

## METHOD FOR PURIFYING LIPIDS

### FIELD OF THE INVENTION

The present invention relates to purifying lipid materials, in particular those  
5 containing phosphorus and metal impurities in high levels. Specifically the present  
invention relates to purifying lipid material to render them suitable for catalytic  
processing.

### BACKGROUND TO THE INVENTION

It is a well-known fact that lipid materials, such as oils and fats, can contain  
10 impurities that need to be removed before catalytic processing as they are  
detrimental to the quality of the final product, and may cause plugging of the reactor,  
deactivation of the used catalyst, as well as fouling of the equipment. However, the  
current methods may not be fully suitable for the most difficult oils and fats as despite  
sufficient removal of at least some of the impurities, they suffer from or even cause  
15 challenges in downstream unit operations such as filtering the purified products.

### BRIEF DESCRIPTION OF THE INVENTION

An objective of the present invention is thus to provide a method so as to overcome  
the above problems. The objectives of the invention are achieved by a method which  
is characterized by what is stated in the independent claims. The preferred  
20 embodiments of the invention are disclosed in the dependent claims.

The invention is based on the surprising realization that lipid feedstocks may be  
successfully purified by a method combining adjusting the total net elementary  
charge of the lipid feedstock and optionally the free fatty acid content to a desirable  
level and removing of solid impurities from the lipid feedstock prior to further  
25 purification steps such as heat treatment followed by bleaching. This allows efficient  
removal of the impurities by enabling smooth downstream phase-separation  
operations following the further purification steps. This further allows efficient  
production of renewable hydrocarbons from said lipid feedstocks.

## BRIEF DESCRIPTION OF THE DRAWINGS

In the following the invention will be described in greater detail by means of preferred embodiments with reference to attached drawings, in which

Figure 1 illustrates the 1st exemplary process flow of the present method;

5 Figure 2 illustrates the 2nd exemplary process flow of the present method;

Figure 3 illustrates the 3rd exemplary process flow of the present method;

Figure 4 illustrates the 4th exemplary process flow of the present method;

Figure 5 illustrates the 5th exemplary process flow of the present method;

Figure 6 illustrates the 6th exemplary process flow of the present method.

10

## DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a method of purifying lipid feedstock, comprising

i) providing the lipid feedstock (a);

15 ii) when the net elementary charge  $Q_1$  of the lipid feedstock (a) is below 0 mmol/kg, adjusting the net elementary charge of the lipid feedstock (a) to total net elementary charge  $Q_t$  of at least 0 mmol/kg, such as from 0 to 10 mmol/kg, preferably at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg, by mixing the lipid feedstock (a) with a charge balancing component comprising, or consisting of, a metal compound; and

20 iii) optionally adjusting the free fatty acid (FFA) concentration of the the lipid feedstock (a) to above 2 wt%, such as 2.5 to 40 wt%, preferably at least 3 wt%, such as from 3 to 30 wt%, more preferably to at least 4 wt%, such as from 4 to 20 wt%, even more preferably from 4 to 10 wt%, of the total weight of the resulting lipid feedstock; and

25 iv) removing solid impurities, and optionally excess water, from the lipid feedstock (a);

to obtain a conditioned lipid feedstock (b) having a total net elementary charge  $Q_t$  within the values defined in step ii);

v) heating the conditioned lipid feedstock (b), preferably in the presence of water, at a temperature from 180 to 325 °C to obtain a heat-treated lipid feedstock (c);

5 vi) optionally further treating the heat-treated lipid feedstock (c) with an acid and/or adsorbent material, preferably under bleaching conditions, to obtain a further treated lipid feedstock (d); and

vii) recovering the heat-treated lipid feedstock (c) and/or the further treated lipid feedstock (d) by phase-separation to obtain a purified lipid feedstock (e).

10 The present invention further provides a process for providing renewable hydrocarbons, comprising x) purifying lipid feedstock by the method contemplated herein to obtain purified lipid material, and y) subjecting the purified lipid material to hydroprocessing to obtain at least one renewable hydrocarbon.

15 In the present description, weight percentages (wt%) are calculated based on the total weight of the material in question (typically a blend or a mixture). Any amounts defined as ppm (parts per million), are based on weight (i.e. mg/kg).

By "molar ratio" is meant the ratio of species a to species b, wherein the amount of species a and species b are determined in terms of moles, converting the concentration in mg/kg to mol/kg ( $n=m/M$ ), in the feedstock, using an appropriate  
20 method thereto, like inductively coupled plasma (ICP) or ICP coupled with atomic emission spectrometry (ICP-AES) or tandem inductively coupled plasma-mass spectrometry (ICP-MS/MS) or inductively coupled plasma optical emission spectroscopy (ICP-OES). The weight amount is calculated as the amount of the species in question in elemental form.

25 The term "renewable" in the context of renewable feed, renewable feedstock or renewable fuel or fuel component refers to one or more organic compounds derived from any renewable source (contrary to source of fossil origin). Thus renewable compounds or compositions are obtainable, obtained, derivable, derived, or originating from plants, animals and/or microbes, including compounds or  
30 compositions obtainable, obtained, derivable, derived, or originating from fungi

and/or algae, in full or in part, whether these compounds or compositions are in their virgin, recycled or reclaimed form. As used herein, renewable compounds or compositions may comprise gene manipulated compounds or compositions. Renewable feedstocks, components, compounds, or compositions may also be referred to as biological feedstocks, components, compounds, or compositions, or as biogenic feedstocks, components, compounds, or compositions. As used herein, the term "fossil" refers to compounds or compositions that are obtainable, obtained, derivable, derived, or originating from naturally occurring non-renewable compositions, such as crude oil, petroleum oil/gas, shale oil/gas, natural gas, or coal deposits, and the like, and combinations thereof, including any hydrocarbon rich deposits that can be utilized from ground and/or underground sources. Thus, the renewable hydrocarbon is based on renewable sources and consequently does not originate from or is not derived from any fossil based material.

The  $^{14}\text{C}$ -isotope content can be used as evidence of the renewable or biological origin of a feedstock or product. Carbon atoms of renewable material comprise a higher number of unstable radiocarbon ( $^{14}\text{C}$ ) atoms compared to carbon atoms of fossil origin. Therefore, it is possible to distinguish between carbon compounds derived from biological sources, and carbon compounds derived from fossil sources by analyzing the ratio of  $^{12}\text{C}$  and  $^{14}\text{C}$  isotopes. Thus, a particular ratio of said isotopes can be used to identify and quantify renewable carbon compounds and differentiate those from non-renewable i.e. fossil carbon compounds. The isotope ratio does not change in the course of chemical reactions. Example of a suitable method for analyzing the content of carbon from biological sources is ASTM D6866 (2020). An example of how to apply ASTM D6866 to determine the renewable content in fuels is provided in the article of Dijs et al., Radiocarbon, 48(3), 2006, pp 315-323. For the purpose of the present invention, a renewable material, such as a feedstock or product, is considered to be of renewable origin if it contains 90 % or more modern carbon (pMC), such as about 100 % modern carbon, as measured using ASTM D6866.

As used herein, "hydrocarbons" refer to compounds consisting of carbon and hydrogen. Examples of hydrocarbons include paraffins, including n-paraffins and i-paraffins, naphthenes, aromatics, and olefins (alkenes). "Oxygenated

hydrocarbons” refer herein to hydrocarbons comprising covalently bound oxygen. Examples of oxygenated hydrocarbons include tri-, di-, and monoglycerides, fatty acids and alkyl-esters of fatty acids (e.g. methyl- or ethyl-ester of fatty acids).

As used herein, “paraffins” refer to non-cyclic alkanes, i.e., non-cyclic, open chain saturated hydrocarbons that are linear (normal paraffins, n-paraffins) or branched (isoparaffins, i-paraffins). In other words, paraffins refer herein to n-paraffins and/or i-paraffins.

In the context of the present disclosure, “olefins” refer to unsaturated, linear, branched, or cyclic hydrocarbons, excluding aromatic compounds. In other words, olefins refer to hydrocarbons having at least one unsaturated bond, excluding unsaturated bonds in aromatic rings.

“Naphthenes” refer herein to cycloalkanes or cycloalkenes containing at least one cyclic structure, with or without side chains, including also compounds having one or more olefinic bonds in the cyclic structure and/or in a side chain, but excluding compounds with any aromatic ring structure(s).

“Aromatics” refers herein to hydrocarbons containing at least one aromatic ring structure, i.e. cyclic structure having delocalized, pi bonds satisfying the Hückel ( $4n + 2$ ) rule.

Term “fuel” refers to both fuels usable as such and/or as fuel components, which fulfill the requirements of standards for the respective use. For example, within the EU, the standard for gasoline is EN 228 (2017), for paraffinic diesel EN 15940 (2019), and for aviation turbine fuel containing synthesized hydrocarbons D7566 (2020).

