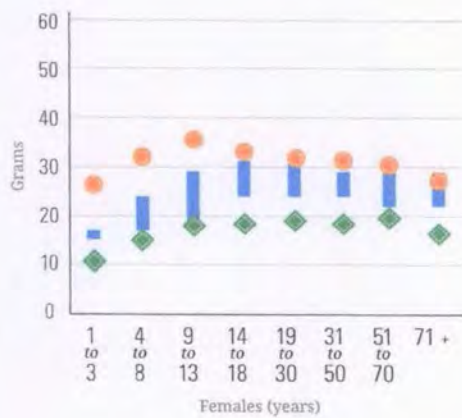
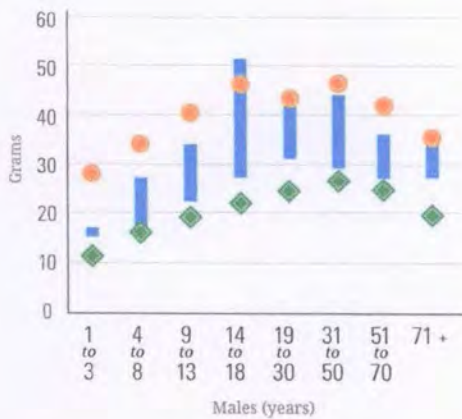
(d) Estimated Consumption of Corn Oil Based Upon Healthy Intake

Oils are fats that contain a high percentage of monounsaturated and polyunsaturated fats and are liquid at room temperature. Although they are not a food group, oils are emphasized as part of healthy eating patterns because they are the major source of essential fatty acids and vitamin E. Commonly consumed oils extracted from plants include canola, corn, olive peanut, safflower, soybean, and sunflower oils. Oils also are naturally present in nuts, seeds, seafood, olives and avocados. The fat in some tropical plants, such as coconut oil, palm kernel oil, and palm oil, are not included in the oils category because they do not resemble other oils in their composition. Specifically, they contain a higher percentage of saturated fats than other oils. The recommendation for oils in the Healthy U.S. – Style Eating Pattern at the 2,000 calorie level is 27 grams (about 5 teaspoons) per day. Oils provide essential fatty acids and vitamin E. Oils are part of healthy eating patterns, but because they are a concentrated source of calories, the amount consumed should be within the AMDR (Acceptable Macronutrient Distribution Range) for total fats without exceeding calorie limits. Oils should replace solid fats rather than being added to the diet. See Figure A. (Dietary Guidelines For Americans 2015 - 2020 Eighth Edition)

Figure A. Average Intakes of Oils & Solid Fats in Grams per Day by Age-Sex Group, in Comparison to Ranges of Recommended Intake for Oils

Average Intakes of Oils & Solid Fats in Grams per Day by Age-Sex Group, in Comparison to Ranges of Recommended Intake for Oils

■ Recommended Oils Intake Range
 ● Average Solid Fats Intake
◆ Average Oils Intake



(e) Purpose for which the substance is used

The substance is for general use in foods

(f) Description of the population expected to consume the substance

The population expected to consume the substance consists of members of the general population who consume at least one of the products described above.

(5) Basis for the GRAS determination

The statutory basis for our conclusion of GRAS status is through scientific procedures in accordance with 21 C.F.R. § 170.30(a) and (b).

(6) Exclusion from premarket approval

The notified substance is not subject to the premarket approval requirements of the FDC Act based on our conclusion that the notified substance is GRAS under the conditions of its intended use.

(7) Availability of data and information

If the Food and Drug Administration (FDA) asks to see the data and information that are the basis for our conclusion of GRAS status either during or after FDA's evaluation of our notice, we will agree to make the data and information available to FDA. Further, upon FDA's request, we will allow the Agency to review and copy the data and information during customary business hours at the above address, and will provide FDA with a complete copy of the data and information either in an electronic format that is accessible for the Agency's evaluation or on paper.

(8) Applicability of FOAI exemptions

None of the data and information in Parts 2 through 7 of our GRAS notice are exempt from disclosure under the Freedom of Information Act, 5 U.S.C. 552.

(9) Certification

We certify that to the best of our knowledge, our GRAS notice is a complete, representative, and balanced submission that includes unfavorable information, as well as favorable information, know to us and pertinent to the evaluation of the safety and GRAS status of the use of the substance.

(b) (6)

[Redacted signature]

Date 4-19-2017

(b) (6)

[Redacted signature]

Date 4-19-2017

Amy Larson

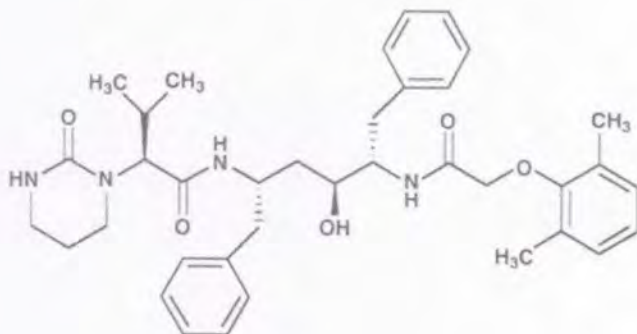
List of Acronyms

CDS	Condensed Distiller's Solubles
CO1	Corn Oil One
COA	Certificate of Analysis
COZ	Corn Oil Zero
CVD	Cardiovascular disease
DCO	Distiller's Corn Oil
DDG	Dried Distiller's Grains
DDGS	Dried Distiller's Grains with Soluble
FAGE	Fatty-Acid Glycerin Esters
FCC	Food Chemical Codex
FFA	Free Fatty Acids
IOM	Investigating Operations Manual
LOD	Limit of Detection
ND	Not Detected
PUFA	Polyunsaturated fats
RBD	Refine, Bleach, Deodorize
TDS	Total Diet Study

§ 170.230 - Part 2 -Identity, method of manufacture, specifications, and physical or technical effect

- (1) Identity of the notified substance
- (a) Common or Usual name
Maize Oil; Corn Oil; Vegetable Oil
- (b) Chemical Abstracts Service (CAS) Registry Number
8001-30-7
- (c) Molecular Weight is 0
- (d) U.S. EPA PC Code
800034
- (e) CA DPR Chemical Code
3626
- (f) Chemical Class
Oil-Vegetable
- (g) Molecular Structure - See Figure B.

Figure B. Molecular Structure of Corn Oil



(h) Characteristic properties

COZ Corn Oil was analyzed for additional parameters including lead, arsenic, moisture & volatiles, insoluble impurities, unsaponifiable matter, MIU, etc., as outlined in Table I.

Table I

<u>STANDARD CORN OIL SPECIFICATIONS</u>	<u>COZ CORN OIL</u>
Arsenic - Not more than 0.5 mg/kg	<0.10 mg/kg
Lead - Not more than 0.1 mg/kg	<0.1 mg/kg
Water - Not more than 0.1%	0.03%
Unsaponifiable Matter - Not more than 1.5%	1.32%
MIU - Total	1.39%
Cold Test, 0°C - Pass	Pass, > 8 hours

COZ Corn Oil was analyzed for residual solvents such as ethanol and methanol which is the basis of the residual solvent specification of <1,000 ppm for ethanol and <40 ppm for methanol (Table II).

Table II.

<u>STANDARD CORN OIL SPECIFICATIONS</u>	<u>COZ CORN OIL</u>
Ethanol Residual - Not more than 1,000 ppm	<10 ppm
Methanol - Not more than 40 ppm	<5 ppm

In addition, Table III lists the primary specifications for corn oil that have been adopted by the Committee on Food Chemicals Codex of the National Academy of Sciences/National Research Council. (Food Chemicals Codex, 2003)

Table III.

<u>FCC SPECIFICATIONS FOR REFINED CORN OIL</u>	<u>COZ CORN OIL</u>
Arsenic - Not more than 0.5 mg/kg	<0.10 mg/kg
Color - Not more than 5.0 red	1.8R
Free Fatty Acids - Not more than 0.10%	≤ 0.05%
Iodine Value - 120-130	122.3
Lead - Not more than 0.1 mg/kg	<0.1 mg/kg
Linolenic Acid - Not more than 2.0%	0.95%
Peroxide Value - Not more than 10 meq/kg	1.2 meq/kg
Unsaponifiable Matter - Not more than 1.5%	1.32%
Water - Not more than 0.1%	0.03%

Determining the quality, purity, and health benefits of edible oils requires a number of evaluations. One of the most important, being fatty acid analysis. Since some fatty acids have more health benefits than others, it is essential to know the full fatty acid makeup of an oil. (Kunitsky, 2014) Table IV provides a comprehensive look at the similarities between COZ Corn Oil and Target Brand Corn Oil and their individual fatty acids profile. The two fatty acid profiles were performed at two different times; hence some fatty acids were not part of the analysis at that particular time and were stated as “n/a.”

Table IV.

FATTY ACID PROFILE	COZ CORN OIL	TARGET™ CORN OIL
C4:0 (Butyric Acid)	<0.01%	<0.01%
C6:0 (Caproic acid)	<0.01%	<0.01%
C8:0 (Caprylic acid)	<0.01%	<0.01%
C10:0 Capric acid)	<0.01%	<0.01%
C11:0 (Undecanoic acid)	<0.01%	<0.01%
C12:0 (Lauric Acid)	0.01%	<0.01%
C14:0 (Myristic acid)	0.04%	0.04%
C14:1 (Myristoleic acid)	<0.01%	<0.01%
C15:0 (Pentadecanoic acid)	0.02%	<0.01%
C15:1 (Pentadecenoic acid)	<0.01%	<0.01%
C16:0 (Palmitic Acid)	10.59%	10.73%
C16:1 Omega 7	0.10%	n/a
C16:1 Total (Palmitoleic Acid + isomers)	0.16%	n/a
C16:2 (Hexadecadienoic Acid)	<0.01%	n/a
C16:3 (Hexadecatrienoic Acid)	<0.01%	n/a
C16:4 (Hexadecatetraenoic Acid)	<0.01%	n/a
C17:0 (Margaric Acid)	0.06%	0.06%
C17:1 (Heptadecenoic Acid)	0.03%	0.03%
C18:0 (Stearic Acid)	1.60%	1.63%
C18:1 (Vaccenic Acid)	0.61%	n/a
C18:1 Omega 9 (Oleic Acid)	25.69%	27.26%
C18:1, Total (Oleic Acid + isomers)	26.41%	27.84%
C18:2 Omega 6 (Linoleic Acid)	51.14%	50.69%
C18:2, Total (Linoleic Acid + isomers)	51.66%	50.87%
C18:3 Omega 3 (Alpha Linolenic Acid)	0.95%	0.91%
C18:3 Omega 6 (Gamma Linolenic Acid)	<0.01%	<0.01%
C18:3, Total (Linolenic Acid + isomers)	0.95%	0.91%
C18:4 Omega 3 (Octadecatetraenoic Acid)	<0.01%	<0.01%
C18:4 Total (Octadecatetraenoic Acid)	<0.01%	<0.01%
C20:0 (Arachidic Acid)	0.36%	0.37%
C20:1 Omega 9 (Gondoic Acid)	0.28%	0.23%
C20:1 Total (Gondoic Acid + isomers)	0.40%	n/a
C20:2 Omega 6	0.04%	0.03%
C20:2 Total (Eicosadienoic Acid)	0.04%	0.03%
C20:3 Omega 3	<0.01%	0.03%

Table IV. Continued

FATTY ACID PROFILE	COZ CORN OIL	TARGET™ CORN OIL
C20:3 Omega 6	<0.01%	<0.01%
C20:3, Total (Eicosatrienoic Acid)	<0.01%	0.03%
C20:4 Omega 3	<0.01%	<0.01%
C20:4 Omega 6 (Arachidonic Acid)	<0.01%	<0.01%
C20:4, Total (Eicosatetraenoic Acid)	<0.01%	<0.01%
C20:5 Omega 3 (Eicosapentaenoic Acid)	<0.01%	<0.01%
C21:5 Omega 3 (Heneicosapentaenoic Acid)	<0.01%	<0.01%
C22:0 (Behenic Acid)	0.11%	0.013%
C22:1 Omega 9 (Erucic Acid)	<0.01%	<0.01%
C22:1 Total (Erucic Acid + isomers)	<0.01%	<0.01%
C22:2 Docosadienoic Omega 6	<0.01%	<0.01%
C22:3 Docosatrienoic, Omega 3	<0.01%	<0.01%
C22:4 Docosatetraenoic Omega 6	<0.01%	<0.01%
C22:5 Docosapentaenoic Omega 3	<0.01%	<0.01%
C22:5 Docosapentaenoic Omega 6	<0.01%	<0.01%
C22:5 Total (Docosapentaenoic Acid)	<0.01%	<0.01%
C22:6 Docosahexaenoic Omega 3	0.03%	<0.01%
C24:0 (Lignoceric Acid)	0.16%	0.17%
C24:1 Omega 9 (Nervonic Acid)	<0.01%	<0.01%
C24:1 Total (Nervonic Acid + isomers)	0.02%	0.05%
Total Omega 3 Isomers	0.98%	0.94%
Total Omega 5 Isomers	<0.01%	n/a
Total Omega 6 Isomers	51.19%	50.71%
Total Omega 7 Isomers	0.71%	n/a
Total Omega 9 Isomers	25.98%	27.51%
Total Monounsaturated Fatty Acids	26.95%	28.38%
Total Polyunsaturated Fatty Acids	52.26%	51.77%
Total Saturated Fatty Acids	12.97%	13.12%
Total Trans Fatty Acids	0.50%	0.19%
Total Fat as Triglycerides	96.90%	97.72%
Total Fatty Acids	92.68%	93.46%

(2) Method of Manufacture Overview

The production of ethanol or ethyl alcohol from starch or sugar-based feed stocks is among man's earliest ventures into value-added processing. While the basic steps remain the same, the process has been considerably refined in recent years, leading to a very efficient process. There are two production processes: wet milling and dry milling. The main difference between the two is in the initial treatment of the grain.

Importantly, companies today are involved in technological innovations such as fractionation, low heat fermentation, methane capture from landfills and biomass gasification that reduce the amount of fossil energy needed to produce ethanol and distiller's grains. The use of pre-treated or "gray water" and other efficiencies are reducing the need for fresh water in ethanol production. A host of new technologies allow ethanol producers to capture additional value by adding corn oil, recoverable carbon dioxide, corn syrup, bio-based chemicals, and other co-products to their traditional output of feed and fuel. (Ethanol Science & Technology Module 2)

Dry Milling

In dry milling, the entire corn kernel or other starchy grain is first ground into flour, which is referred to in the industry as "meal" and processed without separating out the various component parts of the grain. The meal is slurried with water to form a "mash." Enzymes are added to the mash to convert the starch to dextrose, a simple sugar. Ammonia is added for pH control and as a nutrient to the yeast.

The mash is processed in a high-temperature cooker to reduce bacteria levels ahead of fermentation. The mash is cooled and transferred to fermenters where yeast is added and the conversion of sugar to ethanol and carbon dioxide (CO₂) begins.

The fermentation process generally takes about 40 to 50 hours. During this part of the process, the mash is agitated and kept cool to facilitate the activity of the yeast. After fermentation, the resulting "beer" is transferred to distillation columns where the ethanol is separated from the remaining "stillage." The ethanol is concentrated to 190 proof using conventional distillation and then is dehydrated to approximately 200 proof in a molecular sieve system.

The anhydrous ethanol is then blended with about 5% denaturant (such as natural gasoline) to render it undrinkable and thus not subject to beverage alcohol tax. It is then ready for shipment to gasoline terminals or retailers.

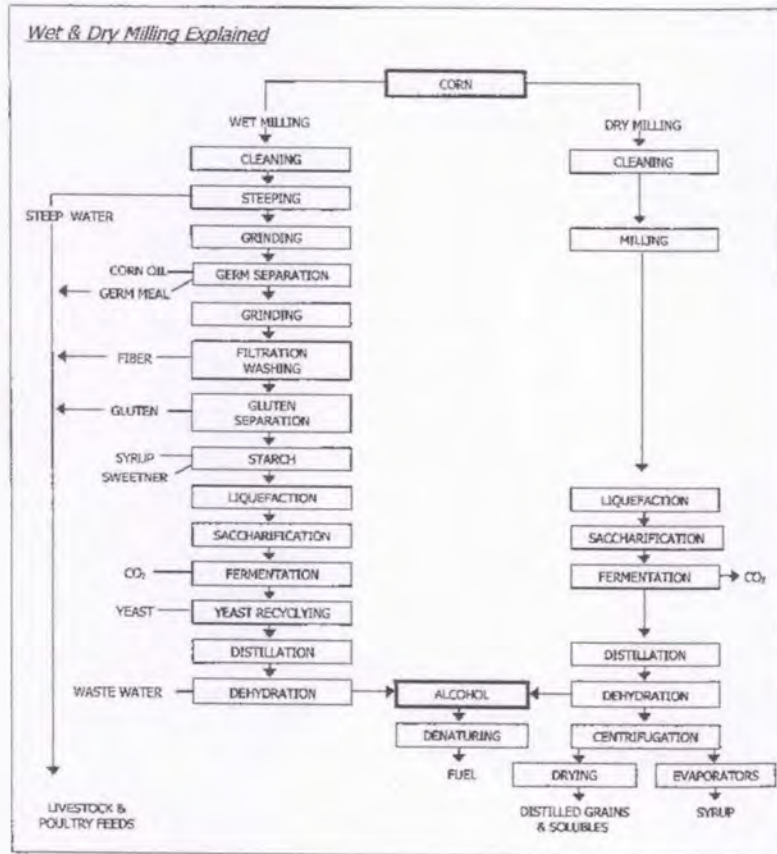
The stillage is sent through a centrifuge that separates the coarse grain from the soluble. The solubles are then concentrated to about 30% solids by evaporation, resulting in Condensed Distiller's Solubles (CDS) or "syrup." The coarse grain and the syrup are then dried together to produce dried distiller's grains with solubles (DDGS).

It also produces “DCO” Distillers Corn Oil, a product high in Free Fatty Acids (FFAs) that is very crude with high R & Y colors and contains impurities. This was almost a nuisance by-product for many years and ultimately fed to cattle as a cheap DDG alternative. Years of research went into how to use this by product or add value. (Fernando Ciaz-Royon, 2012) (Breyer, 2011)

Wet Milling

The wet-milling operation is more elaborate because the grain must be separated into its components. After milling, the corn is heated in a solution of water and sulfur dioxide for 24 to 48 hours to loosen the germ and the hull fiber. The germ is then removed from the kernel, and corn oil is extracted from the germ. The remaining germ meal is added to the hulls and fiber to form corn gluten feed. A high-protein portion of the kernel called gluten is separated and becomes corn gluten meal which is used for animal feed. In wet-milling, only the starch is fermented, unlike dry-milling, when the entire mash is fermented. See Figure C. (Ethanol Science & Technology Module 2)

Figure C. Wet & Dry Milling Explained



(3) Corn Oil One (CO1™) Patented Process

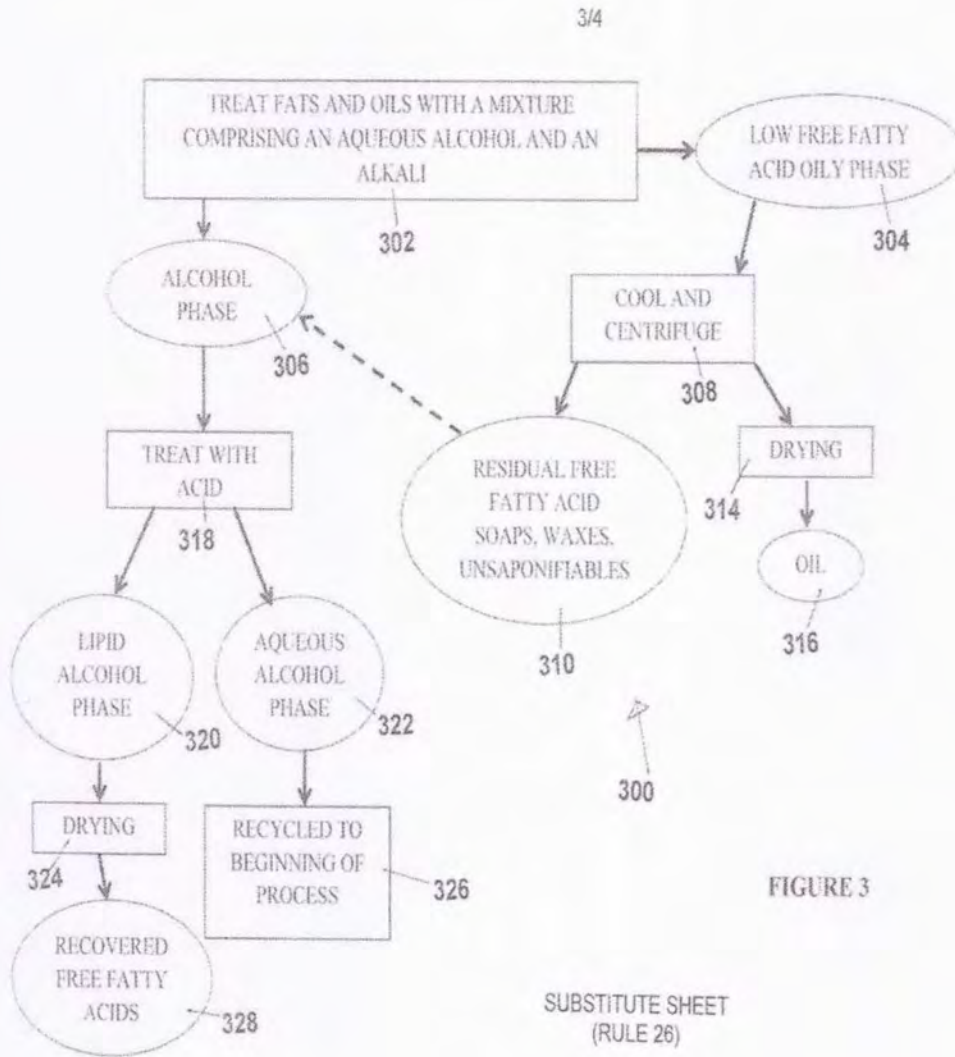
Corn Oil One (CO1™) Patented process was developed by Mohan Dasari and Kidron Knox. (Knox, 2014) See Appendix A.

