(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
5 February 2009 (05.02.2009)

PCT

(10) International Publication Number
WO 2009/018390 A1

(51) International Patent Classification:
C10L 1/00 (2006.01)

(21) International Application Number:
PCT/US2008/071660

(22) International Filing Date: 30 July 2008 (30.07.2008)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/962,690 31 July 2007 (31.07.2007) US

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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(54) Title: VACUUM DISTILLATION PROCESS

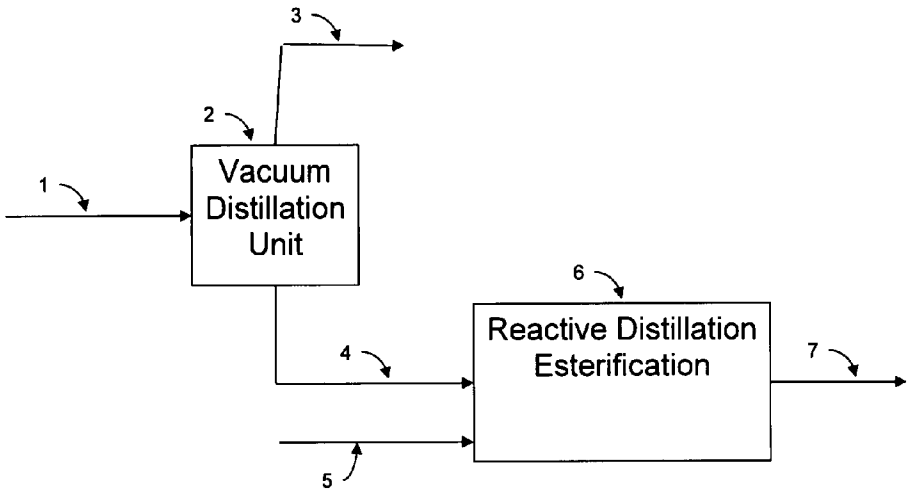


FIGURE 1

(57) Abstract: The present invention relates to a process and apparatus for the production of diesel fuel from feedstocks containing fatty acids, glycerated fatty acids, and glycerin by vacuum distillation followed by esterification. Specifically, the present invention relates to the production of Renewable Diesel having low glycerin, water, and sulfur content. Operation of the distillation system enables production of esters including biodiesel and other biofuels in an economically advantageous manner. The vacuum distillation system is optionally located upstream of an esterification unit or other biodiesel production facility for improvement in production economy

WO 2009/018390 A1

Published:
— *with international search report*

Vacuum Distillation Process

This application claims priority under 35 U.S.C. 119(e) to U.S. provisional application 60/962,690, filed July 31, 2007, the contents of which are incorporated by reference in their entirety.

Field of the Invention

The present invention relates to the field of refining and/or producing hydrocarbons such as biofuels by the method of vacuum distillation.

Background

In an effort to partially replace dependence on petroleum-based diesel, vegetable oils have been directly added to diesel fuel. These vegetable oils are composed mainly of triglycerides, and often contain small amounts (typically between 1 and 10% by weight) of free fatty acids. Some vegetable oils may also contain small amounts (typically less than a few percent by weight) of mono- and di-glycerides.

Triglycerides are esters of glycerol, $CH_2(OH)CH(OH)CH_2(OH)$, and three fatty acids. Fatty acids are, in turn, aliphatic compounds containing 4 to 24 carbon atoms and having a terminal carboxyl group. Diglycerides are esters of glycerol and two fatty acids, and monoglycerides are esters of glycerol and one fatty acid. Naturally occurring fatty acids, with only minor exceptions, have an even number of carbon atoms and, if any unsaturation is present, the first double bond is generally located between the ninth and tenth carbon atoms. The characteristics of the triglyceride are influenced by the nature of their fatty acid residues.

The production of alkyl esters of glycerides by transesterification is a known process. However, transesterification suffers in that the reaction generally requires the addition of an acid or base catalyst which must be neutralized after the reaction thereby generating salts and soaps. In addition, while transesterification results in the separation of fatty acid esters from triglycerides, it also results in the production of glycerin, which must then be separated from the fatty acid esters, glycerin, excess alcohol, salts, and soaps.

