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(54) **METHOD OF PRODUCING BIOFUELS, AND
RELATED APPARATUS**

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(57) **ABSTRACT**
Methods of producing biofuels are provided herein, for
example and without limitation methods of producing
biodiesel from trap grease are provided. Systems and appa-
ratus also are provided for implementing, for example and
without limitation, the methods described herein.

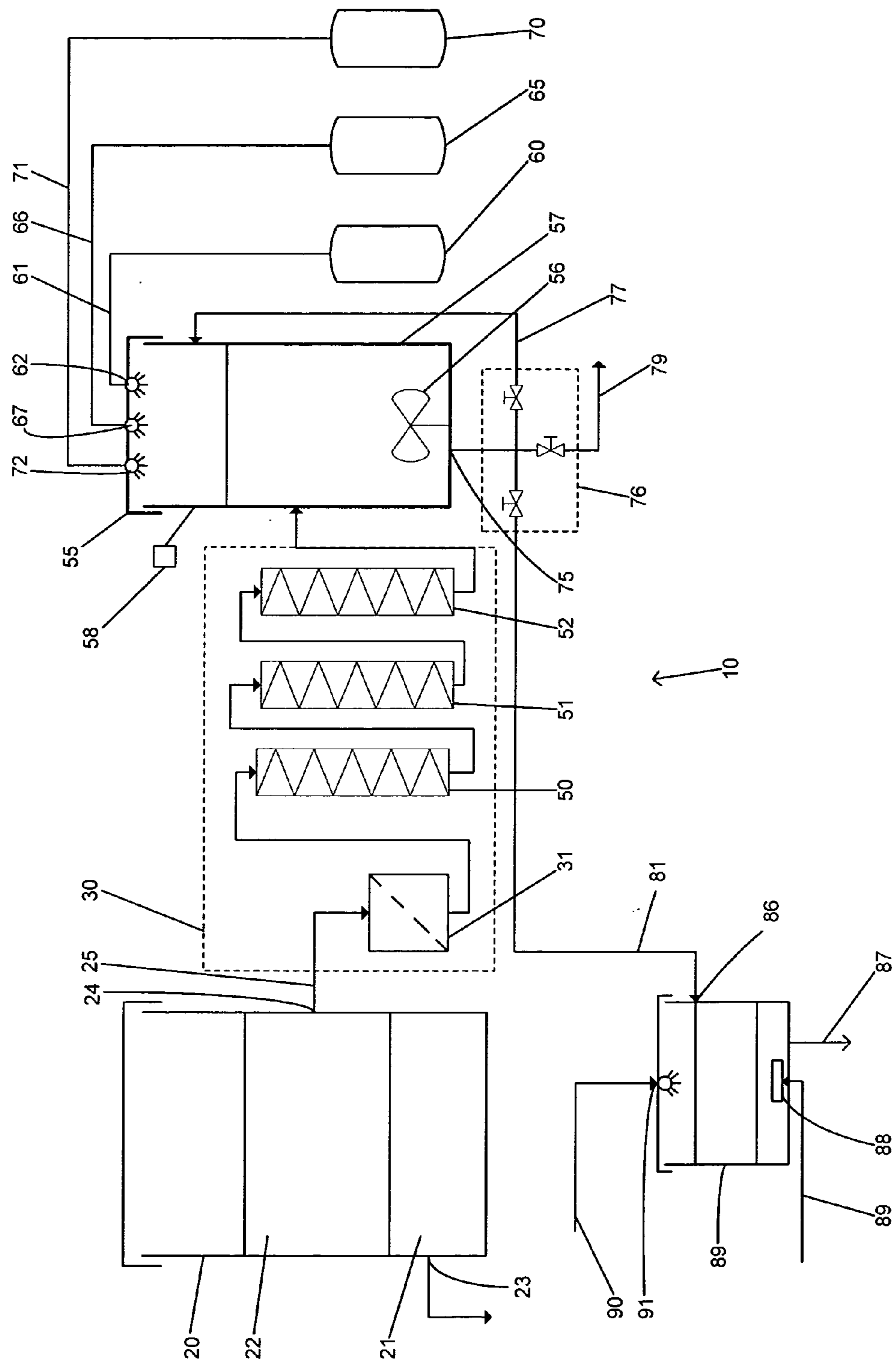


Fig. 1

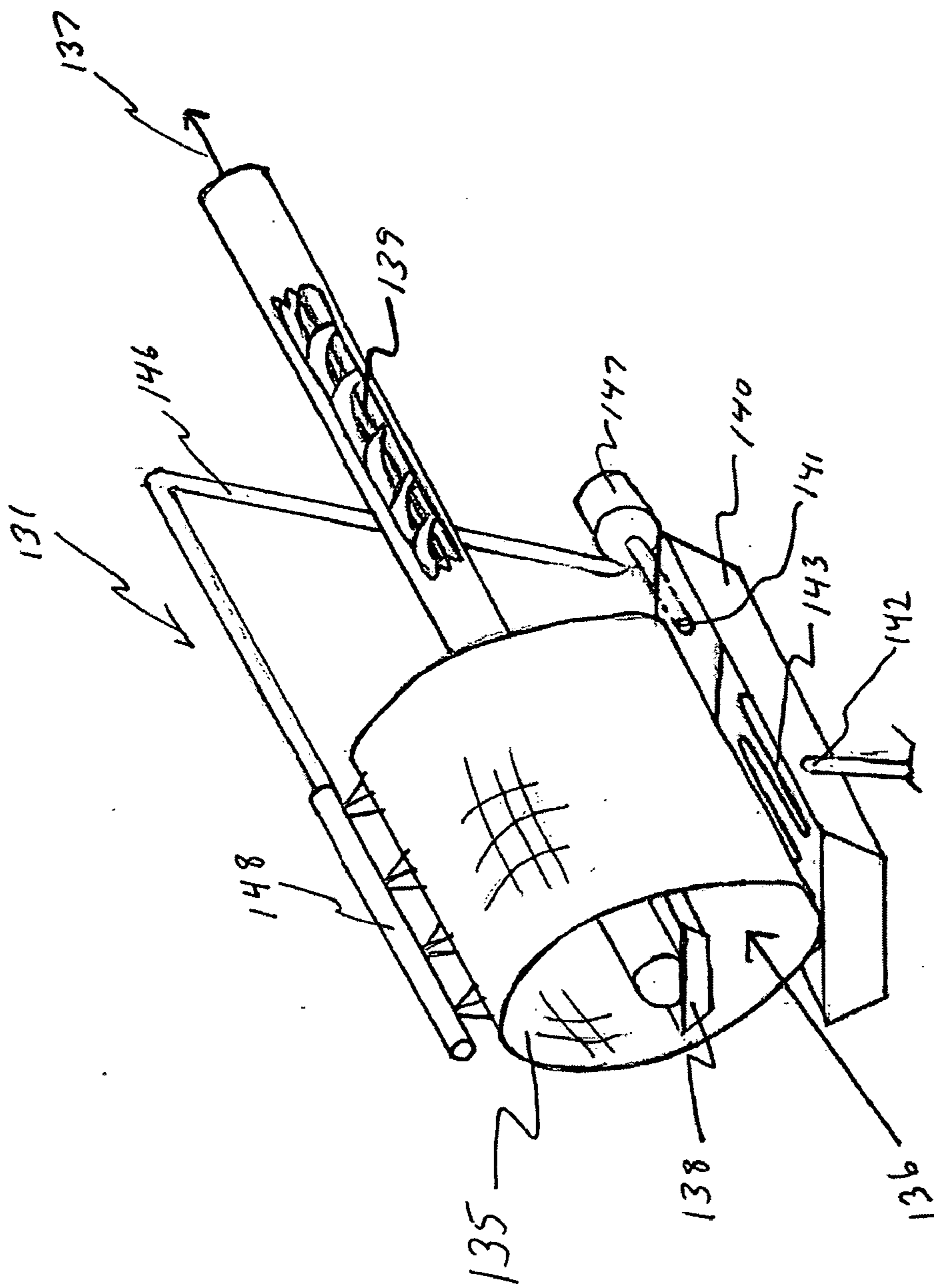


Fig. 2

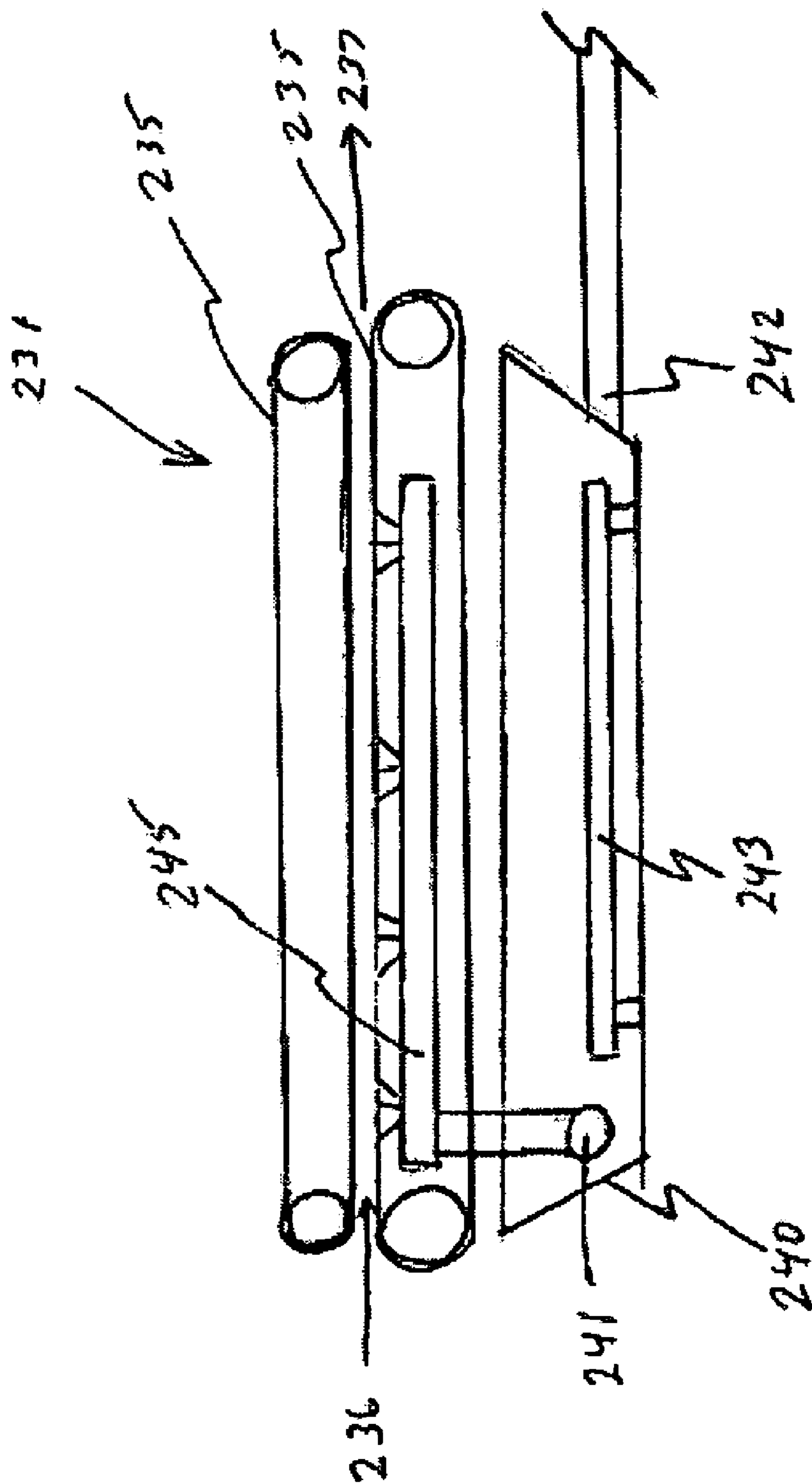


Fig. 3

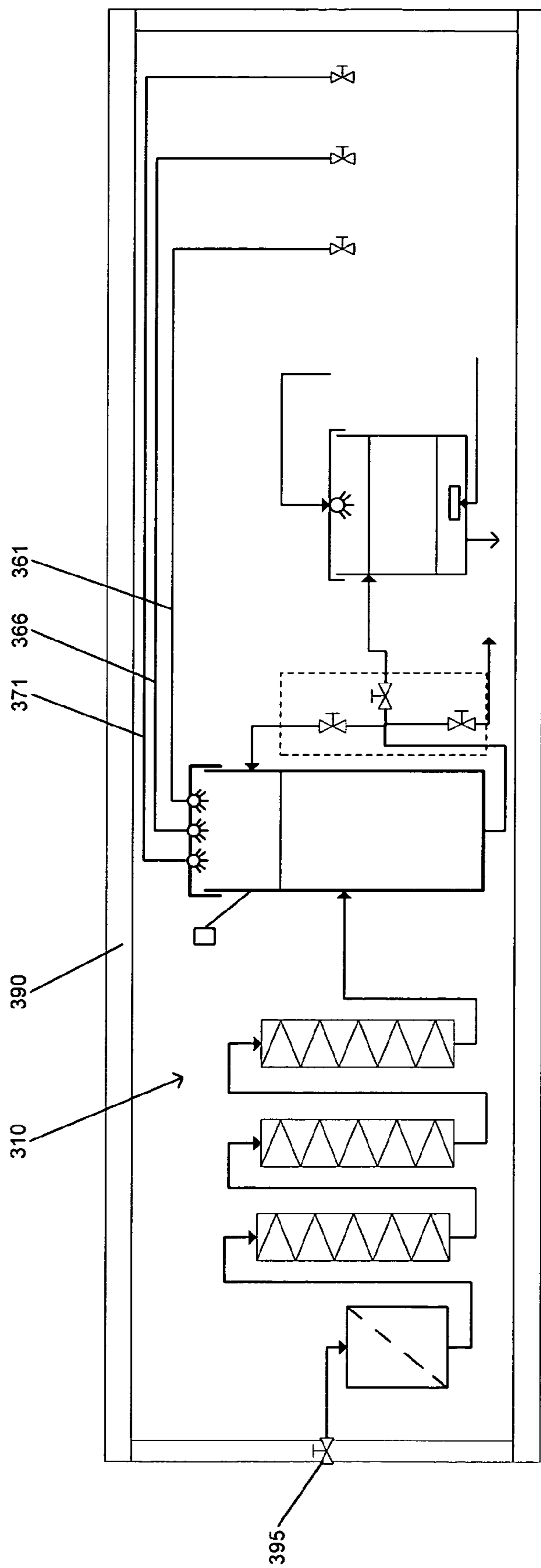


Fig. 4

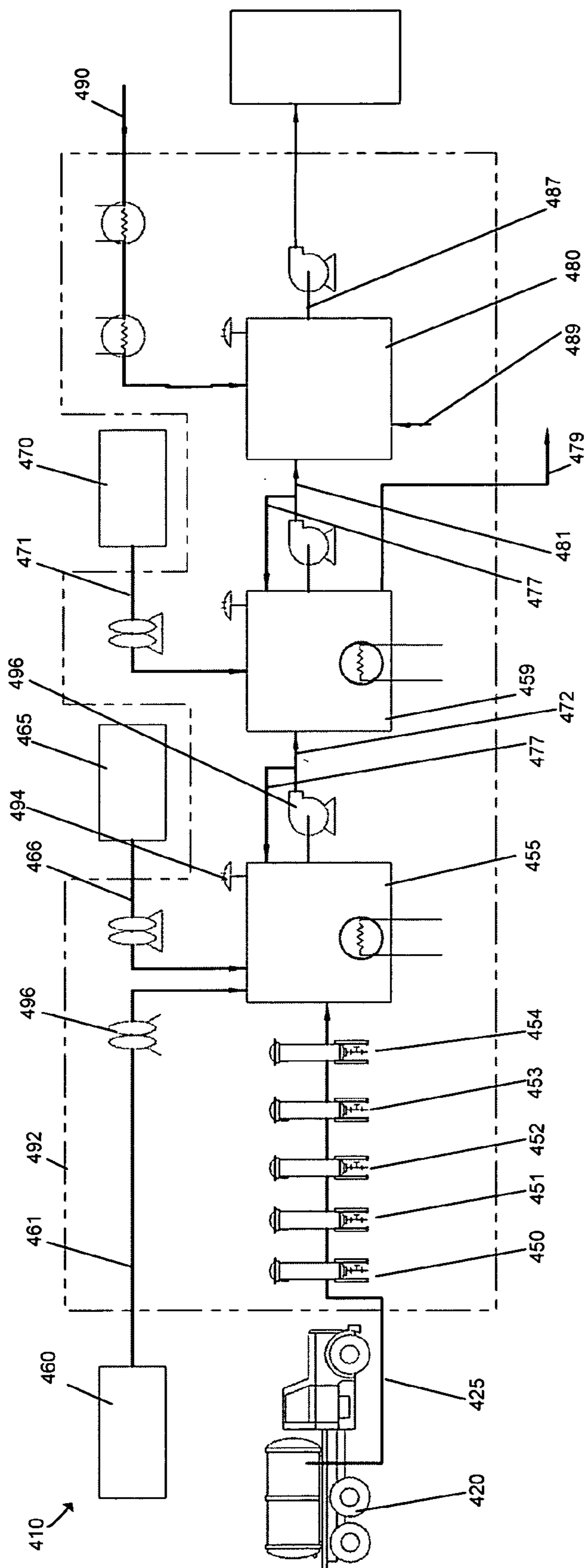


Fig. 5

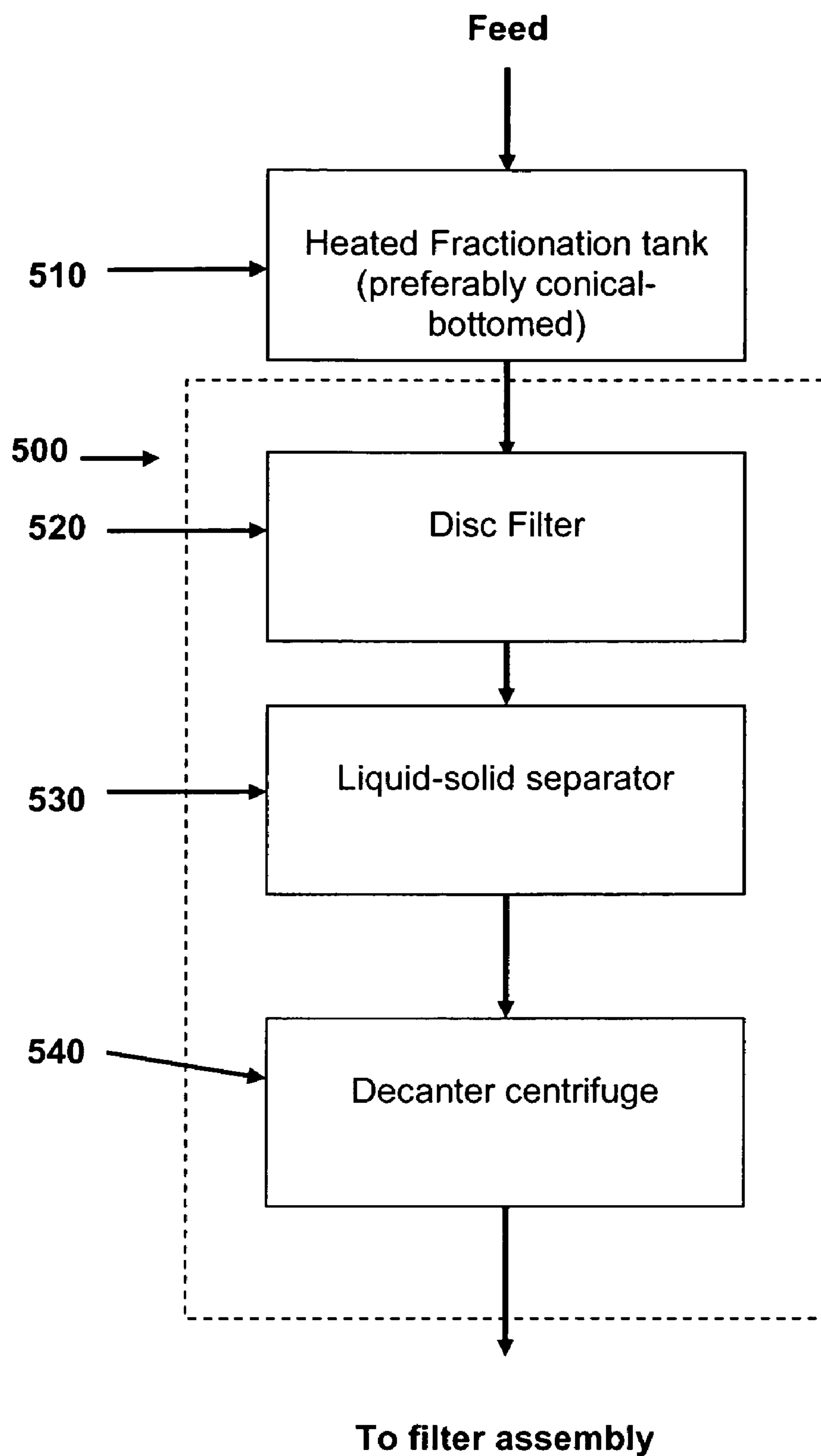


Fig. 6

METHOD OF PRODUCING BIOFUELS, AND RELATED APPARATUS

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit under 35 U.S.C. §119(e) to U.S. Provisional Patent Application Nos. 60/697,047, filed Jul. 6, 2005, and 60/791,771, filed Apr. 13, 2006, both of which are incorporated herein by reference in their entirety.

BACKGROUND

[0002] Today, municipalities are plagued by an increasing amount of food service generated waste oil and grease that accumulate in the sewage network, or is dumped illegally into the ground. As a result, in most dense urban areas, costly fees have been levied on both the restaurants and the liquid waste haulers and carters for the safe disposal of the waste oil and grease. Presently, liquid waste disposal prices have increased by 800% from 2002 to 2004 for carters. Since the waste must be carted on average 185 miles from its point of origin, the costs are even higher. For the fiscal year 2002, restaurants were paying \$65 for the disposal of 1,000 gallons of grease. As of October 2004, the same facilities were paying as much as \$500 per 1,000 gallons.