Background

Some fats and oils contain high free fatty acid content, included but not limited to corn oil and waste fats and oils. As is generally known in the art, fats and oils containing a high percentage of free fatty acids are undesirable. For example, free fatty acids decrease the oxidative stability of oil. Previous methods include the refining of crude oils, which generally result in oil of low free fatty acid content. The crude oil, which has low free fatty acid content is purified by converting the fatty acids to soaps using caustic or alkali and then separates the free fatty acid soaps, commonly referred as soapstock, from the oil. The soapstock is then treated as a waste product or used for animal feed and soap manufacturing. These methods fail to capitalize on the potential of free fatty acids as a valuable product within the fats and oil industry.

Detailed in our patent diagram (See Figure D), number 3, shows the patented process used to convert DCOs (Distiller's Corn Oil) into Corn Oil One (CO1™), a refined corn oil that delivers all the advantages of soybeans. This system transforms a crude commodity into a valuable feedstock for the biodiesel industry. For example, recovering free fatty acids may be used in feed fat supplements and to manufacture industrial products. Moreover, previous methods lead to the formation of an emulsion that entraps neutral oil, thus resulting in a high neutral oil loss. The neutral oil loss is exacerbated in the case of waste fats and oils due to the presence of high free fatty acid content. This is problematic because neutral oil is a valuable product. Accordingly, an ideal method will minimize neutral oil loss. The patented process reduces the color drastically from a very deep dark red to a more desirable color similar to other retail oils. Corn Oil One (CO1™) is consumable in its current refined step but it would not be very palatable due to the amount of waxes in the oil. Figure 3 of the 4 processes used in Corn Oil One (CO1™) patented process shows the treatments of Aqueous Alcohol and an Alkali into the Alcohol phase. It is then treated with acid "318" and ultimately dries "314" refining Distiller's Corn Oil (DCO) into a lower free fatty acid. We have adjusted the process slightly by not centrifuging "308" and just separating the product. The end result is Corn Oil One (CO1™), a low FFA (free fatty acid) product that is much more manageable.

Figure D. Flow Diagram – CO1™ Patented Process



(4) Corn Oil One (CO1™) to Corn Oil Zero (COZ) Method of Manufacture & Overview

Corn Oil One (CO1™) is further refined by removing waxes (winterizing), and refined, bleached and deodorized (RBD) into COZ (corn oil derived from distiller's corn oil.) It is impossible to consume crude vegetable oil in their beginning state. The Corn Oil One process removes FFAs (free fatty acids) and makes it a much more usable product as we strive for a RBD (refined, bleached & deodorized) oil that meets standard corn oil specifications. The Winterization step for Corn Oil One (CO1™) is exactly the same as normal stems as outlined in the CYTA Journal of food. (Nergiz, 2015)

Vegetable Oil Refining

Vegetable Oil Refining is necessary for vegetable oils & fats or animal oils & fats so they can be used for cooking and frying foods. Chemically speaking, oils are Tri-Glycerides or "Fatty-Acid Glycerin Esters" (FAGE). This commonly termed as TriAcylGlyceride or TAG. One Glycerin molecule combines with three Fatty-Acid molecules to form one FAGE. Oils are a blend of different types of FAGES. The Oils can be differentiated on the basis of the length of carbon chain in the Fatty Acids and the number and location of double bonds in the carbon chains of the Fatty Acids. Greater the number of double bonds between two carbon atoms, the greater is the extent of "unsaturated" fatty-acids in the fatty-acid profile of the oils. "Unsaturated" oils and fats are considered more desirable than "saturated" oils and fats from health point of view.

Why is Vegetable Oil Refined?

Vegetable and Animal Oils and Fats have impurities such as Moisture, Solids (Insoluble), Gums (Lecithins), Free-Fatty Acids (FFA), Waxes, and Compounds of Sodium, Potassium, Magnesium, Calcium, and other metals. These impurities must be removed to improve the functionality of the oils. Other characteristics of oils (such as color, odor, and taste) are also considered impurities by modern consumers. These impurities are removed in a series of steps such as degumming (to remove gums), neutralizing (to remove FFA), bleaching (to remove color), deodorizing (to remove odor and taste), and dewaxing or winterization (to remove waxes). Further more, crude vegetable oils are refined to remove gums, Free Fatty Acids (FFA), color bodies such as chlorophylls and beta carotenes, oil decomposition compounds, trace metals and odor bearing compounds.

What is RBD Oil?

Refined Vegetable Oil is commonly called Refined, Bleached, and Deodorized (RBD) oil. As described earlier, these are the three main steps in the refining process; although additional steps are required in most cases.

Chemical Refining

In Chemical Refining, Vegetable Oil is treated with caustic lye for separation of free fatty acids from oil. This is a conventional process that can be applied to all oils. A byproduct of alkali refining is soap-stock, which is used for manufacture of low quality washing soap. Alternately, the soap-stock can be treated with acid to recover fatty acids. The waste-water from refinery requires extensive treatment. This can be avoided by alternate Physical Refining process.

Physical Refining

In Physical Refining, Vegetable Oil is subject to distillation to remove free fatty acids. The alkali treatment is completely avoided. This reduces the amount of waste water and eliminates production of soap. This process is becoming more popular. Physical refining involve 1) Bleaching, and 2) Steam distillation or Deodorization.

Degumming

The purpose of Degumming Vegetable Oils is to remove Gums. All oils have hydratable and non-hydratable gums.

- a. Water Degumming: Hydratable gums are removed by treating oils with water and separating the gums. The gums can be dried to produce lecithin.
- b. Acid Degumming: Non-Hydratable gums are removed by treating oils with acids and separating the gums.
- c. Commercial Lecithin for food application is made from crude soybean oil only. Some products are made from canola oil but they are not that popular. The phospholipids or gums from other oil can be used as industrial lubricants. They primarily go into animal feed.

Neutralizing

The purpose of Neutralizing Vegetable Oils is to remove Free-Fatty Acids (FFAs). Traditionally, FFAs are treated with caustic soda (NaOH). The reaction produces Soaps which are separated from the oil. Because trace amounts of soaps remain in the oil, the oil is either washed with water or treated with Silica.

Some processors prefer not to perform caustic neutralizing. Instead, they prefer Physical Refining is performed on oils that have high FFAs but low gum or phospholipids. These oils are bleached and then processed through FFA stripper and then deodorized.

Physical Refining process is preferred because (a) it does not produce soaps; (b) it recovers fatty acids that provide better cost recovery; (c) there is smaller yield loss compared to caustic refining—especially for oils with higher FFAs; (d) it is a chemical-

free process; and (e) it reduces excess refining loss of caustic refining so the FFA can be recovered better; and (f) there is no plant effluent that has high biological oxygen demand (BOD), and chemical oxygen demand (COD).

Bleaching

The purpose of Bleaching is to remove color pigments contained in Vegetable Oils. The oil is treated with Bleaching Clays that adsorb the color pigments. The clay is filtered and the clean bleached oil is stored for further processing. Furthermore, bleaching reduces trace impurities, such as metals and oil breakdown products, hydrocarbons, etc.; reduces color bodies such as chlorophyll, beta-carotenes and other color bodies; and reduced phospholipids.

Deodorizing

The purpose of Deodorizing Vegetable Oils is to remove odor substances. The oil is subjected to steam distillation under high temperature and vacuum to evaporate all odor substances. The resulting deodorized oil is almost bland and tasteless. Specifically, deodorization reduces FFAs, reduces and removes odor bearing compounds, and heat bleached compounds bearing red color such as beta-carotenes and other chromophoric compounds. Deodorization does not reduce chlorophylls.

FFA Stripping

As mentioned previously, the Deodorizing equipment can be modified to achieve Physical Refining. Since the conditions for deodorization and physical refining are almost identical, the modified equipment can be employed to achieve deodorization as well as physical refining.

Fatty-Acid Recovery Fatty-Acids are evaporated (distilled) in FFA Stripping. The Fatty-Acid vapors are condensed by direct contact with liquid Fatty-Acids to recover the evaporated Fatty-Acids. The distillation and condensation of Fatty-Acids is accomplished under very high vacuum to lower the boiling point of Fatty-Acids and prevent them from oxidizing.

Dewaxing/Winterizing

The purpose of Winterization Vegetable Oils is to remove waxes especially in those oils that contain waxes. Such oils are subjected to chilling and filtration to remove waxes and other high melting point substances. Winterizing is also used to separate the saturated fats from unsaturated fats—especially in Palm Oil or other saturated fats. The chilling process solidifies the saturated fats; thus enabling separation via filtration. The process of removing saturated fat fraction from chilled oil is normally called Dry Fractionation Process.

Hydrogenation Vegetable Oils have varying degrees of saturation levels. Some food products can only be prepared in saturated oils & fats. Hence, some food companies hydrogenate vegetable oils to produce desired level of saturation in the Oils & Fats. Vegetable oils can be hydrogenated under high temperature and pressure in the presence of a catalyst. Baking requires solid fats of different melting characteristics. This can be achieved in seed oils via hydrogenation process where the unsaturated fatty acids in the oil are converted to saturated fatty acids. Hydrogenation process is carried out at elevated temperatures and pressure ranging from atmospheric to several atmospheres (depending on the characteristics required in the hydrogenated oil).

Bakery Fat

The bakery industry uses hydrogenated oil (i.e., shortening). However, the shortening for bakery application should be of smooth texture. Granular texture, while desired in vanaspatighee, leads to bleeding of fat in bakery products. Hence, hydrogenated oil is cooled under controlled conditions to get a product with smooth plastic texture suitable for the bakery industry. Technochem manufactures scraped-surface heat-exchanger to produce bakery fat.

Lecithin Gums (Lecithin) present in Vegetable Oils are valuable emulsifier and an additive in prepared foods. Hence, it may be profitable to recover them for sale to food companies. The degumming process described above produces a mixture of gums, oil, and water. The mixture of gums, oil, and water is introduced in a thin-film evaporator operating under vacuum to remove water. The resulting dry mixture of gums and oil is sold as crude dry gums or lecithin. The main design concerns are that the evaporation of water should be quick and take place under higher vacuum to prevent charring and discoloring of gums.

Soap-Splitting (Acidulation)

The caustic neutralization process described above produces Soap Stock—a mixture of Soaps (Fatty-Acid Salts), Oil, and Water. Soap Stock, by itself, is a low value by-product—usually a waste product. However, it contains considerable amount of Fatty Matter, which may be profitably recovered for sale as a higher-value product. This is accomplished by converting Soaps into Fatty Acids by reacting with an Acid. The resulting product is a mixture of Fatty Acids and Oil (commonly called Acid Oil). (TechnoChem, 2016)

Glycerolysis

The physical refining process described above produces Fatty Acid Distillate—a mixture of Fatty Acids and Oil. The value of the Distillate may be enhanced by converting Fatty Acids into Oils through Glycerolysis. This is accomplished by reacting Fatty Acids with Glycerin to produce Oil. The resulting product is dried to recover oil for sale as a higher-value product.

(5) Final Method of Manufacture of Corn Oil Zero (COZ)

(COZ Corn Oil), corn oil derived from distiller's corn oil, is the end product that meets current industry standards for corn oil. COZ corn oil meets the standard identity of corn oil, analytically and chemically, and would therefore be a substitute for corn oil. All processing aids used in the manufacture of Corn Oil One are used in compliance with appropriate federal regulations. There are also two University studies that extensively tested the corn oil for sensory characteristics as compared to retail corn oil found on the retail shelf. In addition to all of the processes that are involved in producing COZ Corn Oil, The Orthodox Union has certified the facility in Council Bluffs, IA, Corn Oil One, to be OU kosher pareve. A copy of the OU certification is provided in Appendix B.

(6) Specifications and Analytical Data

Product specifications for corn oil derived from distiller's corn oil (COZ Corn Oil) are shown Figure E.

Figure E. Corn Oil Zero Product Specification Sheet



www.cornoilone.com

Corn Oil One, LLC
 4400 E. University Avenue
 Des Moines, IA 50317
 515-868-0030

Corn Oil One Product Data Sheet
 Reviewed SEPT 2016

FINISHED SPECIFICATION FOR DEWAXED, REFINED, BLEACHED AND DEODORIZED CORN OIL ONE=CORN OIL ZERO "COZ"

PRODUCT SPECIFICATIONS		NUTRITIONAL INFORMATION	
RED COLOR	3.0 MAX	CALORIES	860
% FREE FATTY ACIDS	.05 MAX	FAT TOTAL (GRAMS)	100
PEROXIDE VALUE (TRUCK)	.50 MAX	MOISTURE	0
PEROXIDE VALUE (RAIL)	1.0 MAX	PROTEIN	0
FILTERABLE IMPURITIES	7.0 MIN	ASH	0
% MOSTURE	.05 MAX	AVAILABLE CARBOHYDRATES	0
COLD TEST @ 32° F (HRS)	20.0 MIN	CRUDE FIBER	0
IODINE VALUE	120.0 MIN	SUGAR	0
C18:3	1.50 MAX	CALCIUM	0
FLAVOR GRADE	8.0 MIN	IRON	0
		SODIUM	0
		POTASSIUM	0
		VITAMIN A	0
		VITAMIN B-1	0
		VITAMIN B-2	0
		VITAMIN C	0
		NIACIN	0
		CHOLESTEROL	0
		SATURATES	13.00%
		cis-MONOUSATURATES	25.00%
		cis-POLYUNSATURATES	60.50%
		TRANS FATTY ACIDS	(<) 1.00%

SHELF LIFE:

The typical shelf life of bulk oil is generally 30 days after the arrival of shipment if held under optimal controlled conditions. These conditions included but are not limited to protection from light, moisture, correct storage temperature and nitrogen blanketing. Extension of shelf life can be validated through analytical testing.

Analyses of various non-consecutive lots of corn oil derived from distiller's corn oil (COZ Corn Oil) confirm that the materials produced by the manufacturing processes are consistent and comply with the product specifications. The analytical data also demonstrate the absence of any chemical impurities or microbiological contamination.

(7) Data or information bearing on physical or other technical effect

Our GRAS notice does not include data and other information bearing on physical or other technical effect because such data and other information are not necessary to demonstrate safety.

§ 170.235 - Part 3 – Dietary Exposure

(1) Introduction

Corn (Maize) is indigenous to the western hemisphere. It is suggested that corn has been cultivated by man for over 5,000 years. Corn was refined in the mid 1800's into starches, sweeteners and cooking oils. Corn oil was used or blended with animal fats for frying and baking in both the home and institutional business. Corn oil has been accepted by consumers for over 100 years. The accepted definition of corn oil (maize oil) is extracted from the germ of corn (maize). Its main use is in cooking where high smoke point makes refined oil valuable frying oil. (Benson) It is also the key ingredient in some margarine.

Corn Oil Zero (COZ) differs from traditional corn oil only in that it starts as a bi-product of the ethanol process as described in Part 2 (stated earlier in report). Corn Oil is the bi-product used in the Corn Oil One refining (CO1) process as described in Appendix C. After going through the patented refining process, we have added Dewaxing/Winterizing, and the RBD Process described in Part 2 (stated earlier in report). This creates Corn Oil Zero (COZ), a refined corn oil that meets or exceeds the common definition of corn oil. (Corn Oil Refiners Association) We have demonstrated by using chemical and analytical analysis and consumer testing that Corn Oil Zero has similar characteristics and performs similarly to corn oil.

(2) Consumption of corn oil.

Consumption of RBD (Refined, Bleached, and Deodorized Oil) - the principal food users of corn oil as a consumer or institutional product include:

Salad and cooking oil - 100% or in blends with other liquid vegetables oils

Margarine - both 100% corn oil in the oil phase or in blends with other vegetables oils

Blends of butter - 40% butter and 60% corn oil

Mayonnaise and emulsion-type salad dressings

As an oil ingredient in a variety of packaged and restaurant foods, including:

- i. Spaghetti sauce,
- ii. Potato chips and snack foods;
- iii. French fries and breaded fried foods.
- iv. Bakery mixes
- v. Frostings and whipped toppings

vi. Baked goods.

Corn Production – only a minor amount of corn oil production is blended with other vegetable oils except for corn oil in consumer packages such as those listed above where ingredient statements show oil as a part of a blend. (Corn Oil Refiners Association)

(3) Calories from Fats

In Figure 3, recommended calories broken into saturated fat, monounsaturated fat, and polyunsaturated fat as a percentage of the total. In Figure 4, it shows the number of servings recommended by calorie group. (USDA Dietary Guidelines for Americans, 2010) Fats and oil are part of a healthy diet, and it's recommended that a diet not exceed the USDA guidelines for consumption. See Figure 3 & 4.

Exposure Assessments:

- TDS (Total Diet Study) (FDA, 2006-2011) show data on elements in corn oil. The measured analyses for arsenic...zinc show no abnormal levels detected.
- Congender Data (FDA, FDA Food Born Contaminates , 2001-2003) show no abnormal levels detected. In this study, corn oil was found to contain a limit of detection of 0.01 ND, 0.041/2 limit of detection and 0.06 ND=LOD.
- Summary Data (FDA, Non-TSD Food Analyzed for PCDD/PCDFR in Corn Oil) shows no abnormal levels detected.
- Per capita dietary exposure estimates. (FDA, PCDD/DCDF Exposure Estimates point estimate approach from Non-TDS Food analyzed, 2001-2003)
- TDS Perchlorate exposure data for 05-06 vegetable oils contain not detected levels (FDA, Survey Data on Perchlorate food TDS results)

(4) Dietary Fats

Dietary fats are found in corn oil. Fats supply calories and essential fatty acids and help in the absorption of the fat-soluble vitamins A, D, E, and K. The IOM (Investigation Operations Manual FDA) established ranges for total fat intake for 1-3 years are 30-40% of calories; 4-18 years are 25-35%; and adults 19 years and older, 20-35%. (Figure 1) These ranges are associated with reduced risk of chronic diseases, such as cardiovascular disease while providing for adequate intake of essential nutrients. Total fat intake should fall within these ranges. (USDA, 2010) See Figure 1. COZ data falls within the same guidelines as normal corn oil.

(5) Key nutrient contributions

Oils provide essential fatty acids and vitamin E.

a. Oils are part of healthy eating patterns but because they are a concentrated source of calories, the amount consumed should be within the AMDR (Adult Minimum Daily Requirements) for total fats without exceeding calorie limits. Oils should replace solid fats rather than being added to the diet.

b. Intake of saturated fats should be limited to less than 10 percent of calories per day by replacing them with unsaturated fats and while keeping total dietary fats within the age-appropriate AMDR. The human body uses some saturated fats for physiological and structural functions but it makes more than enough to meet those needs. Individuals 2 years and older, therefore, have no dietary requirement for saturated fats.