The production of alkyl esters of fatty acids by acid catalyzed esterification is also known. However, the use a strong acid, such as sulfuric acid, typically leads to higher sulfur content in the resulting ester as the acid reacts with the double bonds in the fatty acid chains. In addition, conversion of the esterification reaction is limited by equilibrium constraints such that either excessive time and equipment size are required or less conversion needs to be accepted. In an effort to overcome some of the problems associated with transesterification, several attempts have been made to employ esterification between fatty acids and alcohols. In these processes, fatty acids are prepared from triglycerides by hydrolysis, followed by catalyzed esterification of the fatty acids with an alcohol, preferably methanol. While this practice is practiced in the production of fatty alcohols and fatty acid esters, as described in U.S. Pat. No. 5536,856 (Harrison et al), it has not been practiced in the production of Renewable Diesel.

Vacuum distillation of hydrocarbons is a refining process that can be utilized to minimize thermal cracking of heavier fractions of hydrocarbons and obtain lighter desired products. Distilling these materials under vacuum, i.e., lower pressure, decreases the boiling temperature of the various hydrocarbon fractions in the feed and therefore minimizes thermal cracking of these fractions.

In conventional vacuum distillation systems, distillation is carried out in a vacuum column under pressures typically in the range of 25 to 100 millimeters of mercury (mmHg). It is important in such systems to reduce pressure as much as possible to improve vaporization. Vaporization is enhanced by various methods such as the addition of steam at the furnace inlet and at the bottom of the vacuum distillation column. Vacuum is created and maintained using cooling water condensers and steam driven ejectors. The size and number of ejectors and condensers used is determined by the vacuum needed and the quantity and quality of vapors handled. While vacuum distillation is practiced in the production of petroleum-based products, it has not been practiced in the production of Renewable Diesel in a continuous process combined with esterification in a reactive distillation process.

Accordingly, one object of the present invention is to utilize vacuum distillation in combination with reactive distillation during esterification to produce a product that meets either ASTM D396 or ASTMD975 specifications or both.

Summary

The present invention provides a vacuum distillation system and method utilizing high free fatty acid feedstock. Operation of the distillation system enables production of esters including biodiesel and other biofuels in an economically advantageous manner. The vacuum distillation system is optionally located upstream of an esterification unit or other biodiesel production facility for improvement in production economy.

In one embodiment, the invention is a process for preparing Renewable Diesel comprising the step of vacuum distillation of oil of vegetable and/or animal origin followed by the step of esterification to yield diesel fuel according to either ASTM D396 or ASTM D975 or

both. In a preferred embodiment, the feedstock for the process is an oil of vegetable and/or animal origin which is selected from Palm Fatty Acid Distillate (PFAD), Palm Acid Oil (PAO), Acid Oil (Acidulated Soap Stock), and mixtures thereof. In one embodiment, the feedstock oil further comprises up to about 20% pre-split tallow and/or up to about 5% tall oil fatty acid. Alternatively, in one embodiment, the feedstock oil is essentially free of tallow or poultry fat.

In one embodiment, esterification is carried out by reactive distillation using a solid catalyst. Preferably, the solid catalyst is an ion exchange resin catalyst comprising -SO3H or -CO2H functional groups. Preferably, the step of esterification is performed with an alcohol selected from methanol, t-butanol, isobutanol, or a mixture thereof.

In one embodiment, a fat splitter may be integrated into the process, optionally between the vacuum distillation unit and the esterification unit.

In one embodiment, the Renewable Diesel is high value and low acidity. In a preferred embodiment, the process is carried out on an industrial scale. Preferably, the process is continuous.

Brief Description of the Drawings

Figure 1 shows one embodiment of the present reaction for the preparation of low acidity Renewable Diesel by vacuum distillation followed by reactive distillation esterification.

Figure 2 shows another embodiment of the present reaction for the preparation of low acidity Renewable Diesel by vacuum distillation followed by reactive distillation esterification.

