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(54) Title: FLUID COMPOSITION COMPRISING LIGNIN AND VINASSE

(57) Abstract: The present invention relates to a fluid composition comprising a solid fraction and a liquid organic fraction, wherein said solid fraction and said liquid fraction are present in a state of being intermixed, wherein said solid fraction comprises a lignin component, wherein said liquid fraction comprises an organic substance. Furthermore, the present invention relates to a process for the manufacture of such fluid compositions, to various uses thereof, and to a process for treatment of a lignocellulosic biomass.



WO 2017/108055 A1

Fluid composition comprising lignin and vinasse

Field of the invention

The current invention relates to the field of industrial biotechnology, in particular to processes and methods related to the provision of bio-products, such as 2nd generation bioethanol from agricultural waste. In particular, methods, products and uses are disclosed relating to fluid compositions comprising a lignin component intermixed with a liquid organic fraction and vinasse (concentrated whole stillage or thin stillage). Furthermore, different uses of such fluid compositions are disclosed, including their use as fuel, as well as methods, processes and systems related to said fluid compositions are disclosed, including their manufacture.

Background of the invention

PCT/DK2015/050242, filed on 14 Aug 2015, said application being herewith enclosed in its entirety, discloses fluid compositions suitable as liquid fuel comprising (i) a lignin component, (ii) an organic fraction in liquid state at 25°C, and optionally (iii) water and/or a (iv) further agent. Surprisingly and unexpectedly, the inventors have discovered that vinasses, such as lignin-depleted or lignin-rich vinasses, said vinasses obtained by removal of water from a stillage can be used for such lignin-component comprising liquid fuels.

Without wanting to be bound by any theory, it is believed that using vinasses, e.g. substituting water and/or the organic fraction at least in part, provides a liquid fuel, which is e.g. (a) environmentally more friendly, and/or (b) possessing a higher heating value, e.g. higher lower heating value (LHV) compared to a similar fuel with water instead of vinasse.

Common process configurations for a 2nd generation bioethanol plant comprise a biogas plant in order to convert waste products such as thin stillage into biogas. In contrast, in the present invention, the concept of vinasse utilization as fuel is disclosed. In particular, lignin-rich or lignin-depleted vinasses are disclosed herein, said vinasses being provided by reducing the water content of e.g. whole or thin stillages, respectively.

A vinasse is believed to comprise the part of the biomass which is not converted into a desired product, such as bioethanol, and has not ended up as a solid biofuel; lignin. Its quality is influenced by the biomass composition as well as the upstream process performance. Different vinasses have been produced and tested as fuel, and
5 different vinasse-based fuel mixtures were tested in a single particle burner (see Examples below).

Surprisingly and unexpectedly, the overall conclusion is that all fuel mixtures were combustible, even when having elevated alkali content in the fuels. Different potassium concentrations were tested and no significant influence on the combustion
10 properties were identified in high K-content fuels. This is surprising, as potassium, often in the form of potassium acetate (acetic acid is e.g. produced during pre-treatment), is used as the active component in fire extinguishers.

Considerable interest has arisen in liquid fuel compositions derived from lignin. A variety of approaches have been reported whereby solid lignin is converted to liquid
15 fuel by pyrolysis or other thermochemical techniques. However, these technologies are capital intensive.

Some authors have previously reported a simple means for converting solid lignin to liquid fuel by including solid lignin particles as a component of a lignin-oil-water emulsion. See Posarac and Watkinson (2000); Thammachote, N. (2000);
20 US5,478,366 "Pumpable lignin fuel."

This simple lignin fuel compositions were never used commercially, in part because they suffered from peculiar viscosity characteristics. These suspensions were only pumpable, that is, able to be transported conveniently to a burner, under conditions of constant agitation. After agitation is discontinued, these suspensions quickly return to
25 a semi-solid, paste-like state. In actual commercial applications for liquid fuels of this sort, it is clearly disadvantageous that high shear mixing be maintained continuously in a fuel storage tank. This requires expensive equipment as well as an increased operating expense.

We have discovered that the problems encountered with this simple lignin
30 emulsion/suspension of the prior art can be solved by using a source of lignin which

has lignin ion exchange capacity less than 0.4 mol/kg, or 0.3 mol/kg. In that case, the viscosity of the suspension is generally reduced, such as to within a range that can be maintained in a storage tank using only gentle agitation, for example, with a recirculation pump.

- 5 Lignin is a complex phenolic polymer which forms an integral part of the secondary cell walls of various plants. It is believed that lignin is one of the most abundant organic polymers on earth, exceeded only by cellulose, and constituting from 25 to 33 % of the dry mass of wood and 20 to 25 % for annual crops.

10 Lignin serves as a strengthening support structure within the plant structure itself in that lignin fills the space in the cell wall between cellulose, hemicellulose and pectin components. Lignin is covalently linked to hemicellulose and therefore crosslinks different plant polysaccharides, conferring mechanical strength to the cell wall by extension in the plant as a whole.

Three major monolignols are involved as phytochemicals in the biosynthesis of lignin.
15 These three monolignols are coniferyl alcohol, sinapyl alcohol and *p*-coumaryl alcohol.

Lignin originating from different plants exhibit varying mutual contents of these three phytochemicals and accordingly, depending on plant species, lignin appears in nature in a high diversity of different structures.

20 Traditionally, lignin has been obtained and isolated as a byproduct in the paper manufacturing industry. Accordingly, in the Kraft process, wood chips are cooked in a pressurized digester in a strong alkaline liquid containing sulfide at 130 – 180 °C. Under these conditions lignin and hemicellulose degrade into fragments that are soluble in the alkaline liquid. The cellulose remains solid and is separated off for
25 further paper making processing, whereas the liquid containing the lignin fragments, denoted black liquor, is evaporated to a dry matter content of approximately 65 – 80%. This concentrated black liquor comprising lignin fragments is burned in order to recover chemicals, such as sodium hydroxide and inorganic sulfur compounds for reuse in the Kraft process and in order to utilize the heat value of the lignin fragments
30 contained in the black liquor.

Lignin is usually not isolated in the Kraft process, but the corresponding lignin fragments are burned in a wet state. However, if the alkaline black liquor is neutralized or acidified with acid, the lignin fragments will precipitate as a solid and may be isolated. A Kraft processing plant may have facilities for isolating the lignin fragments in this way.

Conveniently, the lignin fragments are isolated by solubilizing carbon dioxide, recovered elsewhere in the Kraft process, in the black liquor in order to neutralize/acidify the black liquor resulting in the precipitation of the lignin fragments.

The lignin fragments recovered from the Kraft process have strongly reduced molecular size, and a very high purity compared to the lignin located in the wood chips from which the lignin originates. This reduction of molecular size is due to the fact that the pressurized cooking in the alkaline liquid, takes place in presence of sulfide (S^{2-}) or bisulfide (HS^-) ions, which act as ether bond cleaving reagents, thus cleaving the ether bonds of the lignin and resulting in lignin fragments having strongly reduced sizes. The high purity is due to the fact that Kraft lignin and hemicellulose has been totally solubilized during the cooking process, whereby it has been completely separated from the cellulose fraction, and afterwards only lignin precipitates during acidification.

The energy content (HHV) of Kraft lignin is on average approximately 26 MJ/kg dry matter, slightly below that of coal, and hence lignin represents an interesting alternative source of energy.

Although lignin fragments may be recovered from the black liquor of the Kraft process and may be pelletized and used as a solid fuel for heating, such usage have not been very widespread.

One reason for this may be that solid fuel generally requires specially adapted equipment for loading, feeding and dosing to the boiler in which the pellets are to be burned. Furthermore, as for all kinds of solid fuel, such specially adapted equipment for loading, feeding and dosing of the lignin pellets usually are sensitive as to the pellet size and other fuel qualities, meaning that once a particular fuel having certain specifications has been selected, one cannot easily alter the kind of solid fuel to

another kind having another pellet size, thereby imposing restrictions on the flexibility of such solid fuels.

Accordingly, attempts have been made in converting solid lignin derivatives or fragments to liquid fuel formulations.

5 WO96/10067 A1 discloses a liquid fuel based on a lignin oil slurry. The lignin oil slurry of WO96/10067 is having specified rheological properties thus making it suitable for being poured or pumped. The lignin oil slurry comprises a lignin fragment in an amount of 35 – 60%; water in an amount of 35 – 60%, and oil in an amount of 0.5 – 30%. Furthermore, the slurry comprises a dispersion agent in an amount of at
10 least 50 ppm. The viscosity of the slurry after stirring is 100 – 1,000 mPa.s. According to the description of WO96/10067, the above stated limits of the amounts of the constituents are essential for the slurry to have the desired properties. The lignin fragment is in the description of WO96/10067 and its examples disclosed as being a lignin fragment originating from a Kraft process.

15 Another reason for the limited use of Kraft lignin based oil slurries is the limited storage stability of the dispersion related to both the sedimentation rate of the dispersion and risk of microbial contamination of the product .

US 2011/0239973 discloses a fuel mixture for a combustion engine. The fuel mixture comprises a combustible solid powder and a liquid fuel. The combustible solid
20 powder may be selected from the group comprising lignin, nitrification products of biomass and total biomass powder or any combination thereof. The liquid fuel may be selected from gasoline, kerosene, diesel, heavy oil, emulsified heavy oil, absolute ethanol or any combinations thereof.

US2011/0239973 mentions a range of various forms of lignin to be used in that
25 invention. However, the only lignin fragments exemplified in the working embodiments seems to originate from alkali lignin which according to US2011/0239973 is a lignin fragment obtained from black liquor in a paper making Kraft process.

Furthermore, the inventor of US2011/0239973 is concerned about avoiding having too large particle sizes of the lignin fragments, as these will clog the oil supply line of the engine in which the oil is to be used. According to US 2011/0239973 lignin particles will in the presence of moisture adhere to each other resulting in increasing particles sizes. For this reason, the lignin is subjected to a condensation stabilization treatment. Such treatment according to US2011/0239973 results in smaller particle sizes having increased mixing stability.

Although the above mentioned liquid formulations of fuels comprising lignin fragments solves the problems associated with the solid lignin fuel pellets, i.e. the requirement of specially adapted equipment for loading, feeding and dosing such a solid fuel, these liquid formulations still represent an unsatisfactorily solution especially due to lack of stability and high viscosity of the produced fuel.

Another source of a lignin component may be the biomass refining industry.

In the last decade much effort has been focused on the goal of making cellulose a source of renewable energy. The recent development of highly effective cellulases together with innovative improvements in the pretreatment processes necessary in order to make the cellulose accessible to the cellulase enzymes used in the process, has removed important obstacles in reaching this goal.

In the second generation bioethanol producing process, or the biomass refining process for short, a lignocellulosic biomass comprising cellulose, hemicellulose and lignin may be converted to ethanol. The process involves i) a hydrothermal pretreatment of the lignocellulosic biomass for making the cellulose accessible to catalysts in a subsequent step; followed by ii) a hydrolysis of the cellulose for breaking down the cellulose to soluble carbohydrates and finally iii) a fermentation of the soluble carbohydrates to ethanol.

A fiber fraction and a liquid phase are left behind after the hydrolysis has been performed.

The liquid phase obtained after the hydrolysis step comprises soluble carbohydrates useful for fermentation into ethanol. The remaining fraction obtained after the hydrolysis step comprises a lignin component.

The fiber fraction consist mainly of lignin, cellulose, hemicellulose and ash components. Compared to for example Kraft lignin, the lignin from the 2G bio refining industry is a more complex material, where the physio-chemical properties is only sporadically described. The lignin component may be rinsed, washed, filtered and/or pressed in order to obtain lignin in a more purified state. This will however only remove some of the soluble salts and the carbohydrates with short chain lengths. The rinsed, washed, filtered, dried and/or pressed lignin component obtained this way is usually pressed into pellets and used as a solid fuel.

Lignin fragments originating from a Kraft process are reported to contain only insignificant amount of cellulose and hemicellulose, but up to about 1.5 – 3.0 % sulfur; most being present as organically bound sulfur; however also inorganic sulfur compounds are present in the Kraft lignin fraction as well as minute amounts of elemental sulfur.

With the ever increasing worldwide focus on environmentally concerns, it is undesirably to burn Kraft lignin fragments having such a high sulfur content due to the risk of emission of gaseous sulfur compounds, unless the plant for burning such lignin fragments are equipped with a sulfur filtering device, such as a scrubber or the like.

However, such sulfur filtering devices are not present in most smaller sized heating facilities, such as in household oil burners, in district heating plants etc.

In addition to the merely ethical concerns relating to avoid high sulfur emission to the environment due to the burning of lignin fragments originating from a Kraft process, various governments in different states have provided legislation regulating the maximum allowable emission of sulfur compounds to the environment originating from burning fuel.

Such legislation obviously imposes restrictions to the use of Kraft lignin fragments as a fuel in smaller sized heating facilities not equipped with a sulfur filtering device.

There are several reasons that the liquid fuels disclosed in WO96/10067 and in US2011/0239973 are not fully satisfactory, one is that these fuels have a high sulfur
5 content. Furthermore, the compositions described in these publications tend to be more viscous than desired, thereby reducing the amount of lignin that it is possible to incorporate into the liquid fuel formulation, furthermore the storage stability of the dispersions is less than desired.

Furthermore, in the described fuel formulations in WO96/10067 it is only possible to
10 reduce the viscosity of the fuel by either increasing the amount of dispersant or by reducing the amount of lignin in the fuel.

Hence, there still exists a need for an improved liquid or fluid fuel formulation comprising lignin components which may allow being burned in a fuel burning device or plant not equipped with a sulfur filtering device, and which are having suitable
15 viscosities allowing them to be pumpable.

Furthermore, there is a need for an improved fluid fuel formulation, where lignin of lower purity than the Kraft lignin, can be used.

The above described disadvantages have been overcome with the present invention.

Furthermore, without wanting to be bound by any theory, by combining lignin and
20 vinasse in a fluid composition, where both usually being stillage-derived by-products from 2nd generation bioethanol production, and thus often provided in the same process, it is believed that the present invention provides advantages like: Improved conversion of energy, improved CO₂ footprint, reduced water consumption, more robust process, faster process time, reduced residence time, less waste, no digestate
25 from biogas production, shorter residence time in the waterloop, thus faster water re-use and re-circulation, reduced capital expenditures (CAPEX), reduced operating expenditures (OPEX), increased operational reliability, reduced down time, reduced overall energy demand, reduced external energy demand, increased productivity,

increased flexibility, and/or increased profitability, when compare to a set-up comprising a biogas plant.

Brief description of the invention

In a first aspect (A1), the present invention concerns a fluid compositions comprising
5 a lignin component, an organic fraction in liquid state at 25°C, a vinasse, such as a
vinasse according to any one of aspects B2, or provided e.g. according to aspect B1,
and optionally water and/or optionally a further agent. Such a fluid composition may
comprise (i) 5-60 % (w/w) of a lignin component, (ii) 0-95 % (w/w) of a liquid organic
fraction, (iii) 0-60 % (w/w) vinasse and/or water, and (iv) 0-5.0 or 0-10 % (w/w) of one
10 or more further agents.

In general, these components are intermixed, and the lignin component is different
from e.g. Kraft lignin, characterized e.g. by its lower assessed polarity and other
related features.

In a second aspect (A2), the present invention relates to processes related to the
15 fluid composition according to the first aspect (A1) of the invention, such as methods
and processes for the manufacture of a fluid composition according to the first aspect
(A1) of the present invention. Such a process according to the second aspect (A2)
may comprise the steps of: (i) providing a fraction, preferably a solid fraction
comprising a lignin component; (ii) providing an organic compound to make up at
20 least part of said liquid organic fraction; (iii) providing a vinasse according to e.g.
aspect B2; and (iv) intermixing the fraction provided in step (i) with the organic
compound and/or liquid organic fraction provided in step (ii) and/or vinasse provided
in step (iii). Often, the lignin component in step (i) is a “non-Kraft” lignin with
advantageous features as described herein, including lower LIEC, lower hygroscopy,
25 and/or lower swelling. A process according to the second aspect (A2) may comprise
one or more process steps according to e.g. aspect B1. In some embodiments, both
vinasse and lignin-component may be provided in the same process.

In a third aspect (A3), the present invention concerns to processes related to treatment of lignocellulosic biomasses, such as a process for treatment of a lignocellulosic biomass, said process comprising:

- a) subjecting said lignocellulosic biomass for hydrothermal pretreatment resulting
5 in a hydrothermally pretreated lignocellulosic biomass;
- b) subjecting at least part of said hydrothermally pretreated lignocellulosic biomass obtained in step (a) to a hydrolysis resulting in a liquid fraction comprising soluble carbohydrates, and a fiber fraction comprising a lignin component;
- 10 c) optionally subjecting at least part of the liquid fraction obtained in step (b) to a fermentation in order to ferment at least part of said soluble carbohydrates to a fermentation product, such as ethanol, methane or butanol, thereby obtaining a fermentation broth;
- d) optionally isolating at least part of said fermentation product from the
15 fermentation broth obtained in step (c) e.g. by distillation;
- e) isolating at least part of the lignin component from one or more of: the fiber fraction obtained in step (b); the fermentation broth obtained in step (c); or after isolation of at least a part of the fermentation product in step (d);
- f) converting at least part of the lignin component obtained in step (e) and
20 optionally a vinasse according to aspect B1 or B2 to a fluid composition, such as a fluid composition according to the first aspect (A1) of the invention, by admixing said lignin component with a liquid organic fraction comprising an organic compound or substance.

In some embodiments, both vinasse, often a lignin-depleted vinasse and the
25 lignin-component are provided in the same process.

In a fourth aspect (A4), the present invention relates to uses of a fluid composition according to the first aspect (A1) of the present invention, including a fluid composition provided according to the second (A2) and/or third aspect (A3) of the present invention. This includes uses of the fluid composition as fuel.

30 In a fifth aspect (A5), the present invention relate to the use of lignin or a solid lignin component for a fluid composition, such as a fluid composition according to the first

(A1), second (A2) or third aspect (A3) of the present invention. This includes also uses related to chemical processing of lignin and/or a lignin component or a conversion product thereof. The lignin and/or lignin component can e.g. be, or be provided as described in one of the other aspects of the invention (e.g. A2 and/or
5 A3). For vinasse-comprising fluid compositions, said vinasse can e.g. be provided according to the third aspect (A3) and/or aspect B1.

The current invention concerns also methods, products and uses of vinasses, and the following aspects B1-B5 relate to vinasses (see section B for more details).

In a first aspect (B1), the current invention concerns a method for providing a vinasse,
10 such as a lignin-rich and/or lignin-depleted vinasse, said method comprising the steps of:

- (i) pretreatment of lignocellulosic biomass;
- (ii) hydrolysis of at least a portion of the pretreated lignocellulosic biomass from step (i) to provide one or more products;
- 15 (iii) fermentation of the one or more products from step (ii) to provide a fermentation product;
- (iv) separation/removal of the one or more products from step (ii), and/or the fermentation product from step (iii) to provide a whole stillage;
- (v) solid/liquid separation, whereby a lignin-rich fraction ("lignin") and an
20 aqueous thin stillage are provided;
- (vi) removal of water from the lignin-rich fraction; and
- (vii) removal of water from the whole stillage of step (iv), or from the thin stillage of step (v), to provide said lignin-rich vinasse or said lignin-depleted vinasse;

25 wherein any one of steps (iii), (v) and/or step (vi) are optional; and wherein the lignin-rich vinasse has a dry matter content (dm) of at least 30% (w/w), and the lignin-depleted vinasse has a dm of at least 40% (w/w).

In a second aspect (B2), the current invention relates to a vinasse provided, defined or characterized according to e.g. the first aspect (B1) of the invention. Said vinasse can
30 e.g. be a lignin-rich or a lignin-depleted vinasse.

In a third aspect (B3), the present invention pertains to a fuel comprising a vinasse, such as a vinasse according to the second aspect (B2) of the invention.

In a fourth aspect (B4), the current invention concerns the use of a vinasse - such as a lignin-rich- and/or lignin-depleted vinasse according to the second aspect (B2) of the invention - as a fuel, such as a fuel according to the third aspect (B3) of the invention.

In a fifth aspect (B5), the current invention relates to a system comprising means for providing and/or using a vinasse according to the first (B1) or second aspect (B2) of the invention, and optionally means for converting said vinasse to energy, such as a boiler and/or a combined heat and power plant CHP, adapted for combusting a fuel according to the third (B3) or fourth aspect (B4) of the invention. In particular, embodiments relating to systems or set-ups are disclosed, without anaerobic digestion for biogas production from whole or thin stillage. Furthermore, in some embodiments, said system comprises means relating to e.g. the second (A2), third (A3) or fourth (A4) aspect of the invention.

Brief descriptions of the drawings

Figure 1a shows the sum of ignition delay and ignition time (left) and pyrolysis time (right) of samples at the conditions: 1200 °C, 5.5 % O₂ (dry) and a flue gas velocity of 1.6 m/s. The full drawn lines are linear trendlines of the datasets.

Figure 1b shows the sum of ignition delay and ignition time (left) and pyrolysis time (right) of samples at the conditions: 1200 °C, 2.9 % O₂ (dry) and a flue gas velocity of 1.6 m/s. The full drawn lines are linear trendlines of the datasets.

Figure 1c shows the sum of ignition delay and ignition time (left) and pyrolysis time (right) of samples at the conditions: 990 °C, 5.5 % O₂ (dry) and a flue gas velocity of 1.6 m/s. The full drawn lines are linear trendlines of the datasets.

Figure 9-1: Lignin filter cake; before (left) and after being cut up with the Kenwood machine (right)

Figure 9-2: Schematic of nonylphenol ethoxylate.

Figure 10-1: Viscosity of Lignomulsion (LOW without any additives) at different shear rates and constant temperature (a) and different temperature and shear rates 100 s⁻¹ (b).

Figure 10-2: Viscosity of Lignomulsion (LOW with 5000 ppm Lutensol AP10 and 5000 ppm Sodium benzoate) different temperature and shear rate 100 s^{-1} . The oil content in the formulations is either 0% (a), 10% (b), 20% (c) or 30% (d).

Figure 11-1: Effect of sodium benzoate and Lutensol AP10 on viscosity; measured as a function of shear rate at room temperature.

Figure 11-2: Effect of sodium benzoate and Lutensol AP10 on viscosity; measured as a function of temperature at shear rate 100 s^{-1} .

Figure 11-3: Viscosity of LOW 40-20-40 formulations with the different addition; either with 5000 ppm Lutensol AP10 with a hydrotrope (a), or with 5000 ppm sodium benzoate with a surfactant (b).

Figure 12-1: Paraben structure

Figure 12-1a-b Viscosity as a function of shear rate at room temperature (a) and as a function of temperature at shear rate 100 s^{-1} (b) for emulsions with composition LOW 40-20-40 and 0.5% Lutensol AP10 and various amounts of methylparaben, propylparaben and sodium benzoate.

Figure 13-1: Viscosity of LOW formulations using different oil types, measured at shear rate 100 s^{-1} and at four different temperatures. The formulations are LOW 40-20-40 (a) and LOW 40-30-30 (b), in both cases with 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10.

Figure 13-2: Viscosity of different LOW formulations using unrefined palm oil as a function of shear rate at room temperature (a) and temperature at shear rate 100 s^{-1} (b). The formulations all contain 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10.

Figure 14-1: Viscosity of Lignomulsion with and without heavy fuel oil as a function of shear rate.

Figure 14-2: Viscosity of lignomulsions with and without heavy fuel oil as a function of temperature.

Figure 14-3: Viscosity of emulsions with various diesel:fuel oil ratios as a function of shear rate.

Figure 15-1: Viscosity of Lignomulsion formulations without oil or additives; shown as a function of shear rate measured at room temperature (a); and of temperature measured at shear rate 100 s^{-1} (b).

Figure 15-2: Viscosity of Lignomulsion formulations with 10% diesel oil and without additives; shown as a function of shear rate measured at room temperature (a); and of temperature measured at shear rate 100 s^{-1} (b).

Figure 15-3: Viscosity of Lignomulsion formulations with 10% diesel oil and Lutensol AP10 and sodium benzoate; shown as a function of shear rate measured at room temperature (a); and of temperature measured at shear rate 100 s^{-1} (b).

Figure 15-4: Viscosity of Lignomulsion formulations with different oil content; shown as a function of shear rate measured at room temperature (a); and of temperature measured at shear rate 100 s^{-1} (b).

Figure 15-5: Viscosity of different Lignomulsion formulations with and without additives; shown as a function of shear rate measured at room temperature (a and c); and of temperature measured at shear rate 100 s^{-1} (b and d).

Figure 16-1: Viscosities of different fomulations shown as a function of shear rate measured at room temperature.

Figure 17-1: Lignomulsion (prepared with Indulin, sample ID 140528_001) less than ten minutes after homogenization.

Figure 17-2: Viscosity at room temperature and shear rate 100 s^{-1} as a function of time (for three different formulations).

Figure 18-1: Viscosity of LOW emulsions prepared from lignin filtercake, dried to 99% DM; shown as a function of shear rate measured at room temperature (a) or as a function of temperature measured at shear rate 100 s^{-1} (b).

Figure 18-2: Viscosity of LOW emulsions prepared from lignin filtercake, dried at 50°C ; shown as a function of shear rate measured at room temperature (a) or as a function of temperature measured at shear rate 100 s^{-1} (b).

Figure 19-1: Viscosity as a function of shear rate; first four runs at 85°C for emulsions with the same formulation prepared either at room temperature or at 85°C .

Figure 19-2: Viscosity as a function of shear rate for emulsions with the same formulation prepared either at room temperature or at 85°C .

Figure 19-3: Viscosity as a function of shear rate at different temperatures of LOW 30-00-70 Lignomulsion before (a) and after (b) treatment in the Parr reactor

Figure 20-1: Viscosity of LOW formulations as a function of shear rate at room temperature (a) and as a function of temperature at shear rate 100 s^{-1} (b).

Figure 21-1: Viscosity of LOW formulations as a function of the Indulin fraction

Figure 21-2: Viscosity measured at shear rate 100 s^{-1} and 25°C as a function of klason lignin (a) and the sum of glucan and xylan (b).

Figure 21-3: Correlation between the content of sugar and klason lignin in 12 lignin samples

- 5 **Figure 21-4:** Viscosity at room temperature and shear rate 100 s^{-1} ; divided in to group based on pretreatment severity (a) and hydrolysis/fermentation conditions (b)

- 10 **Figure 21-5:** Viscosity at room temperature and shear rate 100 s^{-1} of 12 lignin samples shown as a function of klason lignin content in the lignin sample. As the lignin content in the formulation was 30%, the mass contribution of klason lignin to the entire formulation is actually in the range 12-21% in the LOW 30-20-50 formulations (a) and 16-28% in the LOW 40-10-50 formulations (b).

Figure 22-1 Viscosity of Lignomulsion prepared with untreated, acid treated or base treated lignin pellets (grinded).

- 15 **Figure 23-1:** Energy consumption of ultra turrax as a function of duration a constant speed of 10000 rpm (a) and as a function of speed at constant durations of 0.5 min and 10 min, respectively (b).

Figure 23-2: Emulsion viscosity as a function of energy consumed by the UT at constant speed (10.000 rpm) and duration from 0.5 to 10 min.

- 20 **Figure 23-3:** Emulsion viscosity as a function of energy consumed by the UT at constant duration; 0.5 min (a) and 10 min (b). The UT duration varied from 3500 to 20000 rpm.

Figure 23-4: Contour plot of viscosity (Pa s) as a function of speed ("stirring" / rpm) and duration/time (min).

- 25 **Figure 24-1:** Viscosity of Lignomulsion samples before (1st run) and after (2nd run) storage.

Figure 25-1 Size distributions of emulsions containing ISK lignin, prepared directly from filter cake with ultra turrax (exp. 1-6, see table 25-1).

Figure 25-2: Size distribution of lignin, milled from dry lignin pellets and separated with different sieves.

- 30 **Figure 26-1:** System for injecting lignomulsion into combustion chamber

Figure 26-2: Modified injection system where the fuel (lignomulsion) is mixed with pressurized air before the nozzle (full cone).

Figure 26-3: Viscosity at shear rate 100 s^{-1} .

Figure 31: Example of a 2nd generation bioethanol process with biogas and lignin production, combined with heat and power plant (CHP). Related configurations may comprise a single or a multi hydrolysis step with or without “C5 bypass”, or a use of the C5-rich e.g. as animal feed. The C5 bypass can also be fed directly to the fermentation step, without a hydrolysis. The pre-treatment may comprise the addition of an acid or base, or not (“auto hydrolysis”).^[A1] **Figure 31B** shows a less complex process.

Figure 32: Example of a 2nd generation bioethanol process, e.g. as disclosed in Figure 31, with provision of lignin-depleted vinasse and lignin, where both lignin-depleted vinasse and lignin are combusted in e.g. a combined heat and power plant (CHP). The CHP can also be replaced by boiler for steam production only. **Figure 32B** shows a less complex process.

Figure 33: Example of a 2nd generation bioethanol process, e.g. as disclosed in Figure 31 or 32, with provision of lignin-depleted vinasse and lignin. The lignin-depleted vinasse can e.g. be combusted directly or co-combusted with oil in e.g. a CHP. The CHP can also be replaced by boiler for steam production only. Lignin can e.g. be dried and used as biofuel or other applications. **Figure 33B** shows a less complex process

Figure 34: Example of a 2nd generation bioethanol process, e.g. as disclosed in Figure 32 or 33 with provision of lignin-depleted vinasse and lignin. In contrast to the set-up as disclosed in Figure 2, a surplus of lignin is provided, which can be used as biofuel or other applications. The CHP can also be replaced by boiler for steam production only. **Figure 34B** shows a less complex process

Figure 5: Example of a 2nd generation bioethanol process, e.g. as disclosed in Figure 32, with a boiler instead of a CHP for combusting lignin-depleted vinasse. The lignin can e.g. used as biofuel or other applications. Other set-ups (not depicted here) are also possible, where the lignin and lignin-depleted vinasse are combusted in a boiler, or some lignin and all lignin-depleted vinasse are combusted in a boiler, thus providing an excess of lignin for use as biofuel or other applications. **Figure 35B** shows a less complex process.

Figure 36: Example of a 2nd generation bioethanol process with provision of lignin-rich vinasse, which is combusted in a CHP or a boiler (not depicted here). The lignin-rich vinasse can be combusted alone, or co-combusted with another suitable fuel.

Figure 36B shows a less complex process.

5 **Figure 37:** Mass loss of samples during heating in N₂.

Figure 38: Mass loss of samples during heating in O₂.

Figure 39: Experimental single particle and droplet combustion setup with high speed camera, sample holder and gas extraction.

Figure 40: Ignition delay for various samples in the single particle burner.

10 **Figure 41:** Pyrolysis for various samples in the single particle burner.

Detailed description of the invention

Definitions and terminologies used herein may refer to SECTION A and/or B. If conflicting, definitions/terms presented in Sections A may refer only to section A, and definitions/terms presented in Sections B may refer only to section B.

15 **SECTION A (Aspects A1-A5)**

The lignin component obtained in the biomass refining process is quite different from the lignin fragments obtained in the Kraft process. First, in the Kraft process, the lignin polymer obtained from the lignocellulosic biomass has been subjected to alkaline liquid containing ether bond cleaving inorganic sulfur species resulting in
20 cleavage of a high number of the ether bonds originally present in the lignin molecules with the consequence that the macromolecular original lignin molecules are cleaved into a larger number of smaller lignin fragments. Secondly, these smaller lignin fragments are solubilized and dissolved in the alkaline liquid in the used in the Kraft process and only re-precipitated upon acidifying the black liquor. Hence, the
25 lignin fragments obtained from a Kraft process comprise relatively small lignin species which have been dissolved in the black liquor and which have a relatively high purity, but also a high sulfur content.

In contrast, it is believed that a lignin component obtained from the biorefining of a lignocellulosic biomass has not been processed in a way that makes it dissolve and/or improve its solubility. Furthermore, such a lignin component has not been subjected to a treatment involving ether bond cleaving reagents which will result in a moderately high increase in sulfur content. Hence, a lignin component obtained from biorefining of a lignocellulosic biomass comprises undissolved lignin, residual hemicellulose, cellulose and ash components. Furthermore the lignin molecules are relatively large and have a relatively low sulfur content.

The present invention according to its various aspects (A1-A5, B1-B5) represents great advantages compared to the prior art.

First aspect (A1)

First of all, the product of the first aspect (A1) is a fluid or a liquid which means that the problems associated with lignin pellets used for fuel as described above is eliminated with the present invention.

Secondly, the fluid composition according to the first aspect (A1) of the present invention is advantageous in that it contains a relatively low content of sulfur which makes it acceptable for use as a fuel in heat producing plants not provided with a sulfur filtering device for its effluent gases.