### **Lipid feedstock**

The term “lipid feedstock” refers to lipid material which is intended to be converted by hydroprocessing into renewable hydrocarbons or other valuable renewable products, including fuels, but which comprises impurity concentration that is too high for the lipid feedstock to be fed to the hydroprocessing processes as such. Lipid feedstock contemplated herein typically comprises at least 5 mg/kg, such as from 5 to 5000 mg/kg, dissolved phosphorus impurities, and at least 10 mg/kg, such as

from 10 to 10000 mg/kg, dissolved metal impurities, measured as elemental phosphorus and elemental metal(s). Preferably the lipid feedstock contemplated herein comprises at least 15 mg/kg, such as 15 to 2000 mg/kg, dissolved phosphorus impurities, and at least 20 mg/kg, such as 20 to 3000 mg/kg, dissolved metal impurities, measured as elemental phosphorus and elemental metal(s). The net elemental charge of the lipid feedstock contemplated herein based on phosphorus and the metal(s) contained in the lipid feedstock may be positive or negative, however the process is particularly beneficial for lipid feedstocks having negative net elementary charge  $Q_1$ , i.e., there are more negative charge from phosphorus impurities than positive charge from metal impurities. The present method is particularly suitable for lipid feedstocks having net elementary charge  $Q_1$  below 0 mmol/kg, such as from -60 to -0.5 mmol/kg, in particular below -0.5 mmol/kg, such as from -30 to -1 mmol/kg.

Accordingly, in an embodiment the present invention provides a method of purifying lipid feedstock, comprising

- i) providing the lipid feedstock (a) having net elementary charge  $Q_1$  below 0 mmol/kg, such as from -60 to -0.5 mmol/kg, in particular below -0.5 mmol/kg, such as from -30 to -1 mmol/kg;
- ii) adjusting the net elementary charge of the lipid feedstock (a) to total net elementary charge  $Q_t$  of at least 0 mmol/kg, such as from 0 to 10 mmol/kg, preferably at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg, by mixing the lipid feedstock (a) with a charge balancing component comprising, or consisting of, a metal compound;
- iii) optionally adjusting the free fatty acid (FFA) concentration of the lipid feedstock (a) to above 2 wt% of the total weight of the resulting lipid feedstock; and
- iv) removing solid impurities, and optionally excess water, from the lipid feedstock (a);

to obtain a conditioned lipid feedstock (b) having a total net elementary charge  $Q_t$  within the values defined in step iii);

- v) heating the conditioned lipid feedstock (b), preferably in the presence of water, at a temperature from 180 to 325 °C to obtain a heat-treated lipid feedstock (c);
- vi) optionally treating the heat-treated lipid feedstock (c) with an acid and/or adsorbent material to obtain a further treated lipid feedstock; and
- 5   vii) recovering the heat-treated lipid feedstock and/or the further treated lipid feedstock (c) by phase-separation to obtain a purified lipid feedstock (e).

In an alternative embodiment, the present invention provides a method of purifying lipid feedstock, comprising

- i) providing the lipid feedstock (a) having net elementary charge Q1 of at least 0  
10   mmol/kg, such as from 0 to 80 mmol/kg, in particular above 1 mmol/kg, such as from 1 to 60 mmol/kg;
- iv) removing solid impurities, and optionally excess water, from the lipid feedstock (a);
- to obtain a conditioned lipid feedstock (b) having a total net elementary charge Qt  
15   within the values defined in step i);
- v) heating the conditioned lipid feedstock (b), preferably in the presence of water, at a temperature from 180 to 325 °C to obtain a heat-treated lipid feedstock (c);
- vi) optionally treating the heat-treated lipid feedstock (c) with an acid and/or adsorbent material to obtain a further treated lipid feedstock (d); and
- 20   vii) recovering the heat-treated lipid feedstock and/or the further treated lipid feedstock (d) by phase-separation to obtain a purified lipid feedstock (e).

The term “dissolved impurities” as used herein refers to impurities which remain in the liquid phase after filtration with 0.45 µm filter, i.e. impurities in solid form that are removed by such filtration are not considered dissolved impurities in this context.

- 25   Phosphorus impurities may be present in the lipid feedstock in various chemical forms in varying phosphorus containing compounds, such as in the form of phospholipids, oil or water soluble or insoluble phosphorus compounds, or inorganic phosphates, diphosphates, and phosphites. Typical phosphorus containing compounds may further comprise glycerophospholipids, sphingolipids, phosphatidic

acid complexes, phosphatidylethanolamines, nucleoside phosphates, apatite or bone meal (calcium phosphate), and the like.

Metal impurities may similarly be present in the lipid feedstock in various chemical forms in varying metal containing compounds, inorganic or organic, in water soluble,  
5 oil soluble, or insoluble forms.

The lipid feedstock may contain both dissolved and undissolved impurities, including dissolved and undissolved phosphorus and/or metal impurities. The amounts of the insoluble impurities are not considered to be part of the dissolved impurities, but are taken into account as solid impurities and are treated accordingly, i.e. removed  
10 together with the other solid impurities (in step iv). Thus, for the determination of the dissolved phosphorus and metal impurities, a sample of the feedstock is filtered to remove any solid particles, and the amounts of various metals and phosphorus as elemental metals and elemental phosphorus are measured thereafter.

The present method may be used to purify any lipid feedstock, such as plant oils,  
15 plant fats, animal fats, animal oils, fish fats, fish oils, waste fats, waste oils, residue fats, residue oils, a fatty acid distillate, acidulated soapstock, mold oils, rapeseed oil, canola oil, colza oil, babassu oil, carinata oil, coconut butter, muscat butter oil, sesame oil, maize oil, poppy seed oil, cottonseed oil, soy oil, laurel seed oil, jatropha oil, palm kernel oil, camelina oil, tall oil, fraction of tall oil, crude tall oil, tall oil pitch,  
20 sunflower oil, corn oil, technical/distillers corn oil, soybean oil, hemp seed oil, olive oil, linseed oil, cottonseed oil, mustard oil, mustard seed oil, peanut oil, castor oil, coconut oil, palm oil, crude palm oil, palm seed oil, palm fatty acid distillate, a sludge originating from plant oil production, palm oil mill effluent, arachis oil, castor oil, coconut oil, archaeal oil, bacterial oil, fungal oil, protozoal oil, algal oil, seaweed oil,  
25 oils from halophiles, poultry fat, dry rendered poultry fat, brown grease, used cooking oil, suet, lard, tallow, blubber, recycled alimentary fats, acid oil, train oil, spent bleaching earth oil, lignocellulosic based feeds, materials produced by genetic engineering, and biological materials produced by microbes, or any combinations or mixtures thereof. The present method is particularly suitable for lipid materials  
30 containing high amounts of phosphorus and/or lipophilic phosphorus, metals, and solid impurities.

The lipid feedstock may be in unprocessed form (e.g. animal fat) or in processed form (e.g. used cooking oil).

Typical lipid feedstocks comprise waste and recycle oils, typically combinations thereof. Such lipid materials typically initially contain high amounts of phosphorus,  
5 typically as phosphorus containing compounds, metals, and solid impurities.

Examples of lipid materials benefitting from the present purification method include soapstock acid oil (SAO), low quality animal fat (LQAF) grades such as choice white grease (CWG) and poultry fat (APF), used cooking oil (UCO), and palm oil mill effluent (POME), as well as mixtures thereof.

10 Soapstock acid oil (SAO) is a by-product from the vegetable oil refining industry obtained by acidification of soapstock. Typically SAO comprises high amounts of phosphorus and metal impurities, such as at least 30 mg/kg, e.g. 50 to 700 mg/kg, phosphorus, at least 10 mg/kg, e.g. 10 to 3000 mg/kg, metals, and at least 0.1 wt%, e.g. 0.1 to 3 wt%, solids. Typically the net elementary charge of SAO is -13 mmol/kg,  
15 such as from -60 to -10 mmol/kg. Typically SAO comprises at least 10 wt%, such as 15 to 80 wt% FFA of the total weight of the composition.

Low quality animal fat (LQAF) is typically inedible lipids derived from animals. Typically LQAF comprises high amounts of phosphorus and metal impurities, such as at least 50 mg/kg, e.g. 100 to 700 mg/kg, phosphorus, at least 50 mg/kg, e.g. 50  
20 to 1200 mg/kg, metals, and at least 0.01 wt%, e.g. 0.01 to 2.0 wt%, solids. Typically the net elementary charge of LQAF is -30 mmol/kg, such as from -50 to -5 mmol/kg. Typically LQAF comprises at least 2 wt%, such as 2 to 10 wt%, FFA of the total weight of the composition.

An example of LQAF, choice white grease (CWG) is an inedible low quality animal  
25 fat obtained from the rendering process of swine in the North American feedstock market. Typically CWG comprises high amounts of phosphorus and metal impurities, such as at least 300 mg/kg, e.g. 300 to 700 mg/kg, phosphorus, at least 600 mg/kg, e.g. 600 to 1200 mg/kg, metals, and at least 0.4 wt%, e.g. 0.4 to 2.0 wt%, solids. Typically the net elementary charge of CWG is -10 mmol/kg, such as  
30 from -30 to -5 mmol/kg. Typically CWG comprises at least 2 wt%, such as 2 to 10 wt%, FFA of the total weight of the composition.

A further example of LQAF, poultry fat (AFP) is fat obtained from poultry rendering and processing. Typically AFP comprises high amounts of phosphorus and metal impurities, such as at least 50 mg/kg, e.g. 100 to 700 mg/kg, phosphorus, at least 10 mg/kg, e.g. 50 to 500 mg/kg, metals, and at least 0.01 wt%, e.g. 0.1 to 1.0 wt%, solids. Typically the net elementary charge of AFP is -30 mmol/kg, such as from -50 to -15 mmol/kg. Typically AFP comprises 5 wt%, such as 2 to 15 wt% FFA of the total weight of the composition.