(6) Saturated Fats and Unsaturated Fats

Strong and consistent evidence shows that replacing saturated fats with unsaturated fats, especially polyunsaturated fats, is associated with reduced blood levels of total cholesterol and of low-density lipoprotein-cholesterol (LDL-cholesterol). Additionally, strong and consistent evidence shows that replacing saturated fats with polyunsaturated fats is associated with a reduced risk of CVD (Cardiovascular Disease) events (heart attacks) and CVD-related deaths. Some evidence has shown that replacing saturated fats with plant sources of monounsaturated fats, such as olive oil and nuts, may be associated with a reduced risk of CVD. However, the evidence base for monounsaturated fats is not as strong as the evidence base for replacement with polyunsaturated fats. Evidence has also shown that replacing saturated fats with carbohydrates reduces blood levels of total and LDL-cholesterol, but increases blood levels of triglycerides and reduces high-density lipoprotein-cholesterol (HDL-cholesterol). Replacing total fat or saturated fats with carbohydrates is not associated with reduced risk of CVD (Cardiovascular Disease). Additional research is needed to determine whether this relationship is consistent across categories of carbohydrates (e.g., whole versus refined grains; intrinsic versus added sugars), as they may have different associations with various health outcomes. Therefore, saturated fats in the diet should be replaced with polyunsaturated and monounsaturated fats.

(7) Consideration & Consumption

The main sources of saturated fats in the U.S. diet include mixed dishes containing cheese, meat, or both, such as burgers, fries, chips, sandwiches; and tacos, pizza, rice, pasta, and grain dishes; and meat, poultry, and seafood dishes. Although some saturated fats are inherent in foods, others are added. Healthy eating patterns can accommodate

nutrient-dense foods with small amounts of saturated fats, as long as calories from saturated fats do not exceed 10 percent per day, intake of total fats remains within the AMDR, and total calorie intake remains within limits. When possible, foods high in saturated fats should be replaced with foods high in unsaturated fats, and other choices to reduce solid fats should be made. (USDA, Dietary Guidelines For Americans, 2010) COZ fat grams from all sources are in line with other RBD corn oil. Using COZ in replacement of traditional corn oil will have zero change in consumption of fats, calories vs. other corn oils.

(8) Trans Fats

Individuals should limit intake of trans fats to as low as possible by limiting foods that contain synthetic sources of trans fats, such as partially hydrogenated oils in margarines, and by limiting other solid fats. Some studies have observed an association between increased intake of trans fats and increased risk of CVD. This increased risk is due, in part, to its LDL-cholesterol-raising effect. Trans fats occur naturally in some foods and are produced in a process called hydrogenation. Hydrogenation is used by food manufacturers to make products containing unsaturated fatty acids solid at room temperature (i.e., more saturated), and therefore, more resistant to becoming spoiled or rancid. Partial hydrogenation means that some, but not all, unsaturated fatty acids are converted to saturated fatty acids; some of the unsaturated fatty acids are changed from a cis to trans configuration. Trans fatty acids produced this way are referred to as “artificial” or “industrially produced” trans fatty acids. Artificial trans fatty acids are found in the partially hydrogenated oils used in some margarines, snack foods, and prepared desserts as a replacement for saturated fatty acids. Although food manufacturers and restaurants have reduced the amounts of artificial trans fats in many foods in recent years, these fats can still be found in some processed foods, such as some desserts, microwave popcorn, frozen pizza, margarines, and coffee creamers. Naturally, ruminant (mammals) animals produce occurring trans fats, known as “natural or ruminant” trans fats. Natural trans fats are present in small quantities in dairy products and meats, and consuming fat-free or low-fat dairy products and lean meats and poultry will reduce the intake of natural trans fats from these foods. (USDA, Dietary Guidelines for Americans, 2010) COZ has the same limitations as other oils, corn oils.

Recommended intake from corn oil and saturated fats as a percent of Calories per day by Age group. (Figure 1). Recommend Macronutrient proportions by age. (Figure 2). Recommends limit of less 10 percent of total calories. Corn oil and COZ are analytically the same. Frying tests performed at two universities found good performance vs. other corn oils and in some testing was actually preferred. See Appendix D & E. See Figure 1 & 2.

(9) Side effects, Safe Dosage, and Toxic Issues

Corn oil is generally not toxic if it has been produced using cold pressed methods. If solvents were used to extract the oil, then it can lead to serious adverse health effects in the long run. Corn oil, however, has a very high amount of linoleic acid (omega-6) with very little alpha-linoleic acid (omega-3). Research has identified that a diet which is rich in omega-6 but lacking in omega-3 can lead to systemic inflammation in the body. In fact, the ratio of omega-6 to omega-3 could hold the key to inflammatory diseases like arthritis and acne. Therefore, one should make sure to eat enough omega-3 as well. It is always about balance. (Corn Oil Refiners Association)

The health benefits of any food item are derived from its nutrients. These are the nutrition facts of corn oil. The most important thing to note about an edible oil is its fatty acid composition. 100 gm of corn oil contains:

- 12.9 gm Saturated Fat
- 54.6 gm Polyunsaturated fats (PUFA)
- 27.5 gm Monounsaturated fats (MUFA)

This is quite different from olive oil, which contains 72 gm Monounsaturated Fatty Acids. (Based on Figure 1, 2 and 4) See Figure 1, 2 & 4.

(10) Vitamins and Minerals in Corn Oil

Corn Oil, COZ does not contain any vitamin or mineral except for Vitamin E, Vitamin K, and choline.

- Vitamin E – 100 gm oil contains 14.3 mg Vitamin E, which is 71 % of the Daily Value DV.
- Vitamin K – 100 gm corn oil contains 1.9 mcg (microgram) Vitamin K making 2 % of the DV.
- Choline – Corn oil contains 0.2 mg choline.

(11) Concern About Trans Fatty Acids

Corn oil is one of the few vegetable oils that contains trans- fats. However, 100 gm of corn oil contains just 0.3 gm of trans-fat. COZ contains 0.02 - 0.05 gm of trans-fat. Based on the January 1, 2006, label guidelines from the FDA, the label value for trans-fat is 0.05 or less per serving that does not require listing. The decision from COA is we will put the trans-fat number regardless of size on the Specification Data Sheet and any label we use.

(12) Aflatoxins

Testing of control, DCO, Corn Oil One, Corn Oil One Winterized, COZ (listed as RBD Corn Oil One) had no detection of aflatoxins. See Appendix F.

(13) Health Benefits

Cholesterol Lowering Effect

Corn oil has received much attention in the recent years for its ability to lower blood cholesterol. It is also being compared with olive oil to find out which oil performs better regarding health benefits. Corn oil contains phytosterols in significant amount. These sterol compounds are derived from plant sources and are similar to cholesterol in structure. Many plant sterols have powerful ability to reduce absorption of cholesterol. Some others help the body to use more cholesterol so that there is less in the serum. In this popular study, it was found that corn oil does, in fact, affect absorption of cholesterol in the body. People were fed a diet rich in cholesterol. There was much higher cholesterol absorption in corn oil from which sterols had been reduced. This effect was observed using 150 mg/kg of body weight corn oil supplementation. This implies that using corn oil in diet, like for cooking or as salad dressing, should lower LDL cholesterol unless corn oil is taken in such high amounts that its saturated fat content increases cholesterol levels. (Ostlund RE Jr, 2002)

Heart Health

Corn oil is good for health of cardiovascular system if taken within limits. Corn oil is rich in linoleic acids and other PUFAs (polyunsaturated fatty acids). A diet, which contains about 8 – 10 % of the total energy from PUFAs is good for heart health. (Dupont J1, 1990) Our regular diet usually contains only about 5 % even though we may be consuming lots of fats. This means that our diet should be rich in foods that contain PUFAs, like corn oil.

PUFAs also have LDL cholesterol lowering activity. This contributes to the earlier cholesterol lowering effect caused by phytosterols.

High Blood Pressure

Consuming polyunsaturated fatty acids lowers high blood pressure in people who are suffering from hypertension. (N Karanja) Corn oil specifically can lower blood pressure levels by about 10 %. However, as always, one should keep the consumption of fats within limits, no matter how healthy.

Corn Oil for Skin

One can apply corn oil for skin as a massage oil. It is a nice emollient, though not as good as the well-known massage oils like olive, coconut or sweet almond. Unrefined corn oil has slight corn-like aroma, which may be preferred by some people. It improves skin functioning because of the presence of linoleic acid and vitamin E in it. Corn Oil is a gentle oil, and one can use it as base oil for household products like lip balms, salves, creams and night oils. Corn oil is about 59 % linoleic acid, and because of it, this oil penetrates quickly into the skin.

Corn Oil for Hair

Corn oil is not conventionally referred to as a “hair oil.” One can use it as a hot oil treatment 1 – 2 times a week. It helps dry and undernourished hair. It conditions the hair and makes them smooth.

For Pets

Corn oil is also used for animals. It is gentle and great for their skin care. Massaging corn oil on a dog’s hair makes the coat healthier. Corn oil is also fed to horses to treat dull coat conditions. Corn oil provides them with healthy fats and more energy. However, their diet should also contain something, which is rich in omega-3 fatty acid, to balance out excess omega-6 from corn oil. (oilhealthbenefits.com/cornoil) (Corn Oil Refiners Association)

(13) Conclusions

Based on the research contained in this submission, a review by a four-person panel that reviewed the data and approved our submission, it is the conclusion of Co-Pack Strategies, LLC, a food consulting company, that Corn Oil Zero (COZ) meets or exceeds the common definition of regular corn oil, and used in a normal diet, is a healthy part of everyday life. The long path to using a bi-product of ethanol, DCO’s into a refined product that is usable in bio diesel and with further refining, as a shelf stable corn oil for consumptions in the general population and foodservice areas. It is a good source of fats in our diet and is a win for US Agriculture and the American farmer.

Futhermore, since Corn Oil Zero, can be used interchangeably with corn oil or other vegetable oils, the overall exposure to Corn Oil Zero would be essentially the same. Therefore, Corn Oil Zero would be considered a substitute for all intended uses of corn oil and thus would not affect exposure.

Figure 1. Average Intakes of Saturated Fats of Calories per Day by Age-Sex Groups

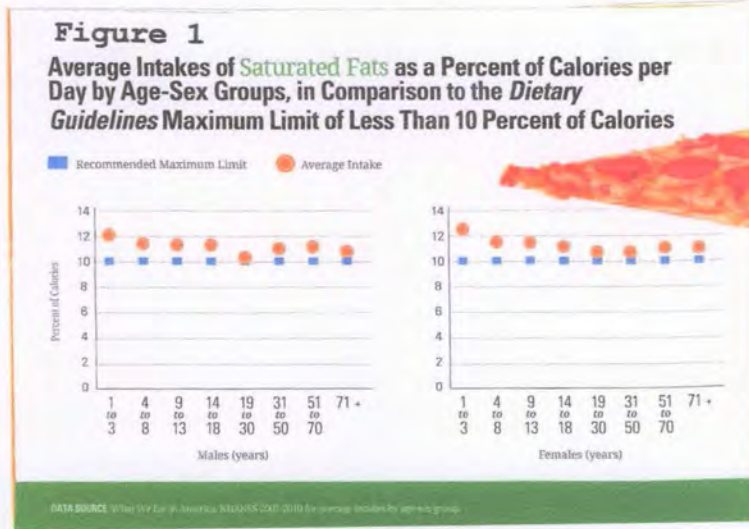


Figure 2. Recommended Macronutrient Proportions by Age

Figure 2 Recommended Macronutrient Proportions by Age

	Carbohydrate	Protein	Fat
Young children (1-3 years)	45-65%	5-20%	30-40%
Older children and adolescents (4-18 years)	45-65%	10-30%	25-35%
Adults (19 years and older)	45-65%	10-35%	20-35%

Source: Institute of Medicine. Dietary Reference Intakes for Energy, Carbohydrate, Fiber, Fat, Fatty Acids, Cholesterol, Protein, and Amino Acids. Washington (DC): The National Academies Press; 2002.

Figure 3. Fatty Acid Consumption of Fats & Oils – Saturated, Monounsaturated, Polyunsaturated

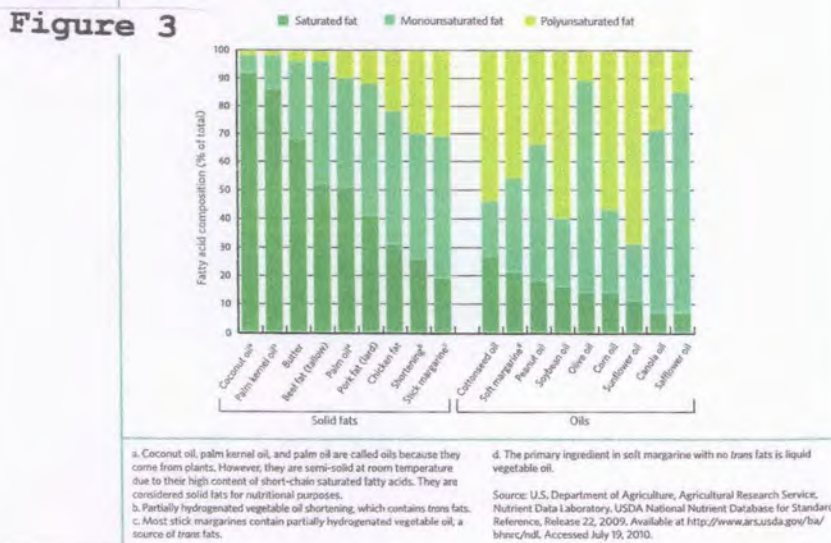


Figure 4. The number of daily servings in a food group

Figure 4

The number of daily servings in a food group vary depending on caloric needs^a

Food Group ^b	1,200 Calories	1,400 Calories	1,600 Calories	1,800 Calories	2,000 Calories	2,600 Calories	3,100 Calories	Serving Sizes
Grains	4-5	5-6	6	6	6-8	10-11	12-13	1 slice bread 1 oz dry cereal ^c ½ cup cooked rice, pasta, or cereal ^c
Vegetables	3-4	3-4	3-4	4-5	4-5	5-6	6	1 cup raw leafy vegetable ½ cup cut-up raw or cooked vegetable ½ cup vegetable juice
Fruits	3-4	4	4	4-5	4-5	5-6	6	1 medium fruit ¼ cup dried fruit ½ cup fresh, frozen, or canned fruit ½ cup fruit juice
Fat-free or low-fat milk and milk products	2-3	2-3	2-3	2-3	2-3	3	3-4	1 cup milk or yogurt 1½ oz cheese
Lean meats, poultry, and fish	3 or less	3-4 or less	3-4 or less	6 or less	6 or less	6 or less	6-9	1 oz cooked meats, poultry, or fish 1 egg
Nuts, seeds, and legumes	3 per week	3 per week	3-4 per week	4 per week	4-5 per week	1	1	½ cup or 1½ oz nuts 2 Tbsp peanut butter 2 Tbsp or ½ oz seeds ½ cup cooked legumes (dried beans, peas)
Fats and oils	1	1	2	2-3	2-3	3	4	1 tsp soft margarine 1 tsp vegetable oil 1 Tbsp mayonnaise 1 Tbsp salad dressing
Sweets and added sugars	3 or less per week	3 or less per week	3 or less per week	5 or less per week	5 or less per week	< 2	< 2	1 Tbsp sugar 1 Tbsp jelly or jam ½ cup sorbet, gelatin dessert 1 cup lemonade
Maximum sodium limit ^d	2,300 mg/day	2,300 mg/day	2,300 mg/day	2,300 mg/day	2,300 mg/day	2,300 mg/day	2,300 mg/day	

§ 170.240 - Part 4 – Self Limiting Levels of Use

The use of corn oil derived from distiller’s corn oil (COZ corn oil) is self-limiting based on the desired level for technical effect. Corn oil is a healthy edible oil which we use commonly in our cooking, like canola oil or safflower oil. Corn oil is primarily a healthy oil because it is composed mainly of polyunsaturated fatty acids (PUFAs) and low on saturated fat. Levels of COZ should not exceed the levels recommended by the FDA of corn oil. See Figure 4.

Figure 4. The number of daily servings in a food group

Figure 4

The number of daily servings in a food group vary depending on caloric needs ^a								
Food Group ^b	1,200 Calories	1,400 Calories	1,600 Calories	1,800 Calories	2,000 Calories	2,600 Calories	3,100 Calories	Serving Sizes
Grains	4-5	5-6	6	6	6-8	10-11	12-13	1 slice bread 1 oz dry cereal ^c ½ cup cooked rice, pasta, or cereal ^d
Vegetables	3-4	3-4	3-4	4-5	4-5	5-6	6	1 cup raw leafy vegetable ½ cup cut-up raw or cooked vegetable ½ cup vegetable juice
Fruits	3-4	4	4	4-5	4-5	5-6	6	1 medium fruit ¼ cup dried fruit ½ cup fresh, frozen, or canned fruit ½ cup fruit juice
Fat-free or low-fat milk and milk products	2-3	2-3	2-3	2-3	2-3	3	3-4	1 cup milk or yogurt 1½ oz cheese
Lean meats, poultry, and fish	3 or less	3-4 or less	3-4 or less	6 or less	6 or less	6 or less	6-9	1 oz cooked meats, poultry, or fish 1 egg
Nuts, seeds, and legumes	3 per week	3 per week	3-4 per week	4 per week	4-5 per week	1	1	½ cup or 1½ oz nuts 2 Tbsp peanut butter 2 Tbsp or ½ oz seeds ½ cup cooked legumes (dried beans, peas)
Fats and oils	1	1	2	2-3	2-3	3	4	1 tsp soft margarine 1 tsp vegetable oil 1 Tbsp mayonnaise 1 Tbsp salad dressing
Sweets and added sugars	3 or less per week	3 or less per week	3 or less per week	5 or less per week	5 or less per week	< 2	< 2	1 Tbsp sugar 1 Tbsp jelly or jam ½ cup sorbet, gelatin dessert 1 cup lemonade
Maximum per day	2,300 mc/day	2,300 mc/day	2,300 mc/day	2,300 mc/day	2,300 mc/day	2,300 mc/day	2,300 mc/day	

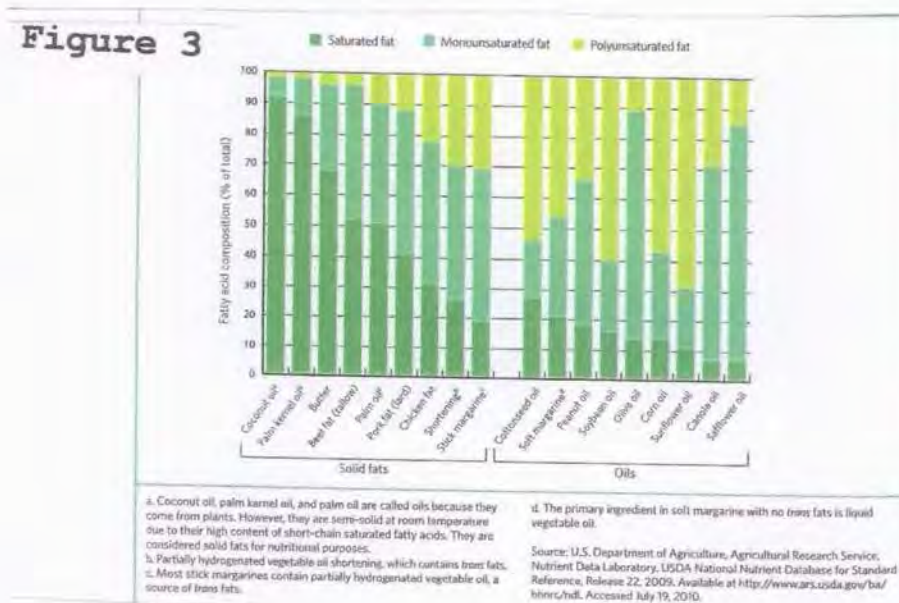
PUFAs also have LDL cholesterol lowering activity. This contributes to the earlier cholesterol lowering effect caused by phytosterols.

The health benefits of any food item are derived from its nutrients. These are the nutrition facts of corn oil. The most important thing to note about an edible oil is its fatty acid composition. 100 gm of corn oil contains:

- 12.9 gm Saturated Fat
- 54.6 gm Polyunsaturated fats (PUFA)
- 27.5 gm Monounsaturated fats (MUFA)

This is quite different from olive oil, which contains 72 grams Monounsaturated Fatty Acids. See Figure 3.