Figure 3 shows a process flow diagram model according to the invention, providing details of various embodiments.

Detailed Description

The present invention provides a process for the production of Renewable Diesel fuels having low acidity, low glycerin, and low sulfur content, from oils of animal and vegetable origin. Renewable Diesel is a material derived from animal or vegetable origin which meets ASTM D396 and/or ASTM D975 specifications. The process of the present invention employs a combination of vacuum distillation and, if desired, reactive distillation esterification to produce fuels that meet either or both of these standards and therefore qualify as Renewable Diesel.

The animal or vegetable oil feedstocks for use according to the invention include, but are not limited to, fatty acids such as decanoic acid, dodecanoic acid, tetradecanoic acid, hexadecanoic,acid, octadecanoic acid, octadecenoic acid, linoleic acid, eicosanoic acid, isostearic acid and the like, as well as mixtures of two or more thereof as well as oils described as coconut oil, rape seed oil, and palm oils, tall oils, lard, bacon grease, yellow grease, tallow and fish oils. Additional oils may be sourced from whale oil and poultry fat, as well as corn, palm kernel, soybean, olive, sesame, and any other oils of animal or vegetal origin not explicitly identified herein. If desired, such mixtures of acids can be subjected to distillation to remove lower boiling acids having a lower boiling point than a chosen temperature (e.g. C_8 to C_{10} acids) and thus produce a "topped" mixture of acids. Optionally, the mixtures can be distilled to remove higher boiling acids having a boiling point higher than a second chosen temperature (e.g. C_{22}+ acids) and thus produce a "tailed" mixture of acids. Additionally, both lower and higher boiling acids may be removed and thus produce a "topped and tailed" mixture of acids. Such fatty acid mixtures may also contain ethylenically unsaturated acids such as oleic acid. Such mixtures may also contain fatty acid esters. In a preferred embodiment, the feedstock for the process is an oil of vegetable and/or animal origin which is selected from Palm Fatty Acid Distillate (PFAD), Palm

Acid Oil (PAO), Acid Oil (Acidulated Soap Stock), and mixtures thereof. In one embodiment, the feedstock oil further comprises up to about 20% pre-split tallow and/or up to about 5% tall oil fatty acid. For example, in one embodiment, about 115 MMlb/yr of pre-split tallow may be used. Use of tall oil fatty acid is envisioned as a cold flow improving blendstock. Alternatively, in one embodiment, the feedstock oil is essentially free of tallow or poultry fat. By essentially free is meant that tallow or poultry fat are not introduced into the feedstock, or in the event of residual contamination from processing of various batches, are not introduced in quantities that affect the processing parameters of the feedstock.

In one embodiment, the purpose of vacuum distillation is to distill all or nearly all fatty acids and those triglycerides not needed for steam generation while leaving a pitch that contains high-boiling material. In one embodiment, additional beneficial effects may be obtained at higher pressures and temperatures, such as additional fat splitting. In a preferred embodiment, four cuts are made during distillation: 1) water, glycerin, fatty acids, and low boiling material; 2) fatty acids and monoglycerides; 3) tri- and di-glycerides; and 4) pitch. In one embodiment, five cuts are made during distillation: 1) water and low boiling material; 2) glycerin and fatty acids; 3) fatty acids and monoglycerides; 4) tri- and di-glycerides; and 5) pitch.

In a preferred embodiment, steam from a pre-existing natural gas turbine or coal power plant is used to reduce costs of construction. In a preferred embodiment, pitch or waste organics generated during vacuum distillation are sold to a third-party to improve the overall economic equation for the process.