Thirdly, the fluid composition according to the first aspect (A1) of the present invention is advantageous in that it can be produced from low purity lignin components.

Fourthly, the stability of the composition according to the first aspect (A1) of the present invention is improved due to the hydrotropic action of the cellulose and hemicellulose.

Fifthly, the viscosity of the composition according to the first aspect (A1) of the present invention can be reduced by adding hydrotropic compounds.

In addition, the idea of converting a solid fuel, such as a lignin component, to a liquid fuel is that the handling, storage and transport of liquid fuels are much more convenient, compared to solid fuels. However, in handling storing and transportation

of liquid fuels also the viscosity of the liquid plays an important role. A fuel having a low viscosity is much easier to handle than a fuel having a high viscosity.

When making fluid compositions, such as dispersions of lignin in various organic substances, the trend has been observed that the higher the concentration of lignin, the higher the viscosity of the resulting dispersion. There has hitherto been no possibility of lowering the viscosity. Unfortunately it has been found that this lowest obtainable limit of viscosity in many liquid formulations of Kraft lignin fragments is undesirably high.

In contrast, when using a lignin obtained from a biorefinery process of a lignocellulosic biomass, involving hydrothermal pretreatment of the biomass followed by a hydrolysis of the biomass, for making a dispersion of a lignin component and an organic substance, much lower viscosities of the dispersion may be encountered. Indeed this is also what the present inventors have found with the fluid composition according to the first aspect (A1) of the present invention.

Hence, a wider range of flexibility relating to viscosity of a fluid composition comprising a lignin component may be obtained with fluid composition according to the invention according to the first aspect (A1) of the present invention, because starting with a lignin component obtained from a biorefinery process of a lignocellulosic biomass, involving hydrothermal pretreatment of the biomass followed by a hydrolysis of the biomass, for making a dispersion of a lignin component and an organic substance, the initial viscosity will be lower than if Kraft lignin has been used.

Moreover, the lignin component obtained from a biorefinery process of a lignocellulosic biomass, involving hydrothermal pretreatment of the biomass followed by a hydrolysis of the biomass, when used for a fluid composition according to the first aspect (A1) of the present invention need not be dried prior to the admixing with the organic substance of the liquid fraction of the fluid, and the lignin component may even be intermixed with this organic substance of the liquid fraction of the fluid in a wet state while still resulting in a stable fluid composition, such as a stable dispersion.

Furthermore, it has surprisingly been found that when using a lignin obtained from a biorefinery process of a lignocellulosic biomass, involving hydrothermal pretreatment of the biomass followed by a hydrolysis of the biomass, for making a dispersion of a lignin component and an organic substance, stable fluids, a more stable dispersion
5 may be obtained due to the content of residual cellulose and hemicellulose even if no dispersion agent is used. Such avoidance of dispersion agents contributes huge savings in the production cost of the fluid composition, not least in case the fluid composition is going to be used as a fuel, where the demand of that specific fuel may be in the order of thousands of metric tons per year.

10 Furthermore, it has surprisingly been found that when using a lignin obtained from a biorefinery process of a lignocellulosic biomass, the viscosity of the fluid composition can be reduced by adding hydrotropes to the composition, this will result in significant cost savings as the lignin content can be increased and still be able to pump the resulting fluid composition.

15 Furthermore, it has surprisingly been found that when using a lignin obtained from a biorefinery process of a lignocellulosic biomass, involving hydrothermal pretreatment of the biomass followed by a hydrolysis of the biomass, for making a dispersion of a lignin component and an organic substance, stable fluids, a more stable dispersion may be obtained even if no dispersion agent is used. This is due to the low tendency
20 of such lignin components to interact with water. This tendency is quantified by the Lignin Ion Exchange Capacity (LIEC), describing as the amount of polar groups per lignin weight unit. Such avoidance of dispersion agents contributes huge savings in the production cost of the fluid composition, not least in case the fluid composition is going to be used as a fuel, where the demand of that specific fuel may be in the order
25 of thousands of metric tons per year.

The above advantages are quite surprising and could not have been foreseen by a person skilled in the art.

In the context of the present invention, the term "fluid composition" as used in the present description and in the appended claims shall be understood to mean a
30 composition which is fluid or liquid in the sense that it exhibits viscosities at various

temperatures falling within ranges as herein, in particular at room temperature, e.g. at 20 or 25°C.

A “liquid” is meant to comprise a near-incompressible fluid that conforms to the shape of its container but retains a (nearly) constant volume independent of pressure at room temperature, e.g. at 20 or 25°C. Being liquid is one of the four fundamental states of matter other than being solid, gas, or plasma.

Thus, as mentioned above, in a first aspect (A1) of the present invention relates to a fluid composition comprising: a solid fraction and a liquid fraction; wherein said solid fraction and said liquid fraction are present in a state of being intermixed; wherein said solid fraction comprises a lignin component; and wherein said liquid fraction comprises an organic substance.

The fluid composition of the first aspect (A1) of the present invention comprises a solid fraction and a liquid fraction.

The solid fraction of the fluid composition of the first aspect (A1) in turn comprises a lignin component often also containing some cellulose and/or hemicellulose.

Whereas the term “lignin” in the present description and in the appended claims refers to the polymer denoted as such and being present in unprocessed lignocellulosic plant material, the term “lignin component” in the present description and in the appended claims has a broader meaning. The term “lignin component” shall the in the present description and in the appended claims be understood to mean a “lignin” that has been subject to various physical and/or chemical treatments imposing minor changes of the lignin polymer structure, however still retaining its polymer character and containing significant amounts of hemicellulose and cellulose.

Hence a “lignin component” as used in the present description and in the appended claims may refer to a lignin that has been subjected to slight structural modifications.

Also a “lignin component” as used in the present description and in the appended claims may refer to a lignin that has been subjected to slight structural modifications and/or comprising an amount of chemical residues originating from its mode of

manufacture, or originating from compounds native for the lignocellulosic material from which it is isolated.

In some embodiments of the various aspects of the present invention a “lignin component” may specifically exclude a Kraft lignin or a Kraft lignin fragment obtained
5 from a Kraft processing of a lignocellulosic biomass.

In some embodiments of the various aspects of the present invention a “lignin component” may specifically exclude a lignosulfonate.

In some embodiments of the various aspects of the present invention a “lignin component” may specifically exclude a soda lignin.

10 In the context of the present invention, the “lignin component” is meant to comprise a by-product from 2nd generation (2G) bioethanol production. There are various different 2nd generation bio-ethanol processes known in the art that may provide such a lignin component, incl. organosolv processes. Schemes for processing lignocellulosic biomass, including specific process steps as well as overall schemes
15 for converting a lignocellulosic biomass to soluble saccharides and a fibrous fraction being or comprising the lignin component, are the subject of numerous published patents and patent applications. See e.g. WO 94/03646; WO 94/29474; WO 2006/007691; US2007/0031918; WO 2008/112291; WO 2008/137639; EP 2 006 354; US 2009/0326286; US 2009/0325251; WO 2009/059149; US 2009/0053770;
20 EP 2 169 074; WO 2009/102256; US 2010/0065128; US 2010/0041119; WO 2010/060050; WO2007009463 A2, WO2007009463 A1; WO2011125056 A1; and WO2009125292 A2, each of which is hereby incorporated by reference in entirety.

According to the first aspect (A1) of the present invention, the solid fraction and the liquid fraction of the fluid composition are present in a state of being intermixed. The
25 term “present in a state of being intermixed” shall in the present description and in the appended claims be understood to mean that the solid fraction and the liquid fraction of the fluid composition have been subjected to some kind of mechanical action which have brought them into an intermixed state.

Hence, in this way, the solid fraction and the liquid fraction of the fluid composition of the first aspect (A1) of the present invention may be present in a state in which the solid fraction is approximately evenly distributed in the liquid fraction of the fluid composition.

- 5 The liquid fraction of the fluid composition according to the first aspect (A1) of the present invention comprises an organic substance.

The term "organic substance" shall in the present description and the amended claims be understood to be any substance which in said liquid fraction upholds a liquid character, meaning that said organic substance at various temperature exhibit
10 viscosities as defined below.

In some embodiments of the various aspects of the present invention, the term "organic substance" shall mean a substance which comprises one or more carbon atoms, wherein at least one of said one or more carbon atoms is bonded to adjacent atoms by forming covalent bonds.

- 15 In some embodiments of the various aspects of the present invention, the organic substance preferably itself is an organic substance which is capable of participating in an exothermal reaction with oxygen, such as an oil or a fuel.

The some embodiments, the invention pertains to a fluid composition comprising a lignin component, an organic fraction in liquid state at 25°C, and a vinasse, such as a
20 vinasse provided according to the aspect B2, and optionally water and/or a further agent. Such a fluid composition may comprise, contain, consist, or consist essentially of: (i) a lignin component ("L"), such as 5-60 % (w/w); (ii) a liquid organic fraction ("O"), such as 0-60 % (w/w), said liquid organic fraction being in a liquid state at room temperature, such as 25 oC; and optionally (iii) vinasse ("W") in the range of 0-
25 95 % (w/w), and optionally (iv) a further agent ("A") and/or water. In some embodiments, said further agent is present in a concentration of 1%, or below. In some embodiments, the further agent is present in the range of 0-0.5 % (w/w). In many, but not alle some embodiments of the first aspect (A1) of the present invention, said liquid fraction of the fluid composition furthermore comprises water.
30 The vinasse can either be a lignin-rich or a lignin-depleted vinasse.

Usually the composition comprises, contains, consists, consists essentially of a fluid composition with a lignin or lignin component, liquid organic fraction, and vinasse content ("L-O-W", expressed as % - % - % (w/w)) of, or around:

- 5 5-0-95, 5-5-90, 5-10-85, 5-15-80, 5-20-75, 5-25-70, 5-30-65, 5-35-60, 10-0-90, 10-5-85, 10-10-80, 10-15-75, 10-20-70, 10-25-65, 10-30-60, 15-0-85, 15-5-80, 15-10-75, 15-15-70, 15-20-65, 15-25-60, 15-30-55, 20-0-80, 20-5-75, 20-10-70, 20-15-65, 20-20-60, 20-25-55, 20-30-50, 25-0-75, 25-5-70, 25-10-65, 25-15-60, 25-20-55, 25-25-50, 25-30-45,
- 10 30-0-70, 30-5-65, 30-10-60, 30-15-55, 30-20-50, 30-25-45, 30-30-40, 35-0-65, 35-5-60, 35-10-55, 35-15-50, 35-20-45, 35-25-40, 35-30-35, 40-0-60, 40-5-55, 40-10-50, 40-15-45, 40-20-40, 40-25-35, 40-30-30, 45-0-55, 45-5-50, 45-10-45, 45-15-40, 45-20-35, 45-25-30, 45-30-25, 50-0-50, 50-5-45, 50-10-40, 50-15-35, 50-20-30, 50-25-25, 50-30-20,
- 15 55-0-45, 55-5-40, 55-10-35, 55-15-30, 55-20-25, 55-25-20, 55-30-15, 60-0-40, 60-5-35, 60-10-30, 60-15-25, 60-20-20, 60-25-15, 60-30-10, 10-35-55, 15-35-50, 20-35-45, 25-35-40, 30-35-35, 35-35-30, 40-35-25, 45-35-20, 50-35-15, 55-35-10, 60-35-5, 5-40-55, 10-40-50, 15-40-45, 20-40-40, 25-40-35, 30-40-30, 35-40-25, 40-40-20, 45-
- 20 40-15, 50-40-10, 55-40-5, 59.9-40-0.1, 5-45-50, 10-45-45, 15-45-40, 20-45-35, 25-45-30, 30-45-25, 35-45-20, 40-45-15, 45-45-10, 50-45-5, 54.9-45-0.1, 5-50-45, 10-50-40, 15-50-35, 20-50-30, 25-50-25, 30-50-20, 35-50-15, 40-50-10, 45-50-5, 49.9-50-0.1,
- 25 5-55-40, 10-55-35, 15-55-30, 20-55-25, 25-55-20, 30-55-15, 35-55-10, 40-55-5, 44.9-55-0.1, 5-60-35, 10-60-30, 15-60-25, 20-60-20, 25-60-15, 30-60-10, 35-60-5, or 39.9-60-0.1 % (w/w), apart from minor amounts of e.g. one or more further agent (usually not more than around 1.0 % or less than 1.0 %, such as around 0.5% or less, i.e. in the
- 30 ppm ranges of around 0-1.000 ppm, or 0-5.000, or (per weight).

Thus, in some embodiment of the fluid composition, the lignin component is, or is around 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w).

5 In some embodiments, the liquid organic fraction is, or is around 0, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w).

In some embodiments, the lignin-rich or lignin-depleted vinasse content of the fluid composition is, or is around 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w). The vinasse content can also be lower, present only in minor amounts - e.g. present
10 in the lignin component and/or liquid organic fraction.

In some embodiments, the water content of the fluid composition is, or is around 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w). The water content can also be lower, present only in minor amounts - e.g. present in the lignin component and/or
15 liquid organic fraction, or be absent.

In some embodiments, the fluid composition according to the invention may comprise a further agent, such as or around 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, 0.50 or more, such as or around 0.55, 0.60, 0.65, 0.70, 0.75, 0.8, 0.85, 0.90, 0.95, or 1.0 % (w/w). Usually, the further agent is not present in an amount of more than 1%
20 (w/w), preferably not more than around 0.5 % (w/w). It has surprisingly been found that addition of further agents, such as hydrotropes can reduce the viscosity of fluid compositions containing lignin components e.g. from biomass refining plants, allowing higher amounts of lignin to be added to the fluid composition.

In the context of the present invention, the terms "about" and "around" can be used
25 interchangeably, and mean variations generally accepted in the field, e.g. comprising analytical errors and the like. Commonly, "around" means variations of +/- 1, 2, or 2.5% (w/w) based on the total composition, or on a specific compound, such as when relating to e.g. one or more further agents. In this case, as this further agent is present in lower amounts, "around" can also mean +/- 0.1, 0.2, or 0.25% (w/w) based
30 on the total composition or the respective agent.

Thus in summary, the first aspect (A1) of the invention concerns a fluid composition comprising a lignin component, an organic fraction in liquid state at 25°C, a vinasse and optionally water and/or a further agent.

- 5 In particular, surprisingly and unexpectedly, the inventors have realized that the source of the lignin component has an influence on the quality of the fluid composition. In particular, a less polar lignin appears more suitable, such as a fluid composition, wherein said lignin component is not lignin from paper and pulp production, such as Kraft lignin, wherein said Kraft lignin being provided from
10 biomass by a process known in the art as Kraft process/method (see e.g. Biermann, Christopher J. (1993) "Essentials of Pulping and Papermaking" San Diego: Academic Press, Inc.).

- Without wanting to be bound by any theory, it is believed that an alkaline treatment
15 has a negative effect on the lignin quality for uses related to the present invention, thus in some embodiments, said lignin component has not been provided by a Kraft method or another method comprising an alkaline treatment, such as by addition of NaOH or another base to provide a pH of around 10 or higher, at or around pH 11 or higher, or at or around pH 12 or higher.

- 20 Furthermore, it is believed that further modifications of the lignin or lignin component are not necessary to obtain a fluid composition according to the present invention, thus some embodiments concern desired a lignin component has not been esterified and/or subjected to an esterification step, e.g. as disclosed in WO2015/094098. It is
25 believed to be an advantage that no further steps are needed, such as said modification of the lignin.

- It appears a complex, if not to say an impossible task to measure polarity of a complex composition such as lignin. However, the inventors have developed a
30 method to assess polarity, based on lignin's ion exchange capacity (LIEC; see e.g. Experimental section for further details). It became apparent that Kraft lignin has a significantly higher LIEC as e.g. 2G lignin that has not been subjected to an alkaline

treatment. It is further believed that any wood, e.g. poplar wood and/or any other wood that e.g. is suitable for the paper industry, if processed without alkaline treatment, such as in a 2G process aiming at bioethanol production, will result in a lignin that is less polar, and thus suitable for providing a fluid composition according to the present invention.

In some embodiments, a fluid composition is provided comprising two or more fractions, wherein (a) the first fraction is an organic fraction in liquid state comprising one or more organic compounds such as one or more fat, and/or one or more oil; and (b) the second fraction comprises a lignin component having a Lignin Ion Exchange Capacity (LIEC) of 0.4 mol/kg dry matter or less. In further embodiments, said lignin component has a Lignin Ion Exchange Capacity (LIEC) of 0.3 mol/kg dry matter (DM) or less, such as 0.25 mol/kg DM or less, such as 0.20 mol/kg DM or less, such as 0.15 mol/kg DM or less, or such as 0.10 mol/kg DM or less. In some embodiments, the LIEC can be in the range of 0.05-0.30, 0.10-0.25, or 0.10-0.15 mol/kg DM. In further embodiments, the lignin component is significantly less polar than Kraft lignin, such as assessed by LIEC measurement, such as having a LIEC at least 0.10, 0.11, 0.12, 0.13, 0.14, 0.15, 0.16, or 0.17 mol/kg DM lower than the LIEC of Kraft lignin.

Without wanting to be bound by any theory, it is believed that it is an advantage that the lignin component according to the invention is less hygroscopic than e.g. Kraft lignin. Thus according to some embodiments, a fluid composition is provided, wherein said lignin component is significantly less hygroscopic, such as binding at least 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 or 100 % (w/w) less water when compared to Kraft lignin.

Without wanting to be bound by any theory, it is believed that it is an advantage that the lignin component according to the invention swells less than e.g. Kraft lignin. Thus according to some embodiments, a fluid composition is provided, wherein said lignin component is swelling significantly less than Kraft lignin, such as swelling at least 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 or 100 % less, and

optionally wherein said swelling is determined as change in particle size upon suspension in water or another suitable medium after 60 min.

Further desired features of the fluid composition according to the present invention concern its improved stability and or pumpability when compared to similar compositions. Thus, according to some embodiments, the fluid composition is significantly more stable and/or pumpable when compared to a similar composition prepared with Kraft lignin.

In the context of the present invention, the term “pumpability” is meant to comprise that the fluid composition has a viscosity of 1 Pa.s or less, such as 0.9 Pa.s or less, such as 0.8 Pa.s or less, such as 0.7 Pa.s or less, such as 0.6 Pa.s or less, such as 0.5 Pa.s, such as 0.4 Pa.s or less, such as 0.3 Pa.s or less, such as 0.2 Pa.s or less, or such as 0.1 Pa.s or less at a shear rate of 100 s^{-1} . According to a preferred embodiment, the viscosity is 0.5 Pa.s, or less, or even around 0.25, again measured at a shear rate of 100 s^{-1} , wherein said viscosity is measured as overage over a time period 10 min. In some embodiments, said time period is 5 or 15 min. It has been observed that viscosity is not constant, and tends to increase with time.

Further desired features of the fluid composition according to the present invention concern its improved stability and or pumpability when compared to similar compositions. Thus, according to some embodiments, the fluid composition possesses a significant short term, medium term, or long term stability and/or pumpability, wherein said short, medium, and long term are periods of time in the range of 1–60 min, >1-24 h, or >24 h, respectively. In particular, in some embodiments a fluid composition is provided with an increased short term, medium term and/or long-term stability and/or pumpability, when compared to a similar composition prepared with Kraft lignin. In further embodiments, said short term time period can be 1, 2, 5, 10, 15, 20, 30, 45, or 60 min. In other embodiments, said medium term time period can be 90 min, 2h, 4h, 6h, 8h, 10h, 12h, 18h, 24h. In embodiments relating to long term stability, said long term time period can be 25h, 30h,

40h, 2d, 3d, 4d, 5d, 6d, 1 week, 2 weeks, 3 weeks, 1 month, 2, months, 3 month, 4 months, 5 months, 6 months, or more than 6 month.

Concerning a definition for stability, in the context of the present invention the term

5 “stability” are meant to comprise that no more than 5.0, 4.0, 3.0, 2.0, 1.0 or 0.5 % (w/w) of any one of the fractions (e.g. water, liquid organic fraction, and/or lignin component) of said fluid composition will separate after said specified period of time. It is understood, that in some embodiments a fluid composition is provided, wherein occasional or constant gentle stirring, agitation, and/or re-circulation but no high
10 shear mixing may be required for maintaining said stability and/or pumpability. “No high shear mixing” can e.g. be defined as requiring significantly less - e.g. at least 5x, 10x, 25x, 50x or 100x less - mixing/shearing energy compared to the mixing provided by an IKA Ultra Turrax T25 high-shear mixer at 3.000 rpm and a volume of around 200 ml for 1 min. “No high shear mixing” can also be defined as mixing with a non-
15 high shear force providing mixer.

According to the first embodiment, the present invention concerns a fluid composition according to any one of the preceding claims comprising two or more fractions, wherein (a) the first fraction is an organic fraction in liquid state at room temperature,
20 said organic fraction comprising one or more organic compounds such as one or more fat, and/or one or more oil; and (b) the second fraction comprises one or more lignin component.

In some embodiments, the fluid composition comprises 5-60 % (w/w) lignin
25 component, 0-40 % (w/w) organic fraction, 0-60 % (w/w) vinasse and/or, and 0-1.0 or 0-0.5 % (w/w) further agent. According to some embodiments, the fluid composition the vinasse and/or water is comprised (i) in the first fraction, such as an oil- and/or fat-water-emulsion, as a homogenous solution of oil and/or fat and water; (ii) as a third, aqueous fraction; or (iii) as a combination of (i) and (ii).

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In some embodiments, the Lignin Ion Exchange Capacity is around 0.40, 0.35, 0.30, 0.25, 0.20, 0.15, 0.10, mol/kg dry matter or less; in the range of 0.10-0.20, 0.20-0.30,

0.30-0.40 mol/kg dry matter; and/or in the range of 0.05-0.40, 0.10-0.30, or 0.10-0.20 mol/kg DM.

As indicated earlier, the fluid composition according to the invention may comprise one or more further agent, such as an agent is selected from the group comprising or consisting of one or more dispersing agent(s), surfactant(s), hydrotropic agent(s), emulsifier(s), preserving agent(s), and any combination thereof. According to one embodiment, said one or more further agent is present in the range of 0.001% to 5% (w/w).

According to invention, the various constituents of the fluid composition are intermixed. Thus, according to some embodiments, the one or more fat, oil, lignin component, water, further agent, dispersing agent, surfactant, hydrotropic agent, emulsifier, preserving agent, and any combination thereof are in a state of being intermixed. In some embodiments, said state of being intermixed is selected from the group comprising or consisting of being intermixed as a solution; being intermixed as a suspension; being intermixed as an emulsion; being intermixed as a dispersion; being intermixed as a slurry; and any combination thereof.

The lignin component comprised in the fluid composition may comprise e.g. cellulose and/or hemicellulose and/or ash. Thus, in some embodiments, said lignin component comprises cellulose in an amount of 2,000 – 300,000 ppm, such as 3,000 – 180,000 ppm, e.g. 4,000 – 160,000 ppm, for example 5,000 – 140,000 ppm, such as 6,000 – 120,000 ppm, 7,000 – 100,000 ppm, for example 8,000 – 80,000 ppm, such as 9,000 – 70,000 ppm, e.g. 10,000 – 60,000 ppm, 12,000 – 50,000 ppm, such as 14,000 – 50,000 ppm, e.g. 16,000 – 40,000 ppm, 18,000 – 30,000 ppm, such as 20,000 – 28,000 ppm, for example 22,000 – 26,000 ppm (w/w) in relation to said fluid composition. In some embodiments, said lignin component comprises hemicellulose in an amount of 2,000 – 200,000 ppm, such as 3,000 – 180,000 ppm, e.g. 4,000 – 160,000 ppm, for example 5,000 – 140,000 ppm, such as 6,000 – 120,000 ppm, 7,000 – 100,000 ppm, for example 8,000 – 80,000 ppm, such as 9,000 – 70,000 ppm, e.g. 10,000 – 60,000 ppm, 12,000 – 50,000 ppm, such as 14,000 – 50,000

ppm, e.g. 16,000 – 40,000 ppm, 18,000 – 30,000 ppm, such as 20,000 – 28,000 ppm, for example 22,000 – 26,000 ppm (w/w) in relation to said fluid composition. In some embodiments, said lignin component comprises ash in an amount of 2,000 – 200,000 ppm, such as 3,000 – 180,000 ppm, e.g. 4,000 – 160,000 ppm, for example
5 5,000 – 140,000 ppm, such as 6,000 – 120,000 ppm, 7,000 – 100,000 ppm, for example 8,000 – 80,000 ppm, such as 9,000 – 70,000 ppm, e.g. 10,000 – 60,000 ppm, 12,000 – 50,000 ppm, such as 14,000 – 50,000 ppm, e.g. 16,000 – 40,000 ppm, 18,000 – 30,000 ppm, such as 20,000 – 28,000 ppm, for example 22,000 – 26,000 ppm (w/w) in relation to said fluid composition.

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Dispersing agents are known in the art, and according to some embodiments, the fluid composition comprises one or more dispersing agent is selected from the group comprising or consisting of non-ionic, anionic, cationic and amphoteric dispersing agent(s) and any combination and/or compatible mixture thereof. Such agents can be
15 present in different concentrations. In some embodiments, said one or more dispersing agent is present in said fluid composition in an amount of 10- 50,000 ppm or 200 – 20,000 ppm, such as 300 – 18,000 ppm, e.g. 400 – 16,000 ppm, for example 500 – 14,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm, 1,200
20 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to said fluid composition. Suitable dispersing agents may be selected from the group comprising: Lutensol AP10, AP8, AP7 and AP6 from BASF. The Lutensol AP series consists of ethoxylated nonylphenols, $C_9H_{19}-C_6H_4O(CH_2CH_2O)_xH$, where x is the
25 numeric portion of the product name. Another suitable dispersing agent may be Tergitol NP-9 from Union Carbide, with essentially the same composition as the Lutensol series.

The above and below referred modes of being intermixed and inclusion of water and
30 dispersing agent have proven advantageous in the goal of obtaining stable fluid compositions according to the first aspect (A1) of the present invention. It should be

noted however, that it is possible to obtain stable fluid composition according to the first aspect (A1) of the present invention without inclusion or separately adding of any further agents, such as dispersing agents or the like. This is quite surprising in view of the prior art teaching.

- 5 Surfactants are known in the art, and according to some embodiments, the fluid composition comprises one or more surfactant selected from the group comprising or consisting of anionic, cationic, zwitterionic and nonionic surfactants, and any combination and/or compatible mixture thereof. In some embodiments, said one or more surfactant is present in said fluid composition in an amount of 10- 50,000 ppm
10 or 200 – 20,000 ppm, such as 300 – 18,000 ppm, e.g. 400 – 16,000 ppm, for example 500 – 14,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm, 1,200 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to
15 said fluid composition.

- Hydrotropes are known in the art, and according to some embodiments, the fluid composition comprises one or more hydrotrope is selected from the group comprising or consisting of: non-ionic, anionic, cationic and amphoteric hydrotropes and any
20 combination and/or compatible mixtures thereof. In some embodiments, said one or more hydrotrope is present in said fluid composition in an amount of 10- 50,000 ppm or 200 – 40,000 ppm, such as 300 – 30,000 ppm, e.g. 400 – 20,000 ppm, for example 500 – 15,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm, 1,200
25 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to said fluid composition. Thus in some embodiments of the first aspect (A1) of the present invention said fluid composition furthermore comprises a hydrotropic agent. In such embodiments, said hydrotropic agent may be selected from the group
30 comprising: non-ionic, anionic, cationic and amphoteric hydrotropic agents and compatible mixtures thereof. Suitable hydrotropes may be selected from the group comprising benzoic acid, alkyl-benzoic acid, Butyldiglycol, butanol, propanol,

lignosulphonates, toluenesulphonates, xylenesulphonates and cumesulphonates, caprionates, caprylates, glucose and sodium benzoate. Suitable hydrotropes may also be selected from the Sokalan CP series from BASF (e.g. Eg. Sokalan CP9 - Maleic acid-olefin copolymer, sodium salt; CP10 - Modified polyacrylic acid, sodium salt, CP10S - Modified polyacrylic acid, sodium salt) comprising polyacrylates and maleic acid - acrylic acid co-polymers and the like.

The above referred modes of being intermixed and inclusion of water and further agent(s), such as one or more hydrotropic agent have proven advantageous in the goal of stabilizing and reducing the viscosity of compositions according to the first aspect (A1) of the present invention.

Emulsifiers are known in the art, and according to some embodiments, the fluid composition comprises one or more emulsifier is selected from the group comprising or consisting of sodium phosphate(s), sodium stearyl lactylate cationic, lecithin, DATEM (diacetyl tartaric acid ester of monoglyceride), and any combination and/or compatible mixture thereof. In some embodiments, said one or more emulsifier is present in said fluid composition in an amount of 10- 50,000 ppm or 200 – 20,000 ppm, such as 300 – 18,000 ppm, e.g. 400 – 16,000 ppm, for example 500 – 14,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm, 1,200 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to said fluid composition.

In some embodiments of the first aspect (A1) of the present invention, said fluid composition furthermore comprises a preservative. Preserving agents are known in the art, and according to some embodiments, the fluid composition comprises one or more preserving agent selected from the group comprising or consisting of one or more carboxylate, benzoate, benzoic acid derivative such as parabene(s), aldehyde(s), thiazine(s), organic acid(s), salt(s) of organic acid(s) and the like, and any combination thereof. In some embodiments, said one or more preserving agent is present in said fluid composition in an amount of 10- 50,000 ppm or 20 – 10,000

ppm, such as 30 – 8,000 ppm, e.g. 40 – 6,000 ppm, for example 50 – 5,000 ppm, such as 60 – 4,000 ppm, 70 – 3,000 ppm, for example 80 – 2,000 ppm, such as 90 – 1,500 ppm, e.g. 100 – 1,200 ppm, 120 – 1,000 ppm, such as 140 – 800 ppm, e.g. 160 – 600 ppm, 180 – 400 ppm, such as 200 – 300 ppm, for example 2,200 – 250 ppm (w/w) in relation to said fluid composition. The above referred modes of being intermixed and inclusion of water and preservative have proven advantageous in the goal of stabilizing and reducing microbial growth according to the first aspect (A1) of the present invention.

As mentioned earlier, the fluid composition according to the invention comprises lignin and/or a lignin component. This lignin and/or lignin component can e.g. be characterized by its dry matter (DM) content. In some embodiments, the dry matter content of said lignin component in said fluid composition is 1.0 - 99% (w/w), 10 - 99 % (w/w) or 20 – 95 % (w/w), such as 21 – 94 % (w/w), e.g. 22 - 93 % (w/w), such as 23 – 92 % (w/w), such as 24 – 91 % (w/w), for example 25 – 90 % (w/w), such as 26 – 89 % (w/w), such as 27 – 88 % (w/w), for example 28 – 87 % (w/w), e.g. 29 – 86 % (w/w), such as 30 – 85 % (w/w), such as 31 – 84 % (w/w), such as 32 – 83 % (w/w), such as 33 – 82 % (w/w), for example 34 – 81 % (w/w), such as 35 – 80 % (w/w), e.g. 36 – 79 % (w/w), such as 37 – 78 % (w/w), e.g. 38 – 77 % (w/w), e.g. 39 – 76 % (w/w), such as 40 – 75 % (w/w), such as 41 – 74 % (w/w), such as 42 – 73 % (w/w), such as 43 – 72 % (w/w), for example 44 – 71 % (w/w), such as 45 – 70 % (w/w), e.g. 46 – 69 % (w/w), such as 47 – 68 % (w/w), e.g. 48 – 67 % (w/w), e.g. 49 – 66 % (w/w), such as 50 – 65 % (w/w), such as 51 – 64 % (w/w), such as 52 – 63 % (w/w), such as 53 – 62 % (w/w), for example 54 – 61 % (w/w), such as 55 – 60 % (w/w), e.g. 56 – 59 % (w/w), such as 57 – 58 % (w/w). These ranges have proven advantageous in reaching satisfactorily fluid compositions according to the first aspect (A1) of the present invention.

The lignin and/or lignin component may also comprise sulfur. In some embodiments, the fluid composition comprises a lignin component, wherein the sulfur content - based on the dry matter content of said lignin component - is 2.0 % (w/w) or less, such as 1.4 % (w/w) or less, such as 1.3 % (w/w) or less, for example 1.2 % (w/w) or

less, such as 1.1 % (w/w) or less, e.g. 1.0 % (w/w) or less, such as 0.9 % (w/w) or less, for example 0.8% (w/w) or less, such as 0.7 % (w/w) or less, e.g. 0.6% (w/w) or less, e.g. 0.5 % (w/w) or less, such as 0.4 % (w/w) or less, for example 0.3 % (w/w) or less, such as 0.2 % (w/w) or less, or 0.1 % (w/w) or less, such as 0.09% (w/w) or less, such as 0.08 % (w/w) or less, e.g. 0.07 % (w/w) or less, e.g. 0.06 % (w/w) or less, such as 0.05 % (w/w) or less, for example 0.04 % (w/w) or less, such as 0.03 % (w/w) or less, e.g. 0.02 % (w/w) or less, such as 0.01 % (w/w) or less. Generally, a low sulfur content seems preferred in view of e.g. environmental and/or economical concerns, in particular when the fluid composition is used as fuel. Thus the above stated low sulfur contents of the lignin component of the fluid composition according to the first aspect (A1) of the present invention contribute in making the fluid composition suitable for use as an environmentally friendly fuel.