Figure 3. Fatty Acid Consumption of Fats & Oils – Saturated, Monounsaturated, Polyunsaturated



**§ 170.245 - Part 5 – Experience based on common use of food before
1958**

Edible oils are often compared by examining their fatty acid profiles. In the 1950's and 1960's, a marketing slogan for corn oil "Mazola" (Vegetables in food technology) was that it was "high in polyunsaturated", which attributed to its high content of the essential fatty acid, linoleic acid. Although corn oil is a minor cooking oil product today because of the extensive use of soy oils, in the 1930's, 40's & 50's, Mazola was the considered the king of oils, a 100% corn oil. During that time, it made multiple unsubstantiated claims, but the one correct claim was "high in polyunsaturates" and relatively low levels of saturated fats which are common in animal lard. The Invention of Crisco® shortening and Mazola® corn oil in 1911, was the first use of vegetable oils in shortening and vegetable oils. The consumer shifted to these new products because of convenience vs. health. Health was later used as reasons to use corn oil in cooking and shortening in baking. Today companies like Mazola® and ACH™ Foods use slogans like "The Plant Sterols Difference" or the comparison to extra virgin olive oil, which Mazola® lowers cholesterol better. We believe that COZ is simply corn oil, and all uses from 1911 until today, are essentially the same.

§ 170.250 - Part 6 – Narrative

The determination that corn oil derived from distiller's corn oil (COZ Corn Oil) is GRAS is based on the scientific procedures. The safety of COZ Corn Oil is based on the natural background occurrence of corn oil in the diet and an estimate of the probable consumption of corn oil as calculated using the most recent publicly-available survey of the U.S. food consumption.

- Detailed information on the notified substance
- Comprehensive analytical data
- Identity of the notified substance
- Methods of Manufacture and Patented Process
- Product Specifications
- Dietary Exposure
- Information of self-limiting levels of use
- Experience based on common use in food before 1958
- Two University sensory & analytical studies comparing COZ to another retail corn oil (Appendix D & E)

Two Universities were asked by CoPack Strategies on behalf of Corn Oil Zero (COZ) to independently evaluate Corn Oil Zero (COZ) and compare COZ to a retail private label corn oil. The private label corn oil was Hy-Vee. CoPack Strategies sent samples of COZ and Hy-Vee corn oil in their original, unopened packaging. Both Universities purchased the remaining materials, such as French fries, etc., from their local grocery stores. Both Universities were instructed to test the cooking properties, smoke points and overall quality of both corn oils with the purpose being to compare Corn Oil Zero (COZ) against another corn oil already on the market. First University study was performed at University of Texas A&M by Dr. Alam M. Phil, Ph.D., head of the Fats and Oils Program. The second University study was performed at South Dakota State University by Dr. Padmanaban Krishnam, Ph.D., Professor Food Science Program. Both University studies approached the same question in different ways but both had similar conclusions. Texas A&M University study provided in Appendix D. South Dakota State University study provided in Appendix E.

Moreover, these data were reviewed by a panel of experts, qualified by scientific training and experience to evaluate the safety of ingredients as components of food, who concluded that the intended uses of oil derived from distiller's corn oil (COZ Corn Oil) are safe and suitable and would be GRAS based on scientific procedures.

Finally, the Expert Panel convened on behalf of CoPack Strategies, LLC, independently and collectively, evaluated the data and information summarized above and concluded that the intended uses of corn oil derived from distiller's corn oil (COZ Corn Oil) produced consistently with cGMP and meeting appropriate food-grade specifications described herein, are safe and suitable. Furthermore, the Expert Panel unanimously concluded that the intended uses of corn oil derived from distiller's corn oil (COZ Corn Oil) are GRAS based on scientific procedures. It is also CoPack Strategies' opinion that other qualified and competent scientists reviewing the same publicly available information would reach the same conclusion. This is supported by the fact that numerous regulatory agencies have reviewed the safety of corn oil, and since corn oil derived from distiller's corn oil (COZ Corn Oil) meets or exceeds standard identity of corn oil and standard analytical, as stated in the product specification, concluded that it is safe for the various intended uses. Therefore, CoPack Strategies, LLC, has concluded that corn oil derived from distiller's corn oil (COZ Corn Oil) is GRAS under the intended conditions of use on the basis of scientific procedures; therefore, the ingredients are excluded from the definition of a food additive and thus may be marketed and sold for the uses designated above in the U.S. without the promulgation of a food additive regulation under Title 21 of the CFR.

§ 170.255 - Part 7 – List of Supporting Data and Information

The following references are all generally available. Data and supporting data in the Appendix are not all generally available but are attached for reference.

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Appendix A

Expert Panel Consensus Statement Concerning the Generally Recognized as Safe (GRAS) Status of Corn Oil Derived from Distiller's Corn Oil (COZ Corn Oil)

**EXPERT PANEL CONSENSUS STATEMENT CONCERNING THE GENERALLY
RECOGNIZED AS SAFE (GRAS) STATUS OF CORN OIL DERIVED FROM DISTILLER'S
CORN OIL (COZ CORN OIL)**

INTRODUCTION

At the request of CoPack Strategies, LLC, of Des Moines, IA, an Expert Panel (the "Panel") of independent scientists, qualified by their relevant national and international experience and scientific training to evaluate the safety of food ingredients, was especially convened to conduct a comprehensive evaluation of the available pertinent data and information, and determine whether, under the conditions of the intended use, COZ Corn Oil derived from distiller's corn oil, as food ingredients for us in conventional foodstuffs would be Generally Recognized as Safe (GRAS), based on scientific procedures. The Panel consisted of the below-signed qualified scientific experts: Professor Padmanaban (Padu) Krishnan, Ph.D. (South Dakota State University), Monoj K. Gupta, President, MG Edible Oil Consulting International, Inc., and Anne Elise Sherod, Student with MS in GRAS Submissions & Food Law; BS in Food Science, Edible Oil (South Dakota State University), Tong Wang, Ph.D. Professor, Director of Graduate Education for Food Science and Technology. Iowa State University.

The Panel, independently, examined a comprehensive package of scientific information and data compiled from the literature and other published sources. This information was presented in a dossier [Documentation Supporting the Evaluation of COZ Corn Oil Derived From Distiller's Corn Oil as Generally Recognized as Safe (GRAS) for Use as Food Ingredients] that was submitted by CoPack Strategies to the Panel. In addition, the Panel evaluated other information deemed appropriate or necessary, including data and information provided by CoPack Strategies, LLC. The data evaluated by the Panel included information pertaining to the method of manufacture and product specifications, analytical data, intended use level, consumption estimates, and a comprehensive assessment of the available scientific literature pertaining to the safety of corn oil derived from distiller's corn oil (COZ Corn Oil).

Following independent, comprehensive evaluation of such data and information, each Panel member, concluded that under the conditions of intended use described herein, corn oil derived from distiller's corn oil (COZ Corn Oil) meeting appropriate food-grade specifications, and manufactured in accordance with current Good Manufacturing Practice (cGMP), are GRAS based on scientific procedures. A summary of the basis for each Panel member's conclusion, excluding confidential data and information, is provided.

SUMMARY AND BASIS FOR GRAS

CoPack Strategies, LLC, intends to market corn oil derived from distiller's corn oil (COZ Corn Oil) as an ingredient for use in conventional foodstuffs the same as all corn oils and/or other edible oils. The standard serving size or reference amount for an edible oil is approximately 15 ml or 1 Tablespoon.*

*RACC - Reference Amounts Customarily Consumed Per Eating Occasion (21 CFR § 101.12) (U.S. FDA, 2015a) When a range of values is reported for a proposed food-use, particular foods within that food may differ with respect to their RACC.

Feed Energy's product derived from feed stock and converted into distiller's corn oil is manufactured using standard manufacturing practices in accordance with cGMP using stable food grade ingredients and processing aids. COZ Corn Oil is a result of converting distiller's oil, a by-product of ethanol production, into Corn Oil 1 (CO1™). Corn Oil 1 (CO1™) is winterized and followed by a final RBD (refine, bleach & deodorize) process which results in a finished, edible corn oil that is consumable. All reagents and processing aids used in the manufacture of the corn oil derived from ethanol production (COZ Corn Oil) are food-grade materials.

Numerous non-consecutive manufactured lots of corn oil derived from distiller's corn oil (COZ Corn Oil) were tested and met the proposed chemical and analytical specifications.

Oils are fats that contain a high percentage of monounsaturated and polyunsaturated fats and are liquid at room temperature. Although they are not a food group, oils are emphasized as part of healthy eating patterns because they are the major source of essential fatty acids and vitamin E. Commonly consumed oils extracted from plants include canola, corn, olive peanut, safflower, soybean and sunflower oils. Oils also are naturally present in nuts, seeds, seafood, olives and avocados. The fat in some tropical plants, such as coconut oil, palm kernel oil, and palm oil, are not included in the oils category because they do not resemble other oils in their composition. Specifically, they contain a higher percentage of saturated fats than other oils. The recommendation for oils in the Healthy U.S. –Style Eating Pattern at the 2,000 calorie level is 27 grams (about 5 teaspoons) per day. Oils provide essential fatty acids and vitamin E. Oils are part of healthy eating patterns, but because they are a concentrated source of calories, the amount consumed should be within the AMDR (Acceptable Macronutrient Distribution Range) for total fats without exceeding calorie limits. Oils should replace solid fats rather than being added to the diet. (Dietary Guidelines For Americans 2015 - 2020 Eighth Edition)

From a comprehensive evaluation of the available scientific data and information, it is concluded that the intended use of corn oil derived from distiller's corn oil (COZ Corn Oil) in traditional foodstuffs is safe. The data and information summarized in this dossier demonstrate that this product, manufactured consistent with cGMP and meeting appropriate food-grade specifications, would be GRAS based on scientific procedures under the intended conditions of use, as described herein.

CONCLUSION

Following independent, comprehensive evaluation of such data and information, each Panel member concluded that corn oil derived from distiller's corn oil (COZ Corn Oil) meets appropriate food-grade specifications, manufactured in accordance with current Good Manufacturing Practice (cGMP), are generally Recognized As Safe (GRAS) for use in Food and produced consistent with current Good Manufacturing Practices (GMP), are safe and suitable.

Each Panel member further concludes that the intended uses of corn oil derived from distiller's corn oil (COZ Corn Oil) in traditional foodstuffs, manufactured consistent with cGMP and meeting appropriate food-grade specifications presented in the supporting dossier are GRAS based on scientific procedures.

It is our opinion that other qualified experts would concur with these conclusions.

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A large rectangular area of the document is redacted with a solid grey fill, obscuring the text underneath.

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Appendix B

ORTHODOX UNION KOSHER CERTIFICATE

Established 1898



Orthodox Union

Union of Orthodox Jewish Congregations of America • איחוד קהילות האורתודוקסים באמריקה
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KASHRUTH DIVISION

MARTIN NACHIMSON
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Chairman

DAVID FUND
Vice Chairman

RABBI MENACHEM GENACK
Rabbinic Administrator, CEO

RABBI ALEXANDER S. ROSENBERG
Rabbinic Administrator (1950-1972)

April 14, 2016

TO WHOM IT MAY CONCERN:

This is to certify that the following products prepared by

Feed Energy, Pleasant Hill, IA, 50327

At the following facility:

Corn Oil One, Council Bluff, IA, 51503

are manufactured under the supervision of the Kashruth Division of the Orthodox Union.
Products designated below are OU certified kosher pareve.

- Corn Oil One

This certification is effective from April 1, 2016 through December 31, 2016, and is subject to renewal at that time.

Sincerely Yours,
UNION OF ORTHODOX JEWISH
CONGREGATIONS OF AMERICA

(b) (6)

Rabbi Dovid Jenkins
Rabbinic Coordinator

Appendix C

**METHOD TO RECOVER FREE FATTY ACIDS FROM FATS AND OILS
PATENT #WO 2014/078387 A1**



US00935331B2

(12) **United States Patent**
Dasari et al.

(10) **Patent No.:** **US 9,353,331 B2**
(45) **Date of Patent:** **May 31, 2016**

(54) **METHOD TO RECOVER FREE FATTY ACIDS FROM FATS AND OILS**

(71) Applicant: **Riverhead Resources, LLC**, Pleasant Hill, IA (US)

(72) Inventors: **Mohan Prasad A. Dasari**, West Des Moines, IA (US); **Kidron Joel Knox**, Ankeny, IA (US)

(73) Assignee: **RRIP, LLC**, Pleasant Hill, IA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 215 days.

(21) Appl. No.: **14/079,059**

(22) Filed: **Nov. 13, 2013**

(65) **Prior Publication Data**
US 2014/0135515 A1 May 15, 2014

Related U.S. Application Data

(60) Provisional application No. 61/725,598, filed on Nov. 13, 2012, provisional application No. 61/793,727, filed on Mar. 15, 2013.

(51) **Int. Cl.**
C11B 3/00 (2006.01)
C11B 3/04 (2006.01)
C11B 7/00 (2006.01)

(52) **U.S. Cl.**
CPC ... *C11B 3/04* (2013.01); *C11B 3/00* (2013.01);
C11B 7/0008 (2013.01); *C11B 7/0066* (2013.01)

(58) **Field of Classification Search**
CPC *C11B 3/04*
USPC 554/212
See application file for complete search history.

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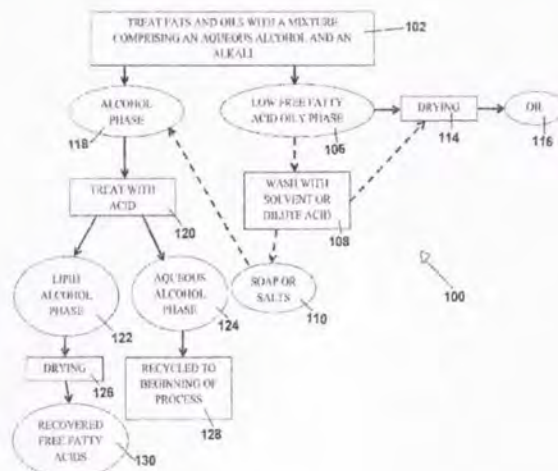
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Primary Examiner — Deborah D Carr
(74) *Attorney, Agent, or Firm* — Brick Gentry PC; Brian J. Laurenzo; Jessica L. Susie

(57) **ABSTRACT**

Methods for recovering free fatty acids from fats and oils having high free fatty acid content are provided. In the method, fats and oils are treated with a mixture having an alcohol to result in a low-free fatty acid oily phase and an alcohol phase. The mixture may also include an alkali. The alcohol may be a monohydric alcohol and an aqueous alcohol, such as an aqueous alcohol having a concentration of at least about 15% alcohol by weight. The alcohol phase may be treated with an acid to form an aqueous alcohol phase and a lipid alcohol phase, wherein the lipid alcohol phase includes free fatty acids, which may be recovered. Fats and oils amenable to such a method may include, but are not limited to, waste fats, waste oils, high acid grease, high acid tallow, and corn oil, such as corn oil produced at an ethanol production plant.

24 Claims, 4 Drawing Sheets



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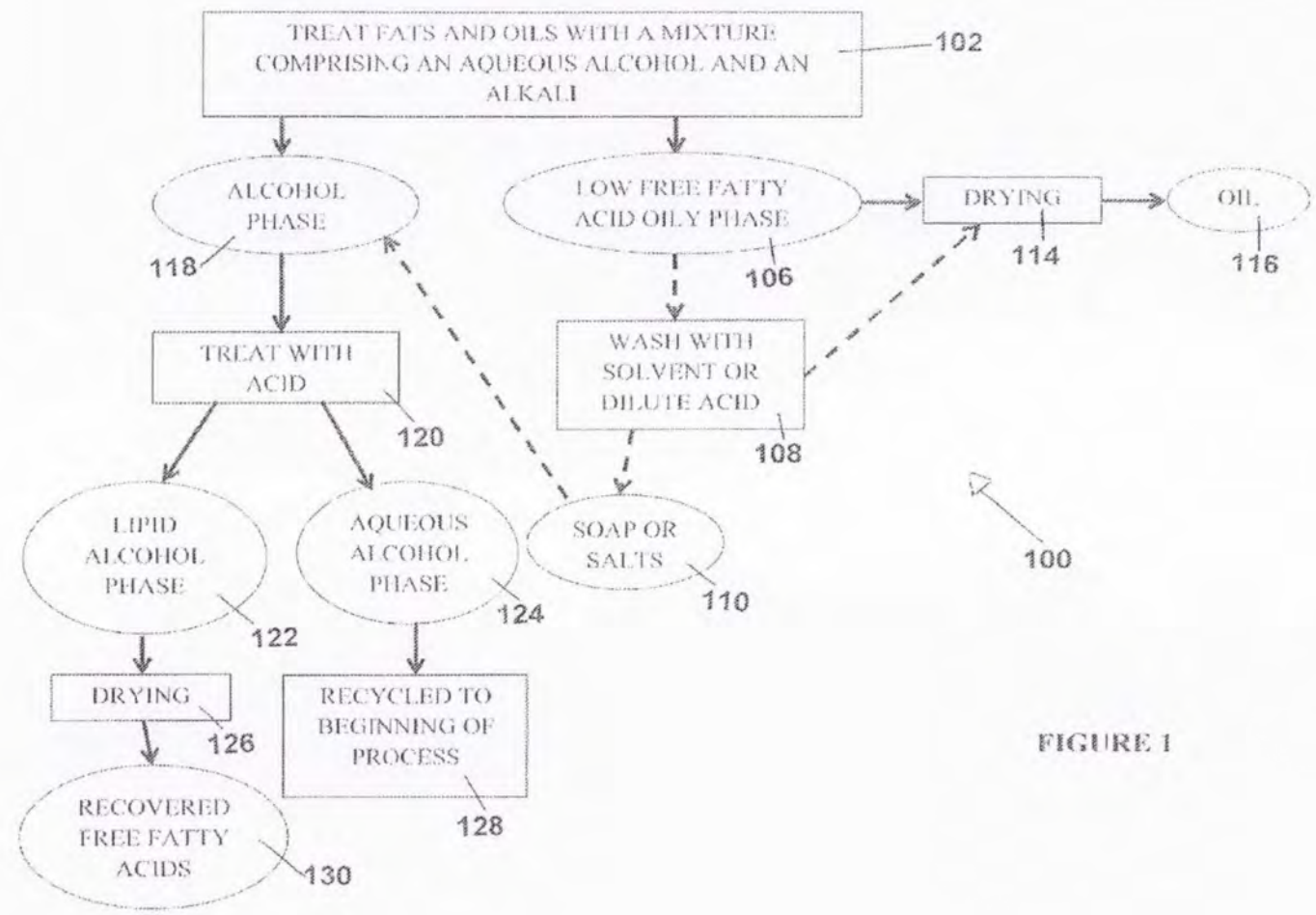