Reactive Distillation Esterification refers to a process taking place in a column so designed such that the vapor stream of the more volatile of the two components, (i.e. the more volatile of the vacuum distillation product component and the alcohol component), flows

countercurrent to the less volatile component such that the vapor stream carries away water produced in the esterification reaction, while advantageously not carrying away a significant quantity of the less volatile component. For this reason it is essential that the boiling point of the vapor mixture exiting the esterification reactor, or of the highest boiling compound present in that vapor mixture, be significantly lower, at the pressure prevailing in the uppermost stage of the esterification reactor, than the boiling point at that pressure of either of the less volatile one of the two components. By the phrase "significantly lower" is meant that the boiling point difference shall be at least about 20°C, and preferably at least about 25°C, at the relevant operating pressure of the column. In practice, the more volatile component of the two will frequently be the alcohol component. For example, methanol will be the more volatile component in the production from fatty acid mixtures obtained by the hydrolysis of triglycerides of methyl fatty acid ester mixtures for subsequent processing, for example for production of detergent alcohols by ester hydrogenation.

In one aspect of the present invention, Renewable Diesel fuels prepared according to the present invention are provided. Sulfur content of the Renewable Diesel fuel is one of many parameters of interest for commercial use. Sulfur is typically present as a result of the use of sulfuric acid catalysts, and can result in increased engine wear and deposits. Additionally, environmental concerns dictate a desired low sulfur content in the Renewable Diesel fuel. Preferably, Renewable Diesels prepared according the methods provided herein have a sulfur content (as measured by ASTM test method D5453) of less than 500 ppm, more preferably less than 200 ppm, less than 100 ppm, less than 50 ppm, less than 25 ppm, less than 10 ppm, and most preferably less than 5 ppm.

The cetane number (i.e., the measure of the ignition quality of the fuel, as measured by ASTM test methods D976 or D4737) is preferably greater than 47, more preferably greater than 50, and most preferably greater than 55.

Cloud points are defined as the temperature at which a cloud or haze of crystals appears in the fuel. Cloud points determine the climate and season in which the Renewable Diesel fuel may be used. Preferably the cloud point of the Renewable Diesel is less than 0°C, more preferably less than -5°C, less than -10°C, less than -15°C, less than -20°C, less than -25°C, less than -30°C, less than -35°C, less than -40°C, and most preferably, less than -45°C.

Total free glycerin in the Renewable Diesel is preferably less than 0.03% by weight, more preferably less than 0.20% by weight, less than 0.018% by weight, less than 0.016% by weight, and most preferably, less than 0.015% by weight. Total glycerin present in the Renewable Diesel fuel is preferably less than 0.25% by weight, more preferably less than 0.24% by weight, less than 0.23% by weight, less than 0.22% by weight, 0.21% by weight, and most preferably, less than 0.20% by weight.

Residual methanol in the Renewable Diesel is desired to be minimized, and is preferably less than 0.2% by weight, more preferably less than 0.18% by weight, and most preferably less than 0.15% by weight.

Water content in the Renewable Diesel fuel produced according the present invention is preferably less than 500 ppm, preferably less than 450 ppm, more preferably less than 400 ppm and most preferably less than 300 ppm.

It can be important to define a minimum viscosity of the Renewable Diesel fuel because of power loss due to injection pump and injector leakage. Preferably, the viscosity of the

Renewable Diesel fuel is between 1.0 and 50 mm^2/s, more preferably between 1.3 and 15.0 mm^2/s, even more preferably between 1.3and 2.1 mm^2/s.

In one embodiment, the Renewable Diesel is produced on an industrial scale. Global biodiesel production is estimated at several million tons per year. In a preferred embodiment, production occurs from 500 kg or more of feedstock per day. Alternatively, production may occur on batches or continuous feeds of 1,000 kg, 5,000 kg, 10,000 kg or more feedstock per day. Alternatively, vacuum distillation may occur on a scale of about 200 tons per day, while esterification may occur on a scale of about 300 tons per day. In one embodiment, a fat splitter with capacity of, for example, about 100 tons per day or about 150 tons per day, may be integrated into the process, optionally between the vacuum distillation unit and the esterification unit. Additionally, a properly scaled glycerin concentration unit assuming feedstock with about 30% free fatty acid may be incorporated into the process. Glycerin recovery is envisioned to account for glycerin carryover from the vacuum distillation process, if present.