Used cooking oil (UCO) is oils and fats that have been used for cooking or frying. Typically UCO comprises high amounts of phosphorus and metal impurities, such as at least 2 mg/kg, e.g. 5 to 300 mg/kg, phosphorus, at least 10 mg/kg, e.g. 20 to 2000 mg/kg, metals, and at least 0.01 wt%, e.g. 0.01 to 1.0 wt%, solids. Typically the net elementary charge of UCO is at least 0 mmol/kg, such as from 0 to 10 mmol/kg. Typically UCO comprises 3 wt%, such as 0.5 to 15 wt% FFA of the total weight of the composition.

Palm oil mill effluent (POME) is oil waste separated from wastewaters generated in palm oil milling. Typically POME comprises high amounts of phosphorus and metal impurities, such as at least 5 mg/kg, e.g. 5 to 150 mg/kg, phosphorus, at least 10 mg/kg, e.g. 10 to 4000 mg/kg, metals, and at least 0.01 wt%, e.g. 0.1 to 2.0 wt%, solids. Typically the net elementary charge of POME is 10 mmol/kg, such as from 0 to 70 mmol/kg. Typically POME comprises 50 wt%, such as 10 to 85 wt% FFA of the total weight of the composition.

The amount of phosphorus and metal impurities may be determined e.g. according to ASTM D5185-18 standard test method or as described herein in the experimental part, preferably as described herein.

## **Adjusting the Net Elementary Charge - Step ii)**

The molar ratio of the dissolved phosphorus impurities to the dissolved metal impurities in the lipid feedstock is reflected in the net elementary charge of the lipid feedstock based on phosphorus and the metal(s) contained in the lipid feedstock.

The term “net elementary charge of a feedstock” means the net elementary charge of phosphorus and the metal(s) contained in the feedstock, while the term “net elementary charge of the feedstock” is used for brevity. Also, when a numerical

value of the net elementary charge of a feedstock is given, the unit is mmol elementary charge/kg of feedstock in question, while also mmol/kg may be used for brevity.

The total net elementary charge of the lipid feedstock is determined by the net elementary charge Q1 based on phosphorus and the at least one metal obtained by equation (I)

$$Q1 = (CPQP) + i(CMiQM_i) \quad (I)$$

wherein

CP is the concentration of dissolved phosphorus in the feedstock in mmol/kg of feedstock,

QP is the elementary charge of dissolved phosphorus in the feedstock,

CM<sub>i</sub> is the concentration of dissolved metal i in the feedstock in mmol/kg of feedstock,

QM<sub>i</sub> is the elementary charge of the dissolved metal i in the feedstock, and

i is the number of dissolved metals taken into account, and i is 1-n.

The total net elementary charge Q<sub>t</sub> is then the sum of the net elementary charges of each lipid feedstock and optional charge balancing component different from the lipid feedstock, i.e.

$$Q_t = Q_1 + Q_2 + Q_3 + \dots + Q_C$$

The elementary charge of phosphorus is -3 e in this equation, as assumed herein to be in the form of a phosphate. The elementary charge of sodium is +1 e, that of potassium +1 e, that of magnesium +2 e, that of calcium +2 e, and that of iron +3 e, depending on the compounds present. In case there are other dissolved metals present in the feed in significant amounts, in an amount higher than 0.1 ppm by weight, those metals are also considered in the calculation of the net elementary charge, with their elementary charge. The same limit of significant amount (0.1 ppm) may also be used for sodium, potassium, magnesium, calcium and iron. The metals include Na, K, Mg, Ca, Fe, Al, Cr, Pb, Mn, Zn, W, Ni and Cu, or any combinations thereof. The elementary charge of a metal is the valence the metal typically has

when forming a metal salt, such as metal phosphate. A person skilled in the art may also use another method for this determination. Only the concentration of dissolved phosphorus and metal(s) (not solid impurities) are used in the net elementary charge calculation. In accordance with the present method, solids are removed from the feedstock to be treated by heat treatment and are thus not considered for determining the net elementary charge  $Q_1$  and/or total net elementary charge  $Q_t$ .

When the net elementary charge  $Q_1$  of the lipid feedstock (a) is below 0 mmol/kg, the net elementary charge of the lipid feedstock is adjusted to total net elementary charge  $Q_t$  of at least 0 mmol/kg, such as from 0 to 10 mmol/kg, preferably at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg, by mixing the lipid feedstock with a charge balancing component comprising, or consisting of, a metal compound, such as an metal hydroxide, an inorganic metal salt, an organic metal salt or any mixture thereof, and/or a first further lipid composition comprising one or more metal compound(s). The adjustment is done by mixing with the charge balancing component(s) to achieve a charge balance. In that case where a first further lipid composition is utilized, the lipid feedstock having high phosphorus content (i.e. negative net elementary charge) needs to be mixed with a first further lipid composition having suitably high metal content, so that there are slightly more metal charge than P charge in the resulting mixture.

The adjustment in step ii) is done prior to the heat treatment step v). The adjustment can be done either before the removal of solid impurities in step iv) or after step iv) and either before, simultaneously, or after the optional adjustment of the free fatty acid (FFA) concentration in step iii), when step iii) is performed. It is required that the net elementary charge  $Q_t$  before the heat treatment in step v) is above 0 mmol/kg, such as from 0 to 10 mmol/kg, preferably at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg, so that there is an excess of metals rather than phosphorus in the feedstock to be treated. The total net elementary charge  $Q_t$  of the feedstock to be treated is preferably slightly positive (meaning a surplus of metals) to ensure the dissolved phosphorus is converted to insoluble phosphorus compounds in the heat treatment step as well as possible. Having a slight surplus of metals in the feed after the

present treatment can be handled, if need be, by removal of the metals with sufficient acid dosage in a subsequent purification step. With a slight surplus of metals over phosphorus (i.e. with slightly positive total net elementary charge), good filtration throughputs are obtained in subsequent bleaching steps as compared to  
5 negative total net elementary charge case.

The amount of added charge balancing component will depend on the quality of the lipid feedstock (a), i.e. the amount of dissolved phosphorus present in the lipid feedstock and the molar ratio of the dissolved phosphorus impurities to the dissolved metal impurities in the lipid feedstock i.e. the net elementary charge Q1 of the lipid  
10 feedstock (a).

The charge balancing component may be used as such or, in particular when the charge balancing component is a metal hydroxide, an inorganic metal salt, and/or an organic metal salt, as an aqueous solution. The aqueous solution contributes in the treatment to easier dosing, mixing, and distribution of the reagent to the lipid  
15 feedstock. In the aqueous solution, the content of the charge balancing component may be from 5 to 50 wt%, such as about 6 wt%, about 10 wt% or about 50 wt% of the total weight of the aqueous solution. Even if the aqueous solution is relatively diluted, the total amount of the aqueous solution remains low. In the following heat treatment (HT), the low water content provides lower pressure HT reactor setup  
20 lowering costs and minimization of triglyceride hydrolysis into free fatty acids. Further advantages are related to wastewater generated and water phase separation when discarding the solid reject.

Examples of suitable metal compounds used as the charge balancing component include, but are not limited to, soaps, alkali metal hydroxide(s), and/or inorganic and  
25 organic alkali metal salt(s). Preferably alkali metal hydroxide, such as NaOH or KOH is used. Use of alkali metal hydroxide, in particular NaOH, is particularly preferred.

Examples of suitable first further lipid components utilized as the charge balancing component include, but are not limited to, palm oil mill effluent (POME), brown grease (BG), crude tall oil (CTO), and any mixtures thereof.

30 When the adjustment of the net elementary charge of step ii) is done before step iv), in particular by addition of a metal hydroxide, such as an alkali metal hydroxide,

the FFA concentration of the lipid feedstock is advantageously adjusted in step iii) to above 2 wt% of the total weight of the resulting lipid feedstock to prevent removal of the added (alkali) metal during the solid removal step iv) as addition of the (alkali) metal hydroxide may induce formation of (alkali) metal containing solids and resultantly reverse the total net elementary charge  $Q_t$  of the lipid feedstock back to undesired level. When both the adjustment of the net elementary charge of the lipid feedstock in step ii) and the adjustment of the FFA concentration of the lipid feedstock in step iii) are both performed, they can be performed in any order in relation to each other or even in a single step, in particular when a single lipid composition is utilized as the second further lipid composition comprising a suitably high amount of one or more free fatty acids of step iii) and as the first further lipid composition of step ii). In an embodiment, adjusting the FFA concentration of the lipid feedstock in step ii) is done before adjusting the net elementary charge of the lipid feedstock in step iii). In an alternative embodiment the adjusting the net elementary charge of the lipid feedstock in step ii) is done before adjusting the FFA concentration of the lipid feedstock in step iii). In a further alternative embodiment adjusting the net elementary charge of the lipid feedstock and adjusting the FFA concentration of the lipid feedstock are performed simultaneously.