[0003] Major metropolitan environmental and sewer authorities have begun to require that all restaurants have approved carters for restaurant liquid wastes. Legislation has been passed in New York City and Long Island, with identical legislation pending in New Jersey, Connecticut and 14 other states. Fines for failing to comply with this law range from \$250 to as high as \$ 1,000 per day in New York City. For the liquid waste carters, the cost of legal disposal has also jumped from \$30 per 1,000 gallons of grease to \$240 per 1,000 gallons. All estimates indicate a continuation of rising disposal prices for 2005 and 2006.

[0004] This sudden increase in price coincides with an historic rise in energy costs. Fuel costs for home heating have increased by 42% nationwide, with increases as high as 60%. Prices for motor vehicle and machine diesel fuel have increased by 125% nationwide.

[0005] Biodiesel is a product derived from natural oils and fatty acids, and typically, though not exclusively, contains methoxylated and ethoxylated free fatty acids, or more generally alkoxyated free fatty acids (methyl, ethyl and alkyl esters of fatty acids).

[0006] Biodiesel typically is made from vegetable oils, mono-, di- and tri-glycerides (mono-, di- and tri-acylglycerides), by transesterification whereby the glycerin is separated from the fat or vegetable oil. The process leaves behind two products—alkyl (for example, methyl) esters of fatty acids and glycerin. An alcohol and a catalyst are required by traditional processes for converting oils to biodiesel. Typical alcohols, due to their low cost and abundance, include methanol and ethanol. Methanol is preferred in many instances because it leads to a more robust transesterification reaction and it is less expensive than ethanol. Methanol, however, dissolves rubber and is toxic. Ethanol is more expensive and often does not lead to as stable and predictable a transesterification reaction as methanol, but it is less toxic than methanol and typically is made from renewable