Concerning the grain and or particle size of the lignin component in the fluid composition of the current invention, this may be of different sizes and/or size distributions. In some embodiments, the lignin component in said fluid composition is having an average grain size of 1-2000 μm , 1-1500 μm , 1-1200 μm , 1-1000 μm , 1-800 μm , 1-600 μm , 1-500 μm , 1 – 450 μm , such as 1.5 - 430 μm , e.g. 2 – 420 μm , such as 3 - 410 μm , for example 4 – 400 μm , e.g. 5 – 390 μm , such as 6 – 380 μm , e.g. 7 – 370 μm , such a 8 – 360 μm , 9 – 350 μm , for example 10 – 340 μm , e.g. 12 – 330 μm , such as 14 – 320 μm , such as 16 - 310 μm , for example 18 – 300 μm , e.g. 20 – 290 μm , such as 22 – 280 μm , e.g. 25 – 270 μm , such a 30 – 260 μm , 35 – 250 μm , for example 40 – 240 μm , e.g. 45 – 230 μm , such as 50 – 220 μm , for example 60 - 210 μm , for example 70 – 200 μm , e.g. 80 – 190, for example 90 – 180 μm , e.g. 100 – 170 μm , such a 110 – 160 μm , 120 – 150 μm , for example 130 – 140 μm . Obviously, the particle size may vary, depending of the time of measurement. It is generally believed, that the particle size may increase upon providing the fluid composition by intermixing the lignin component. An increase in average grain/particle size distribution may be caused by swelling, and without wanting to be bound by any theory, this is believed to also be correlated to the lignin/lignin component's hygroscopy. There are different methods known in the art for determining said particle or grain size. According to some embodiments, said

average grain or particle size being measured before or after providing said fluid composition. In some further embodiments, said grain or particle size being measured by laser diffraction spectroscopy, or e.g. by a Malvern Mastersizer. In some embodiments, dry lignin particle size is measured with a sieve tower (for a standard method, refer to ASTM C136 / C136M – 14) or a Retch Camsizer. In some 5 embodiments, wet samples, such as fluid composition according to the present invention (i.e. where the particles are intermixed with water and/or the liquid organic fraction) particle size is determined using laser diffraction. It is believed that particle size can also be determined in dry or wet samples using microscopy, or other 10 common methods known in the art. The above stated average grain sizes have proven advantageous in the goal of obtaining stable fluid compositions according to the first aspect (A1) of the present invention. One mode of measuring the average grain size of the lignin component is by dynamic light scattering. In one embodiment of the first aspect (A1) of the present invention the lignin component originates from a 15 lignocellulosic biomass having been subjected to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of the hemicellulose present in said lignocellulosic biomass.

As stated herein, the quality and/or physical properties of the lignin and/or lignin 20 component appear important for the invention, and the inventors have discovered that the quality of the fluid composition according to the current invention are improved when not using Kraft lignin or lignin that had been subjected to an alkaline treatment. It is believed that the process used for providing the lignin or lignin component is more important than the biological source of it. Thus, according to 25 some embodiments, the lignin component originates from a lignocellulosic biomass having been subjected to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of the hemicellulose present in said lignocellulosic biomass. According to some embodiment, said lignin component originates from a lignocellulosic biomass having been subject to a hydrothermal 30 pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of the hemicellulose present in said lignocellulosic biomass; and optionally followed by a fermentation, such as an alcohol fermentation. Different hydrolysis methods

appear suitable. Thus, according to some embodiments, said hydrolysis is an acid catalyzed hydrolysis, an enzymatic hydrolysis or a combination of acid/enzyme-catalyzed hydrolysis. Schemes for processing lignocellulosic biomass, including specific process steps as well as overall schemes for converting a lignocellulosic biomass to soluble saccharides and a fibrous fraction comprising a lignin component, are the subject of numerous published patents and patent applications. See e.g. WO 94/03646; WO 94/29474; WO 2006/007691; US2007/0031918; WO 2008/112291; WO 2008/137639; EP 2 006 354; US 2009/0326286; US 2009/0325251; WO 2009/059149; US 2009/0053770; EP 2 169 074; WO 2009/102256; US 2010/0065128; US 2010/0041119; WO 2010/060050; WO2007009463 A2, WO2007009463 A1; WO2011125056 A1; and WO2009125292 A2, each of which is hereby incorporated by reference in entirety.

The above embodiments of the first aspect (A1) of the present invention and relating to the biorefining process of a lignocellulosic biomass as a source to obtain the lignin component of the fluid composition of the first aspect (A1) of the present invention have proven especially advantageous as this process will provide a lignin component having the desired characteristics for obtaining stable fluid compositions having a low sulfur content.

According to the invention, the lignin component may also be characterized by its average molecular weight. According to some embodiments, the a fluid composition is provide, wherein said lignin component is having an average molecular weight (Da) of 1,000 or above, 1,500 or above, 2,000 or above, 2,500 or above, 3,000 or above, such as 3,500 or above, e.g. 4,000 or above, such as 5,000 or above, for example 5,500 or above, such as 6,000 or above, e.g. 7,000 or above, for example 8,000 or above, such as 9,000 or above, for example 10,000 or above, such as 12,000 or above, e.g. 14,000 or above, for example 16,000 or above, e.g. 18,000 or above, e.g. 20,000 or above, such as 25,000 or above, e.g. 30,000 or above, such as 35,000 or above, for example 40,000 or above, such as 45,000 or above, e.g. 50,000 or above, such as 55,000 or above, e.g. 60,000 or above, such as 65,000 or above, e.g. 70,000 or above, such as 75,000 or above, for example 80,000 or above, such

as 85,000 or above, e.g. 90,000 or above, such as 95,000 or above, or 100,000 or above.

As mentioned earlier, a variety of different lignin sources appear suitable in the context of the current invention. According to some embodiments, a fluid composition is provided, wherein said lignin component originates from a lignocellulosic biomass obtained from an annual or a perennial plant. Thus according to some embodiments, the lignin component may originate from a lignocellulosic biomass obtained, obtainable or derived from the group comprising or consisting of one or more of:

cereal, wheat, wheat straw, rice, rice straw, corn, corn fiber, corn cobs, corn stover, hardwood bulk, softwood bulk, sugar cane, sweat sorghum, bagasse, nut shells, empty fruit bunches, grass, cotton seed hairs, barley, rye, oats, sorghum, brewer's spent grains, palm waste material, wood, soft lignocellulosic biomass, and any combination thereof.

It is apparent that in view of different sources of biomass, including different methods of biomass processing, some impurities may be present. According to some embodiments, a fluid composition is provided, wherein said lignin component comprises one or more impurities originating from its mode of production, such as enzyme residues, yeast residues, foam depressant(s), clean in place (CIP) compounds, salts and the like. According to some embodiments, said lignin component comprises impurity/impurities originating from compounds native for the lignocellulosic material, such as cellulose residues, hemicellulose residues, monomeric sugar compounds, dimeric sugar compounds, oligomeric sugar compounds, carbohydrate residues, wax residues, minerals, ash, silica (SiO₂), silica-comprising compounds and/or compositions, salts, organic acids, and the like, and any combination thereof. According to some embodiments, the purity of said lignin component is 50 % (w/w) or more, such as 52 % (w/w) or more, for example 54 % (w/w) or more, such as 56 % (w/w) or more, e.g. 58 % (w/w) or more, such as 60 % (w/w) or more, such as 62 % (w/w) or more, for example 64 % (w/w) or more, such as 66 % (w/w) or more, e.g. 68 % (w/w) or more, such as 70 % (w/w) or more, such as 72 % (w/w) or more, for example 74 % (w/w) or more, such as 76 % (w/w) or more,

e.g. 78 % (w/w) or more, such as 80 % (w/w) or more, such as 82 % (w/w) or more, for example 84 % (w/w) or more, such as 86 % (w/w) or more, e.g. 88 % (w/w) or more, such as 90 % (w/w) or more, such as 92 % (w/w) or more, for example 94 % (w/w) or more, such as 96 % (w/w) or more, e.g. 98 % (w/w) or more. According to an embodiment, the purity of said lignin or lignin component is determined based on content of Klason lignin. According to an embodiment, the purity of said lignin or lignin component is determined based on content of acid insoluble lignin. According to some embodiments, the corresponding percentage constituting impurities may be any one or more impurity as defined earlier in this paragraph.

Apart from lignin or one or more lignin component, and optionally water and/or one or more further agents, the fluid composition according to the present invention comprises an organic fraction. The organic fraction may in turn consist or comprise one or more organic compounds. This organic substance which imparts liquid character to the fluid composition is believed to act as a carrier and/or matrix for the solid fraction comprising the lignin component. In some embodiments, the content of said organic fraction in said fluid composition is at least 2, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, or 95 % (w/w) or more, such as 2 – 95 % (w/w), such as 4 – 78 % (w/w), e.g. 6 – 76 % (w/w), such as 8 – 74 % (w/w), e.g. 10 – 72 % (w/w), such as 12 – 70 % (w/w), e.g. 14 – 68 % (w/w), such as 16 – 66 % (w/w), for example 18 – 64 % (w/w), such as 20 – 62 % (w/w), e.g. 22 – 60 % (w/w), for example 24 – 58 % (w/w), such as 26 – 56 % (w/w), such as 28 – 54 % (w/w), such as 30 – 52 % (w/w), 32 – 50 % (w/w), e.g. 34 – 48 % (w/w), such as 36 – 46 % (w/w), such as 38 – 44 % (w/w), for example 40 – 42 % (w/w). In some embodiments, said organic fraction comprises or consists essentially of an organic solvent, a distillate and/or a residue from a hydrocarbon distillation. In some embodiments, said distillate is selected from the group comprising or consisting of one or more mineral oil, kerosene, diesel, No. 2 fuel oil, No. 3 fuel oil, No. 4 fuel oil, No. 5 fuel oil, No. 6 fuel oil, and No. 7 fuel oil, and any mixture(s) thereof. In other embodiments, the fluid composition may comprise one or more organic compound of plant origin or animal origin. In some embodiments, the one or more organic compound of said liquid organic fraction is an oil of plant origin or a fat of animal origin. In some

embodiments, said one or more organic compound of said liquid organic fraction is an oil originating from pyrolysis of a biomass, such as a cellulosic or lignocellulosic material or wherein said oil is a pyrolysis oil originating from pyrolysis of a lignin component. In some embodiments, the one or more organic compound of said liquid organic fraction is an oil originating from pyrolysis of a polymer, such as a synthetic plastic or synthetic elastomer. In some embodiments, the one or more organic compound of said liquid organic fraction is selected from the group comprising or consisting of glycerol, biodiesel, synfuel, biomass to liquid (BTL) diesel, gas to liquid (GTL) diesel, coal to liquid (CTL) diesel, and any combination thereof. In some embodiments, the one or more organic compound of said liquid organic fraction originates from treatment of a biomass with water and/or other polar liquid(s), such as ethanol or methanol, which may include treatment under supercritical conditions. In an embodiment of the first aspect (A1) of the present invention the biomass which has been treated with water or other polar liquid(s), optionally under supercritical conditions, said biomass may be selected from the group comprising a lignocellulosic material, cellulose and a lignin component.

The above defined characteristics of the organic substance comprised in the liquid fraction of the fluid composition according to the first aspect (A1) of the present invention are very well suited for this fluid composition, especially when used as a liquid fuel.

Concerning biomass treatment in relation to the current invention, in an embodiment, a fluid composition is provided, wherein the said biomass treatment comprises treatment under supercritical conditions. In further embodiments, said biomass which has been treated with water or other polar liquid(s) under supercritical conditions may be selected from the group comprising or consisting of one or more lignocellulosic material, cellulose, lignin component, and any combination thereof.

According to the invention, a fluid composition is provided, wherein said liquid organic fraction or compound of said liquid organic fraction is in itself a mixture of two or more such organic substances, such as three or more such organic substances,

e.g. four or more such organic substances, such as five or six or more of such organic substances.

In mixing different individual organic substances in order to obtain the organic substance to be comprised in the liquid fraction of the fluid composition according to the first aspect (A1) of the present invention it may be possible to impart specific and beneficial physical characteristics to the liquid fraction of the fluid composition not obtainable by using a single component fluid fraction.

10 In some embodiments, the sulfur content of said liquid organic fraction, and/or the one or more organic compound and/or substance of said organic liquid fraction is 5.0 % (w/w) or less, such as 4.5 % (w/w) or less, for example 4.0 % (w/w) or less, such as 3.8 % (w/w) or less, e.g. 3.6 % (w/w) or less, for example 3.4 % (w/w) or less, e.g. 3.2 % (w/w) or less, such as 3.0 % (w/w) or less, for example 2.8 % (w/w) or less, 15 e.g. 2.6 % (w/w) or less, for example 2.4 % (w/w) or less, e.g. 2.2 % (w/w) or less, such as 2.0 % (w/w) or less, for example 1.8 % (w/w) or less, such as 1.6 % (w/w) or less, for example 1.4 % (w/w) or less, e.g. 1.2 % (w/w) or less, such as 1.0 % (w/w) or less, for example 0.8 % (w/w) or less, such as 0.4 % (w/w) or less, such as 0.2 % (w/w) or less, for example 0.1 % (w/w) or less, such as 0.08 % (w/w) or less, e.g. 20 0.06 % (w/w) or less, such as 0.04 % (w/w) or less, e.g. 0.02 % (w/w) or less, for example 0.01 % (w/w) or less, such as 0.008 % (w/w) or less, e.g. 0.006 % (w/w) or less, such as 0.004 % (w/w) or less, e.g. 0.002 % (w/w) or less, such as 0.001 % (w/w), such as 800 ppm or less, e.g. 600 ppm or less, such as 400 ppm or less, e.g. 200 ppm or less, for example 100 ppm or less, such as 50 ppm (w/w) or less. The 25 above stated low sulfur contents of the lignin component of the fluid composition according to the first aspect (A1) of the present invention makes the fluid composition suitable for use as an environmentally friendly fuel.

Seeing that the lignin component originating from biorefining a lignocellulosic biomass by subjecting it to a hydrothermal pretreatment followed by a hydrolysis will 30 have a somewhat hydrophobic character depending on the residual amount of cellulose and hemicellulose, it will in certain formulations be advantageous to make

sure that the organic substance of said liquid fraction of the fluid composition according to the first aspect (A1) of the present invention is an organic substance being immiscible with water thereby itself being hydrophobic. Thus in some embodiments, said liquid organic fraction, organic compound or substance of said liquid organic fraction is immiscible with water.

According to some embodiments, said organic fraction, one or more organic compound or substance at 25 °C is having a viscosity of 0.0005 – 10,000 CSt, such as 0.0010 – 9,000 CSt, e.g. 0.0050 – 8,000 CSt, for example 0.01 – 6,000 CSt, for example 0.05 – 4,000 CSt, such as 0.1 – 2,000 CSt, e.g. 0.5 – 1,000 CSt, such as 1.0 – 800 CSt, e.g. 5.0 – 600 CSt, such as 10 – 400 CSt, for example 50 – 300 CSt, such as 100 – 200 CSt. According to some embodiments, said fluid composition, organic fraction, one or more organic compound or substance, wherein said organic substance at 50 °C is having a viscosity of 0.0004 – 2,000 CSt, such as 0.0010 – 1,500 CSt, e.g. 0.0050 – 1,000 CSt, for example 0.01 – 800 CSt, for example 0.05 – 600 CSt, such as 0.1 – 400 CSt, e.g. 0.5 – 200 CSt, such as 1.0 – 100 CSt, e.g. 5.0 – 80 CSt, such as 10 – 70 CSt, for example 20 – 50 CSt, such as 30 – 40 CSt.

According to some embodiments, said fluid composition, organic fraction, one or more organic compound or substance, wherein said organic substance at 75 °C is having a viscosity of 0.0002 – 200 CSt, such as 0.0001 – 150 CSt, e.g. 0.001 – 100 CSt, for example 0.005 – 80 CSt, such as 0.01 – 60 CSt, e.g. 0.05 – 40 CSt, such as 0.05 – 20 CSt, for example 0.1 – 10 CSt, such as 0.5 – 5 CSt, for example 1.0 – 3 CSt. The above stated viscosities of the organic substance of said liquid fraction of the fluid composition according to the first aspect (A1) of the present invention shall be understood to be independently chosen and shall be understood to define the ranges of viscosities which impart liquid character to said liquid fraction of the fluid composition according to the first aspect (A1) of the present invention.

According to the invention, the fluid composition may or may not comprise water. In some embodiments a fluid composition is provided, wherein the content of said water in said fluid composition is less than 90, 85, 80, 75, 70, 65, 60, 55, 50, 45, 40, 35, 30, 25, 20, 15, 12, 10, 8, 7, 6, 5, 4, 3, 2, 1, 0.5 % (w/w) such as in the range of 2 – 80 %

(w/w), such as 4 – 78 % (w/w), e.g. 6 – 76 % (w/w), such as 8 – 74 % (w/w), e.g. 10 – 72 % (w/w), such as 12 – 70 % (w/w), e.g. 14 – 68 % (w/w), such as 16 – 66 % (w/w), for example 18 – 64 % (w/w), such as 20 – 62 % (w/w), e.g. 22 – 60 % (w/w), for example 24 – 58 % (w/w), such as 26 – 56 % (w/w), such as 28 – 54 % (w/w), such as 30 – 52 % (w/w), 32 – 50 % (w/w), e.g. 34 – 48 % (w/w), such as 36 – 46 % (w/w), such as 38 – 44 % (w/w), for example 40 – 42 % (w/w).

In some embodiments a fluid composition is provided, wherein the ratio lignin component : water is selected from the range of 0.4 – 8.0, such as 0.5 – 7.9, e.g. 0.6 – 7.8, such as 0.7 – 7.6, for example 0.8 – 7.5, for example 0.9 – 7.4, such as 1.0 – 7.3, for example 1.1 – 7.2, e.g. 1.2 – 7.1, such as 1.3 – 7.0, for example 1.4 – 6.9, such as 1.5 – 6.8, such as 1.6 – 6.7, such as 1.7 – 6.6, for example 1.8 – 6.5, for example 1.9 – 6.4, such as 2.0 – 6.3, for example 2.1 – 6.2, e.g. 2.2 – 6.1, such as 2.3 – 6.0, for example 2.4 – 5.9, such as 2.5 – 5.8, such as 2.6 – 5.7, such as for example 2.8 – 5.5, for example 2.9 – 5.4, such as 3.0 – 5.3, for example 3.1 – 5.2, e.g. 3.2 – 5.1, such as 3.3 – 5.0, for example 3.4 – 4.9, such as 3.5 – 4.8, such as 3.6 – 4.7, such as 3.7 – 4.6, for example 3.8 – 4.5, for example 3.9 – 4.4, such as 4.0 – 4.3, for example 4.1 – 4.2, all ratios being based on dry matter content of said lignin component.

The viscosity of the fluid composition according to invention may be e.g. be as follows. In some embodiments said fluid composition at 25, 50 or 75 °C is having a viscosity of 20 – 10,000 CSt, such as 50 – 8,000 CSt, for example 100 – 6,000 CSt, such as 200 – 4,000 CSt, such as 400 – 2,000 CSt, e.g. 500 – 1,000 CSt, such as 600 – 800 CSt. In some embodiments said fluid composition at 25, 50 or 75 °C is having a viscosity of 5 – 2,000 CSt, such as 10 – 1,000 CSt, for example 20 – 800 CSt, such as 50 – 600 CSt, e.g. 100 – 400 CSt, such as 200 – 300 CSt. In some embodiments said fluid composition at 25, 50 or 75 °C is having a viscosity of 2 – 200 CSt, such as 5 – 150 CSt, e.g. 10 – 120 CSt, such as 20 – 100 CSt, for example 30 – 80 CSt, such as 40 – 60 CSt. The above-specified viscosities of the fluid composition are independently examples of what is understood to be a “fluid composition” in the present application and in the appended claims, such as defined according to the first

aspect (A1). These specified viscosities of the fluid composition according to the first aspect (A1) of the present invention have proven advantageous, because such viscosities will ensure that said fluid composition is pumpable. This is especially important in case the fluid composition is going to be used as a fuel.

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According to some embodiments, a fluid composition is provided having a lower heating value of 4 – 37 MJ/kg, such as 5 – 36 MJ/kg,, for example 6 – 35 MJ/kg, such as 7 – 34 MJ/kg, for example 8 - 33 MJ/kg, e.g. 9 – 32 MJ/kg, such as 10 – 31 MJ/kg, for example 11 - 30 MJ/kg, such as 12 – 29 MJ/kg, e.g. 13 – 28 MJ/kg, such as 14 – 27 MJ/kg, such as 15 – 26 MJ/kg,, for example 16 – 25 MJ/kg, such as 17 – 24 MJ/kg, for example 18 - 23 MJ/kg, e.g. 19 – 22 MJ/kg, such as 20 – 21 MJ/kg.

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These lower heating values of the fluid composition make the fluid composition useful as a fuel.

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According to some embodiments, a fluid composition is provided that is stable and/or pumpable for 2 weeks or more, such as 3 weeks or more, e.g. 4 weeks or more, such as 6 weeks or more, for example 7 weeks or more, such as 8 weeks or more, such as 2 months or more, e.g. 3 months or more, for example 4 months or more, such as 5 months or more, or 6 months or more; in the sense that no more than 5.0, 4.5, 4.0, 3.5, 3.0, 2.5, 2.0, 1.5, 1.0 or 0.5 % (w/w) of any one of the fractions (e.g. water and or lignin component fraction) of said fluid composition will separate after said specified period of time. In some embodiments, however, said fluid may require gentle stirring, agitation, and/or re-circulation is required for maintaining said stability and/or pumpability. For the avoidance of doubt, this gentle stirring, agitation, and/or re-circulation is not high-shear mixing.

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According to some embodiments, a fluid composition is provided, wherein the sulfur content of said fluid composition is 3.0 % (w/w) or less, for example 2.8 % (w/w) or less, e.g. 2.6 % (w/w) or less, for example 2.4 % (w/w) or less, e.g. 2.2 % (w/w) or less, such as 2.0 % (w/w) or less, for example 1.8 % (w/w) or less, such as 1.6 % (w/w) or less, for example 1.4 % (w/w) or less, e.g. 1.2 % (w/w) or less, such as 1.0 % (w/w) or less, for example 0.8 % (w/w) or less, such as 0.4 % (w/w) or less, such

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as 0.2 % (w/w) or less, for example 0.1 % (w/w) or less, such as 0.08 % (w/w) or less, e.g. 0.06 % (w/w) or less, such as 0.04 % (w/w) or less. The above stated low sulfur contents of the fluid composition according to the first aspect (A1) of the present invention makes the fluid composition suitable for use as an environmentally friendly fuel.

Second aspect (A2)

In a second aspect (A2), the present invention relates, in the broadest sense, to processes related to the fluid composition according to the first aspect (A1) of the invention, such as methods and processes for the manufacture of a fluid composition according to the first aspect (A1) of the present invention. Such a process according to the second aspect (A2) may comprise the steps of:

- i. providing a fraction, preferably a solid fraction comprising a lignin component;
- ii. providing an organic compound to make up at least part of said liquid organic fraction;
- iii. providing a vinasse, such as a vinasse according to aspect B2 or a vinasse obtainable according to aspect B1;
- iv. intermixing the fraction provided in step (i) with the organic compound and/or liquid organic fraction provided in step (ii) and/or the vinasse provided in step (iii).

In one embodiment, the lignin component in step (i) is a "non-Kraft" lignin with features as e.g. described above, including lower LIEC, lower hygroscopy, and/or lower swelling.

In one embodiment of the second aspect (A2) of the present invention, the lignin component originates from a lignocellulosic biomass, which has been subjected to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of said hemicellulose present in said lignocellulosic biomass. In a further embodiment of the second aspect (A2) of the present invention, said lignin component originates from a lignocellulosic biomass which has been subjected to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of said hemicellulose present in said lignocellulosic biomass,

furthermore followed by a fermentation. In yet a further embodiment, the lignin component originates from a lignocellulosic biomass which has been subjected to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of said hemicellulose present in said lignocellulosic biomass,

5 optionally followed by fermentation and/or hydrolysis.

In a still further embodiment of the second aspect (A2) of the present invention, said lignin component is obtained by pressing said fibrous fraction obtained after subjecting said lignocellulosic biomass to said hydrothermal pretreatment followed by said hydrolysis.

10 In yet a further embodiment of the second aspect (A2) of the present invention, said pressing of said fibrous fraction is preceded by rinsing and/or washing of said fibrous fraction.

In a still further embodiment of the second aspect (A2) of the present invention, said lignin component is obtained by mechanically comminuting said pressed fibrous
15 fraction to a desired extent.

In still yet a further embodiment, the water content of said lignin component may be controlled and/or reduced, e.g. by drying.

The above specified modes of providing the lignin component to be used in the process of the second aspect (A2) of the present invention has proven especially
20 beneficial due to the characteristics of the resulting lignin component.

In one embodiment of the second aspect (A2) of the present invention, said lignin component is as defined above in respect of the first aspect (A1) of the present invention.

In one embodiment of the second aspect (A2) of the present invention, said organic
25 substance of said liquid fraction is having characteristics as defined above in respect of the first aspect (A1) of the present invention.

In one embodiment of the second aspect (A2) of the present invention, the process further comprising admixing of an amount of water.

In one embodiment of the second aspect (A2) of the present invention, the process further comprising admixing of one or more further agent, such as a dispersing agent. In a further embodiment, said fur further agent is selected from the group comprising or consisting of one or more dispersing agent(s), surfactant(s), hydrotropic agent(s),
5 emulsifier(s), preserving agent(s), and any combination thereof.

In one embodiment of the second aspect (A2) of the present invention said lignin component and said organic substance of said liquid fraction, and optionally said amount of water and optionally said dispersing agent may be mixed together using a
10 mechanical stirrer. In one embodiment, the mixing and/or intermixing is performed using one or more mixing device(s), such as a mechanical stirrer, high shear mixer, and/or a pump. In one embodiment of the second aspect (A2) of the present invention said lignin component and an amount of water are separately mixed using a mechanical stirrer; and furthermore, said organic substance of said liquid fraction
15 and an amount of water, optionally also said dispersing agent are separately mixed using a mechanical stirrer; wherein said separately mixed mixtures are mixed and stirred. In a further embodiment, a step of separately intermixing an amount of water and optionally one or more further agent(s) such as a dispersing agent and with (a) said lignin component, (b) said organic compound of said liquid organic fraction,
20 and/or (c) said liquid organic fraction is provided, and optionally wherein said separately mixed mixtures are mixed and stirred.

Third aspect (A3)

In a third aspect (A3), the present invention concerns, in the broadest sense, to
25 processes for treatment of a lignocellulosic treatment comprising the step of converting at least part of the a lignin component obtained in said process to a fluid composition, such as a fluid composition according to the first aspect (A1) of the invention, by admixing said lignin component with a liquid organic fraction comprising an organic compound or substance.

30 In one embodiment, a process for treatment of a lignocellulosic biomass is provided, wherein said process comprises:

- a) subjecting said lignocellulosic biomass for hydrothermal pretreatment resulting in a hydrothermally pretreated lignocellulosic biomass;
- b) subjecting at least part of said hydrothermally pretreated lignocellulosic biomass obtained in step (a) to a hydrolysis resulting in a liquid fraction comprising soluble carbohydrates, and a fiber fraction comprising a lignin component;
- c) optionally subjecting at least part of the liquid fraction obtained in step (b) to a fermentation in order to ferment at least part of said soluble carbohydrates to a fermentation product, such as ethanol, methane or butanol, thereby obtaining a fermentation broth;
- d) optionally isolating at least part of said fermentation product from the fermentation broth obtained in step (c) e.g. by distillation;
- e) isolating at least part of the lignin component from one or more of: the fiber fraction obtained in step (b); the fermentation broth obtained in step (c); or after isolation of at least a part of the fermentation product in step (d);
- f) converting at least part of the lignin component obtained in step (e) to a fluid composition by admixing said lignin component with a liquid organic fraction comprising an organic compound or substance, and/or a vinasse, such as a vinasse according to aspect B2, and/or a vinasse provided according to aspect B1.

Step a) – e) above represent the process of biorefining of a lignocellulosic biomass. This process has itself proven highly beneficial in converting waste biomass into a useful fuel, such as ethanol. It is believed that methods that do not comprise an alkaline treatment step, are beneficial. It is further believed that biorefining methods as outlined above, regardless if e.g. acid, such as H_2SO_4 or the like are added under pretreatment or not, provide a lignin or lignin component suitable for providing a fluid composition according to the first aspect (A1) of the invention. This may include methods comprising a “C5 bypass” or “C5 drain”, in e.g. a two-step pretreatment, wherein a liquid fraction rich in C5 sugars is collected after a first pretreatment step, e.g. by pressing (see e.g. WO2014/019589).

Here, step f) is added to this process making the former known process even more advantageous in that yet another renewable fluid and/or liquid energy product is obtained in the process, such as lignin and/or a vinasse. Thus in some embodiments, the fluid composition obtained in step (f) is a fluid composition according to any one of the preceding claims.

In some embodiments, vinasse is provided by removal of water from a whole stillage (lignin-rich vinasse) and/or from a thin stillage (lignin-depleted vinasse), and optionally, wherein said lignin-rich vinasse and/or lignin-depleted vinasse are provided (i) from the fiber fraction obtained in step (b); (ii) the fermentation broth obtained in step (c); and/or (iii) after isolation of at least a part of the fermentation product in step (d); said vinasse being provided either before (lignin-rich vinasse) or after removal of the lignin component (lignin-depleted vinasse). In further embodiments, at least part of said lignin is isolated from the fiber fraction obtained in step (b) and/or from said fermentation broth obtained in step (c).

In a further embodiment relating to said process for treatment of a lignocellulosic biomass, said process comprises that at least part of said lignin fraction is isolated from the fiber fraction obtained in step (b).

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said process comprises that at least part of said lignin fraction is isolated from said fermentation broth obtained in step (c).

In yet another embodiment relating to said process for treatment of a lignocellulosic biomass, said process comprises that said lignin component is obtained in step (e) by removing an associated liquid phase by using one or more separation device(s), such as a hydraulic press, a vacuum filtration unit, a belt filter, a rotary filter or a centrifuge decanter.

In yet a further embodiment relating to said process for treatment of a lignocellulosic biomass, said process comprises that said lignin component obtained in step (e) is dried to a residual water content at 110 °C of 2 – 20 % (w/w), such as 4 – 18 %

(w/w), for example 6 – 16 % (w/w), such as 8 – 14 % (w/w), e.g. 10 – 12 % (w/w). Alternatively, the lignin component can be dried at 105°C or lower, such as a low as 50°C. Lower temperatures may require a longer drying period. Furthermore, the risk of e.g. microbial contamination and/or growth is increased when drying at lower
5 temperatures. In one embodiment of the invention, the lignin/lignin component is dried at a temperature in the range of 50-150°C.

It is believed that drying the lignin component can be beneficial in terms of obtaining an improved suitable fluid composition, e.g. less viscous and/or more stable, for
10 example, when the lignin/lignin component is, or is derived from a wet filter cake. A 'dry lignin' or 'dry lignin component' possesses usually less than 20 % (w/w) water, preferably around or less than 15 or 10 % (w/w) water. In some embodiments, the residual water content is as low as 5 % (w/w) or lower, such as in the range of 0-5 % (w/w) water, i.e. around 0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, or 5.0 % (w/w).
15 Lignin pellets and/or lignin granulate would usually qualify as 'dry lignin' or 'dry lignin component' as defined above.

In still a further embodiment relating to said process for treatment of a lignocellulosic biomass, said hydrothermal pretreatment of said lignocellulosic biomass is performed
20 at a temperature of 150 – 260 °C, such as 160 – 250 °C, e.g. 170 – 240 °C, such as 180 – 230 °C, for example 190 – 220 °C, such as 200 – 210 °C.

In still another embodiment relating to said process for treatment of a lignocellulosic biomass, said hydrothermal pretreatment of said lignocellulosic biomass is performed
25 in a period of residence time of 2 – 120 min., such as 5 – 110 min., e.g. 10 – 100 min., for example 15 – 90 min., such as 20 - 80 min., such as 25 – 70 min., e.g. 30 – 60 min, such as 35 – 50 min, such as 40 – 45 min.