#### **Adjusting the Free Fatty Acid Concentration**

Optionally the free fatty acid (FFA) concentration of the lipid feedstock may be adjusted to predetermined level in step iii) before the lipid feedstock is subjected to removal of the solid impurities in step iv). Presence of step iii) is particularly desired when adjustment of the net elementary charge in step ii) is accomplished before removal of solids in step iv). Desirably, the free fatty acid (FFA) composition of the lipid feedstock (a) is optionally, yet preferably, adjusted to above 2 wt%, such as 2 to 40 wt%, of the total weight of the resulting lipid feedstock. As demonstrated in Example 3, this enhances the filterability of the lipid feedstock in removal of the solid impurities when step iv) is performed by filtration. Adjustment of the FFA concentration further allows dosing of charge balancing component(s), in particular metal hydroxides, already at the beginning of the process without possibility of process upsets. This further enables flexible use of challenging lipid feedstocks to be purified to low impurities levels required in downstream refining processes.

Preferably the FFA concentration of the lipid feedstock is adjusted to at least 3 wt%, such as from 3 to 30 wt%, of the total weight of the resulting lipid feedstock, preferably to at least 4 wt%, such as from 4 to 20 wt%, of the total weight of the lipid feedstock, more preferably to 4 to 10 wt%, of the total weight of the resulting lipid feedstock. In concentrations of 2 wt% and below clogging of the filter may be observed and loss of charge balancing component. While there appears to be no upper limit for the FFA concentration of the lipid feedstock in regards to observation of the desired effect, it is not economically feasible to adjust the FFA concentration much above the lowest required limit.

- Adjusting the FFA concentration may be achieved by mixing the lipid feedstock with a lipid component comprising, or consisting, of one or more free fatty acid, such as one or more free fatty acids and/or with a second further lipid composition comprising a suitably high amount of one or more free fatty acids. Examples of suitable free fatty acids include, but are not limited to, C<sub>13</sub>-C<sub>21</sub> free fatty acids, such as palmitic acid, stearic acid, oleic acid, and linoleic acid. Examples of second further lipid compositions comprising a suitably high amount of one or more free fatty acids include, but are not limited to, soapstock acid oil (SAO), palm oil mill effluent (POME), brown grease (BG), fatty acid distillate (FAD), and mixtures thereof. The free fatty acid concentration of the lipid composition comprising a suitably high amount of one or more free fatty acids comprises at least 30 wt%, such as from 30 to 100 wt%, free fatty acids of the total weight of the lipid composition, preferably at least 50 wt%, such as from 50 to 80 wt%, free fatty acids of the total weight of the lipid composition. Advantageously POME is utilized as both the first further lipid composition and as the second further lipid composition.
- In an embodiment the desired FFA concentration is achieved by mixing with one or more free fatty acids.

The FFA concentration may be defined according to ISO 660:2020 noting that the potentiometric determination method is preferred.

#### **Removal of the Solid Impurities**

- In step iv) the lipid feedstock is subjected to removing solid impurities, and optionally excess water, from the lipid feedstock (a). Solid impurities removed in step iv)

include e.g. salts, protein residues, bone meal, fibers, carbohydrates, and/or sand. Step iv) and step ii) can be performed in any order in relation to each other. In an embodiment, removal of solid impurities in step iv) can be done before adjusting the net elementary charge of the lipid feedstock in step ii). In an alternative embodiment  
5 adjusting the net elementary charge of the lipid feedstock in step ii) is done before removal of the solid impurities in step iv). When removal of solid impurities is accomplished after adjusting the net elementary charge, step iii) is advantageously performed before removal of the solid impurities.

The removal of solid impurities may be accomplished by any suitable phase  
10 separation method suitable for removing solid impurities from lipid materials, including, but not limited to, settling, centrifuging, filtering, and any combination of those, preferably centrifuging and/or filtering.

It is particularly preferred to accomplish the removal of solid impurities by filtering the lipid feedstock with a filter aid, i.e. filter aid filtration (FAF). Examples of suitable  
15 filter aid materials include, but are not limited to, any material comprising or consisting of mineral, silicon, and/or cellulose based material, e.g. diatomaceous earth, diatomite, perlite, bentonite, palygorskite, kaolin, kaolinite, silica in various crystalline or amorphous configurations, sepiolite, magnesium silicate, silicon, aluminum oxide based materials, zinc oxide based materials, neutral bleaching  
20 earth, activated carbon, activated charcoal, cellulose fibers, or any combinations thereof. The filter aid material may be activated by means known in the art.

Advantageously the lipid feedstock is dried before filter aid filtration to avoid leaching of metal from the filter aid material.

Alternatively, the lipid feedstock is centrifuged in step iv). This is beneficial as both  
25 excess water and solid impurities can be removed from the lipid feedstock.

After the removal of solid impurities the lipid feedstock advantageously comprises less than 0.5 wt%, such as 0 to 0.2 wt%, insoluble impurities of the total weight of the lipid feedstock. Generally lowering the amount of insoluble impurities in the lipid feedstock below 0.1 wt%, renders the lipid feedstock suitable for successful  
30 downstream treatment enabling smooth downstream phase-separation operations

following the further purification steps such as filtration in a subsequent bleaching step.

The amount of insoluble impurities may be determined according to ISO 663:2017.

#### **Heat Treatment, Step v)**

- 5 After accomplishing steps ii) to iv) as described above, the thus obtained conditioned lipid feedstock is subjected in step v) to a heat-treatment at a temperature of at least 180 °C such as from 180 to 325 °C, preferably of at least 200 °C, such as from 200 to 325 °C, preferably of at least 220 °C, such as from 220 to 325 °C, more preferably of at least 270°C, such as from 270 to 300 °C, most  
10 preferably of at least 280 °C, such as from 280 to 290 °C. The heat treatment promotes the separation of impurities, such as phosphorus, as solid precipitates. Temperatures above 180 °C promote formation of solid rejects from the lipid material. In temperatures above 325 °C thermal cracking of the components may start to be observed.
- 15 The heat treatment in step v) may be carried out at a pressure of 100 to 5100 kPa(a), preferably 150 to 2100 kPa(a), more preferably 200 to 600 kPa(a), most preferably 300 to 500 kPa(a). A person skilled in the art will be competent to adjust the time to fit the intended purpose, appreciating that elevating the pressure minimizes losses of lipid material in the heat treatment step.
- 20 The residence time in the heat treatment step v) may be only from a few minutes up to a few hours depending on the temperature. A person skilled in the art will be competent to adjust the time to fit the intended purpose, appreciating that at higher temperatures a shorter residence time is sufficient. In an embodiment the residence time is from 5 to 120 minutes.
- 25 It is necessary that the total net elementary charge  $Q_t$  of the conditioned lipid feedstock treated under the heat treating conditions is in the range required in the step ii) i.e. at least 0 mmol/kg, such as from 0 to 10 mmol/kg, preferably at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg. If any of the pretreatment steps following step ii) and preceding  
30 step v) should alter the total net elementary charge  $Q_t$  it will be necessary to further adjust the total net elementary charge  $Q_t$  before the heat treatment step v) is

commenced. However, it is desired that only a single adjustment of step ii) is required and any changes in the total net elementary charge  $Q_t$  during the intermediate steps is taken into account already at that stage by over-adjusting the net elementary charge as required as far as it is within the therein defined values.

- 5 Advantageously at least a small amount of water is present in the mixture treated in the heat treatment step v). The mixture advantageously contains less than 10 wt%, preferably less than 5 wt%, such as 0.05 wt% to 2 wt%, preferably less than 1.5 wt%, such as from 0.2 wt% to 1.5 wt%, more preferably less than 0.5 wt% water of the total weight of the mixture. Generally the amount of water dissolved in the lipid  
10 feedstock is adequate and no water addition is done. Keeping the water content low contributes to avoidance of emulsions and allows lower processing pressure, in particular when the water content is kept in less than 1 wt%, or preferably in less than 0.5 wt% water of the total weight of the mixture.

- Heat treatment in step v) may be performed in any suitable reactor wherein the  
15 indicated conditions may be achieved. Examples of suitable reactors include mixed reactors and/or tube reactors. Further, the heat treatment in step v) may be performed in one or more batches and/or in continuous mode.

- Generally, it is advantageous to boost the contact and transfer between the charge  
balancing component and the lipid feedstock during the heat treatment. Accordingly,  
20 in preferred embodiments, mixing is provided.

After the heat treatment in step v) the heat-treated lipid feedstock (c) is obtained. The heat-treated lipid feedstock (c) may then be directly subjected to recovery by phase separation in step vii) and/or it may be further purified by treatment with an acid and/or adsorbent material in step vi).

25 **Further treatment, Step vi)**

- In step vi) the heat-treated lipid feedstock (c) is optionally subjected to further purification by treating the heat-treated lipid feedstock (c) with an acid and/or adsorbent material, in particular under bleaching conditions, to obtain a further treated lipid feedstock (d). The further purification of the heat-treated lipid feedstock  
30 (c) is particularly advantageous for lipid feedstock (a) having higher amounts of phosphorous and/or metal impurities.

The further treatment with acid and/or adsorbent material aims at minimizing the content of impurities, such as pigments (e.g. carotenoids and chlorophylls), metals and/or phosphorus, in the treated material. It involves contacting, in particular mixing, the material to be treated with an acid and/or an adsorbent material. Further  
5 treatment in step vi) may be carried out in the presence of an acid, such as citric acid and/or phosphoric acid and/or acidic or acid activated adsorbent. Further, further treatment in step vi) is typically carried out in the presence of a small amount of water. Advantageously bleaching earth or other adsorbent such as silica can be added to the heat-treated lipid feedstock (c) in step vi) to adsorb impurities, such as  
10 remaining metals and/or phosphorus.