Referring now to Figure 1, there is provided an embodiment of a process for the preparation of Renewable Diesel from animal and vegetable oils. Feed Stream **1** contains liquids derived from animal and vegetable sources. Such liquids may contain fatty acids, glycerides of fatty acids, esters, alcohols, and other hydrocarbons. Feed stream **1** could also contain petroleum derived hydrocarbons. Feed stream **1** is fed to a vacuum distillation unit **2.**

Vacuum distillation unit **2** may or may not be equipped to contain a thermal oxidizer for management of tank vapors as well as an emergency MeOH scrubber able to operate without plant power. Preferably, vacuum distillation unit **2** can operate without any fired heaters, but a steam heating and/or hot oil system may optionally be included to allow for distillation at higher temperatures. Vacuum distillation unit **2,** and any coupled separation device such as a glycerin

condensing unit or a fatty acid splitter, is operated in order to, at a minimum, provide free fatty acids to the reactive distillation esterification unit. Vacuum distillation unit **2** and any coupled device therewith incorporated may produce a product **3** consisting of lower boiling hydrocarbons, CO, CO_2, hydrogen, and water and liquid product **4**. Liquid product **4** of vacuum distillation unit **2** may or may not meet all specifications of ASTM D396 and/or D975 at this point, but liquid product **4** may meet the distillation and flash point ranges are near as possible.

In one embodiment, liquid product **4** is fed to Reactive Distillation Esterification Unit **6.** Reactive Distillation Esterification Unit **6** is also fed with an alcohol stream **5**. Within the Reactive Distillation Esterification Unit **6,** acidic components in the liquid product **4** are reacted with the alcohol from stream **5** and converted to esters product **7**. Water of reaction and alcohol are also separated so that excess alcohol used in the reaction can be recycled.

Referring to Figure 2**,** the same process is contemplated with the difference being the feeding of stream **8** along with the feedstock in stream **1.** Stream **8** contains water or steam. Feeding steam or water as stream **8** along with feed stream **1** is intended to help maximize the output of liquid product **4**. In one embodiment, Figure 2 corresponds in all other regards to Figure 1.

Referring to Figure 3, one possible embodiment according to the invention is provided, including a vacuum distillation unit and an esterification unit.

It will be understood by those skilled in the art that the drawings are diagrammatic and that further items of equipment such as reflux drums, pumps, vacuum pumps, temperature sensors, pressure sensors, pressure relief valves, control valves, flow controllers, level controllers, holding tanks, storage tanks, and the like may be required in a commercial plant.

The provision of such ancillary items of equipment forms no part of the present invention and is in accordance with conventional chemical engineering practice.

Modifications and variations of the present invention relating to the selection of reactors, feedstocks, alcohols and catalysts will be obvious to those skilled in the art from the foregoing detailed description of the invention. Such modifications and variations are intended to come within the scope of the appended claims. All numerical values are understood to be prefaced by the term "about" where appropriate. All references cited herein are hereby incorporated by reference in their entirety.

The process and apparatus of the invention can be used in biodiesel refining, and in petrochemical and other industries where vacuum processing of liquid products is required. It is possible to economically integrate the invention process into conventional vacuum distillation systems. It should be noted that various changes and amendments can be made in the details within the scope of the claims set forth below without departing from the spirit of the claimed invention. It should therefore be understood that the claimed invention should not be limited to the specific details shown and described.

Claims

1. A process for preparing Renewable Diesel comprising the step of vacuum distillation of oil of vegetable and/or animal origin followed by the step of esterification to yield diesel fuel according to either ASTM D396 or ASTM D975 or both.

2. The process of claim 1, wherein the oil of vegetable and/or animal origin is selected from Palm Fatty Acid Distillate (PFAD), Palm Acid Oil (PAO), Acid Oil (Acidulated Soap Stock), and mixtures thereof.

3. The process of claim 2, wherein the oil further comprises up to about 20% pre-split tallow and/or up to about 5% tall oil fatty acid.

4. The process of claim 2, wherein the oil is essentially free of tallow or poultry fat.

5. The process of claim 1, wherein esterification is carried out by reactive distillation using a solid catalyst.

6. The process of claim 5, wherein the solid catalyst is an ion exchange resin catalyst comprising $-SO_3H$ or $-CO_2H$ functional groups.