In another embodiment relating to said process for treatment of a lignocellulosic
30 biomass, said hydrothermal pretreatment of said lignocellulosic biomass is performed by subjecting said lignocellulosic biomass to a log severity, $\log(R_0)$ of 2.5 or more, such as a $\log(R_0)$ of 2.6 or more, e.g. a $\log(R_0)$ of 2.7 or more, such as a $\log(R_0)$ of

2.8 or more, for example a $\log(R_o)$ of 2.9 or more, such as a $\log(R_o)$ of 3.0 or more, such as a $\log(R_o)$ of 3.1 or more, for example a $\log(R_o)$ of 3.2 or more, e.g. a $\log(R_o)$ of 3.3 or more, such as a $\log(R_o)$ of 3.4 or more, such as a $\log(R_o)$ of 3.5 or more; such as a $\log(R_o)$ of 3.6 or more; for example such as a $\log(R_o)$ of 3.7 or more, e.g. a
5 $\log(R_o)$ of 3.8 or more, for example a $\log(R_o)$ of 3.9 or more, for example a $\log(R_o)$ of 4.0 or more, such as a $\log(R_o)$ of 4.1 or more, or a $\log(R_o)$ of 4.2 or more; wherein the log severity is defined as: $\log(R_o) = (\text{residence time}) \times (\exp[\text{Temperature} - 100/14.75])$, and where residence time is measured in minutes and temperature in °C.

10 From the pulp and paper industry, it has been understood that the extent of hemicellulose and lignin released to the aqueous phase was a function of the temperature to which the lignocellulose was heated and also of the residence time of the lignocellulose at the actual temperature. A person skilled in the art regularly make
15 use of a “generalized severity parameter” which has shown to provide good comparisons of results obtained using different temperature and time protocols. Cf. Abatzoglou et al., Chemical Engineering Science, Vol. 47, No. 5, p 1109 – 1122, (1992), the teaching of which is hereby incorporated by reference in entirety.

20 In another embodiments relating to said process for treatment of a lignocellulosic biomass, said hydrolysis is an acid catalyzed hydrolysis and/or enzymatic hydrolysis. In further embodiments, said hydrolysis is performed by one or more cellulases, such as by contacting said pre-treated biomass with one or more cellulases, and/or other enzymes, usually commercially available enzyme compositions developed for this
25 specific type of applications. In yet a further embodiment, said one or more cellulases are selected from the group comprising exo-glucanases, endo-glucanases, hemicellulases and beta-glucosidases. In still a further embodiment, said hydrolysis is performed for a period of time of 1 – 200 hours, such as 5 – 190 hours, such as 10 – 185 hours, e.g. 15 – 180 hours, for example 20 – 175 hours, such as 25 – 170 hours,
30 such as 30 – 165 hours, e.g. 35 – 160 hours, for example 40 – 155 hours, such as 45 – 150 hours, such as 50 – 145 hours, e.g. 55 – 140 hours, for example 60 – 135 hours, such as 65 – 130 hours, such as 70 – 125 hours, e.g. 75 – 120 hours, for

example 80 – 115 hours, such as 85 – 110 hours, such as 90 – 105 hours, e.g. 95 – 100 hours.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said said step (b) and step (c) are performed as a separate hydrolysis and fermentation step (SHF), and wherein said hydrolysis is performed at a temperature of 30 – 72 °C, such as 32 – 70°C, e.g. 34 – 68 °C, for example 36 – 66 °C, such as 38 – 64 °C, e.g. 40 – 62 °C, 42 – 60°C, e.g. 44 – 58 °C, for example 46 – 56 °C, such as 48 – 54 °C, e.g. 50 – 52 °C.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said said hydrolysis is performed in a period of time of 70 – 125 hours, e.g. 75 – 120 hours, for example 80 – 115 hours, such as 85 – 110 hours, such as 90 – 105 hours, e.g. 95 – 100 hours.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said step (b) and step (c) are performed as a simultaneous saccharification and fermentation step (SSF), and wherein said hydrolysis is performed at a temperature of 30 – 72 °C, such as 32 – 70 °C, e.g. 34 – 68 °C, for example 36 – 66 °C, such as 38 – 64 °C, e.g. 40 – 62 °C, 42 – 60°C, e.g. 44 – 58 °C, for example 46 – 56 °C, such as 48 – 54 °C, e.g. 50 – 52 °C.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said hydrolysis is performed in a period of time of 1 – 12 hours, such as 2 – 11 hours, for example 3 – 10 hours, such as 4 – 9 hours, e.g. 5 – 8 hours, such as 6 – 7 hours.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said step (b) and step (c) are performed as a simultaneous saccharification and fermentation step (SSF), and wherein said fermentation is performed at a temperature of 25 – 40 °C, such as 26 – 39 °C, e.g. 27 – 38 °C, for example 28 – 37 °C, e.g. 29 – 36 °C, for example 30 – 35 °C, such as 31 – 34 °C or 32 – 33 °C.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said fermentation is performed in a period of time of 100 – 200 hours, such as 105 – 190 hours, such as 110 – 185 hours, e.g. 115 – 180 hours, for example 120
5 – 175 hours, such as 125 – 170 hours, such as 130 – 165 hours, e.g. 135 – 160 hours, for example 140 – 155 hours, such as 145 – 150 hours.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said lignin fraction obtained in step e) is converted to a fluid composition by
10 admixing said lignin fraction with one or more organic substance and/or composition, said organic substance and/or composition constituting a liquid fraction.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said lignin fraction obtained in step e) is converted to a fluid composition by
15 admixing said lignin fraction with an organic substance and with vinasse and/or water, said organic substance constituting a liquid fraction. In a further embodiment, the lignin fraction obtained in step e) is converted to a fluid composition by admixing said lignin fraction with an organic substance and with vinasse, such as a vinasse provided as disclosed above and/or in aspect B1, and/or provided according to
20 aspect B2, and optionally water, said organic substance constituting a liquid fraction.

In another embodiment relating to said process for treatment of a lignocellulosic biomass, said lignin fraction obtained in step e) is converted to a fluid composition by admixing said lignin fraction with an organic substance, with water, and with one or
25 more further agent, such as a dispersing agent, said organic substance constituting a liquid fraction.

The above modes of manufacture of the fluid composition according to the second (A2) and/or third aspect (A3) of the present invention have proved convenient and effective.

30 **Fourth aspect (A4)**

In a fourth aspect (A4), the present invention relates to uses of a fluid composition according to the first aspect (A1) of the present invention, including a fluid composition provided according to the second or third aspect (A3) of the present invention.

- 5 The following embodiments relate to the use of the fluid composition as fuel. Interestingly, lignin itself represent a fairly high heating value but has the disadvantage of being a solid. Therefore, it is highly beneficial to be able to convert such a solid fuel to a liquid fuel which allows easier handling and storing and transportation of the fuel.

- 10 In one embodiment of the fourth aspect (A4) of the present invention the fluid composition is used as a fuel for a household burner.

In one embodiment of the fourth aspect (A4) of the present invention the fluid composition is used as a fuel for a boiler in a district heat plant or in a combined heat and power (CHP) plant.

- 15 In one embodiment of the fourth aspect (A4) of the present invention the fluid composition is used as a fuel for producing steam or other thermal energy products in an industry or factory using such steam or other thermal energy products to power its power consuming facilities.

- 20 In one embodiment of the fourth aspect (A4) of the present invention the fluid composition is used as a fuel in a boiler in a power plant.

In one embodiment of the fourth aspect (A4) of the present invention the fluid composition is used as a fuel in a start-up situation in a boiler in a power plant.

- 25 In a start-up situation in a power plant it is often not possible to use solid fuel, such as coal due to the risk of dust explosions. Hence, for the time being in start-up situations power plants use fuel oil. The fuel oil is rather expensive and may have a high S content and it will be most advantageous to be able to substitute a traditionally and conventionally liquid fuel oil with a liquid fuel in the form of a fluid composition according to the first aspect (A1) or provided according to the second aspect (A2) of the present invention.

Fifth aspect (A5)

In a fifth aspect (A5), the present invention relate to the use of lignin or a solid lignin component for a fluid composition, such as a fluid composition according to any of the previous aspects of the present invention. This includes also uses related to
5 chemical processing of lignin and/or a lignin component or a conversion product thereof. The lignin and/or lignin component can e.g. be provided as described in the first (A1), second (A2), or third aspect (A3) of the invention.

According to one embodiment related to the fifth aspect (A5) of the present invention, said lignin and/or solid lignin component originates from a lignocellulosic biomass
10 which has been subjected to a hydrothermal pretreatment followed by a hydrolysis.

According to one embodiment related to the fifth aspect (A5) of the present invention, said lignin component originates from a lignocellulosic biomass which has been subjected to a hydrothermal pretreatment followed by fermentation and/or distillation.
15

According to one embodiment related to the fifth aspect (A5) of the present invention, said hydrolysis is an acid catalyzed hydrolysis.

According to one embodiment related to the fifth aspect (A5) of the present invention, said hydrolysis is an enzymatic hydrolysis.
20

According to one embodiment related to the fifth aspect (A5) of the present invention, said hydrolysis comprises acid and enzymatic hydrolysis.

According to one embodiment related to the fifth aspect (A5) of the present invention, said fluid composition comprises a solid fraction and a liquid fraction.
25

According to one embodiment related to the fifth aspect (A5) of the present invention, said solid fraction and said liquid fraction are present in a state of being intermixed;
30 said solid fraction comprises said lignin component; and said liquid fraction comprises an organic substance.

It is well-known in the art to use lignin or a lignin component as a feedstock for making organic chemicals, such as toluene.

However, handling of a solid feedstock in a chemical production plant may pose certain challenges as to the handling, transportation and storing of such solid

5 material. Moreover, it is not easy to make a solid feedstock flow in a process line of a chemical manufacturing plant.

The invention of the fluid composition according to the first, second and/or third aspect (A3) of the present invention makes it possible to perform a chemical processing of a lignin component as a fluid or comprised in a fluid. This feature is

10 utilized in the fifth aspect (A5) of the present invention. Thus, in different embodiments of the fifth aspect (A5) of the present invention, the chemical processing of a lignin component may relate to one or more of the following:

- a catalytic processing of said lignin component or a conversion product thereof.
- a non-catalytic processing of said lignin component or a conversion product thereof.
- 15 - an acid and/or base reactions of said lignin component or a conversion product thereof.
- an oxidation reaction of said lignin component or a conversion product thereof.
- a reduction reaction of said lignin component or a conversion product thereof.
- a hydrolysis reaction of said lignin component or a conversion product thereof.
- 20 - a pyrolysis of said lignin component or a conversion product thereof.
- a hydrothermal conversion of said lignin component or a conversion product thereof.
- a supercritical fluid conversion of said lignin component or a conversion product thereof, such as a conversion involving water, methanol, and/or ethanol at
- 25 supercritical conditons.
- hydrogenation of said lignin component or a conversion product thereof.

- hydrodesulfurization of said lignin component or a conversion product thereof.
- hydrodenitrogenation of said lignin component or a conversion product thereof.
- processing involves hydrodeoxygenation and/or hydrogenation of said lignin component or a conversion product thereof.

- 5 - processing involves hydrocracking of said lignin component or a conversion product thereof.
- processing involves hydrodenitrification of said lignin component or a conversion product thereof.
- oxidation of said lignin component or a conversion product thereof.
- 10 - cracking of said lignin component of said lignin component or a conversion product thereof, such as a technical cracking of said lignin component or a conversion product thereof; and/or a catalytical cracking of said lignin component or a conversion product thereof.

- Any use according to the fifth aspect (A5) of the invention may comprise or be
- 15 comprised in a system according to aspect B5.

- In the context of the present invention, the term “conversion product thereof” is meant to comprise the following: One may contemplate situations in which a lignin component is converted to a fluid composition according to e.g. the first aspect (A1) of the present invention. This fluid composition may be used for a chemical
- 20 processing of a lignin component thus leading to reaction products of said chemical processing. However, the reaction product – still being in a fluid mixture – may itself be subject to further chemical processing of the same kind or of another kind. Many of such serial processing steps may be performed starting with a lignin component, leading to a first reaction product in a first chemical processing reaction. This first
- 25 reaction product may be processed further to a second reaction product in a second chemical processing reaction and so forth.

In one embodiment of the fifth aspect (A5) of the present invention the said composition is a fluid composition according to any embodiment of the first aspect (A1) of the present invention as described above.

5 It has surprisingly turned out that a lignin component originating from a lignocellulosic biomass which has been subjected to a hydrothermal pretreatment followed by a hydrolysis may form stable fluids when mixed with a liquid fraction comprising an organic substance, and further impart beneficial properties to such fluids, such as e.g. a relatively low sulfur content and a relatively low viscosity.

10 Accordingly, the above details relating to the manufacture of such a lignin component are advantageous.

SECTION B (Aspects B1-B5)

Section B relates to aspects and embodiments relating to vinasses. The following definitions/terms concern Section B, but may also apply to section A, if non-conflicting.

15 The terms "dry matter" or "dm" can be used interchangeably and are meant to comprise total solids (dissolved and undissolved), usually expressed as weight %, unless indicated otherwise.

"Autohydrolysis" refers to a pretreatment process, wherein it is believed that acetic acid liberated by hemicellulose hydrolysis during pretreatment further catalyzes
20 hemicellulose hydrolysis. This may apply to any hydrothermal pretreatment of lignocellulosic biomass, usually conducted at pH between 3.5 and 9.0.

In the context of the present invention, the terms "active component(s)" or "further components" can be used interchangeably and are meant to comprise e.g.
dispersing agent(s), surfactant(s), hydrotropic agent(s), emulsifier(s), preserving
25 agent(s), anti-foaming agent(s), viscosity modifier(s), and any combination thereof. Such further components may also comprise agents that make a fuel more liquid or solid, also termed "liquefying agent(s)" or "solidifying agent(s)", respectively.

“Solid” is one of the four fundamental states of matter (the others being liquid, gas, and plasma). It is characterized by some structural rigidity and resistance to changes of shape or volume. Unlike a liquid, a solid object or composition does not flow to take on the shape of its container, nor does it expand to fill essentially the entire volume available. In the context of the present, the reference temperature is room temperature, e.g. at 20 or 25°C.

A “solid fuel” according to the invention is a solid composition as defined above, and is usually not pumpable. A solid fuel may comprise solidifying agents.

A “liquid” is meant to comprise a near-incompressible fluid that conforms to the shape of its container but retains a (nearly) constant volume independent of pressure at room temperature, e.g. at 20 or 25°C. Being liquid is one of the four fundamental states of matter other than being solid, gas, or plasma.

In the context of the present invention, the term “fluid composition” as used in the present description and in the appended claims shall be understood to mean a composition which is fluid or liquid in the sense that it exhibits viscosities at various temperatures falling within ranges as herein, in particular at room temperature, e.g. at 20 or 25°C. A fluid composition is usually pumpable.

A “liquid fuel” according to the invention is liquid and/or a fluid composition as defined above and usually pumpable. In some embodiments, it can only be maintained in the fluid or liquid state in e.g. a storage tank when subjected to gentle agitation, for example, with a recirculation pump. A liquid fuel may comprise liquefying agents. It may also be a suspension or emulsion.

In the context of the present invention, the term “pumpable” is meant to comprise that the fluid composition has a viscosity of 1 Pa.s or less, such as 0.9 Pa.s or less, such as 0.8 Pa.s or less, such as 0.7 Pa.s or less, such as 0.6 Pa.s or less, such as 0.5 Pa.s, such as 0.4 Pa.s or less, such as 0.3 Pa.s or less, such as 0.2 Pa.s or less, or such as 0.1 Pa.s or less at a shear rate of 100 s⁻¹. According to a preferred embodiment, the viscosity is 0.5 Pa.s, or less, or even around 0.25, again measured at a shear rate of 100 s⁻¹, wherein said viscosity is measured as average over at time period 10 min. In some embodiments, said time period is 5 or 15 min.

With respect to lignin, lignin-components and/or lignin rich fractions, including methods of their provision and definitions, reference is made to application No. PCT/DK2015/050242, filed on 14 Aug 2015. In the context of the present invention, the term "lignin" is meant to comprise the term "lignin-component" as defined in
5 PCT/DK2015/050242, and both terms may be used interchangeably. Thus, the term "lignin" in the present description and in the appended claims may also refers to the polymer denoted as such and being present in unprocessed lignocellulosic plant material. Furthermore, the term "lignin" shall also be understood to mean a "lignin" that has been subject to various physical and/or chemical treatments imposing
10 changes of the lignin polymer structure, while mostly still retaining its polymer character and containing significant amounts of hemicellulose and cellulose.

Hence, "lignin", as used in the present description and in the appended claims may refer to a lignin that has been subjected to slight structural modifications.

Also, "lignin" as used in the present description and in the appended claims may refer
15 to a lignin that has been subjected to slight structural modifications and/or comprising an amount of chemical residues originating from its mode of manufacture, or originating from compounds native for the lignocellulosic material from which it is isolated.

In some embodiments of the various aspects of the present invention, "lignin" may
20 specifically exclude a Kraft lignin or a Kraft lignin fragment obtained from a Kraft processing of a lignocellulosic biomass.

In some embodiments of the various aspects of the present invention, "lignin" may specifically exclude lignosulfonate, such as a product obtainable from sulfite pulping. As in Kraft pulping, the temperature during sulfite pulping is 130-180°C. Usually,
25 sulfite pulping is conducted at low pH (e.g. 1.5 - 5) in the presence of HSO_3^- and/or SO_3^{2-} ions. During sulfite pulping, lignin is sulfonated, and the resulting lignosulfonate is water-soluble and has a high number of charged groups.

In some embodiments of the various aspects of the present invention, "lignin" may specifically exclude soda lignin, a product obtainable from soda pulping. In this

process, pulping occurs in an essentially sulfur-free medium, e.g. in contrast to the Kraft process, comprising only or predominantly soda.

In some embodiments of the various aspects of the present invention, "lignin" may specifically exclude organosolv lignin, obtainable from a pulping process, where
5 organic solvents and water are used to rid the lignin from cellulose. Temperatures during processing range e.g from 140°C to 220°C. For enhancing solubilization of lignin, sulfuric acid may be added during the process. A number of organic solvents are suitable for such a process, such as acetic acid, formic acid, ethanol, peroxiorganic acids, acetone, methanol, butanol, and ethylene glycol. Organosolv
10 lignin possesses usually lower molecular weight and higher chemical purity. Organosolv lignins are typically hydrophobic and show low water-solubility.

Another process for obtaining lignin is by extraction in ionic liquids (producing ionic liquid lignin). Ionic liquids are salts, which are in liquid state at a relatively low temperature (e.g. below 100°C). Lignin obtained with ionic liquids is believed to
15 possess similar properties as organosolv lignin. However, regeneration of ionic liquid is problematic, and industrial scale production is therefore limited until further progress within this field has been achieved.

In some embodiments of the various aspects of the present invention, "lignin" may specifically exclude ionic liquid lignin.

20 In the context of the present invention, the term "lignin" is meant to comprise a by-product from 2nd generation (2G) bioethanol production. There are various different 2nd generation bio-ethanol processes known in the art that may provide such a lignin component, incl. organosolv processes. Schemes for processing lignocellulosic biomass, including specific process steps as well as overall schemes for converting a
25 lignocellulosic biomass to soluble saccharides and a fibrous fraction being or comprising the lignin component, are the subject of numerous published patents and patent applications. See e.g. WO 94/03646; WO 94/29474; WO 2006/007691; US2007/0031918; WO 2008/112291; WO 2008/137639; EP 2 006 354; US 2009/0326286; US 2009/0325251; WO 2009/059149; US 2009/0053770; EP 2 169
30 074; WO 2009/102256; US 2010/0065128; US 2010/0041119; WO 2010/060050;

WO2007009463 A2, WO2007009463 A1; WO2011125056 A1; and WO2009125292 A2, WO 2014/019589, each of which are hereby incorporated by reference in their entirety.

In some preferred embodiments of the present invention, “lignin” is provided by a solid/liquid separation step from a stillage, e.g. when providing a thin stillage from a whole stillage. The water content of the lignin can be adjusted by methods known in the art, e.g. by evaporation and/or drying.

In the context of the present invention, the terms “two-stage pretreatment” or “two-step pretreatment” can be used interchangeably and are meant to comprise a process comprising two or more stage pretreatment steps, usually designed to provide improved C5 yields, such as processes disclosed in WO2010/113129; US2010/0279361; WO 2009/108773; US2009/0308383; US8,057,639 or US20130029406, each of which are hereby incorporated by reference in their entirety, wherein in these “two stage” pretreatment schemes, some C5-rich liquid fraction is removed by solid/liquid separation after a lower temperature pretreatment, followed by a subsequent, pretreatment of the solid fraction, usually at higher temperature and/or pretreatment severity.

In the context of the present invention, the terms “C5 bypass” or “C5 drain” can be used interchangeably, and are meant to comprise a process, wherein a usually C5-sugar rich liquid fraction is provided, such as through a liquid/solid separation step, e.g. by pressing, after and/or during pretreatment, which can be conducted as a single stage, two-stage, or more than two-stage pretreatment process. For example, PCT/DK2013/050256, filed on 1 Aug 2013, published as WO 2014/019589, herewith incorporated by reference in its entirety, discloses such processes.

In the context of the present invention, the term “whole slurry” is meant to comprise a process, wherein pretreated biomass can be used directly in a subsequent hydrolysis step, such as an enzymatic hydrolysis and/or fermentation, such as e.g. disclosed in PCT/DK2014/050030, filed on 5 Feb 2014, published as WO2015/014364, herewith incorporated by reference in its entirety. “Whole slurry” may also refer to an

enzymatic hydrolysis reaction mixture in which the ratio by weight of undissolved to dissolved solids at the start of enzymatic hydrolysis is less than 3.1 or 2.2:1.

According to the invention, said pretreatment and/or hydrolysis of biomass may be performed with or without addition of one or more acid(s) or one or more base(s),
5 such as H₂SO₄, HCl, NH₃, NH₄OH, NaOH, KOH, Ca₂(OH) and the like.

pH adjustment may be used to provide an appropriate pH for enzymatic hydrolysis and/or fermentation.

In the context of the present invention, the term "cellulase" is meant to comprise enzyme compositions that hydrolyse cellulose (beta-1, 4-D-glucan linkages) and/or
10 derivatives thereof. Cellulases include the classification of exo- cellobiohydrolases (CBH), endoglucanases (EG) and beta-glucosidases (BG) (EC3.2.191, EC3.2.1.4 and EC3.2.1.21). Examples of cellulases include cellulases from e.g. *Penicillium*, *Trichoderma*, *Humicola*, *Fusarium*, *Thermomonospora*, *Cellulomonas*, *Clostridium* and *Aspergillus*. Suitable cellulases are commercially available and known in the art.

15 Commercial cellulase preparations may comprise one or more further enzymatic activities. Furthermore, "cellulase" can also be used interchangeably with "cell-wall modifying enzyme", referring to any enzyme capable of hydrolysing or modifying the complex matrix polysaccharides of the plant cell wall, such as any enzyme that will have activity in the "cell wall solubilization assay" as e.g. described in WO10115754,
20 which is herewith included by reference in its entirety. Included within this definition of "cell-wall modifying enzyme" are cellulases, such as cellobiohydrolase I and cellobiohydrolase II, endo-glucanases and beta-glucosidases, xyloglucanases and hemicellulolytic enzymes, such as xylanases.

Commercially available cellulase preparation(s) suitable in the present context are
25 often optimized for lignocellulosic biomass conversion and may comprise a mixture of enzyme activities that are sufficient to provide enzymatic hydrolysis of pretreated lignocellulosic biomass, often comprising endocellulase (endoglucanase), exocellulase (exoglucanase), endoxylanase, xylosidase and B-glucosidase activities. The term "optimized for lignocellulosic biomass conversion" refers to a product
30 development process in which enzyme mixtures have been selected and/or modified

for the specific purpose of improving hydrolysis yields and/or reducing enzyme consumption in hydrolysis of pretreated lignocellulosic biomass to fermentable sugars.

In the context of the present invention, the term "glucan" is meant to comprise cellulose as well as other gluco-oligomers and other gluco- polymers. Such oligo- or polysaccharides consist of glucose monomers, linked by glycosidic bonds.

"Hydrothermal pretreatment" commonly refers to the use of water, either as hot liquid, vapor steam or pressurized steam comprising high temperature liquid or steam or both, to "cook" biomass, at temperatures of e.g. 120 degrees centigrade (°C) or higher, either with or without addition of acids or other chemicals.

"Solid/liquid separation" - related terms refer to an active mechanical process, whereby liquid is separated from solid by application of force through pressing, centrifugal or other force, whereby "solid" and "liquid" fractions are provided. The separated liquid is collectively referred to as "liquid fraction." The residual fraction comprising considerable insoluble solid content is referred to as "solid fraction." A "solid fraction" will have a dry matter content and will typically also comprise some residual of "liquid fraction." Conventional means for solid/liquid separations comprise e.g. centrifuges, such as decanter centrifuges, filtration units, such as filter presses, chamber filter presses, including membrane filtration technology, either alone or in combination.

The term "lignocellulosic biomass" is meant to comprise any biomass obtained, obtainable or derived from a lignin comprising plant, such as annual or a perennial plants, such as one or more of: cereal, wheat, wheat straw, rice, rice straw, corn, corn fiber, corn cobs, corn stover, hardwood bulk, softwood bulk, sugar cane, sweet sorghum, bagasse, nut shells, empty fruit bunches, grass, straw, cotton seed hairs, barley, rye, oats, sorghum, brewer's spent grains, palm waste material, wood, soft lignocellulosic biomass, algae, and any combination thereof. Suitable biomasses according to the present invention comprise agricultural waste.

In the context of the present invention, the term "soft lignocellulosic biomass" indicates non-wood, or non-wood derived biomass.

The terms "about", "around", "approximately", or "~" indicate e.g. the measuring uncertainty commonly experienced in the art, which can be in the order of magnitude of e.g. +/- 1, 2, 5, 10, 20, or even 50 percent (%), usually +/- 10%.

The term "comprising" is to be interpreted as specifying the presence of the stated parts, steps, features, or components, but does not exclude the presence of one or more additional parts, steps, features, or components. For example, a composition comprising a chemical compound may thus comprise additional chemical compounds.

A "vinasse" according to the present invention can be provided by removal of water from a stillage.

In the context of the present invention, common unit operations known in the art can be used for removal of water, such as evaporation, often facilitated by heat, and/or negative pressure (vacuum), including e.g. consecutive evaporation. Water removal may also be performed uniquely or in combination with membrane separation technology/means, such as nano filtration, ultra filtration (UF), reverse osmosis, cross-flow filtration and the like.

A "stillage" according to the present invention can e.g. be provided by a process providing 2nd generation bioethanol from soft lignocellulosic biomass, usually after removal of EtOH, commonly by distillation. However, it is believed that a vinasse suitable as fuel according to the present invention can also be provided from a process, without fermentation (e.g. yeast to provide EtOH; lactic acid bacteria to provide lactic acid, etc.), and/or distillation. Accordingly, the term "stillage" is meant to comprise the product of a process comprising (i) pretreatment of a biomass, such as, but not exclusively, a soft lignocellulosic biomass, followed by (ii) hydrolysis of at least a part of the pretreated biomass.

Aspect B1

In a first aspect (B1), the current invention concerns a method for providing a vinasse, such as a lignin-rich and/or lignin-depleted vinasse, said method comprising the steps of:

- (i) pretreatment of lignocellulosic biomass;
- (ii) hydrolysis of at least a portion of the pretreated lignocellulosic biomass from step (i) to provide one or more products;
- (iii) fermentation of the one or more products from step (ii) to provide a fermentation product;
- (iv) separation/removal of the one or more products from step (ii), and/or the fermentation product from step (iii) to provide a whole stillage;
- (v) solid/liquid separation, whereby a lignin-rich fraction ("lignin") and an aqueous thin stillage are provided;
- (vi) removal of water from the lignin-rich fraction; and
- (vii) removal of water from the whole stillage of step (iv), or from the thin stillage of step (v), to provide said lignin-rich vinasse or said lignin-depleted vinasse;

wherein any one of steps (iii), (v) and/or step (vi) are optional; and wherein the lignin-rich vinasse has a dry matter content (dm) of at least 30% (w/w), and the lignin-depleted vinasse has a dm of at least 40% (w/w).

In some embodiments, the fermentation product is an alcohol, such as EtOH.

In some embodiments, the fermentation is a fermentation with yeast, optionally a yeast fermenting C5 and C6 sugars.

In some embodiments, the separation of the fermentation product comprises distillation.

In some embodiments, the lignocellulosic biomass is obtained, obtainable or derived from an annual or a perennial plant, such as a cereal, wheat, wheat straw, rice, rice straw, corn, corn fiber, corn cobs, corn stover, hardwood bulk, softwood bulk, sugar cane, sweat sorghum, bagasse, nut shells, empty fruit bunches, grass, cotton seed hairs, barley, rye, oats, sorghum, brewer's spent grains, palm waste material, wood, soft lignocellulosic biomass, and any combination thereof. Usually, the biomass is agricultural waste.

In some embodiments, the pretreatment comprises addition of an acid and/or a base, such as H_2SO_4 and/or ammonia (NH_4OH). In other embodiments, the pretreatment is an autohydrolysis process without addition of an acid and/or a base.

In some embodiments, the pretreatment is a single-stage process.

- 5 In some embodiments, the pretreatment is a two- or more stage process.

In some embodiments, the process comprises a "C5 bypass", wherein a usually C5-sugar rich liquid fraction is provided, such as through a liquid/solid separation step, e.g. by pressing, after and/or during pretreatment, which can be conducted as a single stage, two- or more-stage pretreatment process, such as disclosed in

- 10 PCT/DK2013/050256.

In some embodiments, the process comprises a "whole slurry process", wherein pretreated biomass is used directly in a subsequent hydrolysis step, such as an enzymatic hydrolysis and/or fermentation, such as e.g. disclosed in

- 15 PCT/DK2014/050030. In a further embodiment, the ratio by weight of undissolved to dissolved solids at the start of enzymatic hydrolysis is less than 3.1 or 2.2:1.

In some embodiments, the enzymatic hydrolysis comprises one or more cellulase and/or xylanase, optionally comprising one or more pH adjustment step(s) prior, during and/or after said enzymatic hydrolysis.

- 20 In some embodiments, the hydrolysis is an acid- or base-catalyzed process, usually not comprising the addition of enzymes.

In some embodiments, at least 50-98% of the carbohydrates of the lignocellulosic biomass are converted to mono- or disaccharides during said enzymatic or acid or base-catalyzed hydrolysis.

- 25 In some embodiments, at least 50 - 98 of the carbohydrates of the lignocellulosic biomass are converted to said fermentation product.

In some embodiments, the solid/liquid separation step (v) comprises removal of at least 90% (w/w) of the undissolved solids from the whole stillage from step (iv), e.g. by pressing.

In some embodiments, water is removed from the lignin from step (v), e.g. by evaporation, yielding lignin with a dm of at least 50, 60, 70, 80, 90, 92, 94, 95, 96, 97, 98, 99 or 100 % (w/w).

In some embodiments, at least 60% of water is removed from the whole stillage from
5 step (vii) e.g. by evaporation, such as by consecutive evaporation.

In some embodiments, at least 85% of water is removed from the thin stillage of step (vii), e.g. by evaporation, such as consecutive evaporation.

In some embodiments, at least a fraction of said lignin-rich vinasse and or lignin-depleted vinasse are combusted. In further embodiments, said combusting
10 comprises the use of combusting means, such as a conventional boiler, such as grate boiler, bubbling fluidized bed (BFB), low temperature bubbling fluidized bed (LTBFB), circulating fluidized bed (CFB), low temperature circulating fluidized bed (LT CFB), or oil boiler. Suitable LT CFB technology comprises "Pyrooneer" devices, methods and uses, e.g. as disclosed in any one of WO 1999/032583, WO
15 2014/012556, or WO 2015/007285, each of which are hereby incorporated by reference in their entirety.

Second aspect (B2)

The second aspect (B2) of the current invention relates to a vinasse provided, defined or characterized according to e.g. the first aspect (B1) of the invention. Said vinasse
20 can e.g. be a lignin-rich or a lignin-depleted vinasse.

In some embodiments, the vinasse is a lignin-rich vinasse.

In some embodiments, the lignin-rich vinasse has a lower heating value (LHV) of at least 3.4 or more, 3.6 or more, 3.8 or more, 4.0 or more, 4.2 GJ/t or more, 4.5 GJ/t or more, 5.0 GJ/t or more, 6.0 GJ/t or more, 7.0 GJ/t or more, 8.0 GJ/t or more, 10.0
25 GJ/t or more, 12.0 GJ/t or more, 14.0 GJ/t or more, 16.0 GJ/t or more, or 18.0 GJ/t or more.