The further treatment is preferably accomplished under bleaching conditions. A skilled person will be competent to select the bleaching conditions. Typically the temperature in the further treatment step vi) can be for example in the range from 80 to 120 °C. Further, typically the pressure is close to atmospheric pressure, such  
15 as from 60 kPa(a) to 600 kPa(a), preferably from 80 kPa(a) to 200 kPa(a).

Prior to further treatment in step vi) and after the heat treatment in step v) the heat-treated lipid feedstock (c) may be subjected to solids removal, and impurities containing reject may be discarded. Solid impurities formed in the heat treatment may be phase separated based on the lipid material forming a phase of its own, an  
20 oily phase, from which any impurities containing aqueous and/or solid reject may be removed by ordinary separation unit processes. Accordingly, the term “phase-separating” as used herein and hereafter refers both to liquid-liquid and liquid-solid phase separation. Suitable phase separation means include, but are not limited to, filtration, centrifugation, settling, and any combination thereof.

25 Prior to further treatment in step vi) the heat-treated purified lipid material may additionally or alternatively be subjected to drying, in particular evaporation, under conditions capable of removing water vapor from said heat-treated purified lipid material. However, the evaporation conditions are controlled such that low-boiling components, such as low-boiling fatty acids of the lipid material are not lost.  
30 According to an embodiment containing evaporation, the evaporation may be performed at temperature from 50 to 130 °C, and pressure from 1 to 100 kPa(a). As

an example, a combination of temperature of 105 °C and pressure of 8 kPa(a) could be applied to evaporation.

Preferably the heat-treated feedstock is directly subjected to step vi) and no intermediate recovery is accomplished.

#### 5 **Recovery, Step vii)**

After heat-treatment of step v) and/or after optional further treatment of step vi), the heat-treated lipid feedstock (c) and/or the further treated lipid feedstock (d), respectively, is subjected to phase-separation, preferably filtration, to remove the solids together with the solidified and/or precipitated impurities to provide a purified  
10 lipid feedstock (e). In cases where the phase-separation is accomplished by filtration, the preceding steps allow smooth operation.

Solid impurities formed in the heat treatment step v) and/or further treatment step vi) as well as solid adsorbent material resulting from step vi) may be phase separated based on the lipid material forming a phase of its own, an oily phase, from  
15 which any impurities containing aqueous and/or solid reject may be removed by ordinary separation unit processes. Accordingly, the term “phase-separating” as used herein and hereafter refers both to liquid-liquid and liquid-solid phase separation. Suitable phase separation means include, but are not limited to, filtration, centrifugation, settling, and any combination thereof.

20 The phase-separation is preferably accomplished by filtration. Filtration may be performed by any means found suitable for this purpose by a skilled person.

Prior or after the phase-separation the heat-treated lipid feedstock (c) and/or further treated lipid feedstock (d), respectively, may be subjected to drying, in particular evaporation, under conditions capable of removing water vapor from said lipid  
25 feedstock. However, the evaporation conditions are controlled such that low-boiling components, such as low-boiling fatty acids of the lipid material are not lost. Typically the evaporation could be performed at temperature from 50 to 130 °C, and pressure from 1 to 100 kPa(a). As an example, a combination of temperature of 105 °C and pressure of 8 kPa(a) could be applied to evaporation.

The thus rendered purified lipid feedstock typically comprises residual phosphorus and metals both below 30 mg/kg, preferably below 2.0 mg/kg, more typically close to 1.0 mg/kg. Impurity reduction for phosphorus is typically over 90% and for metals over 95% as compared to the lipid feedstock (a) factoring out any dilution of the lipid feedstock (a) due addition of charge balancing component(s) and/or FFA adjustment with lipid component(s). The actual purification result is dependent on the quality of the initial feedstock.

Figure 1 illustrates a first exemplary process flow of the present method. The process flow exemplified in Figure 1 is particularly suitable for lipid feedstock having the total net elementary charge Q1 of the lipid feedstock below 0 mmol/kg and free fatty acid concentration of 2 wt% or less, in particular below 3 wt%.

Referring to Figure 1, a feed of lipid feedstock 1 is subjected to a step of adjusting the net elementary charge of the lipid feedstock with a charge balancing component 2 to obtain a charge-balanced lipid feedstock 11 as discussed herein for step ii). The charge-balanced lipid feedstock 11 is then subjected to a step of adjusting the free fatty acid concentration of the charge-balanced lipid feedstock 11 with a FFA component 3 to obtain a FFA adjusted and charge balanced lipid feedstock 12 as discussed herein for step iii). The FFA adjusted and charge balanced lipid feedstock 12 is then subjected to as step of removal 30 of solid impurities 39, preferably by filter aid filtration, as discussed herein for step iv) to obtain a conditioned lipid feedstock 13 having a total net elementary charge Qt above 0 mmol/kg, preferably 0.5 mmol/kg, such as from 0.5 to 3 mmol/kg. The conditioned lipid feedstock 13 is then subjected to a step of heating 40 the conditioned lipid feedstock 13, preferably in the presence of water, at a temperature from 180 to 325 °C as discussed herein for step v) to obtain a heat-treated lipid feedstock 41. The heat-treated lipid feedstock may then optionally be subjected to step of further treating 70 with an acid and/or absorbent material 71, such as under bleaching conditions, to further purify the heat-treated lipid feedstock 41 as discussed herein for step vi) to obtain a further treated lipid feedstock 43. Optionally, prior to further treatment 70, the heat-treated lipid feedstock 41 may be subjected to a step of phase-separation 60 to remove solid material 69 comprised in the heat-treated lipid feedstock 41 to obtain a solid-depleted heat-treated lipid

feedstock 42 which is then subjected to further treatment 70 to obtain the further treated lipid feedstock 43. The heat-treated lipid feedstock 41 and/or the further treated lipid feedstock 43 is subjected to a step of phase-separation, preferably filtering, 50 to obtain a purified lipid feedstock 51.

- 5 Figure 2 illustrates a second exemplary process flow of the present method. The process flow exemplified in Figure 2 is particularly suitable for lipid feedstock having the total net elementary charge Q1 of the lipid feedstock below 0 mmol/kg and free fatty acid concentration of 2 wt% or less, in particular below 3 wt%.

Referring to Figure 2, a feed of lipid feedstock 1 is subjected to a step of adjusting  
10 the free fatty acid concentration 20 of the lipid feedstock 1 with a lipid component 3 to obtain a FFA adjusted lipid feedstock 14 as discussed herein for step iii). The FFA adjusted lipid feedstock 14 is then subjected to a step of adjusting 10 the net elementary charge of the FFA adjusted lipid feedstock 14 with a charge balancing component 2 to obtain a FFA adjusted and charge-balanced lipid feedstock 12 as  
15 discussed herein for step ii). The FFA adjusted and charge balanced lipid feedstock 12 is then subjected to a step of removal 30 of solid impurities 39, preferably by filter aid filtration, as discussed herein for step iv) to obtain a conditioned lipid feedstock 13 having a total net elementary charge  $Q_t$  above 0 mmol/kg, preferably 0.5 mmol/kg, such as from 0.5 to 3 mmol/kg. The conditioned lipid feedstock 13 is then  
20 subjected to a step of heating 40 the conditioned lipid feedstock 13, preferably in the presence of water, at a temperature from 180 to 325 °C as discussed herein for step v) to obtain a heat-treated lipid feedstock 41. The heat-treated lipid feedstock may then optionally be subjected to step of further treating 70 with an acid and/or absorbent material 71, such as under bleaching conditions, to further purify the heat-  
25 treated lipid feedstock 41 as discussed herein for step vi) to obtain a further treated lipid feedstock 43. Optionally, prior to further treatment 70, the heat-treated lipid feedstock 41 may be subjected to a step of phase-separation 60 to remove solid material 69 comprised in the heat-treated lipid feedstock 41 to obtain a solid-depleted heat-treated lipid feedstock 42 which is then subjected to further treatment  
30 70 to obtain the further treated lipid feedstock 43. The heat-treated lipid feedstock 41 and/or the further treated lipid feedstock 43 is subjected to a step of phase-separation, preferably filtering, 50 to obtain a purified lipid feedstock 51.

Figure 3 illustrates a third exemplary process flow of the present method. The process flow exemplified in Figure 3 is particularly suitable for lipid feedstock having the total net elementary charge Q1 of the lipid feedstock below 0 mmol/kg and free fatty acid concentration of 2 wt% or less, in particular below 3 wt%.

- 5 Referring to Figure 3, a feed of lipid feedstock 1 is subjected to a step of adjusting 90 the free fatty acid concentration and the net elementary charge of the lipid feedstock 1 with second lipid feedstock 4 representing a FFA component and a charge balancing component as discussed herein for step ii) and iii) to obtain a FFA adjusted and charge-balanced lipid feedstock 14. The FFA adjusted and charge
- 10 balanced lipid feedstock 14 is then subjected to as step of removal 30 of solid impurities 39, preferably by filter aid filtration, as discussed herein for step iv) to obtain a conditioned lipid feedstock 13 having a total net elementary charge Qt above 0 mmol/kg, preferably 0.5 mmol/kg, such as from 0.5 to 3 mmol/kg. The conditioned lipid feedstock 13 is then subjected to a step of heating 40 the
- 15 conditioned lipid feedstock 13, preferably in the presence of water, at a temperature from 180 to 325 °C as discussed herein for step v) to obtain a heat-treated lipid feedstock 41. The heat-treated lipid feedstock may then optionally be subjected to step of further treating 70 with an acid and/or absorbent material 71, such as under bleaching conditions, to further purify the heat-treated lipid feedstock 41 as
- 20 discussed herein for step vi) to obtain a further treated lipid feedstock 43. Optionally, prior to further treatment 70, the heat-treated lipid feedstock 41 may be subjected to a step of phase-separation 60 to remove solid material 69 comprised in the heat-treated lipid feedstock 41 to obtain a solid-depleted heat-treated lipid feedstock 42 which is then subjected to further treatment 70 to obtain the further
- 25 treated lipid feedstock 43. The heat-treated lipid feedstock 41 and/or the further treated lipid feedstock 43 is subjected to a step of phase-separation, preferably filtering, 50 to obtain a purified lipid feedstock 51.