7. The process of claim 1, wherein the process is carried out on an industrial scale.

8. The process of claim 1, wherein the Renewable Diesel is high value and low acidity.

9. The process of claim 1, wherein the process is continuous.

10. The process of claim 1, wherein the step of esterification is performed with an alcohol selected from methanol, t-butanol, isobutanol, or a mixture thereof.

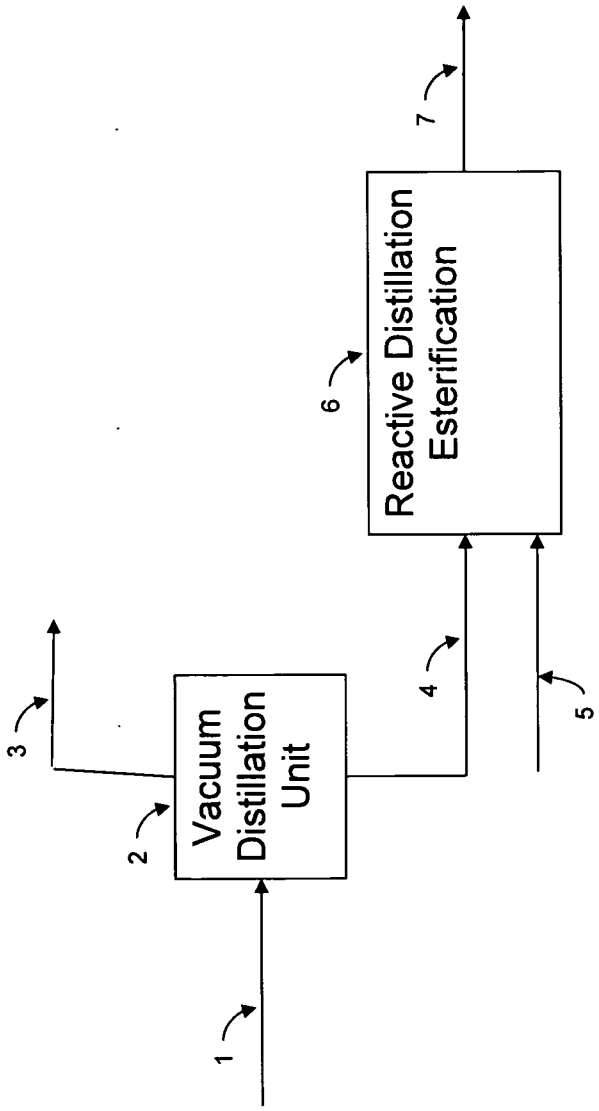

Vacuum Distillation Unit
Reactive Distillation Esterification
1
2
3
4
5
6
7

FIGURE 1

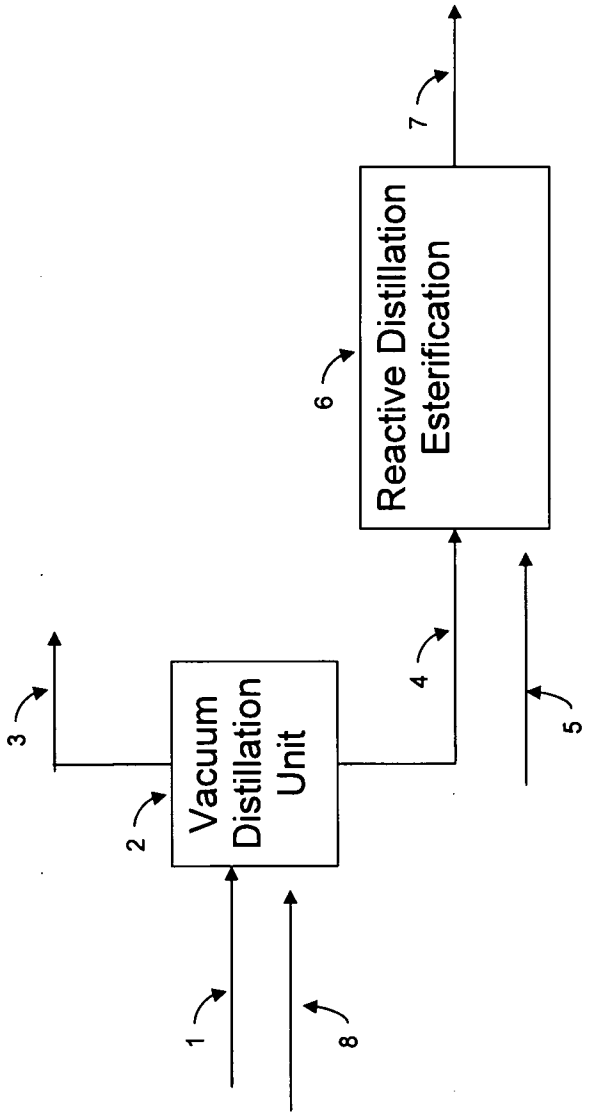

Vacuum Distillation Unit
Reactive Distillation Esterification
1
2
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8