FIGURE 1

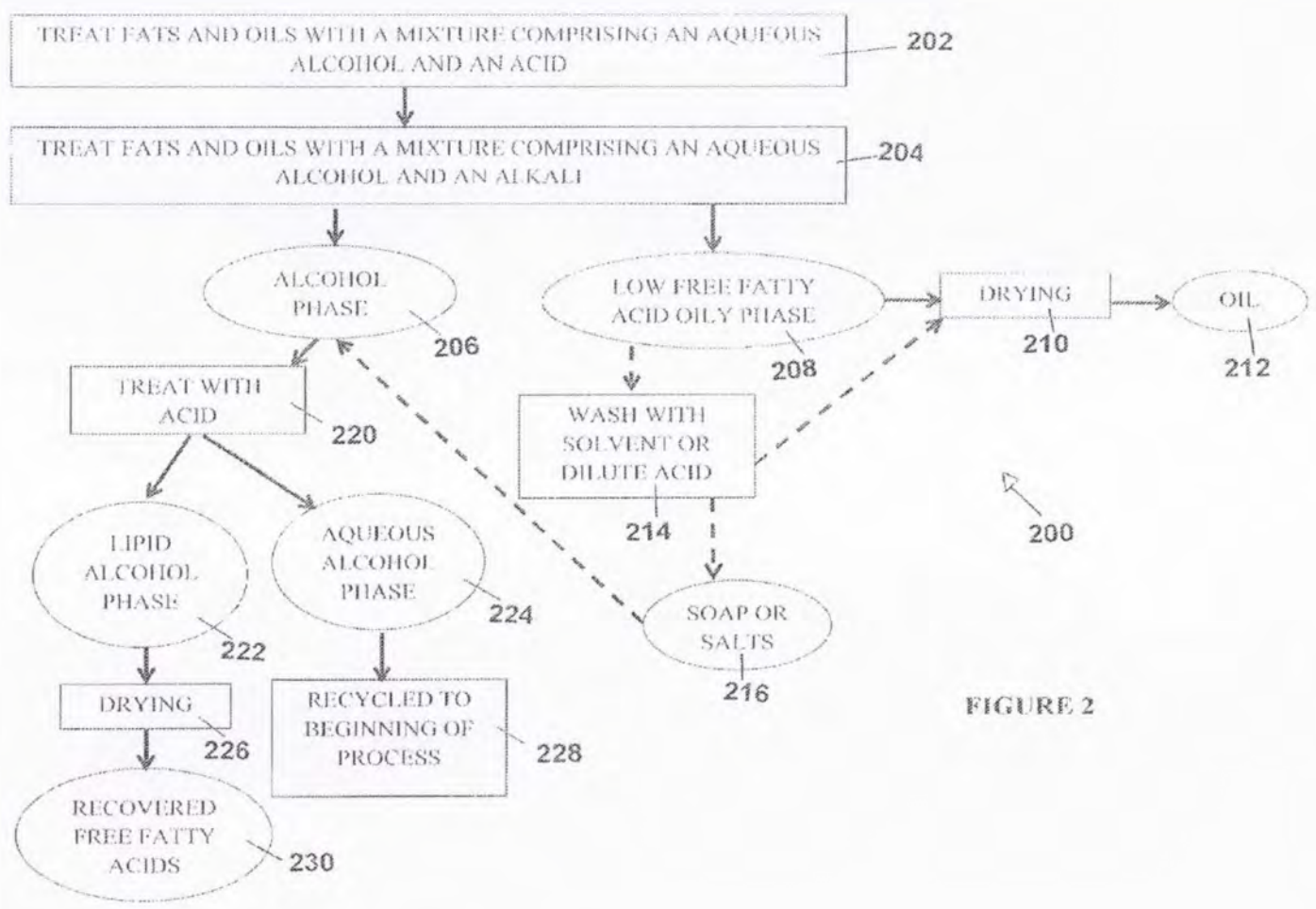


FIGURE 2

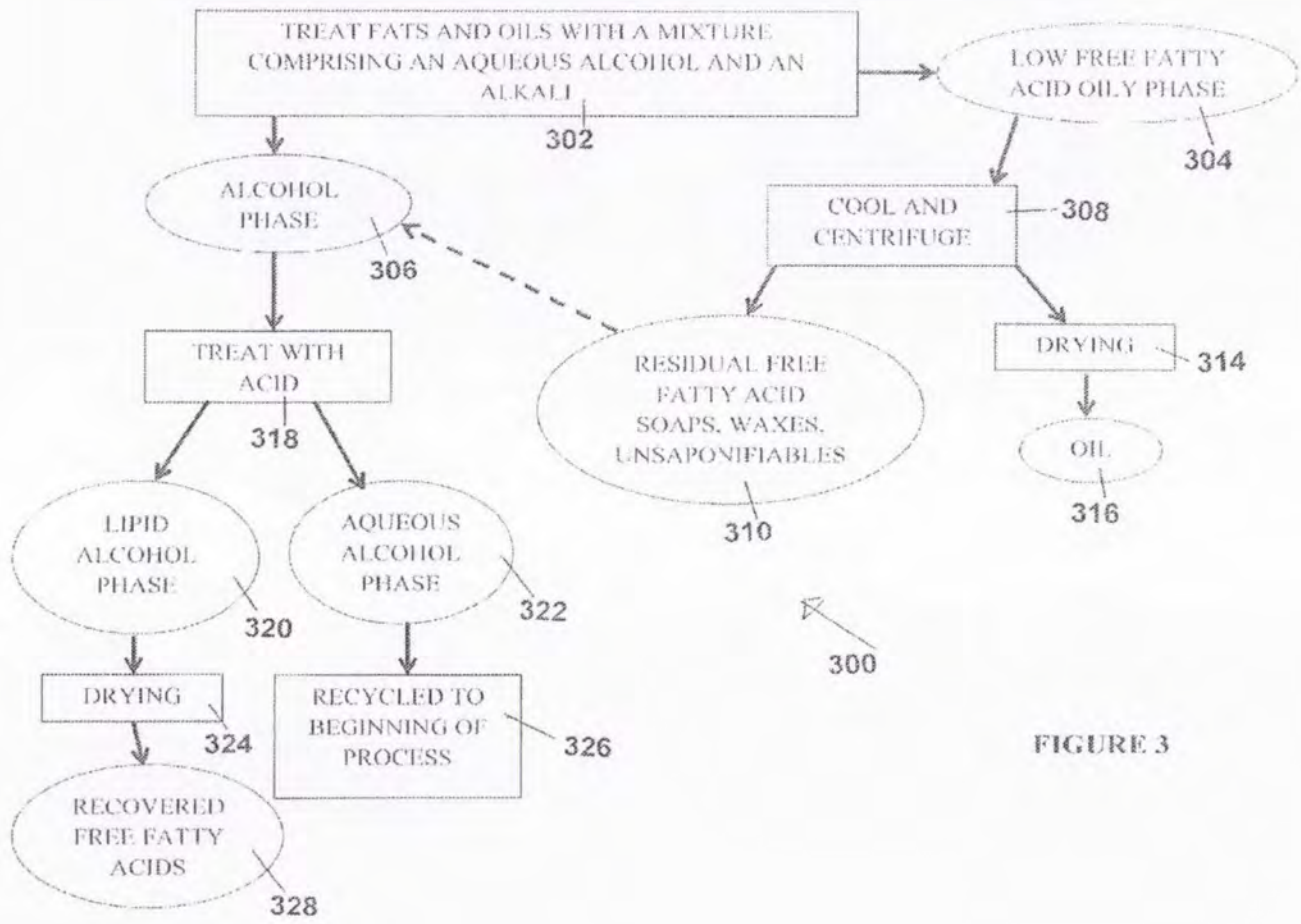


FIGURE 3



FIGURE 4

METHOD TO RECOVER FREE FATTY ACIDS FROM FATS AND OILS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims priority from U.S. Provisional Application Ser. No. 61/725,598 filed Nov. 13, 2012 and entitled METHOD TO RECOVER FREE FATTY ACIDS FROM FATS AND OILS and from U.S. Provisional Application Ser. No. 61/793,727 filed Mar. 15, 2013 and entitled METHOD TO RECOVER FREE FATTY ACIDS FROM FATS AND OILS. The contents of U.S. Provisional Application Ser. No. 61/725,598 and U.S. Provisional Application Ser. No. 61/793,727 are hereby incorporated in their entireties by reference.

FIELD OF THE INVENTION

This invention relates generally to the removal and recovery of free fatty acids from fats and oils and specifically a method for treating high free fatty acid fats and oils to recover free fatty acids wherein the method recovers a high quantity of the free fatty acids while having a low neutral oil loss.

BACKGROUND

Some fats and oils contain high free fatty acid content, including but not limited to corn oil and waste fats and oils. As is generally known in the art, fats and oils containing a high percentage of free fatty acids are undesirable. For example, free fatty acids decrease the oxidative stability of oil. Previous methods include the refining of crude oils, which generally result in oil of low free fatty acid content. The crude oils, which have low free fatty acid content, are purified by converting the fatty acids to soaps using caustic or alkali and then separating the free fatty acid soaps, commonly referred as soapstock, from the oil. The soapstock is then treated as a waste product or used for animal feed and soap manufacturing. These methods fail to capitalize on the potential of free fatty acids as a valuable product within the fats and oils industry. For example, recovered free fatty acids may be used in feed fat supplements and to manufacture industrial products. Moreover, previous methods lead to the formation of an emulsion that entraps neutral oil, thus resulting in a high neutral oil loss. The neutral oil loss is exacerbated in the case of waste fats and oils due to the presence of high free fatty acid content. This is problematic because neutral oil is a valuable product. Accordingly, an ideal method will minimize neutral oil loss.

As provided above, fats and oils with high free fatty acids may include corn oil and waste fats and oils. For example, corn oil, including but not limited to corn oil that is produced as a byproduct of an ethanol production plant, may include at least 4% free fatty acids by weight. Other fats and oils with high free fatty acid content include high acid grease from pork plants, high acid tallow from beef plants, and waste fryer grease. Moreover, a byproduct of biodiesel production may include unreacted fats and oils with high free fatty acid content. Generally, all of these fats and oils are inedible, industrial and fall into secondary or tertiary grade fats and oils. They may have a free fatty acid content of up to 90%. Processing these fats and oils to recover the free fatty acids results in at least two valuable products: neutral oil and free fatty acids. Additionally, other impurities that are removed in the method may be valuable products.

Previous attempts have been made to remove free fatty acids from oil, particularly crude oil having low free fatty acid content. These methods have drawbacks. In particular, these methods are unsuccessful when removing free fatty acids from starting material having high free fatty acid content. For example, the methods are ineffective when recovering free fatty acids from corn oil produced at an ethanol production facility and waste fats and oils. Oftentimes, these methods include adding alkali to the oil to create free fatty acid soaps. However, the addition of alkali to fats and oils having high free fatty acid content results in an emulsion. The emulsion includes fatty acid soaps and neutral oil and must be further processed to remove these valuable substances. Alternatively, if the emulsion is not processed, the recovery of both fatty acids and neutral oil will be reduced, resulting in a loss of valuable products. Moreover, because previous attempts to remove free fatty acids from fats and oils are directed to refining crude oil, the methods fail to capture free fatty acids as a valuable product.

In one example, United Kingdom Patent Specification No. 427,680 discloses a process for refining vegetable and animal oils and fats. The invention described therein relates to the separation of fatty acid soaps formed by free fatty acids and caustic. The disclosed process addresses the problem of an emulsion by treatment with an alcoholic solution of salts sufficiently concentrated to prevent most oil from going into solution. Effective salts include alkali metal salts such as sodium sulfate, chloride, nitrate, formate, and acetate. The reference argues that the salts prevent neutral oil from dissolving in the alcoholic solution. A similar process is disclosed in United Kingdom Patent Specification No. 1,391,906, which discloses a process for the removal of fatty acids from glyceride oils. The process includes mixing the oil with an aqueous alkaline solution including polyhydric alcohol and sulfonate salt.

In another process, United Kingdom Patent Specification No. 430,381 is directed to the recovery of solvents employed during the refining of oils and fats. The reference discloses the process of neutralizing the oil to produce soapstock and drying the fatty acid soaps in a vacuum prior to adding alcohol to the fatty acid soaps. The addition of the alcohol to the dried soapstock forms three layers: neutral oil, soap, and a layer of emulsion. The emulsion layer must then be processed to remove soaps. This process is inefficient in that it requires the steps of drying the fatty acid soaps and processing the emulsion.

Another process, disclosed in United Kingdom Patent Specification No. 596,871 is directed to the refining of vegetable glyceride oils and fats, particularly cottonseed oil. Crude oil having low free fatty acid content is neutralized in the presence of low concentrations of alcohol. The method disclosed therein is particularly applicable to oils having a high content of non-fatty substances, considerable coloring matter, and free fatty acid content around 1-2%. Accordingly, the process is not well-suited for fats and oils having high free fatty acid content and/or low amounts of non-fatty substances and coloring matter. Specifically, the process disclosed therein results in greater neutral oil loss as free fatty acid content increases.

Another reference, U.S. Pat. No. 6,399,802 provides a method for soapstock acidulation. The method includes adding both a monohydric alcohol to soapstock to lower its viscosity and a strong acid which hydrolyzes the fatty acid soaps. The acidulated fatty acids may then be converted to esters utilizing the alcohol already present in the solution, as

well as catalysts already present in the solution. Effective alcohols include isopropanol, n-propanol, isoamyl alcohol, and fusel oil.

None of the above methods provides an efficient means for recovering the free fatty acids found in fats and oils having high free fatty acid content. In addition, the above-described methods fail to result in low amounts of neutral oil loss, particularly as free fatty acid content is increased. Moreover, none of the above methods may be easily integrated into an ethanol production facility or capitalize on the products and byproducts associated with same.

Crude vegetable oils that are food grade typically have free fatty acid content of about 1% in addition to other non-oil impurities. These vegetable oils when refined through traditional alkali refining will result in process loss or neutral oil loss due to physical and chemical binding of oil with the co-products that are generated in the process. Although the neutral oil loss varies with different processes, there are some generally accepted empirical equations that are used by the producers to help estimate the neutral oil loss. American Oil Chemists' Society (AOCS) official methods Ca 9f-57 and Ca-9a-52 form the basis for calculating the neutral oil loss due to processing and inevitable loss due to the presence of free fatty acids, phosphatides and other impurities. L. Strecker et al. developed an equation specific to the process loss during the alkali refining of crude corn oil. According to this given formula, neutral oil loss for alkali refining of crude corn oil with 12% free fatty acid content is about 11% in addition to the inevitable loss due to removal of free fatty acids, impurities etc. Corn oil having 4% free fatty acid content may have neutral oil loss around 4.5% in addition to the inevitable loss due to removal of free fatty acids, impurities etc. Previous methods provide the principle that as free fatty acid content increases, so does neutral oil loss, such as the example immediately above.

Accordingly, there exists a need in the art for a method to recover free fatty acids and other impurities from high free fatty acid fats and oils. The method should have as little neutral oil loss as possible and should further recover as many free fatty acids from the neutral oil as possible in order to maximize the value of both products. Further, the method should remove other impurities from the starting materials, including but not limited to carotenoids, phytosterols, tocopherols, phospholipids and waxes. Such a method should be easily integrated into an ethanol production facility by taking advantage of products and byproducts associated with same.

SUMMARY OF THE INVENTION

Methods to recover free fatty acids from fats and oils are provided. In a first method, fats and oils having high free fatty acid content, such as greater than 4% by weight are treated with a mixture comprising an aqueous monohydric alcohol to form a low-free fatty acid oily phase and an alcohol phase. The mixture may also comprise an alkali. The aqueous monohydric alcohol has a concentration of at least about 15% alcohol by weight. The alcohol phase is treated with an acid, for example to a pH of below 6, to form an aqueous alcohol phase and a lipid alcohol phase, which includes free fatty acids. The free fatty acids may then be recovered, such as by drying, including by evaporation or distillation. The low-free fatty acid oily phase may be further treated with an acid, a monohydric alcohol, aqueous monohydric alcohol, water, or a combination thereof to remove residual free fatty acid soaps. If optionally recovered, the residual free fatty acid soaps may be added to the alcohol phase for treatment with acid.

Monohydric alcohols used in such a method may include methanol, aqueous methanol, ethanol, aqueous ethanol, propanol, aqueous propanol, isopropanol, aqueous isopropanol, butanol, aqueous butanol, isobutanol, aqueous isobutanol, pentanol, aqueous pentanol, and combinations thereof. For example, the alcohol may be an aqueous ethanol including about 15-55% ethanol by weight. The treatment of fats and oils with a mixture comprising a monohydric alcohol and an alkali may occur at about 25 to 75 degrees Celsius, such as at 65 degrees Celsius, and at about atmospheric pressure. The acid may include carbonic acid, which is formed by treating the alcohol phase with carbon dioxide, such as carbon dioxide produced as a byproduct of ethanol production. Other acids may include sulfuric acid, hydrochloric acid, phosphoric acid, citric acid, oxalic acid, and combinations thereof. Fats and oils amenable to the method may include, but are not limited to, waste fats, waste oils, used cooking oil, choice white grade high acid grease, high acid tallow, corn oil, and combinations thereof. In some embodiments, the fats and oils may be further processed to further remove impurities. In one embodiment, the fats and oils may first be treated with a mixture comprising an alcohol and an acid. In some embodiments, the method of the present invention results in neutral oil loss of less than 10%, such as less than 7%, 3%, or, preferably, less than 2%.

In another method to recover free fatty acids from fats and oils, fats and oils having high free fatty acid content are treated with a mixture consisting essentially of an aqueous alcohol and an alkali to form a low-free fatty acid oily phase and an alcohol phase. The aqueous alcohol may have a concentration of at least about 15% alcohol by weight. The alcohol phase may be treated with acid to form an aqueous alcohol phase and a lipid alcohol phase, which includes free fatty acids. The free fatty acids are then recovered from the lipid alcohol phase.

Also provided is a method to recover free fatty acids from corn oil having a free fatty acid content of at least 4%. The corn oil is treated with a mixture comprising an aqueous alcohol and an alkali to form a low-free fatty acid oily phase and an alcohol phase. The aqueous alcohol is selected from the group consisting of aqueous methanol, aqueous ethanol, aqueous propanol, aqueous isopropanol, aqueous butanol, aqueous isobutanol, aqueous pentanol, and combinations thereof. Furthermore the aqueous alcohol has a concentration comprising at least about 15% alcohol by weight. In one embodiment, the aqueous monohydric alcohol is aqueous ethanol comprising about 15-55% ethanol by weight. The alkali may include, but is not limited to, sodium hydroxide, potassium hydroxide, magnesium hydroxide, calcium hydroxide, lithium hydroxide, sodium amide, ammonia, and combinations thereof. The alcohol phase may be treated with acid to form an aqueous alcohol phase and a lipid alcohol phase. The acid may include, but is not limited to sulfuric acid, hydrochloric acid, phosphoric acid, citric acid, oxalic acid, and carbonic acid. Carbonic acid may be formed by treating the alcohol phase with carbon dioxide. Fatty acids are recovered from the lipid alcohol phase.

In addition, a method is provided to recover free fatty acids from corn oil produced in an ethanol production facility. The method includes collecting corn oil produced in an ethanol production facility and having free fatty acid content of at least 4%. The corn oil is treated with a mixture comprising an aqueous ethanol having a concentration of at least 15% ethanol by weight and an alkali. The alcohol phase comprises free fatty acid soaps and may be treated with acid. Accordingly, an

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aqueous alcohol phase and a lipid alcohol phase are formed. The lipid alcohol phase comprises fatty acids, which may then be recovered.

In yet another similar method to recover free fatty acids from corn oil produced in an ethanol production facility and having a free fatty acid content of at least 4%, the corn oil is treated with a mixture comprising an aqueous ethanol having a concentration of at least about 15% ethanol by weight and an alkali to form a low-free fatty acid oily phase and an alcohol phase. The alcohol phase may be treated with carbon dioxide to form an aqueous alcohol phase and a lipid alcohol phase. The lipid alcohol phase comprises free fatty acids which may be recovered.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a flow chart according to one or more examples of a first embodiment of a method to recover free fatty acids from fats and oils of the present invention.

FIG. 2 is a flow chart according to one or more examples of a second embodiment of a method to recover free fatty acids from fats and oils of the present invention wherein the fats and oils are first treated with an alcohol and an acid to remove impurities in the fats and oils.

FIG. 3 is a flow chart according to one or more examples of a third embodiment of a method to recover free fatty acids from fats and oils of the present invention wherein low free fatty acid oil is cooled and centrifuged to remove residual fatty acid soaps, waxes, and unsaponifiables.

FIG. 4 is a flow chart according to one or more examples of a fourth embodiment of a method to recover free fatty acids from fats and oils of the present invention wherein the method takes place at an ethanol production facility to recover free fatty acids from corn oil and also takes advantage of other products of ethanol production, including aqueous ethanol and carbon dioxide.

DETAILED DESCRIPTION

The following is a detailed description of embodiments of a method 100, for recovering free fatty acids from fats and oils. Fats and oils amenable to such a method may include but are not limited to corn oil, such as corn oil produced in an ethanol plant, high acid grease, high acid tallow, bleachable fancy tallow, fancy tallow, A tallow, prime tallow, special tallow, No. 2 tallow, yellow grease, flotation oils/greases from animal processing plant wastewater streams, fatty acid streams from biodiesel plants, acidulated soapstock oils and waste frying grease. These fats and oils are generally inedible. Moreover, fats and oils that have become rancid and unsalable at least in part because of the free fatty acid content may be subjected to this method to create valuable, salable products. The disclosed methods have the advantage of being simple yet highly effective at recovering free fatty acids while minimizing neutral oil loss and emulsion formation. Furthermore, in some embodiments, the disclosed methods have the benefit of capitalizing on products and byproducts of an ethanol production facility. Accordingly, one use of the disclosed method 100 is for the recovery of free fatty acids from corn oil and particularly corn oil obtained as a byproduct of ethanol production. For ease of discussion and understanding, the following detailed description and illustrations often refer to the method for use with corn oil. It should be appreciated that the method 100 of the present invention may be used with any fats and oils of animal or vegetable origin.

Referring to FIG. 1, a method 100 for recovering free fatty acids from fats and oils is provided. As shown by block 102,

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the method begins by treating fats and oils with a mixture comprising an alcohol and an alkali. In the illustrated embodiment, the alcohol is an aqueous alcohol. The alcohol, preferably aqueous alcohol, may also be referred to as the solvent.