- Figure 4 illustrates a fourth exemplary process flow of the present method. The process flow exemplified in Figure 4 is particularly suitable for lipid feedstock having
- 30 the net elementary charge Q1 of the lipid feedstock below 0 mmol/kg and free fatty acid concentration above 2 wt%.

Referring to Figure 4, a feed of lipid feedstock 1 is subjected to a step of adjusting 10 the net elementary charge of the lipid feedstock with a charge balancing component 2 to obtain a charge balanced feedstock 12 as discussed herein for step ii). The charge balanced lipid feedstock 12 is then subjected to a step of removal 5 30 of solid impurities 39, preferably by filter aid filtration, as discussed herein for step iv) to obtain a conditioned lipid feedstock 13 having a total net elementary charge  $Q_t$  above 0 mmol/kg, preferably 0.5 mmol/kg, such as from 0.5 to 3 mmol/kg. The conditioned lipid feedstock 13 is then subjected to a step of heating 40 the conditioned lipid feedstock 13, preferably in the presence of water, at a temperature 10 from 180 to 325 °C as discussed herein for step v) to obtain a heat-treated lipid feedstock 41. The heat-treated feedstock may then optionally be subjected to step of further treating 70 with an acid and/or absorbent material 71, such as under bleaching conditions, to further purify the heat-treated lipid feedstock 41 as discussed herein for step vi) to obtain a further treated lipid feedstock 15 43. Optionally, prior to further treatment 70, the heat-treated lipid feedstock 41 may be subjected to a step of phase-separation 60 to remove solid material 69 comprised in the heat-treated lipid feedstock 41 to obtain a solid-depleted heat-treated lipid feedstock 42 which is then subjected to further treatment 70 to obtain the further treated lipid feedstock 43. The heat-treated lipid feedstock 41 and/or the further 20 treated lipid feedstock 43 is subjected to a step of phase-separation, preferably filtering, 50 to obtain a purified lipid feedstock 51.

Figure 5 illustrates a fifth exemplary process flow of the present method. The process flow exemplified in Figure 5 is particularly suitable for lipid feedstock having the net elementary charge  $Q_1$  of the lipid feedstock below 0 mmol/kg.

25 Referring to Figure 5, a feed of lipid feedstock 1 is subjected to a step of removal of solid impurities 30, preferably by filter aid filtration, as discussed herein for step iv) to obtain a solid-depleted lipid feedstock 15. The solid-depleted lipid feedstock is then subjected to step of adjusting 10 the net elementary charge of the solid-depleted lipid feedstock 15 with a charge balancing component 2 to obtain a 30 conditioned lipid feedstock 13 having a total net elementary charge  $Q_t$  above 0 mmol/kg, preferably 0.5 mmol/kg, such as from 0.5 to 3 mmol/kg. The conditioned lipid feedstock 13 is then subjected to a step of heating 40 the conditioned lipid

feedstock 13, preferably in the presence of water, at a temperature from 180 to 325 °C as discussed herein for step v) to obtain a heat-treated lipid feedstock 41. The heat-treated feedstock may then optionally be subjected to step of further treating 70 with an acid and/or absorbent material 71, such as under bleaching conditions, to further purify the heat-treated lipid feedstock 41 as discussed herein for step vi) to obtain a further treated lipid feedstock 43. Optionally, prior to further treatment 70, the heat-treated lipid feedstock 41 may be subjected to a step of phase-separation 60 to remove solid material 69 comprised in the heat-treated lipid feedstock 41 to obtain a solid-depleted heat-treated lipid feedstock 42 which is then subjected to further treatment 70 to obtain the further treated lipid feedstock 43. The heat-treated lipid feedstock 41 and/or the further treated lipid feedstock 43 is subjected to a step of phase-separation, preferably filtering, 50 to obtain a purified lipid feedstock 51.

Figure 6 illustrates a sixth exemplary process flow of the present method. The process flow exemplified in Figure 6 is particularly suitable for lipid feedstock having the net elementary charge Q1 of the lipid feedstock above 0 mmol/kg and free fatty acid concentration above 2 wt%.

Referring to Figure 6, a feed of lipid feedstock 1 is subjected to a step of removal of solid impurities 30, preferably by filter aid filtration, as discussed herein for step iv) to obtain a conditioned lipid feedstock 13 having a total net elementary charge Qt above 0 mmol/kg, preferably 0.5 mmol/kg, such as from 0.5 to 3 mmol/kg. The conditioned lipid feedstock 13 is then subjected to a step of heating 40 the conditioned lipid feedstock 13, preferably in the presence of water, at a temperature from 180 to 325 °C as discussed herein for step v) to obtain a heat-treated lipid feedstock 41. The heat-treated feedstock may then optionally be subjected to step of further treating 70 with an acid and/or absorbent material 71, such as under bleaching conditions, to further purify the heat-treated lipid feedstock 41 as discussed herein for step vi) to obtain a further treated lipid feedstock 43. Optionally, prior to further treatment 70, the heat-treated lipid feedstock 41 may be subjected to a step of phase-separation 60 to remove solid material 69 comprised in the heat-treated lipid feedstock 41 to obtain a solid-depleted heat-treated lipid feedstock 42 which is then subjected to further treatment 70 to obtain the further

treated lipid feedstock 43. The heat-treated lipid feedstock 41 and/or the further treated lipid feedstock 43 is subjected to a step of phase-separation, preferably filtering, 50 to obtain a purified lipid feedstock 51.

### **Hydroprocessing of the Purified Lipid Feedstock**

- 5 After the lipid feedstock has been purified in accordance with the purification method discussed herein, it may be used as such and/or subjected to further valorization, such as hydroprocessing, to obtain renewable hydrocarbons such as e.g. drop-in renewable fuel(s), renewable fuel component(s) and/or other valuable renewable hydrocarbon products. Such catalytic upgrading processes include, but are not
- 10 limited to, catalytic cracking, catalytic hydrocracking, thermo-catalytic cracking, catalytic hydrotreatment, fluid catalytic cracking, catalytic ketonization, and catalytic esterification. Such processes require the liquid feedstock to be sufficiently pure and free from impurities that may otherwise hamper the catalytic process or deactivate or poison the catalyst(s) present in the process.
- 15 Thus the purified lipid feedstock may be subjected to a post-treatment comprising hydroprocessing.

Consequently, herein is provided a use of the purified lipid feedstock obtained from the purification method as described herein as feed to at least one catalytic hydroprocessing to obtain renewable hydrocarbons. Further, the purified lipid

20 feedstock obtained from the purification method as described in the foregoing may be used as feed to produce fatty acids, and/or soaps.

Further, herein is provided a process for providing renewable hydrocarbons, comprising x) purifying a lipid feedstock to obtain purified lipid feedstock as described in the foregoing, and y) subjecting the purified lipid feedstock to

25 hydroprocessing, preferably catalytic hydroprocessing, to obtain at least one renewable hydrocarbon.

The lipid feedstock is the lipid feedstock as defined herein. Preferably the lipid material may comprise streams known for their high phosphorous, metal and/or solid impurity content, such as SAO, AFP, and LQAF.

The hydroprocessing, preferably catalytic hydroprocessing, may be any upgrading process employing hydrogen and where the lipid material may be used as the process feed, optionally with a co-feed. For example, the hydroprocessing may be an upgrading process to obtain liquid transportation fuel components, solvents, technical fluids, such as electrotechnical fluids, fatty alcohols, cracking feedstocks, such as feedstocks for thermal cracking and/or catalytic cracking, and/or base chemicals for different syntheses. Preferably the hydroprocessing is catalytic hydroprocessing.

According to an embodiment a co-feed of fossil origin is fed to catalytic hydroprocessing.

The hydroprocessing may comprise altering molecular weight, removal of heteroatoms, altering degree of saturation, rearranging molecular structure, or any combination thereof. The hydroprocessing comprises preferably altering molecular weight of the process feed or any intermediate stream or intermediate product derivable or derived therefrom, removal of heteroatoms from the process feed or any intermediate stream or intermediate product derivable or derived therefrom, altering degree of saturation of the process feed or any intermediate stream or intermediate product derivable or derived therefrom, rearranging molecular structure of the process feed or any intermediate stream or intermediate product derivable or derived therefrom, or any combination thereof.

In certain preferred embodiments, the hydroprocessing comprises hydrotreatment, isomerization, and/or cracking, preferably hydrodeoxygenation (HDO), hydroisomerization (HI), and/or hydrocracking (HC), of the process feed or an intermediate stream or intermediate product derivable or derived therefrom, optionally followed by fractionation.