In some embodiments, the lignin-rich vinasse has a dry matter (dm) of at least 30% or more, 35% or more, 40% or more, 45% or more, 50% or more 55% or more, 60%

or more 65% or more, 70% or more 75% or more, 80% or more 85% or more, 90% or more, 95% or more, or 100%.

In some embodiments, the lignin-rich vinasse is liquid and/or pumpable. In another embodiment, said the liquid lignin-rich vinasse comprises one or more further

5 agent(s) in an active amount. In a further embodiment, said further agent is one or more liquefying agent(s), In yet a further embodiment, said liquefying agent(s) is/are present in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

In some embodiments, the lignin-rich vinasse is solid and/or not pumpable. In

10 another embodiment, said solid lignin-rich vinasse comprises one or more further agent(s) in an active amount. In a further embodiment, said further agent is one or more solidifying agent(s), In yet a further embodiment, said solidifying agent(s) is/are present in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

15 In some embodiments, the vinasse is a **lignin-depleted vinasse**.

In some embodiments, the lignin-depleted vinasse has a LHV of at least 3.4 GJ/t or more, 3.6 GJ/t or more, 3.8 GJ/t or more, 4.0 GJ/t or more, 4.2 GJ/t or more, 4.5 GJ/t or more, 5.0 GJ/t or more, 6.0 GJ/t or more, 7.0 GJ/t or more, 8.0 GJ/t or more, 9.0 GJ/t or more, or 10GJ/t or more.

20 In some embodiments, the lignin-depleted vinasse has a dm of at least 30% or more, 35% or more, 40% or more, 45% or more, 50% or more 55% or more, 60% or more 65% or more, 70% or more 75% or more, 80% or more 85% or more, 90% or more, 95% or more, or 100%.

In some embodiments, the lignin-depleted vinasse is liquid and/or pumpable. In

25 another embodiment, said liquid lignin- depleted vinasse comprises one or more further agent(s) in an active amount. In a further embodiment, said further agent is one or more liquefying agent(s), In yet a further embodiment, said liquefying agent(s) is/are present in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

In some embodiments, the lignin- depleted vinasse is solid and/or not pumpable. In another embodiment, said solid lignin-depleted vinasse comprises one or more further agent(s) in an active amount. In a further embodiment, said further agent is one or more solidifying agent(s). In yet a further embodiment, said solidifying agent(s) is/are present in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

Third aspect (B3)

The third aspect (B3) of the current invention pertains to a fuel comprising a vinasse according to the second aspect (B2) of the invention.

- 10 In some embodiments, said fuel comprises 0.01-100, 0.1-100, 1-100, 10-100, 20-100, 30-100, 40-100, 50-100, 60-100, 70-100, 80-100, or 90-100 % of a lignin-rich and/or lignin-depleted vinasse according to the second aspect (B2) of the invention.

- In some embodiments, the fuel is a liquid or fluid, such as a pumpable composition. In a further embodiment, said liquid fuel comprises one or more further agent(s) in an active amount. In a further embodiment, said further agent is one or more liquefying agent(s). In yet a further embodiment, said liquefying agent(s) is/are present in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

- In some embodiments, the fuel is a solid fuel, such as a non-pumpable fuel. In another embodiment, said solid fuel comprises one or more further agent(s) in an active amount. In a further embodiment, said further agent is one or more solidifying agent(s). In yet a further embodiment, said solidifying agent(s) is/are present in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

In some embodiments, the fuel comprising said lignin-rich and/or lignin-depleted vinasse has a lower heating value of 3.4 GJ/ton or more.

- 25 In some embodiments, said lignin-rich and/or lignin-depleted vinasse has a dry matter content of at least 30%, 40% or more than 40%.

In some embodiments, the fuel comprises one or more organic fractions, such as one or more oil, fat, or organic solvent, including any combination thereof.

In some embodiments, the fuel comprises lignin and/or a lignin-rich component, such as lignin provided according to any one of the preceding embodiments.

In some embodiments, the fuel comprises 0.01-100% (w/w) lignin-depleted and/or lignin-rich vinasse, 0-99.9 % (w/w) lignin-rich component; and optionally 0-99.9% (w/w) of other organic fraction, and optionally 0-20% (w/w) of one or more further agent(s).

In some embodiments, the fuel comprises lignin-depleted and/or lignin-rich vinasse comprising 0-70% (w/w) water, 30-100% (w/w) other matter, such as organic and/or inorganic matter.

- 10 In some embodiments, the fuel comprises 50-99.9% (w/w) lignin-depleted and/or lignin-rich vinasse, 0.1-50% (w/w) lignin-rich component.

In some embodiments, the fuel comprises 1-100% (w/w) lignin-depleted and/or lignin-rich vinasse with a dm of 30% or more, 0-99 % (w/w) organic fraction, 0-99% (w/w) lignin-rich component; and optionally 0-20% (w/w) of a further agent.

- 15 In some embodiments, the fuel comprises up to around 50% lignin-depleted vinasse with a dm of around 65% or more, and around 50% lignin or more with a dm of around 90% or more. In a further embodiment, said fuel has one or more of the following features: solid, a LHV of around 10-13 GJ/t, ignition time of > 500 ms, low ash swelling, and suitable for grate boiler, BFB, LTBFB, CFB, LTCFB.
- 20 In some embodiments, the fuel comprises not more than around 60% lignin-depleted vinasse with a dm of around 40% or more, and up to 99.9% lignin with a dm of around 90% or more. In a further embodiment, said fuel has one or more of the following features: solid, a LHV of around 9-13 GJ/t or more, ignition time of > 500 ms, low ash swelling, and suitable for grate boiler, BFB, LTBFB, CFB, LTCFB.
- 25 In some embodiments, the lignin has a dm of e.g. 25% or more, 30% or more, such as 35% or more, such as 40% or more, such as 45% or more, 50% or more, such as 55% or more, such as 60% or more, such as 65% or more, such as 70% or more, such as 75% or more, such as 80% or more, such as 85% or more, such as 90% or more, such as 92% or more, such as 95% or more, such as 96% or more, such as

97% or more, such as 98% or more, such as 99% or more around 100%. In some embodiments, a lignin fraction from a decanter centrifuge provides a lignin with a dm in the range of around 25%-40%. In some embodiments, when using a chamber filter press, a lignin is provided with a dm of around 25-75%, 30-70%, or 40-60%.

- 5 In some embodiments, the fuel comprises at least 95% lignin-depleted vinasse with a dm of around 40% or more, and up to 5% oil. In a further embodiment, said fuel has one or more of the following features: liquid, a LHV of around 9-11 GJ/t, ignition time of < 500 ms, and is suitable for combustion e.g. in an adjusted oil boiler, co-combustion such as fireball co-combustion.
- 10 In some embodiments, the fuel comprises at least 87% lignin-depleted vinasse with a dm of around 65% or more, and up to 13% oil. In a further embodiment, said fuel has one or more of the following features: liquid, a LHV of around 8-11 GJ/t or more, ignition time of < 500 ms, and is suitable for combustion e.g. in an adjusted oil boiler, co-combustion such as fireball co-combustion.
- 15 In some embodiments, the fuel comprises at least 75% lignin-depleted vinasse with a dm of around 65% or more, and up to 25% lignin with a dm of around 90% or more. In a further embodiment, said fuel has one or more of the following features: liquid, a LHV of around 9 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fireball co-combustion.
- 20 In some embodiments, the fuel comprises at least 70% lignin-depleted vinasse with a dm of around 40% or more, and up to 30% lignin with a dm of around 90% or more. In a further embodiment, said fuel has one or more of the following features: liquid, a LHV of around 9 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fire ball co-combustion, BFB or CFB.
- 25 In some embodiments, the fuel comprises 90% or more, or around 100% lignin-depleted vinasse with a dm of around 65%, or 65-100%. In a further embodiment, said fuel has one or more of the following features: liquid, a LHV of around 5-6 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fire ball combustion, BFB or CFB.

In some embodiments, the fuel comprises 80% or more lignin-depleted vinasse with a dm of around 65% or more, and up to 20% wood or coal%. In a further embodiment, said fuel has one or more of the following features: solid, a LHV of around 8 GJ/t or more, and ignition time of < or > 500 ms, suitable for combustion in a conventional solid fuel boiler, e.g. grate boiler, BFB, LTBF, CFB or LTCFB.

In some embodiments, the fuel comprises around 88% or more lignin-rich vinasse with a dm of around 30% or more, and up to 12% oil. In a further embodiment, said fuel has one or more of the following features: liquid, a LHV of around 8 GJ/t or more, and ignition time of < 500 ms, suitable for combustion in an adjusted oil boiler, fire ball combustion, and/or co-combustion.

In some embodiments, the fuel comprises 90% or more, or around 100% lignin-rich vinasse with a dm of around 65% or 65-100%. In a further embodiment, said fuel has one or more of the following features: solid, a LHV of around 7 GJ/t or more, and ignition time of > 500 ms, suitable for combustion in a conventional solid fuel boiler, e.g. grate boiler, BFB, LTBF, CFB or LTCFB.

In some embodiments, the fuel is a liquid fuel and may comprise one or more of: diesel oil, bunker oil, petroleum, petrol, kerosene, paraffin, organic solvent, including any combination thereof.

In some embodiments, the fuel is a solid fuel, and comprises one or more of: lignin, lignin-rich component, coal (such as lignite (brown coal), flame coal, gas flame coal, gas coal, fat coal, forge coal, non-baking coal, or anthracite), wood, (e.g. wood chips or wood pellets), including any combination thereof.

In some embodiments, the fuel is suitable for co-combustion, such as fireball co-combustion.

In some embodiments, the fuel is suitable for combustion in a conventional boiler, such as grate boiler, bubbling fluidized (BFB), low temperature bubbling fluidized bed (LTBF), circulating fluidized bed (CFB), circulating low temperature fluidized bed (LT CFB), oil boiler, or fireball co-combustion.

In some embodiments, the LT CFB comprises "Pyroner" devices, methods and uses, e.g. as disclosed in any one of WO 1999/032583, WO 2014/012556, or WO 2015/007285, said applications being herewith incorporated by reference in their entirety.

- 5 It is conceivable that different combustion technologies are suitable for different fuel mixtures. The parameters taken into consideration when selecting and/or /recommending a suitable combustion technology may comprise (i) whether the fuel is solid or liquid fuel; (ii) Ignition time delay in different fuel mixtures, and/or (iii) alkali content in the fuel.
- 10 Fuels mixtures with only vinasse and lignin could fall within two groups, namely (1) solid fuel or (2) liquid fuel.

Ad (1): Solid fuel: the fuel has long ignition delay (as expected for any solid fuel) and it should be possible to combust using e.g. grate boiler technology, fluidized bed technology as well as fluidized bed technology: In this case, two variants of
15 circulating fluidized bed (CFB) could be required: standard fluidized bed technology and LT (low temperature) fluidized bed technology (e.g. if salt content is high).

Ad (2): Liquid fuel: the liquid fuel consisting of vinasse and lignin only have long ignition delay and is suitable for co-combustion in e.g. fire ball combustion technology.

- 20 Fuels mixtures with only vinasse and oil are liquid fuels and experiments have shown that even a small amount of oil reduces ignition time delay and the mixture starts behaving as heavy fuel oil. Therefore, it is believed that conventional oil-burner technology is suitable for such fuel mixtures.

Also, fuel mixtures with vinasse and solid fuels other than lignin (can e.g. be wood,
25 coal, or the like) can also fall into two groups, namely (1) solid fuel or (2) liquid fuel.

Ad (1): Solid fuel: the fuel has long ignition delay (as expected for any solid fuel) and it is possible to use it in grate boiler technology, fluidized bed technology as well as fluidized bed technology: In this case, two variants of CFB could be required:

standard fluidized bed technology and LT (low temperature) fluidized bed technology (e.g. if salt content is high).

Ad (2) Liquid fuel: the liquid fuel consisting of vinasse and lignin has a long ignition delay and is suitable for co-combustion in e.g. fire ball combustion technology.

- 5 Further examples of fuels according to the third aspect (B3) of the invention are presented in the experimental section, including methods of their provision.

Fourth aspect (B4)

- 10 The fourth aspect (B4) of the current invention concerns the use of a vinasse, such as a lignin-rich- and/or lignin-depleted vinasse according to the second aspect (B2) of the invention as a fuel, such as a fuel according to the third aspect (B3) of the invention.

In some embodiments, conventional combusting means can be used.

- 15 In some embodiments, said use comprises fluidized bed technology, such as circulating fluidized bed (CFB), bubbling fluidized bed (BFB), low temperature fluidized bed technology, such as low temperature circulating fluidized bed (LT CFB), e.g. as disclosed in any one of WO 1999/032583, WO 2014/012556, or WO 2015/007285, and low temperature bubbling fluidized bed LTBFb.

In other embodiments, said use comprises boiler technology, such as grate boiler or oil boiler. In a further embodiment, a conventional boiler can be used.

- 20 In another embodiment, a conventional boiler has to be adapted, modified and/or adjusted, such as to be suitable for combusting a fuel according to the third aspect (B3) of the invention.

Fifth aspect (B5)

- 25 The fifth aspect (B5) of the current invention relates to a system comprising means for providing a vinasse, such as a lignin-rich or a lignin-depleted vinasse according to the first aspect (B1) and/or second aspect (B2) of the invention, and optionally means

for converting said vinasse to energy, such as a boiler and/or a CHP by combusting a fuel according to the third aspect (B3) or fourth aspect (B4) of the invention.

In particular, embodiments relating to systems or set-ups are disclosed, with or without anaerobic digestion for biogas production from whole or thin stillage.

- 5 Furthermore, in some embodiments, said system comprises means relating to e.g. the second (A2), third (A3) or fourth (A4) aspect of the invention.

In some embodiments, the system comprises a boiler and/or fluidized bed means for burning and/or gasifying a vinasse and/or vinasse-comprising fuel, such as a combusting means according to the fourth aspect (B4) of the invention.

- 10 In some embodiments, the boiler and/or fluidized bed means is a boiler and/or fluidized bed means.

In some embodiments, the boiler means comprises or is a grate boiler or oil boiler. In a further embodiment, a conventional boiler can be used. In another embodiment, a conventional boiler has to be adapted and/or modified.

- 15 In some embodiments, the boiler and/or fluidized bed means comprises or is fluidized bed technology, such as circulating fluidized bed (CFB), bubbling fluidized bed (BFB), low temperature fluidized bed technology, such as low temperature circulating fluidized bed (LTCFB) and low temperature bubbling fluidized bed LTBFB.

- 20 In some embodiments, the vinasse-comprising fuel is a fuel according to the third aspect (B3) of the invention.

In some embodiments, the system comprises means for providing energy.

In some embodiments, the system comprises a combined heat and power plant (CHP).

- 25 In some embodiments, the system provides electricity, heat, steam and/or hot water, such as for a biomass processing plant.

In some embodiments, the system comprises means for providing 1st and/or 2nd generation bioethanol.

In some embodiments, the system comprises means for pre-treatment of plant biomass, hydrolysis, removal of water, and combustion means, such as a system disclosed herein in e.g. **Figures 31-36** and/or **Figures 31B-36B**, optionally including the hereto corresponding figure legends and embodiments.

- 5 A 2nd generation bioethanol process includes biogas plant in order to clean the water before re-use and recover the energy from the stillage. Biogas can be produced from lignin thin stillage or vinasses. Often, the biogas produced is usually immediately used for energy production, but can also be cleaned and upgraded and transported to natural gas net. See e.g. **Figures 31 and 31B** for an example of a 2nd generation
10 bioethanol process with biogas and lignin production with combined heat and power plant.

Different process configurations for vinasse containing fuel mixtures are shown in **Figure 32-36** and **Figures 32B-36B**. In **Figures 32 and 32B**, lignin depleted vinasse and lignin are provided as solid biofuel for used onsite for energy production.

- 15 Alternatively, some or all lignin can be exported from the site.

This configuration is promising when taking e.g. the fuel mixture properties into consideration: a high heating value of the fuel and the alkali metals are diluted. This configuration does not require any external fuel source. In **Figures 33 and 33B**, lignin depleted vinasse and oil are mixed together in order to obtain a combustible liquid
20 fuel. In this option, lignin can be utilized for other purposes. The heating value of this fuel depends on the amount of oil added to the vinasses. 5-15%w/w of oil has been tested and all samples were combustible in the single droplet combustion test (see Examples below).

In **Figures 34 and 34B**, lignin depleted vinasse and a part of lignin are mixed into
25 liquid fuel. The amount of lignin mixed into the fuel should ensure a lower heating value of at least 7 to 9 GJ/t of the final liquid fuel. The advantage of this solution is that no addition fuel is needed for the energy production and that the lignin and vinasse consumption fits with the overall energy consumption of the 2nd generation bioethanol plant. In **Figures 35 and 35B**, a process configuration is shown, where
30 lignin depleted vinasse is used directly for steam production. This case is relevant for

a site where electricity is available from the grid and process steam needs to be produced onsite. Whole lignin can be exported in this case. An advantage of this solution is an easy way of self-steam supply. In **Figures 36** and **36B**, a process configuration is shown, where a lignin rich vinasse is produced and used as fuel, optionally in combination with another fuel, e.g. using co-combustion.

Please note that it is believed that the specific configurations of the 2nd generation bioethanol plant as depicted here should not be construed as limiting and/or essential for some embodiments of the present invention.

In some embodiments, essentially all lignin provided is burned, such as burned as a solid lignin biofuel, as lignin-comprising fuel, and/or as lignin-rich vinasse.

In other embodiments, said system provides a surplus of lignin, such as a lignin biofuel or lignin for other use.

In some embodiments, the system does not comprise means for anaerobic digestion. In preferred embodiments, the system does not comprise anaerobic digestion means for methan/biogas production from stillage, such as whole or thin stillage. In further embodiments, the system comprises or does not comprise anaerobic digestion means for treatment and/or purification of condensates, such as condensates from water removal steps of e.g. lignin, stillage, vinasse, lignin-depleted vinasse or lignin-rich vinasse.

Further vinasse-related embodiments according to the present invention are disclosed as numbered embodiments below:

1. A method for providing a vinasse, said vinasse being lignin-rich and/or lignin-depleted vinasse, said method comprising the steps of:

- (i) pretreatment of lignocellulosic biomass;
- (ii) hydrolysis of at least a portion of the pretreated lignocellulosic biomass from step (i) to provide one or more products;
- (iii) fermentation of the one or more products from step (ii) to provide a fermentation product;

- (iv) separation/removal of the one or more products from step (ii), and/or the fermentation product from step (iii) to provide a whole stillage;
- (v) solid/liquid separation, whereby a lignin-rich fraction ("lignin") and an aqueous thin stillage are provided;
- 5 (vi) removal of water from the lignin-rich fraction; and
- (vii) removal of water from the whole stillage of step (iv), or from the thin stillage of step (v), to provide said lignin-rich vinasse or said lignin-depleted vinasse;

wherein any one of steps (iii), (v) and/or step (vi) are optional; and wherein the
10 lignin-rich vinasse has a dry matter content (dm) of at least 30% (w/w), and the lignin-depleted vinasse has a dm of at least 40% (w/w).

2. A method according to embodiment 1, wherein the fermentation product is an alcohol, such as EtOH.
3. A method according to embodiment 1 or 2, wherein the fermentation is a
15 fermentation with yeast, optionally a yeast fermenting C5 and C6 sugars.
4. A method according to any one of the preceding embodiments, wherein the separation of the fermentation product comprises distillation.
5. A method according to any one of the preceding embodiments, wherein the
lignocellulosic biomass is obtained, obtainable or derived from an annual or a
20 perennial plant, such as a cereal, wheat, wheat straw, rice, rice straw, corn, corn fiber, corn cobs, corn stover, hardwood bulk, softwood bulk, sugar cane, sweat sorghum, bagasse, nut shells, empty fruit bunches, grass, cotton seed hairs, barley, rye, oats, sorghum, brewer's spent grains, palm waste material, wood, soft lignocellulosic biomass, and any combination thereof.
- 25 6. A method according to any one of the preceding embodiments, wherein the pretreatment comprises addition of an acid and/or a base, such as H₂SO₄ and/or ammonia (NH₄OH).
7. A method according to any one of embodiments 1 - 5, wherein the pretreatment is an autohydrolysis process without addition of an acid and/or a base.
- 30 8. A method according to any one of the preceding embodiments, wherein the pretreatment is a single stage process.

9. A method according to any one of the preceding embodiments, wherein the pretreatment is a two- or more stage process.
10. A method according to any one of the preceding embodiments, said process comprising a "C5 bypass", wherein a usually C5-sugar rich liquid fraction is provided, such as through a liquid/solid separation step, e.g. by pressing, after
5 and/or during pretreatment, which can be conducted as a single stage, two- or more-stage pretreatment process, such as disclosed in PCT/DK2013/050256.
11. A method according to any one of the preceding embodiments, wherein said process comprises a "whole slurry process", wherein pretreated biomass is used
10 directly in a subsequent hydrolysis step, such as an enzymatic hydrolysis and/or fermentation, such as e.g. disclosed in PCT/DK2014/050030, and optionally, wherein the ratio by weight of undissolved to dissolved solids at the start of enzymatic hydrolysis is less than 3.1 or 2.2:1.
12. A method according to any one of the preceding embodiments, wherein said
15 hydrolysis step (ii) is (a) an enzymatic hydrolysis comprising addition of one or more cellulase and/or xylanase, optionally comprising one or more pH adjustment step(s) prior, during and/or after said enzymatic hydrolysis; or (b) the hydrolysis is an acid- or base-catalyzed process, usually not comprising the addition of enzymes.
- 20 13. A method according to any one of the preceding embodiments, wherein at least 50-98% of the carbohydrates of the lignocellulosic biomass are converted to mono- or disaccharides during said enzymatic hydrolysis.
14. A method according to any one of the preceding embodiments, wherein at least 50 - 98 of the carbohydrates of the lignocellulosic biomass are converted to said
25 fermentation product.
15. A method according to any one of the preceding embodiments, wherein the solid/liquid separation step (v) comprises removal of at least 90% (w/w) of the undissolved solids from the whole stillage from step (iv), e.g. by pressing.
16. A method according to any one of the preceding embodiments, wherein water is
30 removed from the lignin from step (v), e.g. by evaporation, yielding lignin with a dm of at least 50, 60, 70, 80, 90, 92, 94, 95, 96, 97, 98, 99 or 100 % (w/w).

17. A method according to any one of the preceding embodiments, wherein at least 60% of water is removed from the whole stillage from step (vii) e.g. by consecutive evaporation.
18. A method according to any one of the preceding embodiments, wherein at least 85% of water is removed from the thin stillage of step (vii), e.g. by consecutive evaporation.
19. A method according to any one of the preceding embodiments, further comprising combusting at least a fraction of said lignin-rich vinasse and or lignin-depleted vinasse.
20. A method according to embodiment 19, wherein said combusting comprises the use of a conventional boiler, such as grate boiler, bubbling fluidized bed (BFB), low temperature bubbling fluidized bed (LTBFB), circulating fluidized bed (CFB), low temperature circulating fluidized bed (LT CFB - e.g. as disclosed in any one of WO 1999/032583, WO 2014/012556, or WO 2015/007285), or oil boiler.
21. A vinasse provided, defined or characterized according to any one of the preceding embodiments.
22. A vinasse according to embodiment 21, said vinasse being a lignin-rich vinasse.
23. A lignin-rich vinasse according to embodiment 22, having a LHV of at least 3.4 or more, 3.6 or more, 3.8 or more, 4.0 or more, 4.2 GJ/t or more, 4.5 GJ/t or more, 5.0 GJ/t or more, 6.0 GJ/t or more, 7.0 GJ/t or more, 8.0 GJ/t or more, 10.0 GJ/t or more, 12.0 GJ/t or more, 14.0 GJ/t or more, 16.0 GJ/t or more, or 18.0 GJ/t or more.
24. A lignin-rich vinasse according to embodiment 22 or 23, having a dm of at least 30% or more, 35% or more, 40% or more, 45% or more, 50% or more 55% or more, 60% or more 65% or more, 70% or more 75% or more, 80% or more 85% or more, 90% or more, 95% or more, or 100%.
25. A lignin-rich vinasse according to any one of embodiments 22 - 24, wherein the lignin-rich vinasse is liquid, optionally comprising one or more further agents, such as one or more liquefying agent(s), in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.
26. A lignin-rich vinasse according to any one of embodiments 22 - 25, wherein the lignin-rich vinasse is solid, optionally comprising one or more further agents, such

as one or more solidifying agent(s) in an active concentration, in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

27. A vinasse according to embodiment 21, said vinasse being a lignin-depleted vinasse.

5 28. A lignin-depleted vinasse according to embodiment 27, having a LHV of at least 3.4 GJ/t or more, 3.6 GJ/t or more, 3.8 GJ/t or more, 4.0 GJ/t or more, 4.2 GJ/t or more, 4.5 GJ/t or more, 5.0 GJ/t or more, 6.0 GJ/t or more, 7.0 GJ/t or more, 8.0 GJ/t or more, 9.0 GJ/t or more, or 10GJ/t or more.

10 29. A lignin-depleted vinasse according to embodiment 27 or 28, having a dm of at least 30% or more, 35% or more, 40% or more, 45% or more, 50% or more 55% or more, 60% or more 65% or more, 70% or more 75% or more, 80% or more 85% or more, 90% or more, 95% or more, or 100%.

15 30. A lignin-depleted vinasse according to any one of embodiments 27 - 29, wherein the lignin-depleted vinasse is liquid, optionally comprising one or more further agents, such as one or more liquefying agent(s), in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

20 31. A lignin-depleted vinasse according to any one of embodiments 27-30, wherein the lignin-depleted vinasse is solid, optionally comprising one or more further agents, such as one or more solidifying agent(s), in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

32. A fuel comprising 0.01-100, 0.1-100, 1-100, 10-100, 20-100, 30-100, 40-100, 50-100, 60-100, 70-100, 80-100, or 90-100 % of a lignin-rich and/or lignin-depleted vinasse according to any one of the preceding embodiments.

25 33. A fuel according to embodiment 32, wherein said fuel is a liquid, such as a pumpable composition; or a solid fuel, such as a non-pumpable fuel.

34. A fuel according to embodiment 32 or 33, wherein said lignin-rich and/or lignin-depleted vinasse has a lower heating value of 3.4 GJ/ton or more.

30 35. A fuel according to any one of embodiments 32 - 34, wherein said lignin-rich and/or lignin-depleted vinasse has a dry matter content of at least 30%, 40% or more than 40%.

36. A fuel according to any one of embodiments 32 - 35, further comprising one or more organic fractions, such as one or more oil, fat, or organic solvent, including any combination thereof.
37. A fuel according to any one of embodiments 32 -36, further comprising a lignin-rich component, such as lignin provided according to any one of the preceding embodiments.
38. A fuel according to any one of embodiments 32-37, wherein said fuel comprises 0.01-100% (w/w) lignin-depleted and/or lignin-rich vinasse, 0-99.9 % (w/w) lignin-rich component; and optionally 0-99.9% (w/w) of other organic fraction, and optionally 0-20% (w/w) of one or more further agent(s).
39. A fuel according to any one of embodiments 32 - 38, wherein said lignin-depleted and/or lignin-rich vinasse comprises 0-70% (w/w) water, 30-100% (w/w) other matter both organic and inorganic.
40. A fuel according to any one of embodiments 32 - 39, wherein said fuel comprises 50-99.9% (w/w) lignin-depleted and/or lignin-rich vinasse, 0.1-50% (w/w) lignin-rich component.
41. A fuel according to any one of embodiments 32 - 40, wherein said fuel comprises 1-100% (w/w) lignin-depleted and/or lignin-rich vinasse with a dm of 30% or more, 0-99 % (w/w) organic fraction, 0-99% (w/w) lignin-rich component; and optionally 0-20% (w/w) of a further agent.
42. A fuel according to any one of embodiments 32 - 41, comprising up to around 50% lignin-depleted vinasse with a dm of around 65% or more, and around 50% lignin or more with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 10-13 GJ/t, ignition time of > 500 ms, low ash swelling, and suitable for grate boiler, BFB, LTBFB, CFB, LTCFB.
43. A fuel according to any one of embodiments 32 - 42, comprising not more than around 60% lignin-depleted vinasse with a dm of around 40% or more, and up to 99.9% lignin with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 9-13 GJ/t or more, ignition time of > 500 ms, low ash swelling, and suitable for grate boiler, BFB, LTBFB, CFB, LTCFB.

44. A fuel according to any one of embodiments 32 - 43, comprising at least 95% lignin-depleted vinasse with a dm of around 40% or more, and up to 5% oil; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 9-11 GJ/t, ignition time of < 500 ms, and suitable for combustion e.g. in an adjusted oil boiler and/or co-combustion such as fireball co-combustion.
45. A fuel according to any one of embodiments 32 - 44, comprising at least 87% lignin-depleted vinasse with a dm of around 65% or more, and up to 13% oil; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 8-11 GJ/t or more, ignition time of < 500 ms, and suitable for combustion e.g. in an adjusted oil boiler and/or co-combustion such as fireball co-combustion.
46. A fuel according to any one of embodiments 32 - 45, comprising at least 75% lignin-depleted vinasse with a dm of around 65% or more, and up to 25% lignin with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 9 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fireball co-combustion.
47. A fuel according to any one of embodiments 32 - 46, comprising at least 70% lignin-depleted vinasse with a dm of around 40% or more, and up to 30% lignin with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 9 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fire ball co-combustion, BFB or CFB.
48. A fuel according to any one of embodiments 32 -47, comprising 90% or more, or around 100% lignin-depleted vinasse with a dm of around 65%, or 65-100%; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 5-6 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fire ball combustion, BFB or CFB.
49. A fuel according to any one of embodiments 32 - 48, comprising 80% or more lignin-depleted vinasse with a dm of around 65% or more, and up to 20% wood or coal%; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 8 GJ/t or more, and ignition time of < or > 500 ms, suitable

for combustion in a conventional solid fuel boiler, e.g. grate boiler, BFB, LTBFB, CFB or LTCFB.

50. A fuel according to any one of embodiments 32 - 49, comprising around 88% or more lignin-rich vinasse with a dm of around 30% or more, and up to 12% oil; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 8 GJ/t or more, and ignition time of < 500 ms, suitable for combustion in an adjusted oil boiler, fire ball combustion, and/or co-combustion.

51. A fuel according to any one of embodiments 32 - 50, comprising 90% or more, or around 100% lignin-rich vinasse with a dm of around 65% or 65-100%; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 7 GJ/t or more, and ignition time of > 500 ms, suitable for combustion in a conventional solid fuel boiler, e.g. grate boiler, BFB, LTBFB, CFB or LTCFB

52. A fuel according to any one of embodiments 32 - 51, wherein said fuel is liquid fuel and comprises one or more of: diesel oil, bunker oil, petroleum, petrol, kerosene, paraffin, organic solvent, including any combination thereof.

53. A fuel according to any one of embodiments 32 - 51, wherein said fuel is a solid fuel, and comprises one or more of: lignin, lignin-rich component, coal (such as lignite (brown coal), flame coal, gas flame coal, gas coal, fat coal, forge coal, non-baking coal, or anthracite), wood, (e.g. wood chips or wood pellets), including any combination thereof.

54. A fuel according to any one of embodiments 32 - 53, wherein said fuel is suitable for co-combustion, such as fireball co-combustion.

55. A fuel according to any one of embodiments 32 - 54, wherein said fuel is suitable for combustion in a conventional boiler, such as grate boiler, bubbling fluidized (BFB), low temperature bubbling fluidized bed (LTBFB), circulating fluidized bed (CFB), circulating low temperature fluidized bed (LT CFB - LTCFB – e.g. as disclosed in any one of WO 1999/032583, WO 2014/012556, or WO 2015/007285), oil boiler, or fireball co-combustion.

56. Use of a lignin-rich- and/or lignin-depleted vinasse according to any one of embodiments 1 - 31 as a fuel, such as a fuel according to any one of embodiments 32 - 55.

57. Use according to embodiment 56, wherein said use comprises fluidized bed technology, such as circulating fluidized bed (CFB), bubbling fluidized bed (BFB), low temperature fluidized bed technology, such as low temperature circulating fluidized bed (LTCFB – e.g. as disclosed in any one of WO 1999/032583, WO 2014/012556, or WO 2015/007285) and low temperature bubbling fluidized bed LTBF.

58. Use according to embodiment 56, wherein said use comprises boiler technology, such as grate boiler or oil boiler.

59. Use according to embodiment 58, wherein a conventional boiler can be used, or wherein a conventional boiler has to be adapted, modified and/or adjusted.

60. A system comprising means for providing a vinasse according to any one of the preceding embodiments.

61. A system according to embodiment 60, said system comprising a boiler and/or fluidized bed means for burning and/or gasifying a vinasse and/or vinasse-comprising fuel.