resources. The most common catalyst used in production of biodiesel is lye, or NaOH, though KOH and H₂SO₄ have also been used in some instances.

SUMMARY

[0007] Provided are methods and apparatus for producing biofuels, for example, and without limitation, biofuels prepared from trap grease. The methods and apparatus utilize a novel chemistry, as well as, in the case of trap grease and other viscous, dirty sources of fatty acid acyl glycerides, novel heating and separation techniques designed to facilitate use of dirtier, more viscous waste materials. In one embodiment, a fatty acid acyl glyceride-containing material is heated, filtered, esterified with acid and methanol and then treated with a base to transesterify the esterified fatty acid acyl glycerides. This process differs in that both esterification and transesterification are performed. The process is broken in two phases. In the first, esterification phase acid esterification is used to create an initial separation of glycerin and to reduce total free fatty acids of the source feed stock to less the 5%. In the second phase, fatty acid-acyl glycerides are transesterified to produce biofuel and glycerol.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] FIG. 1 is a schematic diagram a biofuels processing system according to one embodiment of the present invention.

[0009] FIG. 2 is a schematic diagram of a cylindrical screening device according to one embodiment of the present invention.

[0010] FIG. 3 is a schematic diagram of a screening device according to one embodiment of the present invention.

[0011] FIG. 4 is a schematic diagram of a biofuels processing system within a commercial shipping container according to one embodiment of the present invention.

[0012] FIG. 5 is a schematic diagram a biofuels processing system according to another embodiment of the present invention.

[0013] FIG. 6 is a flow chart illustrating one embodiment of a water-grease-solids separation assembly.