In certain preferred embodiments, the catalytic hydroprocessing comprises catalytic hydroprocessing converting the lipid material to one or more drop-in liquid transportation fuel(s), one or more liquid transportation fuel component(s) and/or other valuable hydrocarbon product(s) chemicals. The process comprises subjecting the process feed to hydroprocessing comprising hydrodeoxygenation, hydroisomerization, and optionally hydrocracking, followed by fractionation of the

hydroprocessing effluent and recovery of one or more drop-in liquid transport fuel(s), one or more liquid transportation fuel component(s) and/or other valuable hydrocarbon products from the fractionation.

According to an embodiment the catalytic hydrotreating comprises one or more of  
5 hydrodeoxygenation, hydroisomerization, hydrocracking, hydrodenitrogenation, hydrodesulfurization, hydrodehalogenation, hydrodearomatization, and hydrogenation of double bonds. High metal content in the feed tends to deactivate catalysts typically used in these processes. Considering this, it is specifically beneficial that the content of the added metal containing charge balancing  
10 component may be kept relatively low in step ii).

The catalytic hydroprocessing may occur in the presence of a catalyst selected from Pd, Pt, Ni, Co, Mo, Ru, Rh, W, or any combination of these, such as CoMo, NiMo, NiW, CoNiMo, NiMoW or together with SAPO-11, SAPO-41, ZSM-22, ZSM-23, ZSM-12, ZSM-48, ZSM-5, beta zeolites, ferrierite and mixtures thereof, such as  
15 Pt/SAPO-11/Al<sub>2</sub>O<sub>3</sub>, Pt/ZSM-22/ Al<sub>2</sub>O<sub>3</sub>, Pt/ZSM-23/Al<sub>2</sub>O<sub>3</sub>, Pt/SAPO-11/SiO<sub>2</sub>, optionally on a support, wherein the support comprises preferably alumina and/or silica.

## EXPERIMENTAL

FFA content in feed: The FFA concentration was analyzed from all samples using  
20 gel permeation chromatography (GPC) in accordance with ISO 660:2020. Samples were analyzed diluted in tetrahydrofuran. The components of the sample (oligomers, fatty acids, mono-, di- and triglycerides and other compounds such as hydrocarbons are identified by their retention times.

P and metal impurity analysis: The concentration of phosphorus and metals was  
25 analyzed from all samples by first digesting the sample with acids in a microwave oven to obtain a clear water/acid matrix (assessed visually), then diluting it to a known amount and analyzing it against the acid based calibration using ICP-MS/MS (tandem Inductively Coupled Plasma Mass Spectrometry). Metals detected by the method include Li, B, Na, Mg, Al, P, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As,  
30 Mb, Cd, Sn, and Ba.

Example 1: Effect of charge balancing on purification and filtration.

Low Quality Animal Fat (LQAF) feedstock and two different Used Cooking Oil (UCO) samples were subjected to solids removal, heat treatment (with and without adjustment of net elementary charge), followed by filtration or bleaching (treatment  
5 with acid and adsorbent followed by filtration). All feedstock samples were obtained from commercial sources.

Solids removal: either centrifugation (4300 RPM, 80 °C, 30 min) for UCO samples or filter aid filtration for LQAF were used to remove solids from the crude feed. Filter aid filtration was performed with commercial diatomaceous earth filter aid (1 wt%)  
10 added to the sample. A precoat on the filter weave was made with 1 kg/m<sup>2</sup> of the same filter aid and Refined Bleached Deodorized Palm Oil (RBDPO) prior filtration of the sample. Filtering of the sample was performed using a pressure filtration system at 85 °C at 2.5 bar pressure.

Adjusting the net elementary charge of the feed sample was performed by adding  
15 10 wt% aqueous NaOH solution to achieve the positive total elementary charge indicated in the result table 1 (+1 or +5). The other charge values (-7.3, -0.3 and +0.3) refer to the natural net elementary charge of the filtered feeds.

Heat Treatment was performed with a 1 liter pressure reactor with heating programs of 280 °C 30 min (LQAF) or 250 °C 60 min (UCO samples) at initial pressure 300  
20 kPa(a) (nitrogen) with mixing of 500 rpm. The heat treated feedstock was further filtered or bleached (treated with acid and adsorbent and filtered).

After heat treatment the filtration was done by filtering the heat treated feedstock with a 0.45 um filter paper at 80 °C.

Bleaching was performed by adding citric acid, water and bleaching earth adsorbent  
25 to the heat treated feedstock. Typically, for UCO, 800 mg/kg of citric acid (added as a 50 wt% aqueous solution) and 0.9 wt% of water was added to the oil, followed by mixing 8000 rpm at 85 °C for 2 min. Then, the bleaching earth (0.7 wt%) was added and 800 mbar pressure was applied and mixing was continued for 20 min. Next, the sample was dried by reducing the pressure to 80 mbar, increasing temperature to  
30 105 °C, and continuing mixing for 10 min at the elevated temperature. Finally, the reaction mixture was filtered through a precoat of bleaching earth at 105 °C at 350

kPa(a) pressure (nitrogen). For LQAF the procedure was the same but 3300 mg/kg of citric acid and 0.8 wt% of water, and 1.0 wt% bleaching earth was used.

The impurity levels of feeds and samples after solids removal, adjusting the elementary charge, and bleaching as well as bleaching filtration times are shown in

5 Table 1.

Table 1. P and main metals (Al, Ca, Fe, K, Mg, Na) levels after solids removal, adjusting the elementary charge, heat treatment and filtering or bleaching and bleaching filtration time for LQAF and UCO samples.

|              | Crude Feed |                      | Solids Removal Product |                      | Adjustment of elementary charge |                              | Filtered HT Product |                      | Bleached HT Product |                      |                                           |
|--------------|------------|----------------------|------------------------|----------------------|---------------------------------|------------------------------|---------------------|----------------------|---------------------|----------------------|-------------------------------------------|
| Feed         | P<br>mg/kg | Main Metals<br>mg/kg | P<br>mg/kg             | Main Metals<br>mg/kg | Added NaOH<br>mg/kg             | Qt after added Na<br>mmol/kg | P<br>mg/kg          | Main Metals<br>mg/kg | P<br>mg/kg          | Main Metals<br>mg/kg | Filtr. Time<br>min/<br>150 g<br>of sample |
| LQAF         | 403.3      | 756.7                | 257.5                  | 430.3                | 220                             | +1.0                         | 1.2                 | 230.2                | 1.5                 | 2.2                  | 10.0                                      |
| LQAF comp.   | 403.3      | 756.7                | 257.5                  | 430.3                | 0                               | -7.3                         | 5.7                 | 116.7                | 1.1                 | 0.8                  | 59.5                                      |
| UCO #1       | 14.9       | 46.7                 | 10.4                   | 29.6                 | 1200                            | +5                           | 0.8                 | 59.9                 | 1.0                 | 1.0                  | 9.0                                       |
| UCO #1 comp. | 14.9       | 46.7                 | 10.4                   | 29.6                 | 0                               | -0.3                         | 2.6                 | 9.7                  | 1.6                 | 1.3                  | 14.0                                      |
| UCO #2       | 5.5        | 36.1                 | 2.6                    | 11.9                 | 1090                            | +5                           | 0.6                 | 62.5                 | 0.7                 | 2.4                  | 5.5                                       |
| UCO #2       | 5.5        | 36.1                 | 2.6                    | 11.9                 | 0                               | +0.3                         | 1.3                 | 7.2                  | 1.0                 | 1.2                  | 5.5                                       |

10

It can be seen from the results in Table 1 that phase-separation by filtration after heat treatment results in good removal of the phosphorus impurities, however, for best metals removal a bleaching treatment is recommended.

When the feedstock entering the heating step had been conditioned so that the net  
15 elementary charge was on the positive side there was a clear improvement in the

filterability (shorter filtration time in the filtration step) of the acid and adsorbent treated heat-treated feedstock in the bleaching treatment.

Table 2 shows the different purification outcomes with different pretreatment protocols for LQAF. Bleaching protocol in all cases is the same as described earlier.

- 5 Table 2. Comparison of different pretreatment protocols on LQAF purification.

| Feed | Pretreatment                                                         | P /<br>mg/kg | Main Metals /<br>mg/kg | Bleaching Filtering<br>Time / min |
|------|----------------------------------------------------------------------|--------------|------------------------|-----------------------------------|
| LQAF | Crude Feed                                                           | 403.3        | 756.7                  | na                                |
| LQAF | Direct Bleaching (comp.)                                             | 45.4         | 19.8                   | 12                                |
| LQAF | Solids Removal and Bleaching (comp.)                                 | 22.7         | 7.0                    | 12                                |
| LQAF | Solids Removal, Adjusting net elementary charge,<br>HT and Bleaching | 1.5          | 2.2                    | 10                                |

- Table 2 presents a clear improvement in purification after bleaching when the LQAF feedstock had undergone adjustment of net elementary charge and heat treatment prior to bleaching.
- 10

Example 3. Effect of FFA in feed on filterability in solids removal step iv) after charge balancing with NaOH

- 1100 ppm charge balancing component NaOH (added as 50 wt% aqueous solution, ca. 630 ppm added Na) was added to feedstock samples containing different levels of FFA (after adjusting the FFA concentration). The net elementary charge of the feedstock samples were before adding charge balancing component -13 mmol/kg. NaOH addition was followed by mixing with a high shear mixer for 2 min 8000 rpm followed by mixing with a magnetic stirrer for 5 min 250 rpm at 70 °C. Filter aid (diatomaceous earth) 0.5 wt% was added to the sample and the mixture was held at 105 °C, 800 mbar pressure, for 20 min before filtering through a precoat of filter aid (1 kg/m<sup>2</sup>) at 105 °C with 1 bar pressure. The filtration time for filtering 150 g of the sample was recorded. The filtration times for the samples with different FFA
- 15
- 20

content are presented in Table 3. It was observed that the filtration ran smoothly when the FFA concentration of the feed was adjusted to 3 wt% FFA or higher and the charge balancing component was recovered in the filtrate (original Na concentration of blends 40 ppm, added Na ca. 630 ppm) and a conditioned  
5 feedstock was obtained (net elementary charge ca. +10 mmol/kg). The feedstock of 2 wt% FFA, however, had very poor filtration flux, and it was noticed that a major part of the charge balancing component was lost (shown as low Na in filtrate) resulting in a feedstock of elementary charge below 0 mmol/kg (-3.5 mmol/kg).