As mentioned above and discussed in further detail below, the alcohol is advantageous for effecting separation of an alcohol phase 118 and, in some embodiments, residual fatty acid soaps, from a low free fatty acid oily phase 106. The alkali is advantageous for converting free fatty acids to free fatty acid soaps. The treatment results in a low free fatty acid oily phase 106 and an alcohol phase 118. Free fatty acids can also be extracted from crude fats and oils by using aqueous alcohols alone. This is based on the preferential solubility of free fatty acids in the alcohols over neutral oil. In order to sufficiently remove free fatty acids, this method requires a considerably large amount of an alcohol. Alcohols such as methanol, ethanol, propanol, isopropanol, butanol, isobutanol, pentanol, and combinations thereof may be used for this purpose. Laboratory tests show that the process requires about 4-5 times as much weight of alcohol to extract 15% free fatty acids from distillers corn oil than when alkali is also used. When alkali is used, the solvent to oil ratio may be about 0.4-0.6. Recovery of solvent back into the process, although energy intensive, can be easily done with a simple flash distillation due to high difference in the boiling points of the solvent and oil. Moreover, the use of high amounts of solvent also increases the amount of neutral oil loss with the alcohol phase to about 5%, which is likely due to the solubility of oil in high volumes of alcohol. Although this is considerably less than the traditional refining methods, employing an alkali results in even further decreased neutral oil loss, as will be discussed hereinbelow. Accordingly, as provided in FIG. 1, in the preferred embodiment, a mixture comprising both an alcohol and an alkali is employed. Suitable alkalis include, but are not limited to, hydroxides, oxides, carbonates, amines, and amides. For example, sodium hydroxide, potassium hydroxide, magnesium hydroxide, calcium hydroxide, lithium hydroxide, sodium amide, or ammonia may be used. Oftentimes, sodium hydroxide may be used due to its lower cost.

As discussed, above, acceptable alcohols include but are not limited to monohydric alcohols such as methanol, ethanol, propanol, isopropanol, butanol, isobutanol, pentanol, and combinations thereof. Due to the difference in polarity of the aforementioned alcohols and neutral oil, these alcohols are less soluble with oil, leading to decreased neutral oil loss. In general, the alcohol reduces and/or eliminates the emulsion that can be formed when free fatty acids react with alkalis in only water as a solvent, thus effecting clean separation of the low-free fatty acid oily phase and alcohol phase. This provides the advantage of decreasing neutral oil loss while increasing the percentage of free fatty acids that are recovered in the method 100. In some embodiments, the method of the present invention results in neutral oil loss of less than 10%, such as less than 7%, 3%, or, preferably, less than 2%. Ideally, neutral oil loss is as close to 0% as possible. However, some neutral oil loss is often inevitable. As discussed above, previous methods provide a greater neutral oil loss as free fatty acid content of the starting oil increases. As compared to the prior art, methods of the present invention provide a constant, low neutral oil loss for fats and oils with any amount of free fatty acids. Accordingly, while there may be some fluctuation in resulting neutral oil loss among types of oil, neutral oil loss remains generally constant for a particular type of oil. In addition, fluctuation in neutral oil loss for oils with varying contents of free fatty acids is minimized.

The alkali and free fatty acids react in a 1:1 mole ratio. Accordingly, for each mole of free fatty acids, one mole of

alkali should be added. The free fatty acid content of the starting oil may be obtained in the laboratory by methods known in the art, such as titration. In embodiments directed to corn oil obtained from an ethanol plant, it is anticipated that the free fatty acid content will generally be consistent in oils received from the same plant. The solvent to oil ratio is preferably about 0.6 by volume, although it is anticipated that other ratios will be effective. As discussed below in Example 7, lower ratios may result in higher neutral oil loss. On the other hand, employing as little solvent as possible is effective and provides for cost savings in the process. Moreover, if too little solvent is used, then an emulsion will occur, which results in neutral oil loss. Furthermore, this step may occur at temperatures of about 25-75 degrees Celsius and at about atmospheric pressure, such as with the reaction occurring at about 65 degrees Celsius at about atmospheric pressure. To some extent, the temperature range may be limited at the top by the boiling point of the alcohol, such as approximately 78 degrees Celsius at about atmospheric pressure for ethanol, while temperatures below about 25 degrees Celsius may lead to difficulty separating the low-free fatty acid oily phase and the alcohol phase in some circumstances.

In the exemplary embodiment disclosed herein, the method 100 is used for the treatment of corn oil produced at an ethanol plant. Accordingly, ethanol or aqueous ethanol, is used as a solvent. Aqueous ethanol with an ethanol concentration of greater than about 15% by weight is preferred. For example, aqueous ethanols having about 15-55% ethanol by weight are used, such as aqueous ethanol with about 40% by weight ethanol, but it is anticipated that other concentrations will be effective. While an aqueous ethanol with about 40% ethanol is preferred, oftentimes the aqueous ethanol received from an ethanol plant will have a higher ethanol concentration, such as about 55%. This aqueous ethanol is effective in carrying out the claimed methods and can provide cost savings as there is no need to process the aqueous ethanol prior to using same as a solvent. However, it is contemplated that aqueous alcohols with a lower ethanol concentration may be more effective in preventing neutral oil loss. This is because neutral oil is more easily dissolved in aqueous ethanol with higher ethanol concentrations. Moreover, due to the polarity of oil and water, the presence of water reduces the solubility of oil in ethanol. Accordingly, aqueous alcohols with lower ethanol concentrations may result in decreased neutral oil loss. However, alcohol concentrations below 15% may not be effective in breaking the emulsion, and, as a result, neutral loss will increase.

As discussed above, the addition of the alcohol and alkali will result in two phases being formed: an alcohol phase 118 and a low free fatty acid oily phase 106. The low free fatty acid oily phase 106 will include neutral oil but may also include residual impurities, including residual free fatty acid soaps, the optional recovery of which will be discussed below. The alcohol phase 118 will include free fatty acid soaps, ethanol, water, and any impurities present in the oil, such as carotenoids, phytosterols, tocopherols, phytostanols, polyphenols, phospholipids, waxes, and/or other impurities, that have preferential solubility in the aqueous ethanol solvent phase.

The above treatment, which includes a reaction and an extraction, may be exploited in many different fashions, including but not limited to a batch system, a continuous stirred-tank reactor (CSTR), and continuous flow in a tubular or pipe system. For example, the treatment may occur in a continuous tubular system, such as a carbon steel pipe containing at least one static mixer to effect mixing of the alkali and free fatty acids, as well as the free fatty acid soaps and aqueous ethanol. In one laboratory scale example, this step 102 of the method 100 may be carried out in an eleven inch

carbon steel pipe having a one half inch diameter. The pipe includes one static mixer with 12 elements for effective mixing of the substances. It is anticipated that this laboratory reactor is one tenth the size of an industrial system that would be employed at a 50 million gallon per year ethanol plant. The described laboratory reactor will handle oil at 1200 million which will correspond to three gallons per minute rate of oil at the industrial scale.

In the preferred continuous tubular system, the low free fatty acid oily phase 106 and alcohol phase 118 flow into a decanter and are allowed to separate into two layers by settling for 15-30 minutes. Alternatively, the low free fatty acid oily phase 106 and alcohol phase 118 may be separated by any means known in the art, now or in the future, including but not limited to flowing the mixture of low free fatty acid oily 106 and alcohol 118 phases to a liquid-liquid centrifuge to be continuously separated into two phases. When using a decanter, as the layers settle, they are continuously drained or pumped from the decanter.

As discussed above and shown in FIG. 1, after drawing off the low free fatty acid oily phase 106, it may optionally be further processed. In one embodiment, the phase 106 may be washed with solvent or acid, as shown in block 108. Suitable acids include both inorganic and organic acids, such as sulfuric acid, hydrochloric acid, phosphoric acid, citric acid, oxalic acid, and carbonic acid. In one embodiment, carbonic acid is obtained by treating the low-free fatty acid oily phase with carbon dioxide. Advantageously, carbon dioxide is a byproduct of ethanol production. The acid was results in salts and washed, low free fatty acid oil, which may be dried, as shown in block 114. As discussed above, the oil 116 is a valuable product. In another embodiment, the low free fatty acid oily phase 106 may be dried without washing, as shown by block 114 to produce valuable oil 116. In most embodiments, the low-free fatty acid oily phase 106 need not be processed to remove residual free fatty acid soaps, as the oil in the low-free fatty acid oily phase 116 meets many required specifications for sale as a valuable product.

Alternatively, the low free fatty acid oily phase 106 may be washed with the alcohol solvent to remove residual soaps, as shown by block 108. Although water may be effective, its use alone tends to create emulsions. However, the addition of alcohol to the water to create an aqueous alcohol for washing the oil phase reduces or eliminates the emulsion than can be formed when the oil phase is mixed with water alone. As discussed above the alcohol effects clean separation of the oil from free fatty acid soaps. For example, the same solvent that is used in the initial treatment step, such as aqueous ethanol with about 40-60% ethanol, may be used to wash the oil phase. The residual free fatty acid soaps recovered from the oil phase may be added to the alcohol phase 118 for further processing with same. The washed oil may then be processed, such as by drying 114 to remove the solvent, to recover the valuable neutral oil 116. The neutral oil 116 may be used for animal feed, industrial purposes including but not limited to lubricants, biodiesel, polymers, and paints, and potentially food.

As shown by block 120 of FIG. 1, the alcohol phase from the first step is treated with acid to form a lipid alcohol phase 122 and an aqueous alcohol phase 124. In the preferred embodiment, the acid is added until the pH of the mixture is 6 or below, preferably about 2. Suitable acids include both organic and inorganic acids. For example, sulfuric acid, hydrochloric acid, phosphoric acid, citric acid, oxalic acid, acetic acid, and carbonic acid may be used. As discussed above, carbonic acid may be obtained from carbon dioxide, which is produced as a byproduct of ethanol production. As

much as seventeen pounds of carbon dioxide is produced per bushel of corn processed at an ethanol plant. Accordingly, carbon dioxide is an inexpensive or free, readily available substance at ethanol production plants. Some ethanol plants release this carbon dioxide into the atmosphere, while others capture it for sale. As carbon dioxide is a greenhouse gas, using the carbon dioxide in the method such that the release of carbon dioxide into the air is eliminated or reduced helps reduce greenhouse gas emissions and is, accordingly, an environmentally friendly process. Moreover, carbon dioxide in the presence of water acts as carbonic acid. This acid will convert, or acidulate, free fatty acid soaps to free fatty acids and corresponding carbonate salts. When the preferred aqueous ethanol described above is used, water is already present in the alcohol phase 118 for reacting with carbon dioxide to create acid. It is anticipated that other substances could be added at this time as desired. Carbon dioxide acidulation provides the benefit of reducing or eliminating the use of strong acids, such as sulfuric acid, which may otherwise be necessary for acidulation of the free fatty acid soaps.

This step 120 of the method 100 may also be exploited in many different fashions, including but not limited to a batch system, a continuous stirred-tank reactor (CSTR), and continuous flow in a tubular or pipe system. In embodiments employing carbon dioxide, the treatment step with same is preferably carried out in a high pressure reactor, although it is anticipated that other systems may be used. Beneficial to the process, a high pressure reactor is air tight, which prevents the gaseous carbon dioxide from escaping. In one embodiment, carbon dioxide is collected as it is released in the ethanol production process and bubbled to the alcohol phase. After the carbon dioxide treatment step, the resulting lipid alcohol phase and aqueous alcohol phase may be collected in a decanter, where the phases are allowed to settle for 15-30 minutes before being separately drawn off. Alternatively, the separation of the phases may be effected by a liquid-liquid centrifuge or other means known in the art now or in the future, but due to the pH of the output, it is often desirable to use other means to separate the two phases. For example, the low pH of the output may corrode some centrifuges. The aqueous alcohol phase 124 generally includes ethanol, water, and salts. The lipid alcohol phase 122 primarily includes ethanol, free fatty acids, and water.

The lipid alcohol phase may be processed to recover the free fatty acids contained therein. In the preferred embodiment, the lipid alcohol phase 122 is dried, as shown by block 126. Processes such as evaporation or distillation may be used to recover the free fatty acids. Accordingly, the method results in recovered free fatty acids 130. It is anticipated that the disclosed method will result in high recovery of free fatty acid with low neutral oil loss. In some embodiments, neutral oil loss may be 2% or lower. Once the alcohol present in the lipid alcohol phase 122 has been separated from the recovered free fatty acids 130, it may be reused if desired, but may require dilution with water to obtain the appropriate concentration. In addition, the aqueous alcohol phase 124 may be recycled to the beginning of the process, as shown in block 128.

Referring to FIG. 2, a second embodiment of a method 200 to recover free fatty acids from fats and oils is provided. The embodiment begins by treating fats and oils with a mixture comprising an aqueous alcohol and an acid, as shown in block 202. This embodiment is advantageous for waste fats and oils that originate from oils such as soybean oil and canola oil that contain impurities such as phospholipids. The aqueous alcohol and acid effectively hydrates all the phospholipids and separates them from the fats and oils. If fats and oils containing phospholipids are not subjected to an acid treatment pro-

cess, they would interfere with the free fatty acid extraction process and thus increase the neutral oil loss. Specifically, the presence of phospholipids results in an emulsion layer that entraps neutral oil. In the current method, the addition of alcohol reduces or eliminates the need to remove the phospholipids from the resulting mixture or phase containing same prior to proceeding with the process. Rather, the phospholipids are solubilized in an alcohol phase, resulting in better separation from the other valuable products.

As provided in block 204 of FIG. 2, a mixture comprising an aqueous alcohol and alkali is then added to the mixture resulting from step 202. In some embodiments, it may not be necessary to add further alcohol, and only an alkali will be added at this step. As discussed above, the alkali converts the free fatty acids present in the fats and oils into free fatty acid soaps. The alcohol, which is preferably an aqueous alcohol, helps to effect clean separation of an alcohol phase 206 and low-free fatty acid oily phase 208.

The remaining steps of the second embodiment of a method 200 to recover free fatty acids from fats and oils are similar to that of the first-described embodiment of a method of the present invention. Namely, the low free fatty acid oily phase 208 may be washed with acid or solvent, as shown in block 214 to produce salts or soap 216, respectively, and oil 218. The washed, low free fatty acid oil may be dried 210 to produce valuable neutral oil 212. In addition, as shown by block 210 of FIG. 2, the low free fatty acid oily phase 208 may be dried to produce oil 212 without undergoing a wash step. The alcohol phase 206 may be treated with acid 220 to produce a lipid alcohol phase 222 and an aqueous alcohol phase 224. The lipid alcohol phase 222 may be processed, such as by drying 226, to produce recovered free fatty acids 230. The aqueous alcohol phase 224 may be recycled to the beginning of the process, as shown in block 228.

In a third embodiment of a method 300 for recovering free fatty acids from fats and oils, the low free fatty acid oily phase 304 may be further processed to remove waxes, unsaponifiables, and residual fatty acid soaps. The dewaxing method 300 begins by treating fats and oils with a mixture comprising an aqueous alcohol and an alkali, as shown by block 302. This treatment results in an alcohol phase 306 and a low-free fatty acid oily phase 304. The low-free fatty acid oily phase 304 may be cooled and centrifuged, as shown by block 308. By cooling the low-free fatty acid oily phase 304, impurities such as residual free fatty acid soaps, waxes, and unsaponifiables may precipitate out of the mixture. Centrifugation then allows separation of these impurities 310 from the oil 312. The resulting low free fatty acid oil exiting the centrifuge may be dried, as shown by block 314 to produce oil 316. The oil 312 may be processed as discussed above, such as with a dilute acid wash 314 to produce dewaxed oil 316.

The residual fatty acid soaps, waxes, and unsaponifiables shown in block 310 may be mixed with the alcohol phase 306 for further processing or may be processed separately. Namely, the alcohol phase 306 is treated with acid, as shown by block 318. This step 318 creates a lipid alcohol phase 320 and an aqueous alcohol phase 322. The lipid alcohol phase 320 may be processed to recover recovered free fatty acids 328, such as by drying 324. The aqueous alcohol phase 322 may be recycled to the beginning of the process, as shown by block 326.

It will be appreciated by one skilled in the art that a number of other processing steps known in the art, either now or in the future, may be employed in a method of the present invention. In one example, a bleaching agent may be used. Waste fats and oils are generally dark in color due to the presence of impurities. Previous methods to bleach these fats and oils

have included the use of bleaching clays. In methods of the present invention, fats and oils may be treated with a mixture comprising an alcohol, alkali, and bleaching agent. A liquid or dissolved bleaching agent is preferred. The bleaching agent will remove color from the resulting oil. Similar to the above-described methods, this treatment results in an alcohol phase and a low free fatty acid oily phase. The phases may be processed as discussed above to produce oil, recovered free fatty acids, and aqueous alcohol that may be recycled to treat further fats and oils. Suitable bleaching agents include, but are not limited to, hypochlorite, peroxide, chlorite, and peroxyacid. Namely, sodium hypochlorite, benzoyl peroxide, hydrogen peroxide, per-acetic acid, sodium percarbonate, sodium perborate, and sodium borohydride may be used.

Referring to FIG. 4, a fourth embodiment of a method 400 to recover free fatty acids from fats and oils begins with corn 402 at a corn dry milling ethanol plant 404. The corn dry milling ethanol plant 404 process produces at least four products: carbon dioxide 406, ethanol 408, corn oil 410, and dried distillers grains with solubles (DDGS) 412. As discussed above, the method 400 of the present invention may be used to recover free fatty acids from fats and/or oils with high free fatty acid content, and in particular the illustrated corn oil 410. As shown in block 414, the oil is treated with a mixture comprising the aqueous ethanol and an alkali. Suitable alkalis are as discussed above. This treatment results in an alcohol phase 416 and a low-free fatty acid oily phase 418. The low free fatty acid oily phase 418 may be treated to recover valuable neutral oil 428. For example, the low-free fatty acid oily phase 418 may be washed with solvent or dilute acid, as shown in block 420. The wash may produce soaps or salts 422, respectively. Optionally, the soap or salts may be added to the alcohol phase 416. In other embodiments, the low-free fatty acid oily phase 418 may instead be dried 426 immediately to produce valuable neutral oil 428. It is anticipated that in many embodiments, the low-free fatty acid oily phase 418 will be of a high enough quality that only drying 426 is necessary to produce a salable product.

The alcohol phase 416 may be further processed to recover free fatty acids. Specifically, as shown in block 430 the alcohol phase may be treated with carbon dioxide 406 produced by the ethanol plant 404. As discussed above, carbon dioxide dissolves in water to form carbonic acid, thus serving to acidulate the free fatty acid soaps. It is anticipated that in many embodiments, other organic or inorganic acids will be used. This treatment with acid 430 results in a lipid alcohol phase 432 and an aqueous alcohol phase 438. The lipid alcohol phase 432 may be processed, such as by drying 434 to produce recovered free fatty acids 436. The aqueous alcohol phase 438 may be recycled to treat further corn oil, as shown by block 440.

Example 1

This example illustrates the use of a batch reactor to extract free fatty acids from distillers corn oil (DCO) containing 13.2% free fatty acids. A test reaction was performed where 207.8 grams of DCO was added to a 500 ml flask. The corn oil may also be referred to as feedstock. The temperature of the corn oil was raised from ambient temperature to 65 degrees Celsius. A solvent phase was then prepared for use in the reaction. The solvent phase was prepared by initially creating a solution of aqueous ethanol, containing 40% ethanol by weight. Thereafter, 3.9 grams of sodium hydroxide was added to 127.6 grams of aqueous ethanol. In a separate flask, the solvent phase and alkali were mixed and heated from ambient temperature to 65 degrees Celsius. The alkaline solvent was

added to the feedstock and the mixture was then agitated for one minute, after which, the mixture was allowed to separate, in a 65 degree Celsius environment, into two distinct phases. The top phase was collected and dried to yield 179.8 grams of oil with free fatty acid content of 0.2%. 114.6 grams of the bottom solvent phase were collected into a separate beaker to which concentrated sulfuric acid was added until the pH of the mixture was 2. The mixture was then agitated for one minute, after which, it was allowed to separate, in a 65 degree Celsius environment, into two distinct phases. The top phase was separated and dried to yield 27.3 grams of fatty acids. Experimental losses of oil to glassware and other equipment amounted to 4 grams. Yield of free fatty acids may be calculated by measuring the amount of free fatty acids that are recovered as compared to the free fatty acids that are present in the feed stock. Yield of free fatty acids in this example is 98.6%. The neutral oil loss is calculated by measuring the weight of neutral oil in the feedstock minus the weight of neutral oil in the low free fatty acid oil. This example resulted in a 2.1% calculated neutral oil loss.