DETAILED DESCRIPTION

[0014] Provided herein is a method of preparing biofuels, in one non-limiting embodiment, from trap grease or any grease comprising predominantly vegetable oils and free fatty acids that is viscous at room temperature (“vegetable oil grease”). Trap grease is a waste product generated by restaurants. Most restaurants are required to run their waste water through a trap in which waste organics, predominantly viscous oils, including glycerides, predominantly triglycerides, but also containing di- and mono-glycerides, and free fatty acids, collect. These oils are viscous at room temperature and, thus are referred to as greases. Due primarily to the viscosity of trap grease and the fact that it contains significant amounts of particulate matter, it has not been previously considered as a suitable starting material for production of biofuels. As shown herein, use of trap grease requires special handling.

[0015] As mentioned above, methods and apparatus are provided for producing biofuels, for example, and without limitation, biofuels prepared from trap grease. The methods and apparatus utilize a novel chemistry, as well as, in the case of trap grease and other viscous, dirty sources of fatty acid acyl glycerides, novel heating and separation techniques designed to facilitate use of dirtier, more viscous waste materials. In one embodiment, a fatty acid acyl glycerides-containing material is heated, filtered, esterified with acid and methanol and then treated with a base to transesterify the fatty acid acyl glycerides. This process differs in that both esterification and transesterification are performed. The process is broken in two phases. In the first, esterification phase acid esterification is used to create an initial separation of glycerin and to reduce total free fatty acids of the source feed stock to less the 5%.

[0016] Acid, for example and without limitation, H_2SO_4 , is introduced into the heated grease typically at a rate of from about 1.0 ml per liter of feed stock to 1.7 ml/L to a pH of about 2.5. After acid is introduced, the mixture is agitated thoroughly before addition of methanol in order to prevent dimethylether creation. Methanol is then added to from about 10% to about 15% by volume, and the mixture is agitated for about 2 hours. Glycerol is then settled out and is removed.

[0017] Acid is then neutralized by injection of a base, preferably KOH or Potassium methylate to reach a neutral pH of from about 6.2 to about 8.0. Use of potassium methylate or KOH is preferred. Note also in this phase that the potassium and SO_4^{-2} combine to form K_2SO_4 in suspension, which is removed in the wash phase and which can be used as a fertilizer. In this phase (base phase), transesterification occurs, resulting in formation of biofuel (fatty acid methoxide) and glycerol.

[0018] FIG. 1 provides a schematic of one embodiment of a processing system 10 suitable for preparing biofuels from trap grease. System 10 includes fractionating tank 20 in which an aqueous fraction 21 (graywater) is separated from organic fraction 22 (grease). Tank contains drains 23 and 24. Aqueous fraction 21 is drained from tank 20 through drain 23, typically as waste-water to be discarded. Organic fraction 22 is drained from tank 20 through drain 24, fluid conduit 25 and into filter subsystem 30, which includes a pre-filter 31 and filters 50, 51 and 52. In practice, one or more of pre-filters 31 and filters 50, 51 and 52 may be omitted, and additional pre-filters or filters, as are known in the art, may be employed.

[0019] The viscosity of these greases is such that standard biofuels processing techniques cannot be employed. Further, unlike typical processes for producing biofuels, the trap grease contains a good deal of particulate matter that must be filtered out of the grease before the grease is processed into biofuel. As such, in one non-limiting embodiment, pre-filter 31 is a modified screening filter, such as a modified Rotomat. A modified cylindrical screening device 131 is shown in FIG. 2. The device 131 includes a cylindrical screen 135 through which a liquid (filtrate) can pass, leaving within the cylindrical screen 135 any solids that could not pass through the screen 135. Device 131 includes an inlet 136 and an outlet 137. Solids are carried from inlet 136 to outlet 137 by screw 139. In a typical commercial cylindrical screening device 131, such as a Rotamat, inlet 136 is lower

than outlet 137. The liquid carried along screen 135 and is scraped into trough 138, and any materials that could not pass through the screen is carried by screw 139 to outlet 137. Device 131 also comprises trough 140 configured to collect liquid (filtrate) that passes through screen 135. Trough 140 also comprises outlets 141 and 142 for transferring collected filtrate out of trough 140. Trough 140 also comprises a heater element 143, such as, without limitation, a resistance heater for heating filtrate. A recirculation system, comprising a conduit 146, pump 147 and spray head 148 are fluidly connected to outlet 141, and is used to spray heated filtrate onto screen, thereby heating un-filtered trap grease and thereby decreasing its viscosity and dislodging particulate matter from screen 135 with the overall effect of increasing the efficiency of separation of particulate matter from filtrate. Outlet 142 is fluidly connected with down-stream filter units, such as filters 50, 51 and 52 shown in FIG. 1.

[0020] As can be appreciated, a cylindrical screening device 131, as is shown in FIG. 2 is one of many suitable topological configurations for a coarse filter screen. Commercial sources of various cylindrical screening devices that can be modified with a heater as shown in FIG. 2 are available from Huber Technology, Ltd. of Wiltshire, England under the trade name ROTAMAT. Filter screens of this type are typically used in waste-water treatment to remove particulate matter from storm or sewage prior to treatment. In a second embodiment shown in schematic in FIG. 3, device 231, shown in cross-section, includes one or more screens 235 in the configuration of two opposing continuous belts. Trap grease enters inlet 236 of device 231 and particulate retentate passes through device and exits through outlet 237. Filtrate collects in trough 240, which includes outlets 241 and 242 and heater 243. Recirculation system 245 is provided to spray heated filtrate onto screens 235 to heat trap grease and to dislodge particulate matter from screens 235.

[0021] Returning to FIG. 1, trap grease is processed into a biofuel in system 10, shown schematically. Heated trap grease filtrate exiting from pre-filter 31, for example and without limitation, as shown in FIGS. 2 and 3, is then filtered by one or more filter units 50, 51 and 52. In reference to FIG. 1, three filter units 50, 51 and 52 are shown. Filter units 50, 51 and 52 may be the same or different, but filter units are typically, sequentially from filter 50 to filter 51 to filter 52, of a finer filter size. For example and without limitation, filter 50 is a 10-mesh filter, filter 51 is a 20-mesh filter and filter 52 is an 80-mesh filter. Alternately cartridge-based pool filters may be employed, such as Hayward filters. As used herein, the terms "pore size" of a filtering device refers to the minimum size of particles that a filter can stop. Often, the pore size is described as a "mesh size" or in terms of "mesh" units, though it should be recognized that "mesh sizes" may have different values in different countries. For example, 20 mesh refers to a 920 micron (μ) rating in the UK and an 864 μ rating in the US. Reference to mesh size made herein refer to the US mesh standard. It should be recognized by a person of ordinary skill in the art that selection of filter pore size and sequences of serial filters of differing pore size is a matter of design preference and is based on the desired degree of particle removal and how often the filters need to be cleaned or replaced.

[0022] Filtrate exits filter 52 and then passes into reaction tank 55 containing stirrer 56. Reaction tank 55 includes wall

57, which comprises a resistance heating element (not shown) under control of programmable logic controller (PLC) 58 for controlling and maintaining temperature within tank 55. In one non-limiting embodiment, reaction 55 tank is a conical tank. Resistance heating elements and temperature controllers are available from a number of commercial sources, including, for example and without limitation, PolyProcessing Company of Monroe, La.

[0023] Temperature, pH, conductivity and viscosity, as well as other parameters, may be monitored within one or more reaction vessels or transfer lines employed in the systems and processes described herein. By “meters” (for example, and without limitation, pH and temperature meters), it is meant a device that measures one or more parameters. By “controllers” (for example, and without limitation, pH and temperature controllers (e.g., thermostat)), it is meant a device that measures one or more parameters and then can control the operation of a device for adjusting the one or more parameters. In the context of the systems and processes described herein, this means that a pH controller may control, without limitation, a pump or solenoid that controls flow of an acid or base into a reaction vessel, and shuts the flow of the acid off if a specified pH is reached, typically about 2.5 for the acid addition step of the processes described herein. Useful meters and probes for monitoring and/or controlling industrial processes are well-known and are readily available commercially. In one embodiment, Program Automation Controllers (PACs, also known as Programmable Logic Controllers or PLCs) are used. In another embodiment, redundant PACs are utilized. In one embodiment, the PACs are Chameleon Programmable Automation Controllers, commercially available from Fairmount Automation or Phoenixville, Pa.