62. A system according to embodiment 61, wherein said boiler and/or fluidized bed means is a boiler and/or fluidized bed means according to any one of embodiments 57 - 59.

63. A system according to any one of embodiments 60 - 62, wherein said vinasse-comprising fuel is a fuel according to any one of embodiments 32-55.

64. A system according to any one of embodiments 60 - 63, said system comprising means for providing energy.

65. A system according to any one of embodiments 60 - 64, said system comprising a combined heat and power plant (CHP).

66. A system according to any one of embodiments 60 -65, said system providing electricity, heat, steam and/or hot water to a biomass processing plant.

67. A system according to any one of embodiments 60 - 66, said system comprising means for providing 1st and/or 2nd generation bioethanol.

68. A system according to any one of embodiments 60 - 67, said system comprising means for pre-treatment, hydrolysis, removal of water, and combustion means, such as a system disclosed herein in e.g. Figures 31-36 and/or Figures 31B-36B, optionally including the hereto corresponding figure legends and embodiments.

69. A system according to any one of embodiments 60 - 68, wherein essentially all lignin provided is burned, such as burned as a solid lignin biofuel, as lignin-comprising fuel, and/or as lignin-rich vinasse.

70. A system according to any one of embodiments 60 - 68, said system providing a surplus of lignin, such as a lignin biofuel.

71. A system according to any one of embodiments 60 - 70, said system not comprising means for anaerobic digestion, such as means for methan/biogas production from stillage, such as whole or thin stillage.

72. A system according to any one of embodiments 60 - 71, said system comprising or not comprising anaerobic digestion means for treatment and/or purification of condensates, such as condensates from one or more water removal steps from one or more of e.g. lignin, the lignin-rich fraction provided in step (vi) of embodiments 1-20, stillage, vinasse, the lignin-depleted and/or lignin-rich vinasse provided in step (vii) of embodiments 1-20.

Without wanting to be bound by any theory, it is believed that the present invention provides advantages like:

- Improved conversion of energy and CO₂ footprint, as unconverted organic biomass in a biogas process will be combusted in to energy and thereby more energy will be produced per ton of incoming biomass.
- reduced water consumption as salts are found in a dry ash. In a biogas process salts will be in a water solution and this water will leave the process.
- more robust process/ increased operational reliability, e.g. by being independent from anaerobic digestion, and using simpler and more robust unit operations such as water removal and combustion. Furthermore, inhibiting compounds produced in pretreatment will not influence combustion but could influence microorganisms in the anaerobic digestion/biogas process. Potential variations in e.g. biomass (sources, seasonal variations, process parameter changes and the like) are more likely to influence anaerobic digestion than combustion.

- faster process time as combustion will take place in minutes while a biogas process has a residence time of 20-30 days.
- less waste – as e.g. no digestate from aanaerobic digestion/biogas production is produced.
- 5 • no digestate from biogas production is an advantage as undegradable organic matters produced in pretreatment do not have to be identified and verified for bringing out on farm land.
- shorter waterloop/recirculation, as water is not kept for e.g. 20-30 days in the biogas process.
- 10 • reduced capital expenditures (CAPEX), direct vinasse combustion reduces capital cost of the plant because the biogas plant with many days of residence time is excluded from the process. All other process groups are almost unchanged.
- reduced operating expenditures (OPEX), shorter processing time of the whole-
15 and/or thin stillage leads to energy savings.
- reduced down time, the solution with direct vinasse combustion is very robust and not sensitive to variations in the upstream of the process do not lead to down time in the process.
- reduced overall energy demand, is achieved as the process time is reduced
20 and electricity consumption decreases.
- reduced external energy demand, is achieved as the overall plant energy consumption is improved
- increased productivity,
- increased flexibility, the proposed system is less sensitive to changes in the
25 biomass feedstock and/or upstream process variations, and therefore very flexible when feedstock or parameters in to the plant changes

when compared to a set-up comprising a biogas plant for stillage treatment.

Further embodiments relating to the present invention are presented in the following section.

30 **EXAMPLES**

Unless indicated otherwise in the Examples “%” is to be understood as “% (w/w)”.

In some examples, the term "lignomulsion" is used for fluid compositions comprising lignin and/or a lignin component, such as for fluid compositions according to the various aspect of the present invention. Generally, and except when indicated otherwise, the lignin/lignin component samples used in this section were obtained from a second generation (2G) bioethanol manufacturing plant subjecting wheat straw to a hydrothermal pretreatment followed by an enzymatic hydrolysis, usually without addition of acids under pretreatment.

Example 1

This example illustrates a preliminary experiment relating to the manufacture of a fluid composition according to a first aspect (A1) of the present invention.

A lignin component obtained from a second generation bioethanol manufacturing plant subjecting wheat straw to a hydrothermal pretreatment followed by an enzymatic hydrolysis was comminuted, dried and grinded in order to obtain a powder. The lignin component had a dry matter content of 95 - 97%.

This lignin component was exposed to moisture by wetting in order to obtain a lignin component having a dry matter content of 65 % so as to mimic the wet lignin component originally obtained in the manufacturing process.

144.06 g of this lignin component together with 19.66 g diesel and 35.26 g water and 1.0 g Lutensol AP 10 dispersing agent from BASF was used for this fluid composition.

In a separate container 35.26 g water, 19.66 g diesel oil and 1 g Lutensol AP 10 was homogenized using the Ultra Turrax high speed mixer mixing at 10,000 min⁻¹ for 5 min.

The lignin/water mixture was added to the homogenized diesel/water/dispersing agent mixture and homogenized at 10,000 min⁻¹ for 5 min using the Ultra Turrax mixer.

A stable, viscous, liquid substance was obtained. The viscosity of the resulting fluid composition was measured using a "Stresstech HR" apparatus from Reologica Instruments AB, Sweden, in "Cup and Bob CC25" configuration.

Measurements were performed at the shear rates: 50, 100, 150, 200 and 250 min⁻¹ at 25 °C. The viscosity was measured at these shear rates to be 0.33 Pa.s, 0.19 Pa.s, 0.16 Pa.s, 0.14 Pa.s, and 0.12 Pa.s, respectively.

Over the one week observed, the resulting fluid composition showed to be stable without any significant separation of diesel, water or lignin.

Example 2

This example illustrates a second preliminary experiment relating to the manufacture of a fluid composition according to the first aspect (A1) of the present invention.

5 Example 1 was repeated with the same ingredients in the same amounts with the exception that in example 2 all the ingredients were mixed together.

The resulting fluid composition resembled that of example 1 with respect to stability and viscosity.

Example 3

10 This example illustrates a third preliminary experiment relating to the manufacture of a fluid composition according to the first aspect (A1) of the present invention.

Example 1 was repeated with the same ingredients in the same amounts with the exception that in example 3 no dispersing agent was used.

The resulting fluid composition resembled that of example 1 with respect to stability and viscosity.

15 The examples show that it is possible to obtain a stable fluid composition according to the first aspect (A1) of the present invention starting from a lignin component originating from a biorefinery of a lignocellulosic biomass and using diesel as the organic substance of the liquid fraction of the fluid.

20 Most surprisingly, the examples demonstrate that it is possible to obtain a stable fluid composition according to the first aspect (A1) of the present invention starting from a lignin component originating from a biorefinery of a lignocellulosic biomass and using diesel as the organic substance of the liquid fraction of the fluid and without any inclusion of a dispersing agent.

Example 4

25 This example reports studies with Indulin-containing compositions reported in examples from US5,478,366.

205 g of Indulin ATTM, 225 g water, 75 g diesel oil, and two two dispersing agents from BASF: 1.0 g Lutensol AP8 and 1.0 g Lutensol AP10, were used for this fluid composition.

30 In a container, 1.0 g Lutensol AP8, 1.0 g Lutensol AP10, 225 g water and 75 g diesel oil were homogenized with the Ultra Turrax mixer for 5 min at 10,000 min⁻¹. 205 g of Indulin ATTM were added to this mixture in five portions of approximately 40 g; after the addition of each portion the mixture was homogenized with the Ultraturax for 1

min at 10,000 min⁻¹. After the last portion, the mixture was homogenized with the Ultraturrax for 10 min at 20,000 min⁻¹.

The product obtained solidified rapidly. A ball formed by manually rolling the sample remained 3-dimensionally stable for several hours.

- 5 Immediately after homogenizing, the substance was transferred to a Haake ViscoTester VT550 apparatus with a MV-DIN measuring geometry and viscosity was measured over time at shear rate 100 s⁻¹. After 29.4 minutes, viscosity reached 2.1 Pa s, which is the limit of the instrumental capacity, and the apparatus stopped. This duration is defined as the "Stability Index".
- 10 205 g of a lignin component obtained from a second generation bioethanol manufacturing plant (2G lignin, same material as in Example 1), 225 g water, 75 g diesel oil, and two two dispersing agents from BASF: 1.0 g Lutensol AP8 and 1.0 g Lutensol AP10, were used for a similar fluid composition. The components were mixed as described above, and viscosity was measured. The measurement ran for
15 more than 3000 minutes. The viscosity did not reach the limiting value of 2.1 Pa s, and the measurement was stopped manually.

- 20 205 g of Indulin ATTM, 225 g water, 75 g diesel oil, two dispersing agents from BASF: 1.0 g Lutensol AP8 and 1.0 g Lutensol AP10, and 5-50 g glucose (supplied by Merck) were used for a similar fluid composition. The components were mixed as described above, and viscosity was measured. The measurement ran for 75.0-86.3 minutes, as
seen in the table below, before reaching the limiting value of 2.1 Pa s, causing the measurement to stop.

- 25 Finally, Indulin ATTM was washed with 1 M HCl solution and filtered. The filter cake was washed with water, until pH of the filtrate was above 5. The filter cake was then dried. 205 g of this substance was then mixed with 225 g water, 75 g diesel oil, two dispersing agents from BASF: 1.0 g Lutensol AP8 and 1.0 g Lutensol AP10. The components were mixed as described above, and viscosity was measured. The measurement ran for 856 minutes, as seen in the table below, before reaching the limiting value of 2.1 Pa s, causing the measurement to stop.

- 30 All results are summarised in the table below

Experiment	Lignin type	Lignin g	Oil g	Water g	AP8 G	AP10 g	Glucose g	Stability index*
1	Indulin	205	75	225	1	1	0	29.4
2	2G lignin	205	75	225	1	1	0	< 3000
3	Indulin	205	75	225	1	1	5	75.4
4	Indulin	205	75	225	1	1	25	75.0
5	Indulin	205	75	225	1	1	50	86.3
6	Indulin, acid treat	205	75	225	1	1	0	856

As shown, the stability of fluid compositions including Indulin AT™ is much poorer than comparable fluid compositions comprising 2G lignin.

See Example 17 for further detail.

5

Example 5

30 g of Indulin AT™ was mixed with 120 g of a 23 (w/w)% KCl solution. The solution was homogenized with the Ultra Turrax 30 s at ~10,000 rpm. It was shaken for more than one hour at room temperature. The mixture was filtered and the filter cake was washed four times with 4x100 ml water. The filter cake was then dried at 50 C, and the contents of potassium and chloride was measured.

30 g of Alkali lignin (Aldrich, product 471003) and two 30 g samples of lignin obtained from a second generation bioethanol manufacturing plant (2G lignin, as in Example 1) were treated in the same way as above.

Before the KCl treatment, all samples had similar K levels of 0.1-0.2%. However, after KCl treatment, Indulin AT™ and the Alkali lignin from Aldrich had a much higher K content (1.84 and 1.30%, respectively), while the 2G lignin sample had significantly lower K content (0.136 - 0.145%).

This clearly demonstrates the enhanced ability of Kraft lignin to bind K, relative to Inbicon 2G lignin, caused by an increased number of hydrophilic, polar functional groups in Kraft lignin.

The number of these groups can be estimated by calculating the so-called Lignin Ion Exchange Capacity (LIEC), here defined as the number of moles of potassium bound to lignin per kilo sample (unit: mol K/kg sample). This parameter has been calculated and is also given in the table below:

Short name	K1 %	Cl1 %	K2 %	Cl2 %	LIEC mol/kg
Indulin AT™	0.2265	0.0143	1.8424	0.0213	0.471
Alkali lignin	0.1645	0.0082	1.2969	0.0193	0.332
2G lignin 1	0.2286	0.0361	0.5318	0.0178	0.136
2G lignin 2	0.1358	0.0383	0.5672	0.0147	0.145

The low LIEC of 2G lignin demonstrates its low polarity and low hydrophilicity, compared to Alkali lignin and Indulin AT™, is the reason for the high stability of 2G lignin (as demonstrated in Example 4).

Example 6

- 5 Two different mixtures containing lignin (L) from a second generation bioethanol manufacturing plant (2G lignin, as in Example 1), diesel oil (O) and water W were prepared with the following mass percentages: L:O:W 38:30:32 and 48:20:32, also containing 0.5% sodium benzoate and 0.5% Lutensol AP10, supplied by BASF.

10 The first formulation (38:30:32) was sieved through mesh 0,5 mm. The second and third formulations (48:20:32 and 50:30:20) was sieved through mesh 1 mm.

Each fuel composition was placed in a 1-litre container, and using pressurized air (8 bar) it was ejected through a nozzle (either a flat jet nozzle or full cone nozzle) into a combustion chamber and ignited.

After the initial ignition, the fuel was burned independently with a stable flame.

15

Example 7

A long range of different hydrotropic and surface active components were tested formulations containing 40(w/w)% lignin (2G lignin, same as in Example 1), 20(w/w)% diesel oil and 40 (w/w)% water. The additives were present in the concentration 0.1-
20 1%. Many of additives were able to lower viscosity at shear rate 100 s⁻¹. The successful ones among the hydrotropes included lignosulphonate, Pluronic PE 6800¹, Sokalan PA 20 and PA40², Sokalan CP10 (provided by BASF), sodium benzoate, sodium p-toluenesulphonate, sodium benzoate, methylparaben, propyl paraben, glucose and butyldiglycol-

- 25 The successful ones among the surface active compounds included the Lutensol AP, XP³, TO⁴ and ON⁵ series from BASF.

¹ Block copolymers in which the central polypropylene glycol group is flanked by two polyethylene glycol groups.

² Polyacrylates; their average K value (molar mass) is indicated by the numeric code.

- 30 ³ Alkyl polyethylene glycol ethers based on C10-Guerbet alcohol and ethylene oxide, manufactured by reacting the C10-alcohol with ethylene oxide in stoichiometric proportions. The numeric portion of the product name indicates the general degree of ethoxylation.

⁴ RO(CH₂CH₂O)_xH, where R = iso-C₁₃H₂₇ and x = 3, 5, 6, 6.5, 7, 8, 10, 12, 15 or 20

⁵ RO(CH₂CH₂O)_xH, where R = saturated, synthetic, short-chain fatty alcohol, x = 3, 5, 6, 6.5, 7, 8 or 11

- 5 Adding 0.5% of either sodium benzoate, methylparaben or propylparaben to a mixture containing 40 g diesel oil, 80 g water and 80 g 2G lignin was tested as possible preserving agent to avoid microbial activity. All three candidates showed no signs of microbial activity, even after several months of storage at room temperature.

10 Instead of diesel oil, different other oils were tested in mixtures containing 80 g lignin, 80 g water and 40 g oil (with the addition of 1 g Lutensol AP10 and 1 g sodium benzoate). The oils include rapeseed methyl ether (supplied by Emmelev), pyrolysis oil from pyrolysis of wood, unrefined palm oil, unrefined rapeseed oil and a mixture of diesel oil and heavy fuel oil. Measurements of these mixtures were performed at the shear rates: 50, 100, 150, 200 s⁻¹ at 25 °C. The viscosity was measured to be in the
15 range 0-0.3 Pa s.

Over the one week observed, the resulting fluid compositions were stable without any significant separation of diesel, water or lignin observable.

20 Pre-drying of 2G lignin appears advantageous. Preparing a mixture from lignin filter cake (with a dry matter content of 59%), composed of 90 g lignin, 30 g oil and 180 g water (with 1.5 g sodium benzoate and 1.5 g Lutensol AP10) resulted in a viscosity at 10 s⁻¹ of 0.67 Pa s. However, this is still well below the viscosity measured for fluids containing Indulin AT as the lignin source, and over one week observed, the resulting fluid composition showed to be stable without any significant separation of diesel, water or lignin.

- 25 A relation has been observed between the input energy of the ultra turrax (T25) used for homogenizing and the viscosity of the resulting fluid. The input energy is a combination of the duration and speed of the ultra turrax and was determined by measuring the power consumption of the ultra turrax. See Example 23 for further details.

30 **Example 8**

Ignition and combustion properties of five different formulation droplets were tested under well-controlled conditions in high-temperature suspension-fired boilers. Samples were prepared with lignin from a second generation bioethanol manufacturing plant (2G lignin, as in Example 1), water, diesel oil, unrefined palm oil
35 and fuel oil (according to the table below).

Table 1: Overview of sample compositions tested.

DONG label	2G Lignin	Water	Diesel	Palm oil	Fuel Oil
001	45 %	38 %	16 %	0 %	0 %
002	45 %	38 %	0 %	16 %	0 %
003	32 %	57 %	10 %	0 %	0 %
004	52 %	47 %	0 %	0 %	0 %
005	45 %	38 %	8 %	0 %	8 %

Samples (10 mg) placed on a sample holder were inserted into the reactor while shielded by a quartz tube. The tube was subsequently removed and the sample conversion/behavior followed by a high speed camera.

Delay before ignition and pyrolysis time were measured at three different conditions:

- 5 **A:** 1200 °C, 5.5 O₂ and v = 1.6 m/s (standard)
 B: 1200 °C, 2.9 O₂ and v = 1.6 m/s (low oxygen concentration)
 C: 990 °C, 5.5 O₂ and v = 1.6 m/s (low temperature)

Results are summarized below

Label	A		B		C	
	Delay + ignition [ms]	Pyrolysis (10 mg) [ms]	Delay + ignition [ms]	Pyrolysis (10 mg) [ms]	Delay + ignition [ms]	Pyrolysis (10 mg) [ms]
Diesel	60± 15	1290± 230	-	-	-	-
Fuel oil	125± 70	2490± 290	115± 55	2685± 200	410± 175	3670± 365
001	350± 245	4335± 330	380± 105	3945± 45	2870± 835	3840± 570
002	1055± 495	2780± 460	-	-	3135± 610	2960± 835
003	835± 450	3010± 245	-	-	4120± 1340	3055± 760
004	960± 580	2585± 600	-	-	-	-
005	565± 520	3505± 410	-	-	-	-

10 ^{*1} Total time = Delay + ignition + pyrolysis

Different experiments are also summarized in Figure 1.

15 Compared to fuel oil and diesel, the lignomulsion samples are associated with a longer ignition delay, relatively unaffected by a decreased oxygen concentration (from 5.5 % to 2.9 %), while it increases significantly with decreasing temperature. The difference in ignition behavior compared to fuel oil may be connected to water evaporation from the lignin samples. No clear connection between sample mass and

ignition delay/no stable flame is seen. The swiftest ignitions were observed for fuel oil, samples 001 and 005. Samples 002, 003 and 004 performs worst in terms of ignition delay, indicating a positive influence of diesel/oil and low water content.

Following the ignition phase, a stable flame is formed by combustion of pyrolysis gases. The pyrolysis time of 10 mg droplets (app 2.7 mm diameter) ranged from 2585 to 4335 ms at 1200 °C and 5.5 % O₂, which is similar or slightly higher than the fuel oil (2490 ± 290ms). This is surprising, considering the lower heating value of the lignin slurry samples (9.7 to 15.4 MJ/kg) compared to fuel oil (40 MJ/kg).

The total conversion time (delay + ignition + pyrolysis) at 1200 °C and 5.5 % O₂ were generally higher for the lignin slurry samples compared to fuel oil.

Example 9

Materials and experimental

Essentially, lignomulsion consists of three components: Lignin (L), Oil (O) and Water (W). Furthermore, additives capable of lowering viscosity and enhancing stability should be added. These components are all briefly described below

9.1 Lignin

Three different types of lignin were used: 1) Filter cake/centrifugate; 2) Dried, grinded lignin pellets (both supplied by Inbicon) and 3) Commercially available Kraft lignin.

As a standard case, dried lignin pellets were used.

9.1.1 Filter cake

When lignomulsion is to be produced on large scale in the future, it appears advisable that the lignin source is provided through a 2G process, where lignin is isolated by pressing or centrifuging it to a dry matter (DM) content of 50-60%. Such material, referred to as filter cake, has been used in some experiments.

However, storage of large quantities of this material for laboratory test is difficult, as it should be in a freezer to avoid microbial activity. When using filter cake, this material was very tough and hard to break into pieces small enough for suspensions. However, the combination of the blade of a Kenwood machine and an Ultra Turrax did the job, at least on the scale of some kilo. Pictures of lignin filter cake before and after this treatment is shown in Figure 9-1. Particle size of the cut material obtained in this way was studied. This is further described in the following.

Additionally, it should be mentioned that lignin from the 2G Demonstration plant at Kalundborg has sometimes not been pressed to a filter cake but is delivered as centrifugate with a DM of 30-40%. This was highly viscous and not pourable, in spite of the high water content, and appeared less suitable for lignomulsion.

9.1.2 Lignin pellets

Lignin pellets with DM 95% is much less susceptible towards microbes and can be stored at room temperature. In a few case, microbial attacks on lignin pellets/granulate were observed, though.

- 5 Before use, the pellets were grinded to a fine powder (using large scale milling equipment) and sieved (mesh size ~150µm).

9.1.3 Kraft lignin

Two types of commercially available lignin were tested: Indulin AT (supplied by WestVaco) and Alkali Lignin (Sigma-Aldrich, item 249330).

10 9.2 Oil

Different oils have been used: Diesel oil (bought at Q8), heavy fuel oil (only used in a mixture with diesel), pyrolysis oil (from wood), biodiesels (from Emmelev and Daka), unrefined palm oil and rapeseed oil

9.3 Water

- 15 Cold tap water was used to prepare Lignomulsion

9.4 Additives

- A range of additives were used; these can be divided into surfactants and hydrotropes. Surfactants and hydrotropes both contain a hydrophilic end and a hydrophobic end, allowing them to interact with both hydrophilic and hydrophobic compounds. In a water-oil emulsion, this increases the interactions between the two phases, enhances stability and lowers viscosity. The main difference between a surfactant and a hydrotrope is that in the hydrotrope, the contribution of the hydrophobic part is quite small, and the beneficial effects on stability and viscosity are more modest.
- 20

9.4.1 Surfactants

- 25 All tested surfactants were supplied by BASF. The preferred surfactants belonged to the class called Lutensol, ie ethoxylated nonylphenols, see an example figure 9-2.

9.4.2 Hydrotropes

- Sodium benzoate was originally added to lignomulsion as a preserving agent, but testing showed it to have an effect on viscosity as well, identifying it as a hydrotrope. Several other hydrotropes (some commercially available, and some new products supplied by BASF) were tested.
- 30

9.5 Preparing emulsions

- In general, emulsions were prepared by dissolving the hydrotrope in the water and mixing the surfactant in the oil phase (using the ultraturrax for some seconds). The two liquid phases when then mixed, and the lignin phase was added.
- 35

When using grinded, dry pellets, in most cases the lignin was first mixed with water in a 65:35 ratio, before the oil/water phases were added. When using Kraft lignin, all the liquid

components were mixed, and lignin was added in small portions while using the Ultra Turrax. This was necessary to keep the viscosity down.

Viscosity was measured with a Haake ViscoTester VT550. Usually, the instrument was set to measure at shear rates 50, 100, 150 and 200 s^{-1} and at the following temperatures: 298, 318, 338 and 358 K (25-85 °C). Viscosities were measured of different LOW formulations:

1. Formulations with different relative amounts of lignin, oil and water, either with or without the presence of a hydrotrope and/or surfactant.
2. Formulations where different additives (surfactants and hydrotropes) were tested.
3. Formulations with alternative preserving agents, ie parabens.
4. Formulations with different oils, including vegetable oil, biodiesel, pyrolysis oil and fuel oil
5. Formulations with Kraft lignin
6. Formulations with lignin from pellets compared to wet lignin filter cake
7. Formulations with lignin filter cake dried under different conditions
8. Formulations using different types of dried lignin pellets/granulate
9. Formulations using lignin with different sugar contents
10. Formulations with acid washed lignin
11. Formulations prepared with different ultra turrax energy input
12. Formulations stored for different periods of time

These experiments are further described in the following examples.

Example 10

Differences in formulation

To test the importance on the relative contents of lignin, oil and water on viscosity, a number of different Lignomulsion formulations were prepared with a lignin content of 30-55%, and oil content of 0-30%, a water content of 30-55%, and either with or without 5000 ppm sodium benzoate and Lutensol AP10. The presence of these two additives influenced the resistance towards microbial activity as well as lowered viscosity and diminished phase separation.

Figure 10-1a shows viscosity of the formulations *without additives* measured at room temperature at the four different shear rates. Please note that viscosity of LOW 50-20-30 was not measured, since it was too viscous for the range of the available apparatus. In all formulations, viscosity decreases with increasing shear rate, showing Lignomulsion to be a non-Newtonian shear-thinning fluid.

The only exception is the LOW 47-20-33 formulation, which is highly viscous ($> 1 \text{ Pa s}$). The irregularities at high shear rate is therefore ascribed to uncertainties in the measurements.

Figure 10-1b shows viscosity of the formulations without additives measured at shear rate 100 s^{-1} for four different temperatures. Different temperature effects are observed, depending on the formulation:

Formulations containing 20-30% oil show a decrease in viscosity with increasing temperature. This is the most common temperature behaviour in dispersions, suspensions and emulsion, as structuring elements are generally broken down and internal friction decreases at higher temperatures. However, for formulations with 0-10% oil viscosity increases with increasing temperature. When viscosity decrease, it could be an effect of intermolecular structures being built up in the dispersion, or it could be an effect of components of the liquid phase being absorbed by the dispersed phase. In this case, a possible explanation could be that water is taken up by the large structures of the lignin molecules. As lignin contains many hydrophobic groups, this process is facilitated by the presence of the additives, and it should also be enhanced by increasing temperature. The reason why this effect is only seen for small oil contents could be that oil can interfere with these lignin-water interactions, for example by physically or chemically "blocking" potential sites for hydrogen bonding.

In Figure 10-2, the effect of additives is shown by comparing identical LOW formulations, either with or without sodium benzoate and Lutensol AP10. All measurements were performed at 100 s⁻¹.

For all emulsions with a low oil content (0-10%), the effect of additives was modest, as seen in Figure 10-2a and b. At room temperature, the additives had no significant effect on viscosity in the 10% oil case and even increased viscosity in the 0% oil case. These observations are also valid at shear rate 50, 150 and 200 s⁻¹ (data not shown). This is not surprising; the additives are aimed at optimizing the interactions between water and oil and obviously do not work in dispersions with no oil. In the dispersions with only 10% oil, it is likely that lignin - containing both hydrophobic and hydrophilic functional groups and therefore acting as a hydrotrope - was able to act as an adequate emulsifier.

One effect of additives in the low oil formulations is, however, that while increases in viscosity as a function of temperature were observed in formulations without additives, even higher increases are observed for formulations with additives. This observation is in full correspondence with hypothesis described above, since at high temperature lignin restructuring will expose more sites for hydrogen bonding, and the additives facilitates interactions between lignin and water.

For higher oil contents, the additives efficiently reduces viscosity, in some cases with more than 80% (for example LOWs 47:20:43 and 45:20:45 at room temperature). On the other hand, the slight increases in viscosities are observed with increasing temperature. This is contrary to the behaviour observed in emulsions with no additives, and it means that in many cases the viscosity lowering effect of the additives is matched by this temperature effect at 65-85 °C. For emulsions with the lowest lignin content, temperature effects do not seem significant, but in the emulsions with the highest lignin contents (ie 40:30:30, 50:20:30 and 47:20:33) viscosity at high temperature is > 0.3 Pa s, which would make pumping difficult.

Again, the explanation for the temperature effect is that water is transferred from the liquid phase to the lignin phase due to interactions with the hydrophilic groups of lignin. Higher temperature allows water to access more hydrophilic sites within the lignin structure, and

while the high oil content may block or restrict some of these sites, the additives diminished this effect by increasing the favourable interactions between oil and water. Furthermore, the additives may even make it possible for oil to be bound to the hydrophilic groups of lignin - which further transfers mass from the liquid to the dispersed phase, also resulting a lowering of viscosity.

EXAMPLE 11

Surfactants and hydrotropes

In general, surfactants and hydrotropes facilitates interactions between lignin, water and oil making the emulsions less viscous. The favoured additives of lignomulsion include one hydrotrope and one surfactant. The initial choices were sodium benzoate and Lutensol AP10 (note that sodium benzoate was initially chosen because of its anti-microbial properties, with it hydrotropic properties as an added benefit), but several other additives were also tested.

First, the effects of adding AP10 and sodium benzoate in various amounts were evaluated by preparing formulations as shown in Table 11-1.

Table 11-1: Emulsions for studying the effect of additive concentration

LOW	[AP 10] (w/w)%	[NaBenz] (w/w)%	Viscosity (25 °C; 100 s ⁻¹) Pa s
40-20-40	0	0	0.404
40-20-40	0.5	0	0.177
40-20-40	0.5	0.1	0.105
40-20-40	0.5	0.5	0.0650
40-20-40	0.5	1	0.0617
40-20-40	0	0.5	0.335
40-20-40	0.1	0.5	0.322
40-20-40	0.4	0.5	0.188
40-20-40	1	0.5	0.0575

Figure 11-1 shows how viscosity at room temperature and shear rate 50-200 s⁻¹ depends on the concentrations of sodium benzoate and Lutensol AP10, respectively, in a LOW 40-20-40 mixture. This formulation was chosen because additives were found to have large effect on formulations with at least 20% oil, and the formulation with 40% lignin resulted a thin, usable, pumpable liquid when additives were present while still having measurable viscosity without the additives.

Both additives were found to independently lower viscosity. At shear rate 100 s⁻¹, in the case of 5000 ppm sodium benzoate, a reduction of 17% (from 0.40 to 0.34 Pa s), and for 5000 ppm Lutensol AP10 a reduction of 56% (from 0.40 to 0.18 Pa s) were observed. However, in combination (ie 5000 ppm of each) sodium benzoate and Lutensol AP10 lower viscosity by 84% (from 0.4 to 0.065 Pa s), indicative of a synergistic effect between the two additives. There is a clear connection between the concentration of each additive and the decrease in

viscosity, at least up to a concentration of 5000 ppm for the studied formulation of LOW 40-20-40.

Figure 11-2 shows how viscosity at shear rate 100 s^{-1} and temperature $25\text{-}85^\circ\text{C}$ depends on the concentrations of sodium benzoate and Lutensol AP10, respectively, in a LOW 40-20-40 mixture. Adding 5000 ppm sodium benzoate and only 0 or 1000 ppm AP10 does not change the temperature effect observed in Example 10. For these formulations, viscosity decreases as a function of temperature - as was also observed for formulations with no additives present. It is therefore clearly Lutensol AP10 that is responsible for the increase in viscosity as a function of temperature observed for formulations with an oil content of 20-30%

It is also interesting to observe that while viscosity decreases with temperature when no additive is added, the opposite effect is observed when either one or both additives are present, and that this temperature become more pronounced when the amount of additive is increased.

A theory is that the 3D polymeric structure of lignin unfolds and expands when heated. This exposes more sites for H-bonding with water; this process is facilitated and strengthened by the hydrotrope or surfactant additives. Further experiments indicates that this process is to some extent irreversible, as the water partially remains trapped within the lignin structure, even after cooling to room temperature.

Since the viscosity increasing as a function of temperature is not a desired property, several other surfactants and hydrotropes were investigated. A long range of hydrotropes were obtained either from commercial sources (sodium xylanesulphonate, lignosulphonate, sokalan, sodium benzoate, sodium p-toluenesulfonate, BDG and glucose) or from other sources.

As seen in Figure 11-3a, several hydrotropes effectively lower viscosity, but sodium benzoate has added advantage of being a preserving agent. Figure 11-3b shows that several surfactant are as efficient at lowering viscosity as Lutensol AP10 and therefore, other choices could be made. Also, notice that for all efficient hydrotropes and surfactants, viscosity increases as a function of temperature.

Example 12

Parabens

Sodium benzoate is the preferred preserving agent in Lignomulsion, but parabens were studied as alternatives. A generic structure of a paraben is shown in Figure 12-1; in this study -R was either methyl- or propyl.

The potential disadvantage with parabens (ie esters of parahydroxybenzoic acid) is that they are only antimicrobial in the acid form. Above pH 6 the acid is converted to a salt, which is totally inactive. As none of the components in Lignomulsion have acidic properties, this is problematic. Also, parabens function only in the water phase - if they are extracted to the oil phase of lignomulsion, they will become inactive.