Example 2

This example illustrates extraction of free fatty acids from used cooking oil (UCO) containing 11.4% free fatty acids using a batch reactor. A test reaction was performed where 202.8 grams of UCO was added to a 500 ml flask and heated to 65 degrees Celsius. The solvent phase was prepared by initially creating a solution of aqueous ethanol, containing 55% ethanol by weight. Thereafter, 3.3 grams of sodium hydroxide were added to 122.6 grams of aqueous ethanol in a separate flask and heated to 65 degrees Celsius. The alkaline solvent was added to the feedstock, and the mixture was then agitated for one minute, after which, the mixture was allowed to separate into two distinct phases. The top phase was collected and dried to yield 175.9 grams of oil with free fatty acid content of 0.2%. 107.6 grams of the bottom solvent phase were collected into a separate beaker to which concentrated sulfuric acid was added until the pH of the mixture was 2. The mixture was then agitated for one minute, after which, it was allowed to separate, in a 65 degree Celsius environment, into two distinct phases. The top phase was separated and dried to yield 25 grams of fatty acids. Experimental losses of oil to glassware and other equipment amounted to 1.4 grams. Yield of free fatty acids in this example is 92%. The neutral oil loss in this example is 1.9%.

Example 3

This example illustrates extraction of free fatty acids from feed grade crude tallow containing 15.8% free fatty acids using a batch reactor. A test reaction was performed where 203.8 grams of UCO were added to a 500 ml flask and heated to 65 degrees Celsius. The solvent phase was prepared by initially creating a solution of aqueous ethanol, containing 40% ethanol by weight. Thereafter, 4.7 grams of sodium hydroxide were added to 125.6 grams of aqueous ethanol in a separate flask and heated to 65 degrees Celsius. The alkaline solvent was added to the feedstock, and the mixture was then agitated for one minute, after which, the mixture was allowed to separate into two distinct phases. The top phase was collected and dried to yield 159.9 grams of tallow oil with free fatty acid content of 0.2%. 120.8 grams of the bottom solvent phase were collected into a separate beaker to which concentrated sulfuric acid was added until the pH of the mixture was 2. The mixture was then agitated for one minute, after which, it was allowed to separate, in a 65 degree Celsius

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environment, into two distinct phases. The top phase was separated and dried to yield 42.5 grams of fatty acids. Experimental losses of oil to glassware and other equipment amounted to 5.4 grams. Yield of free fatty acids in this example is 96%. The neutral oil loss in this example is 6.6%.

Example 4

This example illustrates extraction of free fatty acids from distillers corn oil that is being produced at a commercial corn dry milling ethanol production facility. Distillers corn oil is continuously produced at a rate of 3 gal/min with an average of 15.5 wt % free fatty acids at the ethanol production facility. The corn oil is heated to 65° C. and is passed through a tubular reactor where it is mixed with 1.8 gal/min of 40 wt % ethanol solution that is premixed with 0.3 gal/min of 50 wt % sodium hydroxide. After mixing, the reaction mixture is allowed to mechanically separate into two phases. The top phase of low free fatty acid corn oil is pumped out at a rate of 2.6 gal/min, and the bottom solvent phase is pumped into another tubular reactor where it is mixed with concentrated sulfuric acid until the pH of the mixture is 2. The reaction mixture is further separated into two phases. The top free fatty acid phase is recovered and further dried to remove residual solvent to produce 0.5 gal/min of free fatty acids. Yield of free fatty acids in this example is 96%. The neutral oil loss in this example is 1.4%.

Example 5

Several experiments were conducted in order to determine the effect of alcohol proof. With the exception of the ethanol concentration in the solvent, the experimental procedure followed was similar to that described in the above examples. Ethanol concentrations from 5 wt % up to 100 wt % (absolute alcohol) were tested to determine the impact on reaction, separation, neutral oil loss, and free fatty acid yield. In general, different ethanol proofs did not have an impact on the reaction. However, with respect to the separation, when using gravity, ethanol solutions between 15 wt % and 55 wt % resulted in the lowest neutral oil loss along with high yield of free fatty acids. A middle emulsion layer was formed when ethanol solutions below 15 wt % were used. This resulted in higher oil loss due to the entrainment of the oil in the emulsion layer. Using a centrifugal separator, in place of gravity, may eliminate the possibility of forming a middle emulsion layer. Ethanol solutions between 60 wt % and 70 wt % caused convolution of oily phase and alcohol phase due to similar densities. As a result, efficient separation of two phases becomes impossible. Ethanol solutions above 70 wt % resulted in efficient phase separation but resulted in high neutral oil loss due to higher solubility of oil in alcohol phase.

Example 6

Experiments were conducted in order to determine the role of temperature on the reaction, separation, neutral oil loss, and free fatty acid yield. With the exception of the target temperature, the experimental procedure followed was similar to that described in the above examples. The neutralization reaction and the acidulation have been performed between 25° C. and 75° C. at atmospheric pressure. Results indicated that the temperature had minimal impact on the completion of the reaction. However, it was observed that temperature above 50° C. resulted in a quicker and cleaner separation of the two phases resulting in minimal oil loss to the alcohol phase.

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Example 7

With intent to use less amount of solvent, several experiments were conducted in order to determine the effect of solvent to oil ratio on reaction, separation, neutral oil loss and free fatty acid yield. With the exception of the amount of solvent used, the experimental procedure followed was similar to that described in the above examples. Solvent to oil ratios from 0.2 up to 0.6 were tested. Test results indicated that solvent to oil ratios below 0.4 impacted reaction, separation, and neutral oil loss. Specifically, solvent ratio of below 0.4 failed to completely extract the free fatty acids from the oil due to incomplete reaction. This could be a result of not enough mixing between solvent and oil phases. This problem can be overcome by using high shear mixers. However, the use of high shear mixers can result in stable emulsions between oil and solvent phases increasing the high neutral oil loss. At solvent ratios below 0.2, in addition to incomplete reaction and fatty acid extraction, a middle emulsion layer was formed which resulted in higher oil loss due to the entrainment of the oil in the emulsion layer. At this ratio there may not be enough ethanol to assist in effective separation of two phases. Using a centrifugal separator, in place of gravity, may eliminate the possibility of forming a middle emulsion layer.

Although various representative embodiments of this invention have been described above with a certain degree of particularity, those skilled in the art could make numerous alterations to the disclosed embodiments without departing from the spirit or scope of the inventive subject matter set forth in the specification and claims. Accordingly, the invention should only be limited as required in the claims below. Joinder references (e.g. attached, adhered, joined) are to be construed broadly and may include intermediate members between a connection of elements and relative movement between elements. As such, joinder references do not necessarily infer that two elements are directly connected and in fixed relation to each other. In some instances, in methodologies directly or indirectly set forth herein, various steps and operations are described in one possible order of operation, but those skilled in the art will recognize that steps and operations may be rearranged, replaced, or eliminated without necessarily departing from the spirit and scope of the present invention unless otherwise required by the claims. It is intended that all matter contained in the above description or shown the accompanying drawings shall be interpreted as illustrative only and not limiting. Changes in detail or structure may be made without departing from the spirit of the invention as defined in the appended claims.

Although the present invention has been described with reference to the embodiments outlined above, various alternatives, modifications, variations, improvements and/or substantial equivalents, whether known or that are or may be presently foreseen, may become apparent to those having at least ordinary skill in the art. Accordingly, the invention should only be limited as required in the claims below. Listing the steps of a method in a certain order does not constitute any limitation on the order of the steps of the method unless otherwise required by the claims. Accordingly, the embodiments of the invention set forth above are intended to be illustrative, not limiting. Persons skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention. Therefore, the invention is intended to embrace all known or earlier developed alternatives, modifications, variations, improvements, and/or substantial equivalents consistent with the claims.

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The invention claimed is:

1. A method to recover free fatty acids from fats and oils comprising:

treating fats and oils having a high free fatty acid content with a mixture comprising a monohydric alcohol to form a low-free fatty acid oily phase and an alcohol phase wherein said monohydric alcohol is a solvent;

said monohydric alcohol having a concentration of at least about 15% alcohol by weight;

treating said alcohol phase with an acid to form an aqueous alcohol phase and a lipid alcohol phase, wherein said lipid alcohol phase comprises free fatty acids; and recovering fatty acids from said lipid alcohol phase.

2. The method of claim 1 wherein said mixture comprising a monohydric alcohol further comprises an alkali.

3. The method of claim 2 wherein said low-free fatty acid oily phase is further treated with at least one of a monohydric alcohol, aqueous monohydric alcohol, water, acid, or a combination thereof.

4. The method of claim 2 wherein said monohydric alcohol is selected from the group consisting of methanol, aqueous methanol, ethanol, aqueous ethanol, propanol, aqueous propanol, isopropanol, aqueous isopropanol, butanol, aqueous butanol, isobutanol, aqueous isobutanol, pentanol, aqueous pentanol, and combinations thereof.

5. The method of claim 4 wherein said alcohol is an aqueous alcohol comprising about 15% to 55% ethanol by weight.

6. The method of claim 2 wherein said the step of treating said fats and oils with a mixture comprising an alcohol and an alkali to form a low-free fatty acid oily phase and an alcohol phase is carried out at about 25 to 75 degrees Celsius and about atmospheric pressure.

7. The method of claim 6 wherein the step of treating said fats and oils with a mixture comprising an alcohol and an alkali to form a low-free fatty acid oily phase and an alcohol phase is carried out at about 65 degrees Celsius.

8. The method of claim 2 wherein said acid is selected from the group consisting of carbonic acid formed by treating said alcohol phase with carbon dioxide, sulfuric acid, hydrochloric acid, phosphoric acid, citric acid, oxalic acid, and combinations thereof.

9. The method of claim 2 wherein said fats and oils are selected from the group consisting of waste fats, waste oils, high acid grease, high acid tallow, corn oil, used cooking oil and combinations thereof.

10. The method of claim 2 wherein said fats and oils are first treated with a mixture comprising an alcohol and an acid.

11. The method of claim 2 wherein said fatty acids are recovered by using a process selected from the group consisting of drying, evaporation, distillation, and combinations thereof.

12. The method of claim 2 wherein said acid is added to said alcohol phase until the pH of said alcohol phase is at least 6.

13. The method of claim 2 wherein said acid is added to said alcohol phase until the pH of said alcohol phase is 2.

14. The method of claim 2 wherein said fats and oils have a free fatty acid content of at least 4%.

15. The method of claim 2 wherein the neutral oil loss of the process is less than 10%.

16. The method of claim 15 wherein the neutral oil loss of the process is less than 3%.

17. The method of claim 2 wherein the ratio of aqueous monohydric alcohol to oil is about 0.4-0.75.

18. A method to recover free fatty acids from fats and oils comprising:

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treating fats and oils having a high free fatty acid content with a mixture consisting essentially of an aqueous alcohol and an alkali to form a low-free fatty acid oily phase and an alcohol phase and wherein said aqueous alcohol is a solvent;

treating said alcohol phase with an acid to form an aqueous alcohol phase and a lipid alcohol phase, wherein said lipid alcohol phase comprises free fatty acids; and recovering fatty acids from said lipid alcohol phase; wherein said aqueous alcohol has a concentration of at least about 15% alcohol by weight.

19. A method to recover free fatty acids from corn oil comprising:

said corn oil having a free fatty acid content of at least 4%; treating said corn oil with a mixture comprising an aqueous alcohol and an alkali to form a low-free fatty acid oily phase and an alcohol phase and wherein

said aqueous alcohol is a solvent; treating said alcohol phase with an acid to form an aqueous alcohol phase and a lipid alcohol phase, wherein said lipid alcohol phase comprises free fatty acids; and recovering fatty acids from said lipid alcohol phase;

wherein said aqueous alcohol is selected from the group consisting of aqueous methanol, aqueous ethanol, aqueous propanol, aqueous isopropanol, aqueous butanol, aqueous isobutanol, aqueous pentanol, and combinations thereof; and

wherein said aqueous alcohol has a concentration comprising at least about 15% alcohol by weight.

20. The method of claim 19 wherein said aqueous alcohol is an aqueous ethanol comprising about 15% to 55% ethanol by weight.

21. The method of claim 19 wherein said alkali is selected from the group consisting of sodium hydroxide, potassium hydroxide, magnesium hydroxide, calcium hydroxide, lithium hydroxide, sodium amide, ammonia, and combinations thereof.

22. The method of claim 19 wherein said acid is selected from the group consisting of sulfuric acid, hydrochloric acid, phosphoric acid, citric acid, oxalic acid, carbonic acid produced by treating said alcohol phase with carbon dioxide, and combinations thereof.

23. A method to recover free fatty acids from corn oil produced in an ethanol production facility comprising:

collecting corn oil produced in an ethanol production facility, said corn oil having a free fatty acid content of at least 4%;

treating said corn oil with a mixture comprising an aqueous ethanol having a concentration of at least about 1.5% percent ethanol by weight and an alkali to form a low-free fatty acid oily phase and an alcohol phase and wherein said aqueous alcohol is a solvent;

said alcohol phase comprising free fatty acid soaps; treating said alcohol phase with an acid to form an aqueous alcohol phase and a lipid alcohol phase, said lipid alcohol phase comprising free fatty acids; and recovering said free fatty acids.

24. A method to recover free fatty acids from corn oil produced in an ethanol production facility comprising:

collecting corn oil produced in an ethanol production facility, said corn oil having a free fatty acid content of at least 4%;

treating said corn oil with a mixture comprising an aqueous ethanol having a concentration of about at least 15% percent ethanol by weight and an alkali to form a low-free fatty acid oily phase and an alcohol phase and wherein said aqueous alcohol is a solvent;

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said alcohol phase comprising free fatty acid soaps;
treating said alcohol phase with carbon dioxide to form an
aqueous alcohol phase and a lipid alcohol phase, said
lipid alcohol phase comprising free fatty acids; and
recovering said free fatty acids.

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Appendix D

UNIVERSITY OF TEXAS A&M STUDY



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Experiment and Results:

A study to evaluate the deep frying quality of 2 corn oils with French fries and a sensory evaluation (consumer panel only) of the fried product was conducted at the Food Protein R & D Center of Texas A& M University. Frying was conducted at a temperature of 370 °F for 4 days (160 batches of frying). Each batch was fried for 5 minutes along with 2 minutes drained time. Sensory evaluation of the product was carried out on the first day of frying. Various chemical analyses including smoke point, moisture, free fatty acid, peroxide value, total polar material and the fatty acids profile were performed according to the AOCS standard procedures (American Oil Chemist Society methods) on the fresh and day 4 oil. Oil usage rate was determined by the ratio of fries fried by per pound of oil.

Materials:

Oils: Frying oils were provided by CoPack Strategies, LLC.

	Code
1) HyVee 100% Pure Corn Oil	A
2) Co-Pack Strategies, LLC Corn Oil	B

French Fries: Were purchased from Sam's Club, College Station, TX (Valley Select Traditional Premium French Fry 3/8" Regular Cut).

Testing Protocol:

Testing Entity: Texas A&M University, Food Protein R&D Center
(<http://foodprotein.tamu.edu/fatsoils>).

Blind Testing: The consumer sensory panel participants were given only the codes for each sample. They were not told the identities or type of oils that they are testing.



Results:

Food to Oil Ratio: Weight of oil (Initial oil in the fryer plus make-up oil plus oil samples removed for chemical analyses) used to cook the weight of uncooked French fries placed in the fryer. The weight of fries presumed to be 5 lbs/bag as stated by the manufacturer.

Table 1:

OIL	End of Day 2	End of Day 4
A	16.6	19.23
B	17.0	18.52

A = HyVee 100% Corn Oil
 B = Co-Pack Strategies, LLC Corn Oil

Table 2. Major Fatty Acid Profile of Fresh Oils (Gas Chromatographic Analyses).

Fresh Oil

Oil	Fatty Acid Methyl Esters (%)										
	C16:0	C16:1	C18:0	C18:1	C18:2	C18:3	C19:0	C20:0	C20:1	C22:0	Others
A	11.61	0.00	1.81	29.06	55.79	1.07	0.00	0.42	0.25	0.00	0.00
B	11.87	0.00	1.90	30.29	52.95	0.76	0.25	0.45	0.48	0.00	1.19

A = HyVee 100% Corn Oil
 B = Co-Pack Strategies, LLC Corn Oil

C16:0, Palmitic Acid., C16:1, Palmitoleic acid., C18:0, Stearic acid., C18:1, Oleic acid., C18:2, Linoleic acid., C18:3, Linolenic acid., C19:0, Nonadecanoic acid., C20:0, Arachidic acid., C20:1, Gadoleic acid., C22:0, Behenic acid



Table 3. Major Fatty Acid Profile of day 4 oil (Gas chromatographic analyses).

Day 4 Oil

Oil	Fatty Acid Methyl Esters (%)										
	C16:0	C16:1	C18:0	C18:1	C18:2	C18:3	C19:0	C20:0	C20:1	C22:0	Others
A	12.30	0.11	2.45	29.82	51.32	1.64	0.24	0.45	0.46	0.17	1.04
B	12.62	0.00	2.49	29.53	52.29	1.73	0.00	0.44	0.39	0.17	0.22

A = HyVee 100% Corn Oil
 B = Co-Pack Strategies, LLC Corn Oil

C16:0, Palmitic Acid., C16:1, Palmitoleic acid., C18:0, Stearic acid., C18:1, Oleic acid., C18:2, Linoleic acid., C18:3, Linolenic acid., C19:0, Nonadecanoic acid., C20:0, Arachidic acid., C20:1, Gadoleic acid., C22:0, Behenic acid

Note: No significant changes were seen in the fatty acid profile of both oils after 4 days of frying.

Table 4. Total Polar Material Percent (TPM %) Data of fresh oil and day 4. (24% TPM is considered the end point of any frying oil to be used, if the oil crosses that number it should be discarded.

TPM (%)	A	B
Fresh Oil	4.71	8.38
Day-4	14.71	18.08

A = HyVee 100% Corn Oil
 B = Co-Pack Strategies, LLC Corn Oil

Note: Based on TM numbers both oils seemed to be still left with long lasting fry life.

Table 5. Smoke Point

Smoke Point	A	B
Fresh Oil	430	418
Day 4	378	360

A = HyVee 100% Corn Oil
 B = Co-Pack Strategies, LLC Corn Oil

Note: Smoke point of the testing oil (B) seemed to have lower numbers compared to oil A indicating more prone to oxidation

Table 6. Moisture

Moisture	A	B
Fresh Oil	0.02	0.02
Day 4	0.02	0.06

A = HyVee 100% Corn Oil
 B = Co-Pack Strategies, LLC Corn Oil

Sensory Evaluation Results of the Frying Project

Consumer Sensory Evaluation: Consumer sensory evaluation was conducted on day 1 of frying with **n=22**. Various participants were recruited from the Texas A&M Engineering Experiment Station. Participants differed in jobs, education-level, sex, and age. Consumer testing was conducted at the Food Protein R&D Center at Texas A&M University Riverside Campus. Consumers were seated in a room where they were given access to distilled water. These were used as pallet cleansers before the first sample and between samples. A moderator was present throughout the study to reduce communication between panelists and to answer consumer questions. Consumers were served up to four almost equally shaped and sized French fries in a pre-heated glass custard dish. French fries for each consumer were randomly selected from a batch of French fries cooked in an oil and served immediately. Consumers evaluated each French fry sample for appearance like/dislike, color like/dislike, crispiness like/dislike, greasiness like/dislike, flavor like/dislike and overall like/dislike using 10-point hedonic scales where 1 = dislike extremely and 10 = like extremely. After evaluation, consumers were given a drink and candy bar of their choice for participating in the study.

Graph. 1. Graphic representation of sensory evaluation of French fries fried in oils A & B on a 10-point hedonic scale (where 1 = dislike extremely and 10 = like extremely)

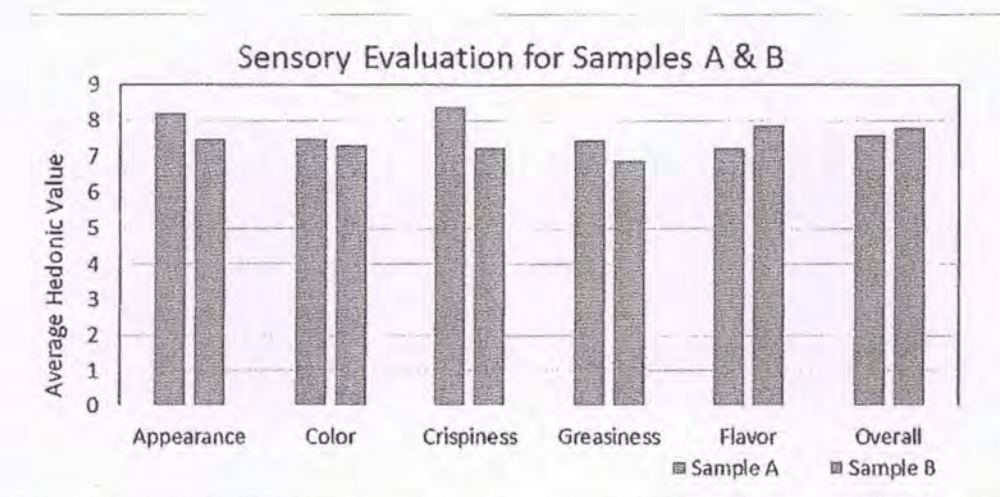




Table 4. Sensory evaluation of French fries fried in oils A & B on a 10-point hedonic scale (where 1 = dislike extremely and 10 = like extremely).