The blends were after the filter aid filtration treated by heat treatment followed by  
10 bleaching. The treatment of the blends with net elementary charge above 0 mmol/kg resulted in excellent impurity removal, whereas the blend where the charge balancing component was lost in filtration (blend of FFA 2 wt%) resulted in significantly lower removal of P and metals.

Table 3.

| FFA concentration in feedstock sample (wt%) | Filtration time (min/ 150 g sample) | Na in filtrate (mg/kg) | Final net elementary charge of filtrate (mmol/kg) |
|---------------------------------------------|-------------------------------------|------------------------|---------------------------------------------------|
| 2                                           | 131                                 | 260                    | -3.6                                              |
| 2 (repetition)                              | 126                                 | 230                    | -3.9                                              |
| 3                                           | ~1.5                                | 560                    | 9.8                                               |
| 4                                           | ~1.5                                | 590                    | 10.2                                              |
| 6                                           | <1                                  | 620                    | 11.4                                              |
| 10                                          | <1                                  | 590                    | 10.8                                              |
| 32                                          | ~1.5                                | 580                    | 10.3                                              |
| 57                                          | ~1.5                                | 610                    | 11.3                                              |

## CLAIMS

1. A method for purifying lipid feedstock (a), the method comprising
- i) providing the lipid feedstock (a);
  - ii) when the net elementary charge Q1 of the lipid feedstock (a) is below 0 mmol/kg, adjusting the net elementary charge of the lipid feedstock (a) to total net elementary charge Qt of at least 0 mmol/kg, such as from 0 to 10 mmol/kg, preferably at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg, by mixing the lipid feedstock (a) with a charge balancing component comprising, or consisting of, a metal compound;
  - iii) optionally adjusting the free fatty acid (FFA) concentration of the the lipid feedstock (a) to above 2 wt%, such as 2.5 to 40 wt%, preferably at least 3 wt%, such as from 3 to 30 wt%, more preferably to at least 4 wt%, such as from 4 to 20 wt%, even more preferably to 4 to 10 wt%, of the total weight of the resulting lipid feedstock; and
  - iv) removing solid impurities, and optionally excess water, from the lipid feedstock (a);
- to obtain a conditioned lipid feedstock (b) having total net elementary charge Qt within the values defined in step ii);
- v) heating the conditioned lipid feedstock (b), preferably in the presence of water, at a temperature from 180 to 325 °C to obtain a heat-treated lipid feedstock (c);
  - vi) optionally further treating the heat-treated lipid feedstock (c) with an acid and/or adsorbent material, preferably under bleaching conditions, to obtain a further treated lipid feedstock (d); and
  - vii) recovering the heat-treated lipid feedstock (c) and/or the further treated lipid feedstock (d) by phase-separation, preferably by filtration, to obtain a purified lipid feedstock (e).
2. The method as claimed in claim 1, wherein the lipid feedstock comprises at least 5 mg/kg, such as from 5 to 5000 mg/kg, preferably at least 15 mg/kg, such as 15 to 2000 mg/kg, dissolved phosphorus impurities, and at least 10 mg/kg, such as from

10 to 10000 mg/kg, preferably at least 20 mg/kg, such as 20 to 3000 mg/kg mg/kg, dissolved metal impurities, measured as elemental phosphorus and elemental metal(s).

3. The method as claimed in claim 1 or 2, wherein the lipid feedstock is selected  
5 from a group consisting of soapstock acid oil (SAO), low quality animal fat grades (LQAF), used cooking oil (UCO), palm oil mill effluent (POME), and mixtures thereof.

4. The method as claimed in any one of claims 1 to 3, wherein the lipid feedstock has net elementary charge below 0 mmol/kg, such as from -60 to -0.5 mmol/kg, in  
10 particular below -0.5 mmol/kg, such as from -30 to -1 mmol/kg.

5. The method as claimed in claim 4, wherein the method comprises the step of

i) providing the lipid feedstock (a) having net elementary charge below 0 mg, such as from -60 to -0.5 mmol/kg, in particular below -0.5 mmol/kg, such as from -30 to -1 mmol/kg;

15 ii) adjusting the total net elementary charge Q1 of the lipid feedstock (a) to total net elementary charge Qt of at least 0 mmol/kg, such as from 0 to 10 mmol/kg, preferably at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg, by mixing the lipid feedstock (a) with a charge balancing component comprising, or consisting of, a metal compound;

20 iii) optionally adjusting the free fatty acid (FFA) concentration of the the lipid feedstock (a) to above 2 wt%, such as 2.5 to 40 wt%, preferably at least 3 wt%, such as from 3 to 30 wt%, more preferably to at least 4 wt%, such as from 4 to 20 wt%, even more preferably to at least 5 wt%, such as from 5 to 10 wt%, of the total weight of the resulting lipid feedstock; and

25 iv) removing solid impurities, and optionally excess water, from the lipid feedstock (a);

to obtain a conditioned lipid feedstock (b) having a total net elementary charge Q1 within the values defined in step ii);

v) heating the conditioned lipid feedstock (b), preferably in the presence of water, at  
30 a temperature from 180 to 325 °C to obtain a heat-treated lipid feedstock (c);

- vi) optionally further treating the heat-treated lipid feedstock (c) with an acid and/or adsorbent material, preferably under bleaching conditions, to obtain a further treated lipid feedstock (d); and
- vii) recovering the heat-treated lipid feedstock (c) and/or the further treated lipid feedstock (d) by phase-separation to obtain a purified lipid feedstock (e).
- 5 6. The method as claimed in any one of claims 1 to 5, wherein the FFA concentration is adjusted to the predetermined level in step iii) before the lipid feedstock is subjected to removal of the solid impurities in step iv).
7. The method as claimed in any of claims 1 to 6, wherein the FFA concentration is  
10 adjusted by mixing the lipid feedstock with one or more free fatty acids and/or with a second further lipid composition comprising a suitably high amount of one or more free fatty acids, preferably with C<sub>13</sub>-C<sub>21</sub> free fatty acids, such as palmitic acid, stearic acid, oleic acid, and linoleic acid and/or with a lipid composition selected from soapstock acid oil (SAO), palm oil mill effluent (POME), brown grease (BG), and  
15 fatty acid distillate (FAD), and mixtures thereof.
8. The method as claimed in claim 7, wherein the second further lipid composition is selected from soapstock acid oil (SAO), palm oil mill effluent (POME), brown grease (BG), fatty acid distillate (FAD), and mixtures thereof.
9. The method as claimed in any of claims 1 to 8, wherein the charge balancing  
20 component is selected from a group consisting of metal hydroxide(s) and/or metal salt(s), preferably alkali metal hydroxide(s) and/or alkali metal salt(s), more preferably from NaOH or aqueous solution of NaOH.
10. The method as claimed in any of claims 1 to 8, wherein the charge balancing component is the same as the second further lipid composition.
- 25 11. The method as claimed in any of claims 1 to 10, wherein the net elementary charge of the conditioned lipid feedstock (b) is at least 0.5 mmol/kg, such as from 0.5 to 5 mmol/kg, more preferably at least 1 mmol/kg, such as 1 to 3 mmol/kg, determined as described in the description.

12. The method as claimed in any of claims 1 to 11, wherein removing solid impurities in step iv) is accomplished by settling, centrifuging, and/or filter aid filtration.
13. The method as claimed in any of claims 1 to 11, wherein removing solid  
5 impurities in step iv) is accomplished by filter aid filtration.
14. The method as claimed in any of claims 1 to 13, wherein vi) the heat-treated lipid feedstock (c) is treated with an acid and/or adsorbent material, preferably under bleaching conditions, to obtain a further treated lipid feedstock (d).
15. The method as claimed in claim 14, wherein step iv) is accomplished at  
10 temperature from 80 to 120 °C and at pressure from 60 kPa(a) to 600 kPa(a), preferably from 80 kPa(a) to 200 kPa(a).
16. A method for producing renewable hydrocarbons, comprising
- x) purifying a lipid feedstock (a) as claimed in any one of claim 1 to 15 to obtain purified lipid feedstock (d); and
- 15 y) subjecting the purified lipid feedstock (d) to hydroprocessing to obtain at least one renewable hydrocarbon.