In all formulations presented in this section, it was necessary to heat water to more than 60 °C and stir for several minutes before the entire amount had been dissolved. Hopefully, it did not precipitate when the other ingredients were added, but due to the dark colour of Lignomulsion there was no way to actually ascertain this.

- 5 The advantage is that parabens are active when used in much smaller quantities than sodium benzoate.

Two parabens, methyl- and propylparabene, were used to prepare six different formulations, as specified below in Table 12-1.

Table 12-1

Parabene ID	Parabene amount	Lignin	Oil	W	AP10	<i>Masses added / g</i>				
						<i>Parabene amount</i>	<i>Lignin</i>	<i>Oil</i>	<i>W</i>	<i>AP10</i>
Methylparabene	0.05	40	20	40	0.5	0.1535	185.6	60	55	1.5
Propylparabene	0.05	40	20	40	0.5	0.1499	185	60	55	1.5
Methylparabene	0.01	40	20	40	0.5	0.0323	185.1	60	55	1.4951
Propylparabene	0.01	40	20	40	0.5	0.0319	185.5	59.9	55.8	1.4927
Methylparabene	0.005	40	20	40	0.5	0.0157	185.7	60.1	55.2	1.4988
Propylparabene	0.005	40	20	40	0.5	0.0149	185.2	60	55	1.4932

10

- Figure 12-1a-b shows that the presence of a paraben lowers viscosity; i.e. it acts as a hydrotrope, and at temperatures above room temperature, it is even more efficient than sodium benzoate (which, as was shown above, is among the most efficient hydrotropes). However, there does not seem to be a straightforward relation between the added paraben quantity and viscosity suppression. This could be due to the difficulty with dissolving paraben in water - possibly in some formulations, the entire amount of added parabens was not dissolved.

15

As a preserving agent, parabens also seem efficient in suppressing microbial activity.

Example 13

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Different oils

As a general case, diesel oil is used in Lignomulsion. However, other oils have been tested, including biodiesel (from either fish products or rapeseed) and vegetable oils (sunflower oil, unrefined palm or rapeseed oil and oil from wood pyrolysis), see Table 13-1. Other oils are believed to be suitable, too.

25

Table 13-1: Oils used in Lignomulsion formulations

Type	Source/producer	Sample ID	LOW formulations*	Viscosity** Pa s
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Fish diesel (Fish)	Pronova biopharma	131107_001	40-20-20	0.055
		131108_001	40-30-30	0.079
Heavy bio diesel, FAME (Daka)	Daka	NA	40-20-20	NA
		NA	40-30-30	NA
Rapeseed methyl ether (RME1)	Emmelev	131204_001	40-20-20	0.171
		131220_003	40-30-30	0.136
Rapeseed methyl ether, secunda (RME2)	Emmelev	131205_001	40-20-20	0.231
		131217_001	40-30-30	0.153
Sunflower oil (Sunflower)			40-20-20	
		131104_001	40-30-30	0.648
		130320_001	30-30-40	0.062
Pyrolysis oil (Pyro)	B21st, WP4	131115_001	40-20-20	0.217
		131115_002	40-30-30	NA
Unrefined palm oil (Palm)	Neste Oil	140502_001	40-20-40	
		140505_001	40-30-30	
		140505_002	45-15-40	
Rapeseed oil (RSO)	Neste Oil	NA	40-20-40	0.046
		NA	40-30-30	0.158
		NA	45-15-40	NA

* Formulations also contained 5000 ppm Lutensol AP10 and 5000 sodium benzoate

** Measured at room temperature and shear rate 100 s⁻¹

- 5 Figure 13-1 shows viscosity measured at 100 s⁻¹ and four different temperature for the two LOW formulations 40-20-40 and 40-30-30 (with 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10) using different oils. The viscosity of LOW 40-30-30 with pyrolysis oil was too high to be measured by the Viscotester, so results for this formulation are not available. Pyrolysis oil has a low pH which can result in corrosion of tanks and equipment (as well as high cost), and using this oil was never preferable for large-scale lignomulsion production.
- 10 Unrefined palm oil has a melting point close to (or slightly above) room temperature, and is semi-solid at room temperature. It produces formulations which are quite viscous at room temperature. The decrease in viscosity from 25 °C and 45 °C observed in both of the displayed palm oil formulations is therefore partially due to a softening/melting of the oil.

The viscosities of formulations prepared with DAKA FAME and Emmelev RME1 and -2 do not change significantly with temperature, whereas viscosities of formulations prepared with unrefined rapeseed oil and the fish diesel increase with increasing temperature; similar to what was observed for conventional diesel oil.

- 5 Except for the pyrolysis oil and palm oil at room temperature, replacing conventional diesel with biodiesel or bio-oil seems perfectly possible from a viscosity point of view.

As unrefined palm oil or rapeseed oil has been suggested as the component in a "non-fossil" version of Lignomulsion, such formulations are studied further in Figure 13-2, including a third formulation, LOW 45-15-40. The formulations with 40-45% lignin and 15-20% palm oil
10 have viscosity $\sim 0.3 \text{ Pa s}$ at shear rate 100 s^{-1} and room temperature, with viscosity decreasing slightly when temperature is increased to 45°C (as mentioned above). When temperature increases further to 65 and 85°C , viscosity increases. This is most likely related to expansion of the lignin structure and subsequent water uptake, as also mentioned above.

15 In formulations with 40 % lignin, increasing palm oil content from 20% to 30% results in a massive viscosity increase. However, viscosity decreases as a function of temperature to an extent that viscosity of the LOW 40-30-30 formulation is actually lower than the LOW 40-20-40 formulation at 65 and 85°C .

Similar quantitative behaviours are seen in Figure 13-2 for the RSO formulations, except that RSO results in a much lower viscosities. This is probably because the RSO sample has a
20 lower melting point than palm oil and is liquid at room temperature.

EXAMPLE 14

Fuel oil

In a series of experiments, Lignomulsion formulations with a mixture of diesel oil (DO) and heavy fuel oil (FO) were studied, see Table 14-1.

25 Table 14-1: Formulations of lignomulsion prepared with diesel and fuel oil

ID	Lignin	OD:FO	Water	Sodium benzoate	AP10	Viscosity*
	%	%	%	Ppm	ppm	Pa s
130617_002	45	5:5	45	5000	5000	0.0549
130610_002	43	5:5	47	5000	5000	0.0578
130612_001	45	5.5:5.5	44	5000	5000	0.0728
130611_001	39	9:9	44	5000	5000	0.0391
130610_003	35	9:9	47	5000	5000	0.0420
130619_001	40	10:10	40	5000	5000	0.0575
130617_003	33	15:15	37	5000	5000	0.0537
130617_001	30	15:15	40	5000	5000	0.0338
130619_002	40	8:12	40	5000	5000	0.1143

130620_001	40	4:16	40	5000	5000	0.1642
130619_001	40	10:10	40	5000	5000	0.0575

* measured at room temperature and shear rate 100 s⁻¹

In Figure 14-1 and -2, viscosity as a function of shear rate and temperature, respectively are shown for Lignomulsion formulations prepared with a 1:1 mixture of diesel and fuel oil; these were compared to formulations containing only diesel oil. Note, that in Figure 14-1, the y-axis is logarithmic to better distinguish between the different data sets.

Replacing half of the diesel oil with heavy fuel oil (which is a highly viscous substance) significantly lowers viscosity of the formulation at all shear rates and all temperatures. As the most extreme example, in LOW 45-10-45 at shear rate 100 s⁻¹ and room temperature, replacing half of the diesel with heavy fuel oil, results in a 80% reduction in viscosity. However, the viscosity reduction is smaller for the other formulations.

In Figure 14-3, viscosity measurements are shown as a function of shear rate and temperature. The formulations all have 40% lignin, 40% water and 20% oil, with the ratio between diesel oil and heavy fuel oil varying between 4:16 and 10:10. For comparison, a formulation with no fuel oil (only diesel oil) is also shown. For the 40-20-40 formulation, replacing half of the diesel with fuel oil causes a small decrease in viscosity (of 17 % at room temperature and shear rate 100 s⁻¹), as also described above. However, increasing the amount of heavy fuel oil causes a significant increase in viscosity.

In conclusion; for a variety of different LOW formulations, it is possible to replace a large fraction of diesel oil with heavy fuel oil and still have thin, pumpable liquids. However, there appears to be some limit to how much of diesel it is possible to replace.

Example 15

Filter cake

Lignin filter cake has a water content of 40-50%. It is perfectly possible to prepare a thin, pumpable formulation of lignomulsion with a water content of less than 40% and a lignin content of more than 50%, when using grinded pellets. The fact that lignin filter cake is a hard and solid material gives the first indication that there may be some differences between filter cake and pellets/granulate.

Lignin filter cake is a hard material and is prepared for lignomulsion by cutting it into smaller pieces with the cutting function of the Kenwood machine. This results in a material with texture similar to earth, which can potentially be dried and milled to a fine powder.

Formulations were prepared using lignin filter cake from wheat straw treated at Inbicon in Kalundborg, Denmark, see Table 15-1. In some cases, lignin was dried at 50 °C to DM 82%.

Sample name	L	O	W	AP10	NaBen z.	Lignin %	DM /	Viscosity* / Pa s
130517_004	20	0	80	0	0	59.07 ± 0.04		0.099
130530_001	25	0	75	0	0	59.07 ± 0.04		0.33

130522_001	30	0	70	0	0	59.07 ± 0.04	0.91
130522_002	30	10	60	0	0	59.07 ± 0.04	0.76
130523_001	30	10	60	0.5	0.5	59.07 ± 0.04	0.91
130529_001	35	20	45	0	0	59.07 ± 0.04	NA
130611_003	16	0	84	0	0	Dried (82%)	0.011
130613_001	20	0	80	0	0	Dried (82%)	0.022
130604_002	25	0	75	0	0	Dried (82%)	0.068
130610_001	25	10	65	0	0	Dried (82%)	0.057
130611_002	25	10	65	0.5	0.5	Dried (82%)	0.095
130612_002	30	10	60	0.5	0.5	Dried (82%)	0.18
130614_001	30	10	60	0	0	Dried (82%)	0.55
130621_002	25	20	55	0.0	0.0	Dried (82%)	0.47
130628_001	25	20	55	0.5	0.5	Dried (82%)	0.13
130628_002	29	20	51	0.5	0.5	Dried (82%)	0.41

* viscosity at 25 °C and shear rate 100 s⁻¹

Working with not-dried lignin filter cake was difficult, as it resulted in highly viscous liquids, as is seen in Figure 15-1. For example, at shear rate 100 s⁻¹, LOW 30-00-70 has a viscosity of 0.91 Pa s - for comparison a LOW formulation of 55-00-45 (ie almost twice as much lignin!) made from grinded pellets has a viscosity of 0.44 Pa s.

When, instead, lignin filter cake was pre-dried at 50 °C to a dry matter content of 82%, the result was a completely different material, very suitable for preparing low-viscosity emulsions - also shown in Figure 15-1 (the difference between wet and dry lignin filtercake is further studied below). For example, the viscosity of LOW 25-00-75 formulation decreased from 0.33 Pa s when "not-dried" lignin filter cake was used - to 0.068 Pa s when instead using dried lignin filter cake, ie by almost 80%.

In Figures 15-2 and -3, the effect of lignin content is studied in the presence of 10% diesel oil when using dried filter cake, either with or without additives present. Again, an increase in lignin content results in an increase in viscosity. Also notice that in the absence of oil and/or additives, viscosity decreases with temperature (Figure 15-1 and 2), whereas in the presence of oil *and* additives, viscosities increases with temperature (Figure 15-3). This is similar to what was observed for grinded pellets.

The effect of increasing the oil content was studied for both wet and dried (ie DM 82%) filtercake (Figure 15-4), and an increase in oil content results in an increase in viscosity, similar to what was observed for grinded pellets.

The effect of adding sodium benzoate and Lutensol AP10 was studied in two cases with different LOW composition (30:10:60 and 25:10:65, respectively); see Figure 15-5. For the first composition (LOW 30:10:60; 15-5a and b), additives effectively decreased viscosity, whereas for the second composition (LOW 25:10:55; 15-5c and d) a slightly increase was observed when additives was added. Previously it was observed that for grinded pellets, an oil content of more than 10% was necessary to fully see the effects of additives.

Thus, using wet (and dried) filter cake is very similar to using grinded pellets in Lignomulsion, except that wet filter cake results in significantly higher viscosity formulations, and that lower lignin levels (and thereby lower fuel value) are needed to produce low viscosity liquids.

EXAMPLE 16

5 Optimizing the use of filter cake

Since the use of wet filter cake (or decanter cake) are very relevant with regard to the overall economy of the process, there has been focus on how much "not-dried" lignin it is possible to suspend in an oil/water emulsion and still have low viscosity, and a different series of experiments were carried out, this time using a different stock of lignin filter cake (Filterkage fiber stillage IKA; 2013-09-02) with DM 54%. The details are given below in Table 16-1 and Figure 16-1. First, a "standard" case was made with a LOW composition of 30:10:60 and with 5000 ppm of each of the additives Lutensol AP10 and sodium benzoate (additives 1A and 1B), mixed by using the ultraturrax at 10.000 rpm for 5 minutes.

The second sample had the same formulation, but the ultraturrax was only used for 1 minute. This did not change viscosity, and for the remaining samples, the ultraturrax was used for 5 minutes.

In the third sample, the concentrations of the additives were increased 5 fold, but this did not lower viscosity either.

In the fourth sample, different additives; RD193295 and Lutensol TO15 (called 2A and 2B) were used. From the results in Example 11, this was expected to lower viscosity. This was also the case.

In the fifth sample, concentrations of additives 2A and 2B were increased 5 fold, but with no decrease in viscosity compared to the fourth sample.

In the sixth sample, oil content was increased to 30% (by lowering water content), but this resulted in a fluid too viscous for measuring viscosity.

In the seventh sample, half of the (diesel) oil content was replaced by heavy fuel oil, which did not affect viscosity.

Finally, in the 8th sample a different lignin stock with a lower dry matter (DM 36,5 %) was used, resulting in a much higher viscosity. This was an example of decanter cake from Inbicon.

Table 16-1 List of formulations for optimisation the use of wet lignin filter cake in Lignomulsion

	Sample	L	O	W	Add. A	Add. B	Viscosity *	Comment
1	140326_001	30	10	59	0.5	0.5	0.67	Ultraturrax 5 min additive 1A.

2	140326_002	30	10	60	0.5	0.5	0.72	1B Ultraturrax 1 min; additive 1A. 1B
3	140326_003	29	10	59	1.2	1.2	0.64	Extra additive 1A. 1B
4	140326_004	29	10	60	0.5	0.5	0.55	Additive 2A. 2B
5	140328_001	29	10	58	1.3	1.2	0.52	Extra additive 2A. 2B
6	140328_002	29	29	40	1.2	1.2	NA	Different LOW; additive 2A. 2B
7	140328_003	29	10	58	1.2	1.2	0.57	1:1 diesel-fuel oil mixture. additive 2A. 2B
8	140411_002	29	10	59	1.2	1.2	1.25	Different lignin; additive 2A. 2B

* viscosity at 25 °C and shear rate 100 s⁻¹

In conclusion; wet filter cake is quite difficult to work with, but a formulation of LOW 30-10-60 seems to result in a thick, pourable liquid. Drying filter cake completely changes the material, making it possible to add more lignin and still obtain a thin fluid. The effect of drying is studied below.

Example 17

Comparison with Kraft lignin

Lignomulsion was prepared from Indulin (supplied by MeadwestVaco), according to Table 17-1.1

10 Table 17-1: Formulation of Lignomulsion prepared with Indulin (DM 98%), LOW 40-15-45

Sample ID	Lignin type	Lignin %	Oil %	Water %	AP8 %	AP10 %	Glucose %	TO15 %	RD193295 %	Stability index*
140528_001	Indulin	40	15	45	0.2	0.2	0	0	0	29.4
140604_001	Indulin	40	15	45	0	0	0	1	1	7.9
140603_001	Indulin	40	15	45	0.2	0.2	1	0	0	75.4
140617_001	Indulin	40	15	45	0.2	0.2	5	0	0	75.0
140616_001	Indulin	40	15	45	0.2	0.2	10	0	0	86.3
140617_002	Grinded pellets	40	15	45	0.2	0.2	0	0	0	< 3000

*Defined as the number of minutes at shear rate 100 s⁻¹ necessary for the viscosity to reach 2.1 Pa s.

Sample 140528_001 is a reproduction of an example from Patent WO96/10067 (Title: Lignin water oil slurry fuel); see the extract and link below:

Example 4

160 g of Terigtol™ NP-9 together with 35.2 kg of water and 12 kg of fuel oil No. 2 were homogenised in a tank to form an oil;-in-water emulsion. 32.65 kg of Indulin AT™ lignin (sold by Westvaco.) containing 2% by weight moisture was added gradually (in portions) to the oil-in-water emulsion and mixed under high shear conditions at a temperature between 18 and 25°C. The resultant stable slurry contained 40% lignin; 15% fuel oil no. 2; 44.8% water; and 0.2% (i.e. 2,000 ppm) surfactant. This produced an 80 kg sample for testing in a lime kiln.

Note that the "Terigtol NP" series are Nonylphenol ethoxylate of various molecular size, similar to the "Lutensol AP" series. Lutensol AP8 and AP10 were therefore mixed to obtain an additive with the same hydrophilic-lipophilic balance.

- 5 In samples 140603_001, 140616_001 and 140617_001, 1-10% glucose was added in an attempt to make Indulin more comparable to Inbicon lignin, which has a ~10% carbohydrate content composed mainly of glucan. Indulin has a glucan content of 0.11% and a xylan content of 0.37%. In sample 140604_001, different additives were used. These are among those tested in the above examples and found to be among the most optimal for lowering viscosity. Finally, a LOW formulation with grinded lignin pellets from Inbicon was prepared for comparison.

Oil, water and additive was mixed and homogenized with an ultraturrax to prepare an emulsion to which Indulin was added gradually in portions of 50-100 g. This was homogenized with an ultraturrax for 10 minutes at 20,000 rpm.

- 15 Immediately after homogenizing sample 140528_001 (the reproduction of the patent example), it could be poured into another container, although it was quite viscous. However, 10 minutes after, a spoonful of matter was scooped out of the container. As seen in Figure 17-1a, the liquid character of the slurry is much reduced.

- Figure 17-1b shows how a handful of the slurry could be formed to a small ball (diameter of 5 cm). The ball was left for several hours, and during this time, no change in physical appearance was observed. There is every reason to assume that the ball could have kept its shape for any duration.

- Viscosity was measured of the slurries shown in Table 17-1; 40 ml slurry was poured into the cup of the Viscotester VT550 to measure viscosity at shear rate 100 s^{-1} . The results are shown in Figure 17-2.

Initially, viscosity of the formulations prepared with Indulin decreases over time until reaching a minimum. Then viscosity increases until the slurry is so viscous (viscosity = 2.1 Pa s) that the instrument stopped. This duration (in minutes) is defined as the "Stability index".

- Analysis of Sample 140528_001 stopped after 30 minutes. Sample 140604_001 showed the "optimal" additives were not very efficient for lowering viscosity of Indulin Lignomulsion, and a

viscosity of 2.1 Pa s beyond the capacity of the instrument was reached in less than 10 minutes. Using glucose as an additive was slightly more efficient. Viscosity decreased slowly, and the of viscosity 2.1 Pa s, at which the instrument could no longer measure was reached, after more than 75 minutes.

- 5 However, in all cases the formulation was far from liquid and very difficult to pour or pump. As Lignomulsion prepared from Kraft lignin went from liquid and pourable to highly viscous and semi-solid within minutes, Kraft lignin is not suitable for preparing a lignin based lignin fuel.

Example 18

10 Effects of drying

- The difference between lignin filter cake and pellets, is that the pellets have been dried (to 95% DM), pelletized and grinded. Observations indicate that drying could be conceived as an important step, altering the physical and chemical properties of lignin. Therefore, different samples of lignin filter cake and decanter cake have been dried under different conditions, and their properties in Lignomulsion have been assessed.

Temperature effect

Lignin filtercake was dried to DM > 95% at four different temperatures (30-100°C) according to Table 18-1. Drying at low temperature took several days.

- Formulations were prepared with the composition LOW 30-20-50 and the addition of 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10, see Table 18-1. Viscosity was measured at the following shear rates: 50, 100, 150 and 200 s⁻¹, at the following temperatures: 25, 45, 65 and 85 °C.

Table 18-1:

ID	L-O-W	Drying T / °C	DM / %	Viscosity* / Pa s
130705_001	30-20-50	30	97.4 ± 0.4	0.232
130705_002	30-20-50	50	99.6 ± 0.2	0.234
130704_001	30-20-50	80	100.5 ± 0.2	0.228
130704_002	30-20-50	100	101.3 ± 0.1	0.141

* Measured at room temperature and shear rate 100 s⁻¹.

- 25 Figure 18-1 shows viscosity as a function of shear rate (a) and of temperature (b). At room temperature, here appears to be a connection between viscosity and drying temperature, as the emulsions prepared with lignin dried at the lowest temperatures (30-50 °C) are more viscous than emulsions prepared from lignin dried at the highest temperatures (80-100 °C).

- This is also correlated to the temperature dependency of the viscosity measurements: Viscosity of LOW formulations from filter cake dried at 80-100 °C does not change as a

function of temperature. This indicates that the transformations in the emulsions causing viscosity to change is some sort of heat initiated change of lignin.

DM effect

Lignin filter cake was dried in various degrees (DM = 59-100%) at 50 °C, according to table 18-2.

Formulations were prepared with the composition LOW 30-20-50 with the addition of 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10, see table 18-2. Viscosity was measured at the following shear rates: 50, 100, 150 and 200 s⁻¹, at the following temperatures: 25, 45, 65 and 85 °C.

Table 18-2: Formulations

ID	L-O-W	Drying T / °C	DM / %	Viscosity* / Pa s
130710_002	30-20-50	50	59.07 ± 0.04	0.327
130709_001	30-20-50	50	65.4 ± 0.2	0.307
130710_001	30-20-50	50	73 ± 1	0.271
130711_001	30-20-50	50	82 ± 2	0.139
130709_002	30-20-50	50	99.6 ± 0.2	0.238

* Measured at room temperature and shear rate 100 s⁻¹.

The object of these measurements was to find out if the change in properties between wet and dry material, as noted above, appears gradually or suddenly. Lignin was therefore dried for different durations resulting in different dry matter contents, all at the same temperature. It is not possible to completely characterize the drying conditions through temperature and duration, as it also depends on for example the exposed surface properties of the wet material (eg particle size and the shape of the container of the wet lignin). Some uncertainty is therefore expected.

This is also reflected in Figure 18-2, showing viscosity as a function of shear rate (a) and of temperature (b). No clear connection between dry matter content and viscosity can be observed, although the three wettest materials (DM 59%, 65% and 73%) result in more viscous emulsions than the two driest materials (82% and 99%). However, this effect is not significant enough to draw any conclusions.

Example 19

Heated lignin experiment

As lignin dried at high temperatures resulted in emulsions with low viscosities, a study was made to determine if preparing lignomulsion at high temperature resulted in different properties. 85 °C was selected as the maximum temperature that could be safely handled in the lab.

A formulation with LOW 40-20-40 (with grinded pellets as the lignin source) and 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10. All ingredients were heated to 85 °C and mixed (using the ultraturrax for 5 minutes and 10.000 rpm) while in a 85 °C water bath. Viscosity was measured, first at 85 °C at shear rates 50, 100, 150 and 200 s⁻¹; this was repeated a total of four times (see Figure 19-1). Then the formulation was cooled to 25 °C and viscosity was re-measured. This measurement was repeated at 25 °C the next day.

These sets of measurements were compared to Lignomulsion with the same formulation (40-20-40 and 5000 ppm Sodium benzoate and Lutensol AP10), see Figure 19-2. No variation in viscosity is observed above room temperature, but the viscosity at 25 °C is higher in the Lignomulsion prepared at 85 °C. It seemed to decrease after one day of storage.

Finally, a LOW 30-00-70 formulation using lignin filter cake was treated in the Parr reactor at 10 min at 120 °C and then cooled to room temperature. As drying lignin at elevated temperature was found to reduce viscosity, it was tested if it was the drying or the high temperature that caused the change. Viscosity was therefore measured before and after this treatment, see Figure 19-3. Viscosity decreases with increasing shear rate and increasing temperature, which is also expected for a formulation with no oils and no additives. At low temperature (25-45 °C), the treatment of the Parr reactor caused viscosity to increase significantly, whereas at high temperature (65-85 °C) viscosity decreased slightly.

In conclusion, from the Parr reactor experiments and the "85 °C preparation" experiments there is no advantage to exposing lignin or lignomulsion to high temperatures under wet conditions.

Example 20

Lignin, dried types

As drying conditions and dry matter content was found to be highly important for Lignomulsion prepared from lignin filter cake, we tried to find out if the same effects applied to pellets. Lignin pellets from the same campaign (spring, 2012) and different dry matter contents (84%, 89% and 95%) were tested, as well as pellets and granulate with the same dry matter contents (~95%). In the following, these materials are referred to as "84% pellets", "89% pellets", "95% pellets" and "95% granulate".

Dry matter content was re-analyzed, and the results are given below in Table 20-1.

Table 20-1: Properties of raw material

	Type	DM(1) / %	DM(2) / %
A	Pellets	84	91.84 ± 0.04
B	Pellets	89	94.22 ± 0.03
C	Pellets	95	96,08 ± 0.05
D	Granulate	95	93.92 ± 0.06

DM(1) is the dry matter content determined first. DM(2) lists the results after re-analysis. The pellets were drier than expected, whereas the granulate had taken up a little water. The discrepancies could be due to water uptake/evaporation during storage or grinding, and it gives a first indication that pellets and granulate are two fundamentally different materials.

"Stock emulsions" of grinded lignin pellets/granulate and water (65:35 by mass) were prepared and used for preparing emulsions of three different formulation, see Table 20-2. 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10 were added.

Table 20-2: Formulations with different lignin types

Sample ID	L %	O %	W %	AP10 %	NaBenz %	Lignin type	Viscosity* Pa s
131202_001	40	20	40	0.5	0.5	Pellets, 84%	0.174
131202_002	40	30	30	0.5	0.5	Pellets, 84%	0.111
131202_003	30	30	40	0.5	0.5	Pellets, 84%	0.090
131203_001	40	20	40	0.5	0.5	Pellets, 89%	0.097
131203_002	40	30	30	0.5	0.5	Pellets, 89%	0.096
130312_001	40	20	40	0.5	0.5	Pellets, 95%	0.065
131216_001	40	30	30	0.5	0.5	Pellets, 95%	0.849
130308_001	30	30	40	0.5	0.5	Pellets, 95%	0.047
131127_001	40	20	40	0.5	0.5	Granulate, 95%	0.059
131127_002	40	30	30	0.5	0.5	Granulate, 95%	0.123
131127_003	30	30	40	0.5	0.5	Granulate, 95%	0.023

5 Viscosity was measured with a Haake ViscoTester VT550 at shear rates 50, 100, 150 and 200 s⁻¹ and the following temperatures: 298, 318, 338 and 358 K (25-85 °C).

First, the four emulsions with LOW composition 40-20-40 (see also Table 20-2) were compared, see figure 20-1a-b: Overall, there is not much difference between pellets and granulate. At room temperature, viscosity of emulsions from granulate and pellets dried to RH 95% are very similar, at all four measured shear rates and all four temperatures. The pellets dried to RH 84 and 89% result in slightly more viscous formulations at room temperature. It has previously been shown for filter cake that drying prior to making Lignomulsion results in a significant reduction in viscosity. It is therefore not surprising that the level of dryness is correlated to viscosity. When temperature is increased, in all cases, viscosity increases with increasing temperature (probably due to additive facilitated lignin-water interaction, resulting in water uptake), and the temperature gradient is slightly greater for 95% pellets than for granulate, whereas the temperature gradient is minimal for the 84% pellets. There is no clear relation between viscosity and original lignin dry matter content at temperatures above room temperature.

Then, these results were compared to the four formulations with LOW composition 40-30-30 to evaluate the effect of replacing 10% of water with 10% oil. This has a different effect on viscosity for the four different lignin types: For the pellet types with the lowest DM (ie 84% and 89%) at room temperature, the extra oil causes viscosity to decrease, but for the driest material (95% pellets and granulate) it causes increases viscosity. At higher temperatures, increasing oil content causes viscosity to decrease in the formulations prepared from the three pellet types, but viscosity increases in formulations prepared from granulate.

Finally, the four formulations with LOW composition 30-30-40 were evaluated. Please note, that there is no data for LOW 30-30-40 containing lignin dried to RH 89%. By comparing this set of measurements with the LOW 40-20-40 formulations, we evaluate the effect of replacing 10% of lignin with 10% oil at a constant water content. The conclusion is that when lignin content goes down, so does viscosity. This is seen for all lignin types, temperatures and shear rates.

By comparing LOW 30-30-40 with LOW 40-30-30, we can evaluate the effect of replacing 10% of lignin with 10% water at a constant oil content. Again, it is seen that when lignin content goes down, so does viscosity (for all lignin types, temperatures and shear rates).

Evaluating viscosity of the four formulations with the same LOW composition, it is seen that at room temperature the different lignin materials result in different viscosities, with the 84% pellets resulting in the highest viscosity, but also with a big difference between the 95% pellets and granulate.

However, it is noted that on an absolute scale, the viscosities of the studied emulsions appear quite low.

In terms of viscosity, it is apparently an advantage that the lignin is as dry as possible, since the 95% material generally results in emulsions of viscosities lower than those prepared from the 84% or 89% material.

2) Using granulate in Lignomulsion result in low viscosity, comparable to formulations prepared from pellets. Increasing the amount of oil in Lignomulsion may have different effects on lignomulsion viscosity.

EXAMPLE 21

Sugar content

It was postulated that the presence of sugars and other residues from the Inbicon 2G process has beneficial properties for Lignomulsion, especially with regard to viscosity and stability. This should be seen in contrast to for example Kraft lignin.

In an initial, three different types of lignin were studied: One was Indulin AT from Westvaco. The other two were from ISK (13-R4-23-8, 8/5 11:20 Fiber mash - designated "Inbicon lignin") - they had undergone same pretreatment, hydrolysis and fermentation (A), but one of them was then given an extra hydrolysis (with approximately 300 g enzyme per kg sugar) and fermentation (B). The resulting material was dried at 50 °C to a dry matter content (DM)

of >99.5%. The composition was then analysed (according to the NREL procedure) with the following results:

Table 21-1: Chemical composition of 13-R4-23-8 with single and double hydrolysis

	Glucan	Xylan	Arabinose	Klason lignin	Ash	Total
13-R4-23-8 (A)	6.89%	2.94%	< 0.5%	61.8%	10.5%	82.10%
13-R4-23-8 (B)	2.52%	2.77%	< 0.5%	72.7%	9.9%	87.88%
ISK 2012-03-28 (C)	27.4%	9.9%	0.8%	50.9	6.5	95.5%

Also included in the table above is a third type of filtercake which for some reason has a very high content of sugar (C). To increase the range of sugar concentrations in the prepared formulations, the different lignin types were mixed, according to Table 21-2:

ID	L (%) Indulin	L (%) 1 hydro.	x	L (%) 2 hydro.	x	O (%)	W (%)	NaBenz Ppm	AP10 Ppm
130702_001	30	0		0		10	60	5000	5000
130702_002	15	15		0		10	60	5000	5000
130702_003	15	0		15		10	60	5000	5000
130702_004	7.5	22.5		0		10	60	5000	5000
130702_005	7.5	0		22.5		10	60	5000	5000
130701_002	0	30		0		10	60	5000	5000
130701_003	0	0		30		10	60	5000	5000

Figure 21-1 shows that viscosity of a LOW 30-20-50 emulsion where the lignin content is solely made up of Indulin is much lower than emulsions where the lignin content is either partially or completely composed of Inbicon lignin. This definitely demonstrates that there is some difference between Inbicon lignin and Kraft lignin.