Sensory Attributes	Sample A Average Value	Sample B Average Value
Appearance Like/Dislike	8.23	7.5
Color Like/Dislike	7.5	7.32
Crispiness Like/Dislike	8.41	7.23
Greasiness Like/Dislike	7.45	6.90
Flavor Like/Dislike	7.23	7.86
Overall Like/Dislike	7.59	7.81

Sample A = HyVee 100% Corn Oil

Sample B = Co-Pack Strategies, LLC Corn Oil

Conclusions:

The results of the deep frying experiment conducted at the food protein R&D Center showed more or less very similar performance between the two oils (A and B). However, there was a significant difference in the total polar material (TPM) between the two oils before and after the frying experiment. Oil B had higher TPM even before the experiment started (8.38%) compared to oil A (4.71%) indicating oil B seemed to be more susceptible to oxidation compared to A. However, it is generally considered used oil below 24% TPM is still considered suitable for frying and in this experiment the TPM numbers both oils are below the cut off number (Table 4). It was surprising to note that there were no significant changes occurred in the C18:1 and C18:2 fatty acids in both oils even after for 4 days of frying. Oils used to cook French fries resulted no significant differences in sensory attributes either. Regardless of statistical analysis, it is apparent that sensory differences were minimal between the oils and that very minor changes occurred during repeated heating and usage of the oils. In fact the flavor and overall like and dislike of oil

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B was higher (7.81) compared to oil A (7.59)
(Graph1.). Based on these results it was concluded that both oils (A&B) seemed to be comparable and can be used
for deep frying purpose.

Sincerely,

(b) (6)



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Appendix E

SOUTH DAKOTA STATE UNIVERSITY STUDY

Executive Summary

The purpose of the study was to determine the quality of a Refined oil (provided by CoPack Strategies, LLC) relative to a commercially available vegetable oil sample. The two products were evaluated for chemical and nutritional composition. Sensory analyses of food products prepared with the two oils were compared for acceptability and preference by a trained panel. Taste, Aroma, Color and Overall Appearance were evaluated using a 5-point hedonic scale. A Triangle Test was also performed to determine if the expert panel (comprised of students enrolled in a Food Chemistry Course) could discern differences between the Refined Oil and Commercial Vegetable Oil. Sensory analyses was also done on the resultant oils to determine the aroma of post heat-treatment oils.

The effects of heat treatment were also evaluated. The two samples of oil were subjected to heat in a deep fat fryer at 375 ° Fahrenheit in preparing frozen French fries for taste testing. Two such heat treatments were carried out followed by 4-week room temperature storage prior to chemical analyses. Chemical analyses were done by Medallion Laboratories, MN. Compositional and functional traits of the four samples (Commercial and Refined oil, before and after heat treatment) were analyzed using officially accepted procedures used in the food industry. Lovibond Color values for yellow and red color were determined for characterizing appearance. Free fatty acids (FFA), Fatty acids composition, Triglycerides, Hexanal Content, Peroxide Value (PV), Oxidative Stability Index (OSI) and Trans Fatty Acids content (TFA) are provided in summary Tables 1 and 3 and in the Appendix.

Table 1 provides comparisons of a commercial vegetable oil and the refined oil. Refined oil showed a deeper red color and yellowness relative to the commercial control oil. Chromatographic analysis revealed hexanal levels less than 1% in both samples. Changes in the oils are due mostly to the composition of unsaturated fatty acids content and their susceptibility to physical treatments and change over the life of the oil. Free fatty acids content (FFA) also revealed the level and degree of refining of the oils. Both samples contained minimal levels of FFA at 0.04% (Control) and 0.056 % (refined oil). No appreciable increase was noted in heat processing as noted in Table 3 where FFA levels of less than 0.1% were noted post treatment.

The highest magnitude of differences between the fresh oils samples was determined in Peroxide Value Test with Refined Oil showing 15 meqv/Kg and 20.7 meqv/Kg. This value is high given the low values (2.96 to 6.56%) noted for PV of heat treated oils noted in Table 3. One would expect PVs to be higher in heat processed oils as opposed to unheated oils. Peroxide values of fresh oils are generally less than 10 meqv/kg. When the peroxide value is between 30 and 40 meqv/kg, a rancid taste is noticeable. While chemical indices such as PV, FFA,

Hexanal content, etc., are related to oil degradation, the most definitive test of oil quality is that of rancidity odor as perceived by the consumer.

Table 3 provides compositional and functional information on vegetable oils that were subjected to high temperature and ambient temperature storage. Heating at 375 °F and two frying experiments subjected the oils to slight changes in fatty acids composition.

Lovibond Color

Lovibond color was measured on the 4 samples. Differences in yellowness was noted between all samples. Heat treatments reduced the color differences between both commercial oil and Refined Oil. Both oils yielded a yellowness of 70 units at the end of the shelf stability experiment. Refined oil showed 50 units of Yellowness prior to heat treatment while the commercial sample yielded 38 units. However, heated oils (both commercial and Test oil) showed identical yellowness at the end of the heat treatments. Increased deepening of redness was noted in oils subjected to heat and room temperature storage for 4 weeks. A wide range of yellowness is noted in vegetable oils depending on origin. However, the color was aesthetically pleasing as noted in the Figure 1

Hexanal Value

In determining Hexanal value (HV), a homogenous portion of the sample is mixed with water containing an internal standard. It is heated in a heating block for a specified amount of time, after which a sample of the headspace over the mixture is taken and injected into the gas chromatograph. The hexanal released into the headspace is quantitated via comparison of the hexanal gas's chromatographic response to that of the internal standard. Hexanal is a by-product of fatty acid oxidation and this method measures the amount of hexanal in food products and ingredients. Therefore, this tests provides a measurement of the level of oxidative rancidity for oil in a food product. Hexanal Values for the untreated Control Oil and Refined Oil were 0.3ppm and 0.993ppm, respectively. Heat treatment increased the HV considerably of the Control and Refined Oils to 5.27ppm and 9.71 ppm, respectively.

Oxidative Stability Index

This method determines the oxidative stability index in fats and oils. The oil sample is heated at 110°C (unless another temperature is specified), and air is bubbled through it. The effluent air from the oil or fat sample is then bubbled through a vessel containing ultrapure water. The conductivity is continually measured in the adjoining test tube. The effluent air contains volatile organic acids, swept from the oxidizing oil, that increase the conductivity of the water as oxidation proceeds. Formic acid is the predominant organic acid formed. A computer monitors the conductivity of the water. The Oil Stability Index (OSI) is defined as the point of maximum change of the rate of oxidation, or mathematically of the second derivative of the conductivity with respect to time. In the case of Control Commercial oil, OSI was determined to be 10 hours. Heat used in processing in

general reduced OSI to 3.5 to 5.2 hours as noted in Tables 1 and 3 whether it was from heat employed in refining or that which was involved in cooking.

The Peroxide Value (PV) is measured in milliequivalents/Kg units. Ideally, quality food grade oils need to have values under 10 meq/Kg. Peroxides are produced by the oxidation reaction and formation of free radicals in the unsaturated fatty acids of oils. Sensory characteristics are not noticeable until the oil reaches 30-40 meq/Kg. The initial Peroxide values for the samples were 2.47 meq/kg and 15.1 meq/Kg. Control and Refined Oils, respectively. Heat treatment increased PV of Control from 2.47 to 6.56 meq/Kg. The peroxide value is a quality criterion for judging the freshness (or lack thereof) of edible oils. The lower the figure, the fresher is the oil. Crude pressed oils have a PV of 5 to 20 meq/Kg.

Free Fatty Acids Content (%)

Free fatty Acids (FFA) content of all 4 oil samples was determined to be less than 1%.

Trans Fatty acids reported in the tested oils ranged from 0.17% to 2.7%. There was a ten-fold difference in TFA between commercial and processed oils. Such levels will need to be placed within the context of commonly consumed serving sizes and exposure level through a typical or average diet. Oil uptake by French fried potatoes were 9.55% and 3.83%, for Control and Refined oils, respectively when the commercially purchased frozen french fries were cooked under identical conditions of temperature and time.

Tzzuki et al 2010 reported a 1-2% (grams/100 g oil) level of trans fat in corn oil when potatoes were fried in the oil ten times under normal frying conditions (160 to 180C). Yang et al (2012) reported up to 7.3% TFA in corn oil heated in excess of 180C. The literature shows that heating can significantly induce Cis to Trans isomerization of unsaturated triglycerides, particularly at high temperatures.

Sensory Tests of fresh oils

Evaluation of the fresh oils (as received) was done by a sensory panel made up of 7 judges. The panel members were students enrolled in a Food Chemistry course. The two oils were evaluated by taste-testing of frozen French fries that were fried under identical temperatures and times. The evaluation therefore, was of a food product prepared using the oils. Several parameters were evaluated including appearance, color, taste, aroma, texture (mouth feel) and Overall quality. Rating was done using a numeral score of 1 through 5 with 5 being the score for the highest liking.

The results from the triangle test showed that out of eight panelists, three panelists identified the odd sample given for testing. In a triangle test, two samples are similar and one (odd sample) is dissimilar. The panel members were asked to pick out the odd sample of the three samples. Two of the three panel members who correctly picked out the difference, indicated that they guessed their responses as it

was difficult to discern between the three coded oil samples. This test showed that the panel had difficulties distinguishing between the two oil samples (Control and Refined Oil). *French fries prepared using refined oil showed no differences from the Control Fries (prepared using Commercial vegetable oil).*

The Hedonic test (see table 2) which evaluated sensory traits on a scale of 1 to 5, showed that Refined Oil received higher scores than the Control oil in all categories with the exception of aroma. Appearance, color, taste, texture and Overall quality were rated higher for the Refined Oil over the control. Lower aroma scores are reflective of the aroma of French fries and indirectly to the aroma of frying oil.

Conclusion:

The quality of Refined Oil compared well with a commercial standard. Sensory test used to evaluate oil quality traits on a scale of 1 to 5, showed that Refined Oil received higher scores than the Control oil in all categories with the exception of aroma. With the exception of Hexanal and Peroxide Value tests the refined oils showed similar characteristics to commercial vegetable oil. Stability tests for vegetable oils need to be evaluated within the context of intended use. There are a wide range of results on chemical indices of oil stability based on heating temperatures and cooking times. Heating at 375 °F (twice), frying food products, and storage for 28 days provided baseline empirical information to define quality as judged by Peroxide Value, Oxidative Stability Index, Free Fatty acids, and trans fatty acids. These tests simulate extended storage testing and will need to be interpreted in that context. Sensory quality, however, is an excellent indicator of quality as judged by the consumer.

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Table 1. Comparison of Physical Traits of As Is Oils (As Received)

Test	Units	Private Label Oil (Control, #431)	Refined Oil (Sample #440)
FFA	(%)	0.04	0.056
Peroxide Value	(meq/Kg)	2.47	15.1 (20.7, reanalysis)
Hexanal	(ppm)	<0.3	0.993
Oxid Stab. Ind.	(hrs)	10	3.5 (3.3, reanalysis)
Lovibond Color			
Yellow		38 Units	50 Units
Red		2.3 Units	2.8 Units
Sat Fat	(%)	13.59	13.88
Mono	(%)	26.42	28.63
Cis-Cis PU	(%)	56.51	49.74
Trans FA	(%)	0.17	2.43

Table 2. Preference/Acceptability Scores for French Fried cooked in Vegetable Oil.

Trait	(Control, #382)	(Refined Oil, #541)
Appearance	3.7	3.8
Color	3.5	3.8
Taste	4.0	4.1
Texture	4.4	5.0
Aroma	4.6	3.0
Overall Quality	3.6	3.7

1=dislike extremely, 5=like extremely

Table 3. Comparison of Physical Traits of Heat Treated Oils (Self Stability, 2 heat treatments @ 375 °F and 28 days storage)

Test	Units	Private Label Oil* (Sample #634)	Refined Oil* (Sample #235)
FFA	(%)	0.07	0.09
Peroxide Value	(meq/Kg)	6.56 (5.83)	2.96 (3.2, reanalysis)
Hexanal	(ppm)	5.27	9.71 (2.97)
Oxid Stab. Ind.	(hrs)	5.2 (3.3)	4.3
Lovibond Color			
Yellow		70 Units	70 Units
Red		4.0 Units	4.8 Units
Sat Fat	(%)	13.98	14.06
Mono	(%)	27.19	28.7
Cis-Cis PU	(%)	54.51	47.9
Trans FA	(%)	0.26	2.36 (2.7)

*Subjected to cooking and 28 days of ambient temp storage.

Values within parenthesis were generated by reanalysis of the samples.

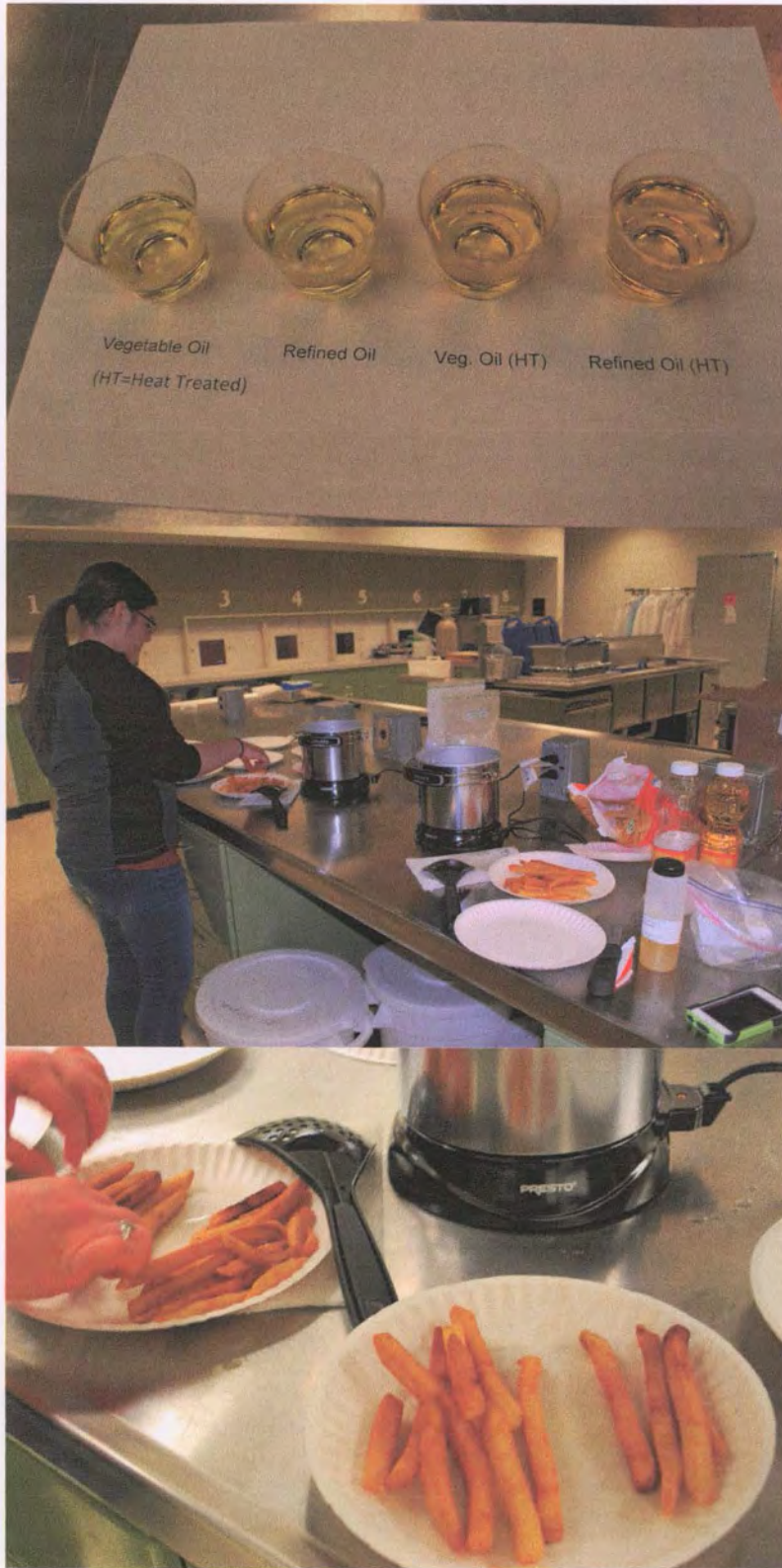


Figure 1. Taste panel evaluation of fries cooked in vegetable oils.

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Appendix F

AFLATOXIN ANALYTICAL RESULTS

REPORT NUMBER
16-285-9098
REPORT DATE
Oct 11, 2016
RECEIVED DATE
Oct 06, 2016

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6506



PAGE 1/2
ISSUE DATE
Oct 11, 2016

FEED ENERGY CO
MOHAN DASARI
4400 E UNIVERSITY AVE
PLEASANT HILL IA 50327

REPORT OF ANALYSIS
For: (6506) FEED ENERGY CO
ING CRUDE CORN OIL
R&D
SHA02 CRUDE
LIQUID

Analysis	Level Found		Reporting			Analyst- Date	Verified- Date
	As Received	Units	Limit	Method			
Sample ID: SHA02 CRUDE	Lab Number: 12715004	Date Sampled: 2016-10-05					
Aflatoxin B1	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin B2	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin G1	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin G2	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin summation	n.d.	ppb	1.00	Calculation *	Auto-2016/10/11	Auto-2016/10/11	

All results are reported on an AS RECEIVED basis., n.d. = not detected , ppb = parts per billion

For questions please contact:

(b) (6)

Heather Ramig
Account Manager
heather.ramig@midwestlabs.com (402)829-9891

The result(s) issued on this report only reflect the analysis of the sample(s) submitted.

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16-285-9100

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**FEED ENERGY CO
MOHAN DASARI
4400 E UNIVERSITY AVE
PLEASANT HILL IA 50327**

REPORT OF ANALYSIS
For: (6506) FEED ENERGY CO
TOTAL 4 65% STOICHIOMETRY
R&D
SHA04 RBD COI
LIQUID

Analysis	Level Found		Reporting			Analyst- Date	Verified- Date
	As Received	Units	Limit	Method			
Sample ID: SHA04 RBD COI	Lab Number: 12715006	Date Sampled: 2016-10-05					
Aflatoxin B1	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	Kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin B2	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	Kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin G1	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	Kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin G2	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	Kia9-2016/10/11	tp8-2016/10/11	
Aflatoxin summation	n.d.	ppb	1.00	Calculation *	Auto-2016/10/11	Auto-2016/10/11	

All results are reported on an AS RECEIVED basis., n.d. = not detected , ppb = parts per billion

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(b) (6)

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REPORT NUMBER
16-285-9097

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Oct 11, 2016

**FEED ENERGY CO
MOHAN DASARI
4400 E UNIVERSITY AVE
PLEASANT HILL IA 50327**

REPORT OF ANALYSIS

For: (6506) FEED ENERGY CO
WINTERIZED CORN OIL ONE
R&D
SHA01 WINTERIZED
LIQUID

Analysis	Level Found		Reporting			Analyst- Date	Verified- Date
	As Received	Units	Limit	Method			
Sample ID: SHA01 WINTERIZED	Lab Number: 12715003	Date Sampled: 2016-10-05					
Aflatoxin B1	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	klg-2016/10/11	tpg-2016/10/11	
Aflatoxin B2	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	klg-2016/10/11	tpg-2016/10/11	
Aflatoxin G1	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	klg-2016/10/11	tpg-2016/10/11	
Aflatoxin G2	n.d.	ppb	1.00	AOAC 2008.02 (mod) *	klg-2016/10/11	tpg-2016/10/11	
Aflatoxin summation	n.d.	ppb	1.00	Calculation *	Auto-2016/10/11	Auto-2016/10/11	

All results are reported on an AS RECEIVED basis., n.d. = not detected , ppb = parts per billion

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