[0024] In one embodiment, conductivity of the oil- or biofuel-containing fractions is measured to evaluate the completeness of the separations. Prior to addition of acid, conductivity of the fatty acid acyl-glyceride-containing composition may be evaluated to determine the completeness of any dewatering step. After acid and methanol treatment, subsequent settling and removal of glycerol, the completeness of glycerol removal can be determined by conductivity. Likewise, the conductivity of pre-wash biofuel, post-wash biofuel and the final product may be evaluated.

[0025] As used herein, the term “fluidly connected” means connected by a fluid transfer conduit or any other method that permits fluid transfer, with or without intervening elements, such as, without limitation, containers, filters, devices, pumps, valves, etc. A non-limiting example, two reaction vessels may be “fluidly connected” if they are connected to each-other through a pipe or tube, even if a pump, manifold, valve or other device is placed in-line between the vessels. Likewise, a pre-filter assembly is considered to be fluidly connected to a reaction vessel, even though one or more filters lay in-line between the pre-filter and the vessel. Two elements are considered to be “fluidly connected” even though there is no pipe or tubing making the connection if the first element spills or otherwise drains, overflows, siphons or transfers into the second element, though there may be no actual physical connection between the two elements in the form of a pipe or tube. In reference to a process system, the term “downstream” means a later in the direction of general process and/or fluid flow and

“upstream” means earlier in the direction of general process and/or flow. Thus, acid feed tanks and acid feeds are upstream of the reaction vessel into which the acid feed drains because acid feeds from the acid feed tank through the acid feed and into the reaction vessel. Likewise, as shown in the figures, a pre-filter assembly is upstream of the reaction vessel because fluid and the overall process flows in the direction from the pre-filter assembly to the reaction vessels.

[0026] Feed tanks 60, 65 and 70 are shown in FIG. 1. Methanol storage tank 60 is fluidly connected to methanol transfer conduit 61 and methanol spray head 62 for delivering methanol to reaction tank 55. Potassium methylate storage tank 65 is fluidly connected to potassium methylate transfer conduit 66 and potassium methylate spray head 67 for delivering potassium methylate to reaction tank 55. Sulfuric acid storage tank 70 is fluidly connected to sulfuric acid transfer conduit 71 and sulfuric acid spray head 72 for delivering sulfuric acid to reaction tank 55. Typically pumps (not shown) are inserted in-line in conduits 61, 66 and 71 in order to generate sufficient fluid pressure at spray heads 62, 67 and 72. Alternately, tanks 60, 65 and 70 are placed above spray heads 62, 67 and 72 and the chemicals are gravity-fed. Depending on the choice of reactants and catalysts, the feed tanks may be substituted with feed tanks for other chemicals or compositions, such as, without limitation, sodium hydroxide and sodium methylate, as is applicable.

[0027] In use, filtrate is reacted with the compositions delivered from feed tanks 60, 65 and 70, for example and without limitation, as described herein. Reaction tank 55 comprises drain 75 connected to four-way manifold 76. As is shown, liquid contained within reaction tank 55, is stirred while the reaction within reaction tank 55 proceeds. As an alternative to stirring or in addition to stirring, for example use of stirrer 56, liquid may be recirculated through recirculation conduit 77 by a pump, not shown. Acid, methanol and base are added to reaction tank 55 typically in the order of acid, methanol and base. Acid is mixed thoroughly before addition of methanol to avoid production of hazardous dimethyl ether. The reaction can be conducted in a single tank, as shown in FIG. 1, or in two tanks, as shown below.

[0028] Upon completion of the conversion reaction, the reaction mixture is allowed to settle and, first, glycerol fraction 78, is removed through drain 75, manifold 76, and through glycerol drain conduit 79. Once glycerol is drained, the biofuel fraction is drained through drain 75, manifold 76 and biofuel conduit 81 and transferred to wash tank 85. Wash tank contains inlet 86, outlet 87 aerator 88 with air supply line 89 and water supply 90 with water spray head 91. In one embodiment, aerator is a limewood air-stone, though it may be any air bubble source. Other air diffusers may be employed, as are commercially available, such as, without limitation, the Airstation products, commercially available from Vertex water Features of Pompano Beach, Fla. After a desired length of air-biofuel contact time, the biofuel is allowed to settle and water is removed. This washing step may be repeated any number of times. After washing, the biofuel is drained and is ready for shipping.

[0029] All tanks or vessels used in the described process, including, without limitation fractionating tank 20, reaction tank 55, wash tank 85 or any other vessels described herein may have any physical configuration, which in many cases may be a matter of design choice, while in other cases,

facilitate the process(es) carried out in the particular vessel. In the Figures, the vessels are shown schematically as having cylindrical shapes. In practice, that is but one of many possible vessel configurations. The vessels may be cubic, conical, frustoconical, ovoid, spherical and any combination thereof. The vessels may have, without limitation, a tapered bottom (having a larger horizontal cross-sectional area at the top and a smaller horizontal cross-sectional area at the bottom as in the case of a conical or frustoconical bottom) or a flat bottom. In practice, practical implementation of the systems and processes described herein involves selection of vessels that are readily available and are least expensive while retaining functionality. Cylindrical tanks are commonly and relatively inexpensively available as well as conical tanks having a cylindrical upper portion and a conical lower portion. In one non-limiting embodiment, in reference to the Figures, one or more of fractionating tank 20, reaction tank 55, wash tank 85 have a conical or frustoconical bottom, or a tapered bottom. Use of vessels having a circular horizontal cross-section, such as cylindrical, conical, frustoconical or spherical vessels may be preferred in many instances as facilitating stirring of the contents of the vessel. A conical, frustoconical or otherwise tapered bottom may be preferred as facilitating the settling and separation processes carried out in each vessel, where applicable. The interior of the one or more vessels may contain ribs or other protuberances in order to facilitate mixing of the liquids present in the container. One example of a tank useful in the processes described herein is a tank having a conical bottom is a 510 gallon, cross-linked polyethylene, vertical, closed heard 15 degree cone bottom tank, typically with a support stand, commercially available, along with other suitable tank configurations, from PolyProcessing Company of Monroe, La.

[0030] In all embodiments, fluids are transferred either by gravity feed or by pumping. Where gravity feed is insufficient to provide enough pressure to feed the fluid to its destination pumps are required. For clarity, pumps are not shown in FIGS. 1 and 4, though they may be necessary in-line in one or more feeds, including, without limitation: chemical supply feeds, recirculation feeds (for example, to the spray heads) and drains. Of course, the relative configuration of the components will dictate whether a pump is needed or if siphoning or draining is all that is needed. Factors involved in the determination of whether a pump is needed in any given fluid conduit and the specifications of the pump include, without limitation: the distance between components, the fluid conduit diameter and composition (friction), the viscosity of the fluid to be transferred, the vertical orientation of the inlet end of the fluid conduit relative to the outlet end of the fluid conduit, and the required pressure at the outlet end of the fluid conduit.

[0031] In one embodiment, shown in schematic in FIGS. 4 and 5, system, or any suitable system, may be configured within a shipping container, such as a standard 8'x8'9'x20' or 8'x8' to 9'x20' commercial shipping container, such as, without limitation, a SeaLand container. Distribution of the biofuels processing system, when configured within a commercial transport container, as described herein may be accomplished by standard routes. In use, one or more of the containers can be stacked and/or lain side-by-side to produce a larger facility. The commercial shipping container may be a stock shipping container as are commonly available in the trade that has been retrofitted with suitable processing

equipment, or a customized or turnkey shipping container fabricated with suitable processing equipment inside and having the dimensions of a standard commercial shipping container, facilitating shipment and handling by truck, train or boat.

[0032] In FIG. 4, biofuel processing system 310 is embodied within a commercial container 390. Configuration of system 310 within a commercial container has significant advantages, including standardization of processes, ease of installation and ease of moving. Moving of the processing system literally can take place overnight. In one embodiment, in reference to FIG. 1, the shipping container does not contain a fractionating tank 20, but includes a coupling 395, permitting transfer of liquids into system 310. The processing system 310 includes fluid conduits 361, 365 and 371 and couplings at the inlet end of conduits 361, 365 and 371 that permit fluid attachment of reactant chemicals, such as methanol, sulfuric acid or methylate, to system 310 through conduits 361, 365 and 371.

[0033] FIG. 5 provides a second embodiment of the biofuel processing system 410 contained within a commercial transport container. In this embodiment, transport truck 420 delivers grease to system 410 through fluid conduit 425. The grease is first filtered through filters 450, 451, 452, 453 and 454. Filtrate exits filter 454 and is transferred to heated reaction tank 455. Methanol is transported from methanol storage tank, 460, through methanol conduit 461, and into reaction tank 455. Pumps 496 are used to facilitate transfer of liquids from one vessel to another. As with methanol, sulfuric acid is transported from sulfuric acid storage tank 465 through sulfuric acid transfer conduit 466 and into reaction tank 455 in order to titrate the pH of the reaction mixture. The reaction mixture within reaction tank 455 is mixed by the circulation through circulation conduit 477.