FIGURE 2

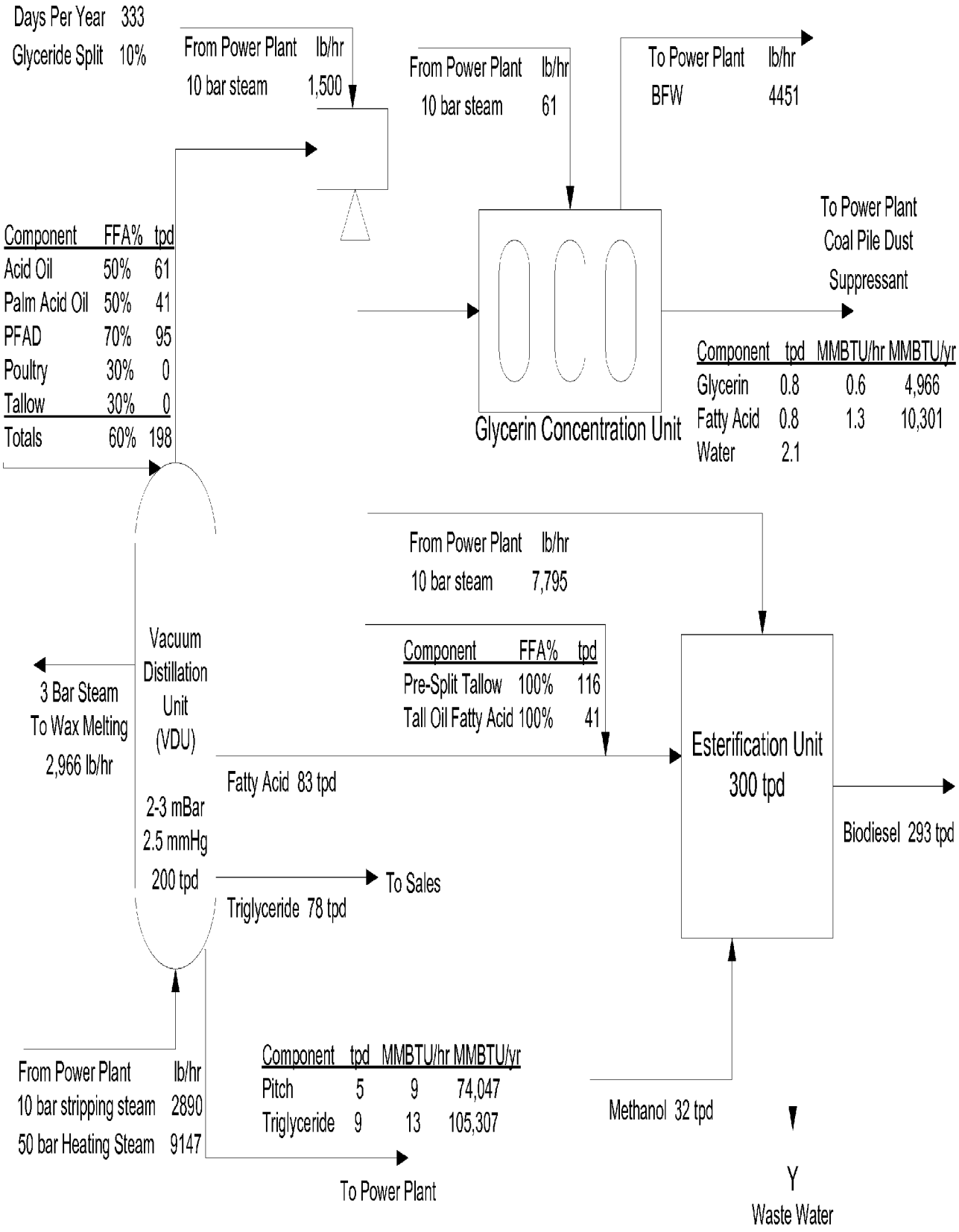

Days Per Year 333
Glyceride Split 10%
From Power Plant lb/hr
10 bar steam 1,500
From Power Plant lb/hr
10 bar steam 61
To Power Plant lb/hr
BFW 4451
Component FFA% tpd
Acid Oil 50% 61
Palm Acid Oil 50% 41
PFAD 70% 95
Poultry 30% 0
Tallow 30% 0
Totals 60% 198
To Power Plant
Coal Pile Dust
Suppressant
Component tpd MMBTU/hr MMBTU/yr
Glycerin 0.8 0.6 4,966
Fatty Acid 0.8 1.3 10,301
Water 2.1
Glycerin Concentration Unit
From Power Plant lb/hr
10 bar steam 7,795
Component FFA% tpd
Pre-Split Tallow 100% 116
Tall Oil Fatty Acid 100% 41
Vacuum Distillation Unit (VDU)
3 Bar Steam To Wax Melting 2,966 lb/hr
Fatty Acid 83 tpd
2-3 mBar 2.5 mmHg 200 tpd
Triglyceride 78 tpd
To Sales
Esterification Unit 300 tpd
Biodiesel 293 tpd
From Power Plant lb/hr
10 bar stripping steam 2890
50 bar Heating Steam 9147
Component tpd MMBTU/hr MMBTU/yr
Pitch 5 9 74,047
Triglyceride 9 13 105,307
To Power Plant
Methanol 32 tpd
Y
Waste Water

Figure 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 08/71660

A. CLASSIFICATION OF SUBJECT MATTER
IPC(8) - C10L 1/00 (2008.04)
USPC - 44/307, 310
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
USPC - 44/307, 310

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
PubWest, GoogleScholar