[0034] Once the pH of the reaction mixture is adjusted within reaction tank 455, the reaction mixture is transported through conduit 477 and two second heated reaction tank 459. Sodium methylate is fed from sodium methylate storage tank 470, through sodium methylate transfer conduit 471, and into reaction tank 459. The reaction mixture is recirculated within reaction tank 459 through recirculation conduit 477. Once the reaction with sodium methylate is completed, glycerol is transferred from reaction tank 459 to a storage container (not shown) through conduit 479.

[0035] Raw biofuel is then transferred through conduit 481 and into wash vessel 480. Air is forced into the biofuel mixture through conduit 489 and water is transferred into wash vessel 480 through conduit 490. Reaction tank 455 and 459, as well as wash vessel 480 each contain a vent 494 and one. Biofuel is transferred from system, 410, through conduit 487.

[0036] FIG. 6 illustrates schematically an alternate embodiment of a liquid-solids separation assembly 500 that is particularly suited to treating trap grease or any grease product that contains significant water and/or solids contamination. The assembly is preceded by a fractionation 510 tank in which water settles and is removed. Separation assembly 500 may be placed in line in a biofuels processing system, such as, in reference to system 410 shown in FIG. 5, in line in fluid conduit 425. The fractionation tank preferably is heated, without limitation by resistance heating or by a water coil. In one embodiment, heated water pro-

duced from the washing step or from other steps in the biofuel production process is passed through the water coil in order to recycle the heat generated in the production process. The raw grease typically is heated to greater than 95° F., and typically to about 125° F. In one preferred embodiment, the fractionation tank has a conical bottom to facilitate phase separation and, thus more complete removal of water. Traditional fractionation tanks have flat bottoms, which prevent good separation of the water from the grease, and which require regular scraping to clean debris from their bottom.

[0037] Referring again to FIG. 6, after the grease is fractionated, the grease is separated from solids using a disc filter 520, preferably a vibratory disc filter. An example of such a filter is the Eco Separator, commercially available from Russell Finex, Inc. of Pineville, N.C. Other mesh disc filters, disc screening filters or screening filters, and preferably self-cleaning filters, for example and without limitation, a Rotamat or like devices, are available commercially. After the disc filter, a liquid-solid separator 530 is employed to remove additional, smaller solids from the grease. A non-limiting example of a liquid-solid separator is the Russell Finex Liquid solid separator, which is a form of a cylindrical screening device having a smaller mesh size than disc filter 520, or other filter used as a substitute for disc filter 520, as is described in the materials. As a final step in preparing grease for filtration and processing, the grease is passed through a decanter centrifuge 540, for example and without limitation, from Alfa Laval. The combination of the removal of larger particles by the disc filter and smaller particles by the liquid solid separator yields a clean grease product that is more amenable to filtration using, for example and without limitation, the sequential bag filters described above. The water-grease-solids separation assembly 500 may be assembled from individual components, or may be contained within a pre-assembled unit, containing the disc separator, the liquid solids separator and the decanter centrifuge fluidly attached in-line and ready for fluid attachment to a fractionation tank at an inlet of the disc filter, and to additional filters at the outlet of the decanter centrifuge. The assembly may be housed and distributed as part of a larger assembly, within a commercial shipping container.

[0038] As will be apparent to those of skill in the process engineering field, a large number of commercially available alternatives to the series of devices of assembly 500. Assembly is intended to provide a pre-filtering system that removes solids from the trap grease or other oil-containing materials to be filtered, thereby taking the load off of the finer filters downstream in the process, preferably requiring minimal human intervention in the form of requiring cleaning of screens, etc. Disc filter 520 and liquid solid separator 530 may be any combination of screening filters, though self-cleaning devices are preferred. A device is self-cleaning if it provides a method by which retained solids can be continuously, or in batch, removed from the screen without manual removal or cleaning of the screen. For example and without limitation, the screen can be scraped, flushed or otherwise de-fouled by a mechanism built into the device, such as a spray head, screw, paddles, etc. A device is considered to be self-cleaning if the retentate (retained solids) cleaning process either is continuous in the separation process, automatically timed or otherwise built into the separation process, or can be initialized manually at a desired time, so long as the screen does not need to be removed and manually cleaned of

retentate, as is the case with, for example, bag filters. Although the devices are described as having “screens,” this is a generic term referring to any fixed filtering substrate, such as a screen, a perforated metal or plastic sheet, or other porous material that generally has a pore/hole size of larger than about 40 microns (μ). Equivalents of disc filter 520 typically remove solids of 150 microns or larger and equivalents of liquid solid separator 530 typically remove solids of larger than about 40 μ to about 50 μ .

[0039] A number of processes for production of biofuel may be implemented favorably using the process system described herein. For example and without limitation, biofuel production processes utilizing methanol, lye, sulfuric acid and sodium or potassium methylate, as are known (for example and without limitation at www.cubiodiesel.org/howtomakebiodiesel.php and www.journeytoforever.org) and/or as described herein. Nevertheless, the automated processes described herein are far superior to the manual processes described in the prior art. The prior art processes require use of good quality vegetable oil in order to achieve high quality conversion of the oil to biodiesel. According to one aspect of the present invention, titration of acid addition has the benefit of reduced requirements for acid, and less by-product production due to imbalances in the reactants. Another added feature according to one embodiment of the described processes is the addition of sulfuric acid and thorough mixing of the sulfuric acid to substantial homogeneity prior to addition of methanol to the reaction mixture. This prevents formation of dangerous dimethyl ether in the heated reaction mixture, which would result if sulfuric acid were added to the mixture containing significant amounts of methanol.

[0040] A number of additional process steps can be implemented in order to add further value to the general biofuel production processes described herein. Glycerol produced by the acid/methanol/methylate treatment, as drained from the process tanks retains a surprisingly large amount of biofuel and/or oils. By allowing the glycerol to settle for sufficient periods of time (typically 1 hour or more), and preferably in a conical-bottomed tank, additional acyl-fatty acid glycerides and biofuel can be retrieved from the glycerol fraction and circulated back into the processing tank(s), generating higher yields of biofuel.

[0041] Glycerol can be converted to higher value propylene glycol by heat and pressure treatment. In one embodiment, the glycerol is converted to propylene glycol by treatment of the glycerol at 200° C. at about 200 psi for a time sufficient to convert substantially all glycerol to propylene glycol. Propylene glycol also can be prepared from the glycerol by heating in the presence of NaOH, followed by distillation according to known protocols. Water (grey water) obtained from the washing step not only can be re-circulated through a heating coil in the grease fractionation tank, but can be used as a fertilizer due to its high K_2SO_4 content.

[0042] Occasionally, the biofuel product from the above-described process contains excess water and some triglycerides remain. This can be remedied by additional filtration steps, such as, without limitation, filtration through one or more filters, with the final filter being, for example and without limitation, about a treated 2 μ filter. The treated filter, for example and without limitation, treated with Teflon

(polytetrafluoroethylene or PTFE), is treated with a composition that excludes acyl-fatty acid glycerides, yet passes biofuel. To remove water, a dewatering device, such as, without limitation a fluid-fluid coalescing filter (for example and without limitation, commercially available from Racor of Modesto, Calif. or Pall Corporation of East Hills, N.Y.) may be employed. In one embodiment, a biofuel polishing filter assembly is provided which includes, sequentially, a 30 μ filter, a 10 μ filter, a coalescing filter and a 2 μ filter.

[0043] The examples provided below are merely exemplary and are not intended to limit the scope of the present invention.

EXAMPLE 1

[0044] A Sealand container is provided, containing 3 large polyethylene, resin lined reactors—Tanks 1, 2 and 3. These tanks are rotomolded high density cross linked polyethylene, with an embedded 120 volt, 27 amp heating mesh on tanks 1 and 2. The heating mesh is controlled by an analog process controller, with a high temp limit preset at 150 degrees Fahrenheit. Tanks 1 and 2 are processing tanks; tank 3 is a wash tank. All tanks vent to an activated carbon vapor control system. There are no atmospheric vents. Three Hayward filters are used to filter the trap grease prior to conversion.

[0045] The process is carried out as follows. One hundred gallons of grease is pumped from a 10,000 gallon fractionation tank, through five sequential Hayward bag filters and into a processing tank. The processing tank has an electric resistance heater. The heater on the tank then is used to heat the contents of the tank to 95° F. for 2 hours. Thirty six gallons of methanol (8% by volume) is then transferred from a 55 gallon drum into processing tank using a pneumatic diaphragm pump. The contents of the processing tank is then mixed for three hours at 95° F. The pH of the mixture is determined using a Hydrion pH indicator and about 407 gallons (typical) of sulfuric acid is transferred from a 55 gallon drum using a pneumatic diaphragm (titrate to about pH 2.5). The mixture is then transferred to a second processing tank, also having a resistance heater. Twelve gallons of pre-mixed potassium methylate, prepared as described above, is transferred into the mixture using a diaphragm pump. The reaction mixture is then heated to 95° F. and maintained at 95° F. for about 15 minutes. The mixture is then mixed for an additional 25 minutes. At 25 minutes mixing is halted and glycerin is drained into storage. Mix for another ten minutes and then drain glycerol after halting mixing. Repeat ten minute mixing and draining until no glycerol forms. Glycerol is then shipped out for methanol recovery.

[0046] The biodiesel is then transferred to the wash tank. Forty 40 gallons of warm water is then added through a mist system, and the bubble aerator is turned on. The mixture is aerated for 1-2 hours and grey water is drained. The washing step is repeated three times and is tested for soaps and glycerol. If no soaps or glycerol is formed, the product is finished.

EXAMPLE 2

Lye Plus Acid

[0047] The chemicals used in the process are methanol, sulfuric acid, and sodium methoxide (Methanol premixed with lye at 33% concentration by DOW Chemical—avoids powdered lye handling).

[0048] Chemicals are stored in original drums, in hooded spill containment. Hooded spill containment systems are (2) drum systems, labeled Containment 1 and Containment 2. Containment 1 contains 1 drum (55 gallons) of sodium methoxide and 1 drum (55 gallons) of methanol. Containment 2 contains (2) drums of sulfuric acid, 66 degree Baume (93-96 percent concentration).

[0049] The process is carried out as follows. One hundred gallons of grease is pumped from a 10,000 gallon fractionation tank, through three sequential Hayward filters and into a processing tank. The processing tank has an electric resistance heater. The heater on the tank then is used to heat the contents of the tank to 95° F. for 2 hours. Eight gallons of methanol is then transferred from a 55 gallon drum into the processing tank using a pneumatic diaphragm pump. The contents of the processing tank is then mixed for one hour at 95° F. The pH of the mixture is determined using a Hydrion pH indicator and about three gallons (typical) of sulfuric acid is transferred from a 55 gallon drum using a pneumatic diaphragm (titrate to pH 2.5) The mixture is then transferred to a second processing tank, also having a resistance heater. Two 2 gallons of premixed sodium methoxide, prepared as described above, is then transferred into second processing tank using a pneumatic diaphragm pump. The reaction mixture is then heated to 95° F. and maintained at 95° F. for about 15 minutes. The mixture is then mixed for an additional 25 minutes. At 25 minutes mixing is halted and glycerin is drained into storage. Mix for another ten minutes and then drain glycerol after halting mixing. Repeat ten minute mixing and draining until no glycerol forms. Glycerol is then shipped out for methanol recovery.

[0050] The biodiesel is then transferred to the wash tank. Forty 40 gallons of warm water is then added through a mist system, and the bubble aerator is turned on. The mixture is aerated for 1-2 hours and grey water is drained. The washing step is repeated three times and is tested for soaps and glycerol. If no soaps or glycerol is formed, the product is finished.

EXAMPLE 3

[0051] The chemicals used in the process are methanol, sulfuric acid, and potassium methoxide. Grease is filtered through five sequential Hayward Filters ranging from 80 Mesh (US) to 1 micron (μ) and into a first processing tank. The first processing tank has an electric resistance heater. The heater on the tank then is used to heat the contents of the tank, 400 gallons of filtered grease, to 125° F. for 1 hour. Fluid levels in the tanks are monitored by sonar and radar and process parameters, such, without limitation temperature and pH, are controlled and/or monitored by redundant Chameleon Programmable Automation Controllers.

[0052] In the process, Sulfuric acid is added to the reaction mixture until the pH reaches 2.5. Afterwards 10-15% by volume of methanol is then transferred into the processing tank using a pneumatic diaphragm pump. The contents of the processing tank are then mixed by recirculation for one hour at 125° F. Some glycerol is produced by this process and can be drained from the first processing tank. The mixture is then transferred to a second processing tank, also having a resistance heater. Approx 20 g to pH of about 7 gallons of premixed potassium methylate (to pH), prepared as described above, is then transferred into second process-

ing tank. The reaction mixture is maintained at 125° F. for about 15 minutes. The mixture is then mixed by recirculation for an additional 25 minutes. At 25 minutes, mixing is halted and glycerin is drained into storage. The reaction is mixed for another ten minutes and then glycerol is drained after halting mixing. This mixing/glycerol draining process is repeated until no glycerol forms.

[0053] The glycerin obtained from the first and second processing tanks can be further settled in a conical tank to recover fuel that is not effectively separated from the glycerol in the second processing tank. The fuel can be added back to the first processing tank for further processing.

[0054] Glycerol may then be shipped out for methanol recovery. Alternately, the glycerin may be heated to about 200° C. at about 200 psi (pounds per square inch) to produce higher-value propylene glycol, thereby producing additional value.

[0055] The biodiesel is then transferred to the wash tank. Forty 40 gallons of warm, deionized water is then added through a mist system, and a limestone bubble aerator is turned on. The mixture is aerated for 1-2 hours and grey water is drained. The washing step is repeated three times and is tested for soaps and glycerol. If no soaps or glycerol are present, the product is finished. The water fraction contains Potassium Sulfate and can be used as a fertilizer.

[0056] Once the biofuel is washed, it can be further refined to remove any residual triglycerides. The fuel is filtered through a 30 μ KVHS filter, a 10 μ filter, a fluid-fluid coalescer (for example and without limitation a PhaseSep® Coalescer from Pall Corporation of East Hills, N.Y.) followed by a 2 μ coated filter, such as a Teflon-coated filter.

[0057] Having now fully described this invention, it will be understood to those of ordinary skill in the art that the same can be performed within a wide and equivalent range of conditions, formulations and other parameters without affecting the scope of the invention or any embodiment thereof. All publications, patents and patent applications mentioned in this specification are herein incorporated by reference into the specification to the extent of their technical disclosure, to the same extent as if each individual publication, patent or patent application was specifically and individually indicated to be incorporated herein by reference.

I claim:

1. A method of producing biofuel from a fatty acid triglyceride-containing material, comprising:

- (a) heating the material to a temperature greater than about 25° C. to decrease its viscosity;
- (b) filtering and dewatering the material at a temperature greater than about 25° C.; and
- (c) converting the material to glycerol and biofuel by:
 - i) adjusting the pH of the starting material to about 2.5 and substantially completely mixing the acid into the material to produce a reaction mixture;
 - ii) adding methanol to the reaction mixture to esterify free fatty acids; and
 - iii) adding sodium or potassium methylate or hydroxide to the reaction mixture in an amount effective to

bring the pH of the reaction mixture to from about 6.2 to about 8.0, thereby converting fatty acid acyl-glycerides to glycerol and biofuel.

2. The method of claim 1, further comprising separating the glycerol and biofuel and washing the methoxylated fatty acids by mixing the methoxylated fatty acids with water and removing the water.

3. The method of claim 2, wherein the water contains phosphoric acid.

4. The method of claim 2, wherein the water is sprayed over the methoxylated fatty acids.

5. The method of claim 2, wherein the methoxylated fatty acids are aerated during washing.

6. The method of claim 5, wherein the methoxylated fatty acids are aerated using a limewood airstone.

7. The method of claim 2, wherein the methoxylated fatty acids are washed at least two times with water.

8. The method of claim 2, wherein the methoxylated fatty acids are washed three times with water.

9. The method of claim 1, comprising filtering the trap grease at a temperature of from about 25° C. to about 65° C.

10. The method of claim 9, comprising filtering the trap grease at a temperature of from about 50° C. to about 55° C.

11. The method of claim 9, comprising filtering the trap grease at a temperature of about 52° C.

12. The method of claim 1, comprising filtering the trap grease in a screen filter comprising a filter screen, a filtrate collection container, a heater within or integral to the filtrate collection container, a pump fluidly connected to the filtrate collection container and a spray head fluidly attached to the pump and directed onto the filter screen for pumping warmed filtrate onto the screen.

13. The method of claim 12, wherein the screen is a belt.

14. The method of claim 12, wherein the screen is cylindrical, the screen filter further comprising a screw within the cylindrical screen for advancing material through the filter.

15. The method of claim 1, comprising adding potassium methylate to the reaction mixture to convert fatty acid acyl-glycerides to glycerol and biofuel.