search terms: Diesel, distillation, esterification, animal, vegetable, vacuum distillation, process, method, reactive distillation, solid catalyst

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 7,045,100 B2 (Ergun et al.) 16 May 2006 (16.05.2006) (abstract; col 1, ln 20-33; col 2 ln 30-37, 40-42; col 4, ln 26-40); (col 6, ln 3-6). (col 7, ln 41-43).	1-10
Y	US 2005/0039384 A1 (Gormley) 24 February 2005 (24.02.2005) para [0032]	1-10
Y	US 5,399,731 A (Wimmer) 21 March 1995 (21.03.2005) (col 3, ln 33-55).	2-4, 10
Y	US 2007/0033865 A1 (Caprotti et al.) 15 February 2007 (15.02.2007) para [0010]	3
Y	Lotero et al. "The Catalysis of Biodiesel Synthesis." Catalysis, Volume 19. 2006. (p.75, par. 2); (p.75, par. 4).	5, 6

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

* Special categories of cited documents:
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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
14 October 2008 (14.10.2008)	20 OCT 2008
Name and mailing address of the ISA/US Mail Stop PCT, Attn: ISA/US, Commissioner for Patents P.O. Box 1450, Alexandria, Virginia 22313-1450 Facsimile No. 571-273-3201	Authorized officer: Lee W. Young PCT Helpdesk: 571-272-4300 PCT OSP: 571-272-7774

Form PCT/ISA/210 (second sheet) (April 2007)