16. The method of claim 1, comprising adding sodium methylate to the reaction mixture to convert fatty acid acyl-glycerides to glycerol and biofuel.

17. The method of claim 1, wherein the starting material is trap grease.

18. The method of claim 1, comprising converting the material to glycerol and biofuel at a temperature ranging from about 25° C. to about 65° C.

19. The method of claim 18, comprising converting the material to glycerol and biofuel at a temperature ranging from about 45° C. to about 55° C.

20. The method of claim 18, comprising converting the material to glycerol and biofuel at a temperature of about 52° C.

21. The method of claim 1, wherein the filtering, dewatering and conversion of the material to glycerol and biofuel are performed using processing equipment configured within a commercial shipping container.

22. The method of claim 21, wherein the container is about 40' by about 8' by about 8'9".

23. The method of claim 1, wherein the fatty acid triglyceride-containing material is trap grease.

24. The method of claim 1, wherein during the heating of the material to a temperature greater than about 25° C. to decrease its viscosity, the material is fractionated to separate water and grease.

25. The method of claim 24, wherein the material is fractionated in a conical-bottomed tank.

26. The method of claim 1, wherein the material is passed through one or more self-cleaning screening devices.

27. The method of claim 26, wherein the material is passed through two self-cleaning screening devices.

28. The method of claim 27, wherein one of the self-cleaning screening devices is a disc filter.

29. The method of claim 28, wherein the disc filter is a vibratory disc filter.

30. The method of claim 27, wherein one of the self-cleaning screening devices is a liquid solid separator comprising a cylindrical screen.

31. The method of claim 26, wherein the material is passed through a decanter centrifuge after it is passed through the one or more self-cleaning screening devices.

32. The method of claim 26, wherein the material is passed through, in sequence, a vibratory disc filter, a liquid solid separator comprising a cylindrical screen and having a finer mesh than the disc filter, and a decanter centrifuge.

33. The method of claim 1, further comprising separating the glycerol from the biofuel.

34. The method of claim 33, further comprising fractionating the separated glycerol into a glycerol fraction and a residual fraction comprising one or both of biofuel and fatty acid acyl-glycerides.

35. The method of claim 34, wherein the fractionating is performed by settling.

36. The method of claim 35, wherein the settling is performed in a conical-bottom container.

37. The method of claim 34, wherein the fractionating is performed in a centrifugal decanter.

38. The method of claim 1, further comprising dewatering the washed biofuel.

39. The method of claim 1, wherein the washed biofuel is dewatered in a water-oil separating coalescing filter.

40. The method of claim 1, further comprising filtering the washed biofuel with a filter that excludes fatty acid acyl-glycerides, but passes biofuel.

41. The method of claim 40, wherein the filter that excludes fatty acid acyl-glycerides, but passes biofuel is a PTFE-coated filter membrane.

42. The method of claim 41, wherein the filter is about a 2 μ filter.

43. The method of claim 40, wherein the filter is about a 2 μ filter.

44. The method of claim 1, further comprising filtering the washed biofuel with, in sequence, about a 30 μ filter, about a 10 μ filter, a water-oil separating coalescing filter and a filter that excludes fatty acid acyl-glycerides, but passes biofuel.

45. The method of claim 1, further comprising heating the glycerol under pressure to make propylene glycol.

46. The method of claim 45 wherein the glycerol is heated to about 200° C. at about 200 psi to produce propylene glycol.

47. A biofuels processing system comprising, in sequence and fluidly connected:

a pre-filter assembly comprising one or more self-cleaning screening devices;

one or more reaction vessels comprising a heater and a mixing subsystem;

a wash tank; and

one or more of a methanol feed, an acid feed and a methylate feed fluidly connected to the one or more heated reaction vessels and a water feed fluidly connected to the wash tank.

48. The system of claim 47, wherein pre-filter assembly comprises two self-cleaning screening devices.

49. The system of claim 48, wherein one of the self-cleaning screening devices is a disc filter.

50. The system of claim 49, wherein the disc filter is a vibratory disc filter.

51. The system of claim 48, wherein one of the self-cleaning screening devices is a liquid solid separator comprising a cylindrical screen.

52. The system of claim 47, further comprising a decanter centrifuge downstream to the one or more self-cleaning screening devices and upstream to the one or more reaction vessels.

53. The system of claim 47, the pre-filter assembly comprising in sequence, a vibratory disc filter, a liquid solid separator comprising a cylindrical screen and having a finer mesh than the disc filter, and a decanter centrifuge.

54. The system of claim 47, further comprising between the pre-filter assembly and the one or more reaction vessels two or more filter units of differing pore size connected in series, wherein the mesh size of the filter units decreases in a downstream direction.

55. The system of claim 54, wherein the filter units are bag filters.

56. The system of claim 54, comprising from three to five filter units.

57. The system of claim 47, comprising one reaction vessel and a methanol feed, an acid feed and a methylate feed fluidly connected to the reaction vessel.

58. The system of claim 47, comprising a first reaction vessel downstream to the pre-filter assembly and a second reaction vessel downstream to the first reaction vessel.

59. The system of claim 58, in which a methanol feed and an acid feed are fluidly connected to the first reaction vessel and a methylate feed is fluidly connected to the second reaction vessel.

60. The system of claim 47, comprising an acid feed fluidly connected to the one or more reaction vessels and further comprising a pH controller having a probe within the same reaction vessel and a pump or solenoid fluidly connected to the acid feed upstream of the reaction vessel under control of the pH controller.

61. The system of claim 47, in which one or more of the methanol feed, the acid feed, the methylate feed and the water feed are fluidly connected to a spray head within the one or more reaction vessels and located in a portion within the one or more reaction vessels above where liquid collects during operation of the system.

62. The system of claim 47, further comprising a temperature controller having a probe within the one or more reaction vessels.

63. The system of claim 47, housed within a commercial shipping container.

64. The system of claim 47, wherein dimensions of the container are about 40' by about 8' by about 8' to about 9'.

65. The system of claim 47, wherein dimensions of the container are about 20' by about 8' by about 8' to about 9'.

66. The system of claim 47, further comprising a fractionation container downstream to one or more of the reaction vessels to receive glycerol from the one or more reaction vessels.

67. The system of claim 66, wherein the fractionation container is a conical-bottom container.

68. The system of claim 47, further comprising a centrifugal decanter downstream to one or more of the one or more reaction vessels.

69. The system of claim 47, further comprising a dewatering device downstream to the wash tank for dewatering the washed biofuel.

70. The system of claim 69, wherein the dewatering device is a water-oil separating coalescing filter.

71. The system of claim 47, further comprising a filter that excludes fatty acid acyl-glycerides, but passes biofuel downstream to the wash tank.

72. The system of claim 71, wherein the filter that excludes fatty acid acyl-glycerides, but passes biofuel is a PTFE-coated filter membrane.

73. The system of claim 72, wherein the filter is about a 2 μ filter.

74. The system of claim 71, wherein the filter is about a 2 μ filter.

75. The system of claim 47, further comprising downstream to the wash tank: about a 30 μ filter, about a 10 μ filter, a water-oil separating coalescing filter and a filter that excludes fatty acid acyl-glycerides, but passes biofuel.

76. The system of claim 47, the pre-filter assembly containing a triglyceride.

77. A pre-filter assembly for a biofuels processing station comprising a pre-filter assembly comprising two self-cleaning screening devices fluidly attached.

78. The pre-filter assembly of claim 77, wherein one of the self-cleaning screening devices is a disc filter.

79. The system of claim 78, wherein the disc filter is a vibratory disc filter.

80. The system of claim 77, wherein one of the self-cleaning screening devices is a liquid solid separator comprising a cylindrical screen.

81. The system of claim 77, further comprising a decanter centrifuge downstream to the one or more self-cleaning screening devices and upstream to the one or more reaction vessels.

82. The system of claim 77, the pre-filter assembly comprising in sequence, a vibratory disc filter, a liquid solid separator comprising a cylindrical screen and having a finer mesh than the disc filter, and a decanter centrifuge.

83. A screen filter apparatus, comprising: a filter screen, a filtrate collection container, a heater within or integral to the filtrate collection container, a pump fluidly connected to the filtrate collection container and a spray head fluidly attached to the pump and directed into the filter screen for pumping warmed filtrate onto the screen.

84. The screen filter apparatus of claim 83, wherein the screen is a belt.

85. The screen filter apparatus of claim 83, wherein the screen is cylindrical, the filter further comprising a screw within the cylindrical screen for advancing material through the filter.

86. The modified screening filter of claim 83, wherein the heater is an electric resistance heater.

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