Some additional formulations were prepared, see table 21-2:

Name	Lignin (%)				Contribution to dry matter					Viscosity
	13-R4-23-8 A	ISK 2012-03-28 B		Oil	Water	Klason lignin	Glucan	Xylan	Glucan + xylan	s ⁻¹
130701_002	30	0	0	10	60	18.54	2.07	0.88	2.95	0.17
130701_003	0	30	0	10	60	21.81	0.76	0.83	1.59	0.41
130719_001	15	15	0	10	60	20.18	1.41	0.86	2.27	0.29
130719_002	7.5	22.5	0	10	60	20.99	1.08	0.84	1.93	0.20
130722_002	0	22.5	7.5	10	60	20.18	2.62	1.37	3.99	0.28
130723_002	22.5	0	7.5	10	60	17.72	3.61	1.40	5.01	0.23
130723_003	15	0	15	10	60	16.91	5.14	1.93	7.07	0.14
130724_001	0	7.5	22.5	10	60	16.91	6.35	2.44	8.79	0.25
130724_002	7.5	0	22.5	10	60	16.09	6.68	2.45	9.13	0.10
130724_003	0	15	15	10	60	18.54	4.49	1.90	6.39	0.35
130723_001	0	0	30	10	60	15.27	8.22	2.97	11.19	0.15

Since the lignin and sugar content is different in each sample, viscosity is displayed in Figure 21-2 as a function of content of klason lignin and sugar (determined as the sum of xylan and glucan). Viscosity is measured at room temperature and shear rate 100 s^{-1} .

As a rule, viscosity increases with klason lignin content and decreases with sugar content. However, since the trends in Figure 21-2 are mainly observed for the formulations containing the same lignin samples, a new series of lignin samples were prepared from three different materials pretreated at three different severities; these either underwent a "normal" hydrolysis (60 g/kg glucan) or hydrolysis with a five times extra enzyme dosis, and in some cases fermented. The effect of pretreatment is clearly seen from the measured xylan, but not in the measured glucan. On the other hand; the effect of different enzyme dosages and fermentation is mainly reflected in the measured glucan content. Both of these effects are reflected in the lignin content, which is correlated to the combined sugar content; see Figure 21-3.

The samples were dried at 50°C and milled to ensure a homogeneous particle size distribution below 2 mm. This material was used to make Lignomulsion with LOW formulations 30-20-50 and 40-10-50, both with 5000 ppm Lutensol AP10 and sodium benzoate, respectively. In all cases, viscosity decreased with shear rate and only the measurements at shear rate 100 s^{-1} will be shown in the following (see also Table 21-3).

Based on the assumption that sugar decreases viscosity, it would be expected that formulations with high severity, high enzyme dosage and/or with fermented lignin samples should be the most viscous. In figure 21-4 viscosity measurements are shown for the twelve samples; divided into groups based on pretreatment severity (21-4a) and hydrolysis fermentation conditions (21-4b). No clear trends are observed. For the LOW 30-20-50 formulations prepared from "high severity" lignin, viscosity decreases with increasing sugar content (as predicted), but the opposite is observed for the "low" and "middle" severity conditions. For the unfermented samples produced with normal enzyme dosage (ie with the highest sugar content) viscosity increases with increasing xylan number (ie decreasing pretreatment severity), contrary to the prediction. On the other hand, for the fermented samples with normal enzyme dosage (ie the "standard" Inbicon conditions), viscosity increases with decreasing xylan number (ie increasing pretreatment severity).

A similar chaotic behaviour is observed for the LOW 40-10-50 formulations.

Finally, in Figure 21-5, viscosity is depicted as a function of klason lignin for both sets of formulations, without any clear connection being observed.

Table 21-3: Details of the lignin samples

Name	Severity	Hydrolysis	Fermentation	Glucan	Xylan	Klason lignin	Viscosity*
13-62-R6 F1	Low (13.5%)	Normal	Yes	8.45%	6.64%	54.07%	0.104
13-62-R6 F3	High (4.6%)	Normal	Yes	14.80%	1.20%	61.11%	0.145

13-62-R6 F2	Low (13.5%)	High	Yes	11,.12%	5,.56%	61,.99%	0,.138
13-62-R6 F4	High (4.6%)	High	Yes	7.64%	1.17%	70.48%	0.235
13-62-R6 F5	Middle (9.1%)	Normal	Yes	8.32%	3.01%	60.40%	0.123
13-62-R6 F6	Middle (9.1%)	High	Yes	3.42%	2.87%	67.61%	0.112
13-62-R6 1	Low (13.5%)	Normal	No	20.25%	4.19%	41.72%	0.142
13-62-R6 2	Low (13.5%)	High	No	14.16%	4.16%	52.67%	0.219
13-62-R6 3	High (4.6%)	Normal	No	15.42%	1.74%	57.57%	0.127
13-62-R6 4	High (4.6%)	High	No	10.48%	0.87%	62.12%	0.131
13-62-R6 5	Middle (9.1%)	Normal	No	17.35%	2.82%	48.64%	0.084
13-62-R6 6	Middle (9.1%)	High	No	12.71%	2.84%	55.71%	0.198

* Viscosity (in Pa s) of a LOW 30-10-50 with 5000 ppm Lutensol AP10 and sodium benzoate; measured at room temperature at shear rate 100 s⁻¹.

In conclusion; although a relation between viscosity and sugar content was initially suspected, based on experiments with a specific lignin material being diluted with an alternative low-sugar material, designing different lignin materials with different sugar content did not reveal any clear relation between sugar content and viscosity.

Example 22

Acid wash

Grinded lignin pellets were suspended in a ~1M solution of HCl (pH = 1). The pellets were washed repeatedly with water until pH ~ 5. Lignin was then separated through filtration; DM of the filtered material was 79%. The material was then dried to DM 100%.

Simultaneously, grinded lignin pellets were suspended in concentrated NH₄OH (pH = 10), washed until pH = 8, and filtered. The filtration was quite difficult, with the filter getting clogged easily. This resulted in material with DM 56%, which had a very unpleasant, ammonia-like smell. The material was dried to DM 100%.

Different LOW formulations were prepared with the acid treated lignin, which was compared to formulations prepared with untreated lignin, shown in Figure 22-1 and Table 22-1. In the 40-20-40 formulation. Whereas the base treated lignin did not result in lower viscosity Lignomulsion, acid treated lignin resulted in significant decreases in viscosity of the two tested formulations.

Table 22-1: Formulations of acid-, base- and untreated lignin material

Name	Lignin type	L	O	W	AP10	SoBenz
140331_001	Acid	40	20	40	0.5	0.5
130312_001	Untreated	40	20	40	0.5	0.5
140331_003	Base	40	20	40	0.5	0.5
140331_002	Acid	45	10	45	0.5	0.5
130307_002	Untreated	45	10	45	0.5	0.5
140331_004	Base	50	10	40	0.5	0.5
130627_001	Untreated	50	10	40	0.5	0.5

EXAMPLE 23

Ultraturrax power input study

Based on visual inspection, there was an expectation that the duration of mixing LOW formulations with an ultra turrax (UT) affected emulsion viscosity, and experiments were designed to quantify this effect.

LOW 40-20-40 formulations (with 5000 ppm sodium benzoate and 5000 ppm Lutensol AP10) were prepared and mixed with the Ultra Turrax (UT). The lignin came from dried, grinded pellets and the oil was diesel oil. UT speed and duration was varied. Energy consumption of the UT was estimated with an "energy-meter". This number was compared to viscosity, measured at four different shear rates (50 s^{-1} , 100 s^{-1} , 150 s^{-1} , 200 s^{-1}), all at room temperature. Details are shown in Table 23-1:

Table 23-1 Energy input and viscosity of LOW 40-20-40 formulations for first run of experiments

ID	UT time min	UT speed rpm	Energy J	Viscosity			
				Pa s			
				50 s^{-1}	100 s^{-1}	150 s^{-1}	200 s^{-1}
130624_002	0.5	10.000	1704	0.107	0.086	0.079	0.075
130624_003	1	10.000	3331	0.175	0.124	0.107	0.099
130624_004	10	10.000	28760	0.318	0.204	0.166	0.150
130624_005	0.5	3.500	895	0.208	0.148	0.123	0.111
130624_006	0.5	5.000	1200	0.246	0.193	0.161	0.153
130624_007	0.5	20.000	3522	0.208	0.117	0.100	0.095
130709_004	10	3.500	17295	0.154	0.121	0.121	0.126
130709_005	10	5.000	19603	0.194	0.146	0.136	0.134
130710_003	10	10.000	33410	0.207	0.145	0.123	0.107
130710_004	10	20.000	56583	0.799	0.454	0.316	0.274

Figure 23-1 shows how the energy input is related linearly to ultra turrax duration and speed, respectively. Viscosity is shown as a function of the UT energy input in Figures 23-2 and 3.

Figure 23-2 shows a clear dependence between emulsion viscosity and UT duration at the four shear rates, ie that when UT duration is increased, the emulsion becomes more viscous. The effect does not seem linear; it could be for low durations and then reaching a constant level.

- 5 Figure 23-3 shows viscosity as a function of UT speed at two different durations, 0.5 min and 10 min. At 0.5 min, no dependency between emulsion viscosity and UT speed is observed, but this is probably due to the short time scale and low energy input (resulting in very little variation). At a duration of 10 min., a clear correlation between viscosity and the ultra turrax effect is seen.
- 10 In conclusion, increasing UT duration from 0.5 min to 10 min (ie a factor of 20) increases viscosity at shear rate 100 s^{-1} with a factor of 2.4. Since low viscosity and low UT energy consumption are desired, the UT duration should be as short as possible, only long enough to ensure homogeneity of the emulsion. An effect of UT speed was also observed; increasing the UT speed from 3500 to 20000 rpm (a factor of 5.7) increases viscosity with a factor of
- 15 3.8. To test which of the factors - duration or speed - was the more significant, a new set of experiments was designed, see Table 23-2. On the basis of these results, Figure 23-4 is constructed, showing viscosity as a function of the combined effects of speed ("stirring") and duration ("time"). Within the tested conditions (duration 0.5 - 20.5 min; speed 3500-20000 rpm), the minimum is seen at the lowest possible energy input (ie at 3500 rpm for 0.5 min).
- 20 This may, however, not be enough to ensure a homogenous liquid - especially when working with wet lignin filter cake, which has not been pre-grinded, and has to be milled under wet conditions.

Within the tested conditions, the two factors - duration and speed - seem to be of equal importance.

- 25 Table 23-2 Energy input and viscosity of LOW 40-20-40 formulations for second run of experiments

ID	UT time	UT speed	Energy	Viscosity
	min	rpm	J	Pa s
				100 s^{-1}
20140306_001	0.5	3500	1070.6	0.030
20140306_002	15.5	15875	75338.9	0.153
20140306_003	0.5	3500	1334.7	0.029
20140310_002	20.5	20000	133173.9	0.227
20140310_003	20.5	20000	123947.1	0.233
20140310_004	0.5	20000	9156.6	0.078
20140317_001	5.5	15875	35883.3	0.134
20140317_002	5.5	7625	15096.1	0.044
20140317_003	10.5	11750	41072.4	0.132
20140317_004	20.5	3500	27701.4	0.044

20140317_005	20.5	11750	71361.6	0.190
20140317_006	10.5	20000	77471.7	0.198
20140317_007	10.5	3500	16099.1	0.037
20140317_008	20.5	3500	28428.5	0.048
20140317_009	15.5	7625	35326.6	0.101
20140317_010	0.5	20000	6964.2	0.055
20140317_011	0.5	11750	3378.7	0.037
20140317_012	10.5	11750	39174.1	0.127

EXAMPLE 24

Stability

Stability was studied in two different ways. In the first case, different lignomulsion formulations were exposed to high G-forces in a centrifuge, and the degree of phase separation was quantified. In the second case, lignomulsion was stored at room temperature or at 5 °C for several months, and viscosity was measured.

Storage stability

Most of the formulations presented in the sections above were stored for some time; then viscosity was measured again. If the formulations contained a preserving agent (sodium benzoate or paraben), they were stored at room temperature. Otherwise, they were stored at 5 °C. In all emulsions, phase separation was observed after some days of storage. But even after several months of storage, the formulations containing Inbicon lignin (in contrast to Indulin) were easily re-homogenized by manual shaking. It was not necessary to use the ultra turrax or other forms of high-shear mixing.

Formulations with grinded lignin pellets, diesel oil and water (in the absence of any additives) were prepared. In general, there is good correspondence between the viscosity measurements before and after the storage period, see Figure 24-1a. In most cases, storage causes viscosity to decrease. This would therefore not be a problem with the "pumpability" of Lignomulsion - however, as no surfactants were added in these experiments, they are not representative for the final Lignomulsion formulation.

Formulations with grinded pellets, diesel oil, water, and different hydrotropes and surfactants were prepared. These were stored for approximately 3 months at room temperature. In all studied cases, storage increases viscosity measured at 25 °C, see figure 24-1b. However, at temperatures of 45-65 °C the viscosity is not changed by the storage.

Formulations were prepared using grinded pellets, diesel oil, fuel oil and water and Lutensol AP10 and sodium benzoate. Emulsions were stored for approximately 3 months at room temperature. In some studied cases, storage increases viscosity measured at 25 °C, and in others it increases viscosity. There does not seem to be any relation between the viscosity changes and oil content.

Formulations prepared with different types of filter cake, diesel oil, water, Lutensol AP10 and sodium benzoate were prepared and stored at room temperature. For example, the filter cake samples which were 2dried at different temperatures are shown in Figure 24-1d. The "not dried" sample (LOW 30-20-50) shows decrease in viscosity due to the storage period, whereas it increases for the dried samples. On the other hand, the LOW 30-10-60 formulations (see Figure 24-1e) all show decreases in viscosity over time, although it is unclear why this is the case. The lignin samples had all been dried to >99% dry matter, and based on the results from 24-1d a decrease would have been expected. The difference could be related to the difference in oil content (although a relation between oil content and viscosity changes were not detected in Figure 24-1a and -c).

Example 25

Determining particle size distribution

Size distributions of particles in emulsions made from lignin filter cakes and water, prepared by "milling" the lignin while wet with an ultraturrax, were characterized, to determine any correlation between particle size and ultraturrax effect (ie time).

Approximately 350 g lignin filter cake was taken from freezer (dry matter content ~50 %). The cake was manually broken into smaller pieces and placed in the vegetable chopper of the Kenwood. The cut filter cake was then mixed with water and milled with an ultra turrax, operated at 10.000 rpm. Four different samples were prepared with different ultra turrax time: Sample 001 with 1 min, 002 with 5 min, 003 with 10 min and 004 with 30 min.

Particle sizes were measured with a Malvern Mastersizer at Asnæsværket. In the instrument, particles (administered either as a dry powder or an emulsion) were suspended in either water or ethanol. Particle size and concentration were determined from laser diffraction.

For comparison, particle size distributions were also determined from samples of grinded lignin pellets (dry matter content ~5%) collected with different mesh sizes.

An overview of the measurements is given in table 25-1.

Table 25-1:

Nr	Lignin type	Ultraturrax time / min	Mesh size	Liquid	Phase	Ultra sound
1	ISK 12-R6-44	1 min	--	Water	Dispersion	No
2	ISK 12-R6-44	5 min	--	Water	Dispersion	No
3	ISK 12-R6-44	10 min	--	Water	Dispersion	No
4	ISK 12-R6-44	30 min	--	Water	Dispersion	No
5	ISK 12-R6-44	1 min	--	Ethanol	Dispersion	No
6	ISK 12-R6-44	30 min	--	Ethanol	Dispersion	No
7	Grinded pellets	--	< 150	Water	Solid powder	60 s

8	Grinded pellets	--	150	Water	Solid powder	60 s
9	Grinded pellets	--	250	Water	Solid powder	60 s
10	Grinded pellets	--	710	Water	Solid powder	60 s
11	Grinded pellets	--	< 150	Ethanol	Solid powder	60 s
12	Grinded pellets	--	250	Ethanol	Solid powder	60 s
13	Grinded pellets	--	150	Water	Solid powder	60 s
14	Grinded pellets	--	150	Water	Solid powder	60 s

Figure 25-1 shows number distributions of wet milled filter cake. The maximal concentration is found below 100 μm , but the shoulder found above 100 μm means that larger particles makes the major contribution to the mass (data not shown). No direct correspondence
5 between particle size and ultra turrax time is observed.

In some cases, ethanol was used as the suspension medium in the instrument. As seen in Figure 25-1, there is a quite large difference in the resulting particle size. This is partially explained by the fact that the particles can take up liquid and swell to a bigger size, and partially by the fact that material can be extracted in the liquid phase. This was especially
10 relevant for ethanol, which clearly changed to a darker colour after the addition of lignin.

Figure 25-2 shows grinded pellets separated into different sizes by sieving, also showing the effect of suspension in water contra ethanol. More importantly, the figure does not show any clear connection between the dry particle size and the particle size measured by the instrument. This is mainly due to swelling of the particles, clouding the difference in dry size.
15 Comparison with figure 25-1 demonstrates the efficiency of wet milling, as a higher proportion of the particles are found below 100 μm in figure 5.3-1b than in Figure 25-2b

Example 26

Burner tests

In a very exiting set of experiments, it was tested if lignomulsion could actually ignite and
20 burn. Three different emulsions containing lignin (L), diesel oil (O) and water W were prepared with the following contents: 38:30:32 - 48:20:32 and 50:30:20, also containing 5000 ppm of sodium benzoate and 5000 ppm of Lutensol AP10. Due to lack of time, only the first two emulsions were tested.

The first emulsion (38:30:32) was sieved through mesh 0.5 mm. However, due to some
25 unfortunate, experimental circumstances the sieving process was slow, and a fraction lignin particles agglomerated to some rather large clumps (diameter of ~ 5 cm). The actual composition of this emulsion is therefore unknown. The second and third emulsions (48:20:32 and 50:30:20) was sieved through mesh 1 mm. This sieving went much faster, and lignin agglomeration was negligible.

In the first few burning experiments, the emulsions were heated to 80 °C. However, this was actually unnecessary, and it was not done in the last (and most successful, see below) burner experiments.

Figure 26-1 shows the system for injecting the fuel: Lignomulsion was placed in an approximately 1 l container and using pressurized air (up to 8 bar) it was ejected through a nozzle into a combustion chamber. Initially, the lignomulsion was ignited using a weed burner.

Different nozzles were used with varying degrees of success: The first nozzle used was a hollow cone nozzle (RTX 0250 B1) which had too small inner dimensions and was quickly clogged. A homemade nozzle (no name) could not atomize the fuel, and also clogged easily. The three last nozzles (flat and full jet nozzles, respectively) were, however, all capable of administering the fuel well enough for ignition and combustion.

The first emulsion tested was the 38:30:32 formulation using a RTX 0250 nozzle with a nozzle diameter of 1 mm. However, the inner dimensions of the nozzle were significantly smaller and the nozzle clogged within seconds. No real flames were seen in the combustion chamber.

A larger nozzle (2 mm) of the same type was also used, but with the same negative result. Three homemade "nozzles" (basically just tiny holes with diameters of 1-2 mm) were then used. They did not clog as easily, but did not atomize the fuel either. Instead it was "shot" out of the combustion chamber - like water through a hose.

Then, flat jet nozzles were used. First, KGW 1120 and 1190 was used. Initially, it atomized the fuel nicely and flames were seen, but the nozzle also had a tendency to clog. Instead the full cone nozzle (FEEBQ1550B3 - "pigtail nozzle"), resulting in the first truly successful burn. The remaining experiments were conducted on the emulsion with the composition 48-20-32. Finally, a modified injection system was designed, using the full cone nozzle, and adding a secondary stream of air, see figure 26-2. This resulted in the best atomization and the most stable combustion.

While experimenting with several different nozzles, the inside of the combustion chamber was repeatedly coated with lignomulsion. This was on several occasions ignited and burned independently for some time.

Using this setup, different Lignomulsion formulations were tested, see Table 26-1 below:

Nr	Lignin type	Oil type	Lignin	Oil	Water	NaBenz	AP10	AP8	Name
1	Pellets	Diesel. 10%	50	10	40	0.5	0.5	0	130702_002
2	Pellets	Diesel. 15%	50	15	35	0.5	0.5	0	130702_003
		1:1 Diesel:Fuel							130702_004
3	Pellets	oil	40	20	40	0.5	0.5	0	
4	Filter cake	Diesel. 20%	30	20	50	0.5	0.5	0	130701_001
5	Indulin	Diesel	40	15	44.8	0	0.1	0.1	130701_005

None of the formulations were neither sieved nor heated prior to combustion.

Since a low viscosity is essential for successfully pumping the fuel, an initial study of the sample viscosities was performed and presented below. Figure 26-3 shows viscosity at shear rate 100 s⁻¹ as a function of temperature for the first four formulations from Table 26-1 (note, however that the formulation 2 used in the viscosity test had a slightly higher water content - LOW 48-15-37 - than the emulsion used in the burner test - LOW 50-15-35).
5 Formulation 5 (with Indulin) was strongly thixotropic. Under static conditions, it was almost solid, but with a lot of stress from shaking, stirring and ultra turrax'ing the viscosity decreased to a degree where it could actually be poured.

Testing of the five formulations is described below.

10 **10% diesel**

The formulation was pumped and atomized in the setup, but could not burn even with auxiliary firing - 10% diesel is apparently below the lower limit.

Since the formulation could be pumped, a viscosity of ~0.4 Pa s (slightly) below the upper limit for pourability.

15 **15% diesel**

This emulsion was quite viscous and was not very well atomized, however it did seem to burn well. In a future experiment, a little lignin should be replaced with water - or the viscosity should be lowered with viscosity modifying additives. Alternatively, the formulation may atomize better on a more optimized setup.

20 **Fuel oil**

It has previously been demonstrated that a formulation with 20% diesel burns well. We therefore replaced half of the diesel with heavy fuel oil, but the emulsion did not burn without auxiliary firing. It should probably have been preheated.

Filter cake

25 The formulation was prepared using filter cake, which had been dried to > 90% DM and wet milled with an ultra turrax. However, the formulation contained some large particles which clogged the nozzle. The emulsion was therefore not successfully pumped and atomized - and consequently did not burn.

30 It is believed that such problems could be overcome, e.g. by a more careful wet milling, and avoiding larger particles, i.e. particles of a size close to the nozzle size, or somewhat smaller, could be avoided, e.g. by including a size separation step such as sieving. Alternatively, or in combination, larger nozzles should be used.

Emulsion with Indulin

35 It has been demonstrated and documented that lignomulsion burns very well. However, choosing the injection system is not trivial. Pumping the fuel with pressured air works very well, and full cone and flat jet nozzles work well. Ensuring sufficient air during combustion appears also important.

Example 31 - Provision of lignin-depleted vinasse and lignin from wheat straw and corn

Lignin-depleted vinasse 1 (= ISK vinasse) and lignin 1 (= ISK lignin):

ISK vinasse is produced at DONG Energy's Inbicon pilot plant in Skærbæk,

- 5 Denmark. Wheat straw is hydrothermal pretreated by use of steam (180-200°C in 10-20 minutes). Cooked fibres are separated from solubles, adjusted to pH 5.0-5.5 and hydrolysed by cellulolytic enzymes (for example Cellic Ctec3 from Novozymes) to a conversion degree of cellulose and xylan in the area of 65-85%. The hydrolysate is combined with the solubles and then fermented by use of C5 yeast consuming
- 10 glucose and xylose, producing ethanol. Ethanol is distilled off and the stillage is separated by a frame filtration unit into a solid fraction called "lignin" and a liquid fraction called "thin stillage". Thin stillage is evaporated providing lignin depleted ISK vinasse. The ISK lignin is dried at 105°C in a laboratory oven.

Lignin-depleted vinasse 2 (= IKA vinasse) and lignin 2 (= IKA lignin):

- 15 IKA vinasse is produced at DONG Energy's Inbicon demonstration plant in Kalundborg, Denmark. Wheat straw is hydrothermally pretreated by use of steam (180-200°C in 10-20 minutes). Cooked fibres are separated from solubles, adjusted to pH 5.0-5.5 and hydrolysed by cellulolytic enzymes (for example Cellic Ctec3 from Novozymes) to a conversion degree of cellulose and xylan in the area of 65-85%.
- 20 The hydrolysate is combined with the solubles and then fermented by use of C5 yeast consuming glucose and xylose, producing ethanol. Ethanol is distilled off and the stillage is separated by a frame filtration unit into a solid fraction called lignin and a liquid fraction called thin stillage. Thin stillage is evaporated into the lignin depleted IKA vinasse. The IKA lignin is dried in a tube bundle dryer at 70-75°C and pelletized.

- 25 *Lignin-depleted vinasse 3 (= corn vinasse) and lignin 3 (= corn lignin):*

Corn vinasse is produced at DONG Energy's Inbicon pilot plant in Skærbæk,

- Denmark. Corn stover is hydrothermally pretreated by use of steam (180-200°C in 10-20 minutes). Cooked fibres are separated from solubles, adjusted to pH 5.0-5.5 and hydrolysed by cellulolytic enzymes (for example Cellic Ctec3 from Novozymes)
- 30 to a conversion degree of cellulose and xylan in the area of 65-85%. The hydrolysate is combined with the solubles and then fermented by use of C5 yeast consuming

glucose and xylose, producing ethanol. Ethanol is distilled off and the stillage is separated by a frame filtration unit in to a solid fraction called lignin and a liquid fraction called thin stillage. Thin stillage is evaporated in to the lignin depleted corn vinasse. The corn lignin is dried at 105°C in a laboratory oven.

5 *Lignin-rich vinasse*

Lignin rich vinasse can be produces by the following steps: suitable biomass such as wheat straw or corn stover is processed essentially as disclosed above. However, after removal of the fermentation product (EtOH) e.g. by distillation, water is removed from the whole stillage, e.g. by evaporation, whereby the lignin-rich vinasse is provided.

Alternatively, the solubles from the solid liquid separation are not combined with the hydrolysate, and/or a yeast not capable of fermenting C5 sugars is used. In such cases, it is believed that the resulting lignin-depleted vinasses and/or lignin-rich vinasses possess higher heating values, which make them even more suitable for use as fuel according to the present invention.

Lignin, lignin-depleted vinasse, and/or lignin-rich vinasse are dried to desired values by methods known in the art. Lignin-rich vinasse is e.g. dried to 30, 65 or 100% dm, and lignin-depleted vinasse can is e.g. dried to 40, 65 and 100% dm.

Often, lignin is dried to a dm in excess of 80% dm, such as 85, 90, 95 or 100% dm.

20 **Example 32** – Analysis of lignin-depleted vinasse and lignin samples

Samples provided according to **Example 31** were analyzed as disclosed in **Table 31**.

Table 31 *Composition and properties of lignin-depleted vinasse*

	Unit	ISK vinasse	IKA vinasse	Corn vinasse	Method
Ash	%of dm	22	38	26	DS/EN 14775 (2009)
Acetic acid	%of dm	10	15	7,2	HPLC
Formic acid	%of dm	-	6,1	-	HPLC
Lactic acid	%of dm	4,0	16	5,1	HPLC
Succinic acid	%of dm	-	-	3,5	HPLC
Glycerol	%of dm	7,0	-	8,2	HPLC
Total sugars	%of dm	14	2,7	7,0	HPLC
Ca	%of dm	5,7	3,0	3,6	DS/EN 15290

					(2011), ICP -OES)
K	%of dm	3,9	13	5,7	DS/EN 15290 (2011), ICP -OES)
Na	%of dm	0,08	0,3	0	DS/EN 15290 (2011), ICP -OES)
Si	%of dm	-	0,12	-	DS/EN 15290 (2011), ICP -OES)
Gross calorific value	MJ/kg	10,1	5,76	-	DS/EN 14918 (2010)
Net calorific value	MJ/kg	5,56	3,9	-	DS/EN 14918 (2010)
Dry matter	% of total	60	60	60	Dried at 105°C until stable

HPLC

Sugar and acid quantification was done on a Dionex Summit High Performance Liquid Chromatography (HPLC) system equipped with a Shodex RI detector. Sugars (glucose, xylose and arabinose) and acids were separated in a Phenomenex Razex ROA column at 80 C with 5 nM H₂SO₄ as eluent at a flow rate of 0.6 ml min⁻¹.

Table 32 Composition and properties of lignin

	Unit	Lignin 1	Lignin 2	Lignin 3	Method
Dry matter	%of dm	95	90	90	dried at 105°C until stable
Gross calorific value	MJ/kg	20,3	18,7	20,0	DS/EN 14918 (2010)
Net calorific value	MJ/kg	19,1	17,5	19,0	DS/EN 14918 (2010)
Ash	%of dm	10,4	14,1	15,0	DS/EN 14775 (2009)

Example 32 Test of combustion properties

- Combustion properties of a variety of different samples comprising vinasse and lignin provided according to **Example 31** were determined by thermogravimetric analysis (TGA) and a single particle burner.

Thermogravimetric analysis (TGA)

- Thermogravimetric analysis was performed using a Netzsh STA 449 instrument to determine properties such as mass and heat flow as a function of temperature. For this experiment, small samples of 10 to 40 mg were prepared by mixing of the fuel components which has been provided as described in Example 31. The following temperature settings were used:

1. Samples were heated to 105°C (5 K/min) in N₂
2. 60 min isotherm at 105°C in N₂

3. Heating to 600°C (5 K/min) in N₂
4. 10 min isotherm at 600°C in N₂
5. Cooling to 200°C (50 K/min) in N₂
6. Heating to 1100°C (5 K/min) in 5% O₂
- 5 7. 15 min isotherm at 1100°C (10% O₂)

It is assumed that evaporation below 105°C is mainly due to water/diesel, while evaporation in the range 105-600°C is due to organic volatiles.

Table 33 shows the fuel mixtures analyzed by TGA, including reference samples like oil, straw, carbon black, diesel and lignin; lignin emulsions (lignin, oil/diesel and water mixtures). The vinasse and lignin samples are produced as described in Example 31 and are characterized in Example 32. Potassium acetate has been added to some of the samples in order to investigate the effect of it on combustion properties.

Table 33 Overview of sample composition, potassium content, ash and estimated heating values.

Sample	Lignin	Water	Diesel	Oil	CH ₄ , CO ₂	Unburnt	#	Ash	1200/1000
							(%dm)	(%dm)	(H ₂ O/kg)
Diesel	-	-	100 %	-	-	-	-	-	~ 61/45
Fuel oil	-	-	-	100 %	-	-	-	-	~ 39/42
Carbon black	-	-	-	-	-	-	-	-	-
Emulsion 1 (Reference)	45 % ¹²	38 %	10 %	0 %	-	-	-	-	~ 1/16
Emulsion 2 (Palm oil)	45 % ¹²	38 %	9 %	10 % ¹³	-	-	-	-	~ 1/16
Emulsion 3 (High water)	31 % ^{12,14}	57 %	10 %	0 %	-	-	-	-	~ 1/8
Emulsion 4 (Two oil/diesel)	50 % ¹²	47 %	0 %	0 %	-	-	-	-	~ 1/10
Emulsion 5 (Diesel + oil)	45 % ¹²	38 %	8 %	8 %	-	-	-	-	~ 1/16
IKA vinasse (0116-001)	-	-	-	-	-	100 % ¹⁵	13.8%	38%	~ 1/8
ISK vinasse (0116-002)	-	-	-	-	-	100 % ¹⁵	4.0%	20%	~ 6/10
IKA vinasse filtered (0116-003)	-	-	-	-	-	100 % ¹⁵	~13.8%	-	~ 1/8
IKA vinasse diluted (0116-004)	-	80 %	-	-	-	20 % ¹⁵	13.8%	-	-
IKA vinasse diluted (0116-005)	-	91 %	-	-	-	9 % ¹⁵	13.8%	-	-
ISK vinasse diluted (0116-006)	-	80 %	-	-	-	20 % ¹⁵	4.0%	-	-
ISK vinasse diluted (0116-007)	-	50 %	-	-	-	50 % ¹⁵	4.0%	-	-
IKA vinasse diluted (0116-008)	-	50 %	-	-	-	50 % ¹⁵	13.8%	-	-
ISK vinasse diluted (0116-009)	-	91 %	-	-	-	9 % ¹⁵	4.0%	-	-
ISK vinasse & lignin (0116-012)	47 % ¹²	-	-	-	-	53 % ¹²	2.3 %	15.7 %	~ 1/15
ISK vinasse & lignin (0116-013)	32 % ¹²	-	-	-	-	68 % ¹²	2.6 %	17 %	~ 1/14
ISK lignin (0116-014)	100 % ¹²	-	-	-	-	-	0.3 %	10.9 %	~ 20/21
IKA lignin (0116-017)	100 % ¹²	-	-	-	-	-	-	-	-
Straw (0116-018)	-	-	-	-	-	-	-	-	-
Corn vinasse vinasse (0116-019)	-	-	-	-	-	100 % ¹⁵	5.7 %	-	-
Corn vinasse vinasse conc. (0116-020)	-	-	-	-	-	100 % ¹⁵	-	-	-
ISK vinasse & lignin (0324-001)	50 % ¹²	-	-	-	-	50 % ¹²	2.2 %	16.5 %	12/15.5
Corn vinasse & lignin (0324-002)	50 % ¹²	-	-	-	-	50 % ¹²	3.3 %	-	12/
ISK vinasse & lignin (0324-003)	32 % ¹²	-	-	-	-	68 % ¹²	2.8%	18.5 %	10/
Corn vinasse & lignin (0324-004)	35 % ¹²	-	-	-	-	65 % ¹²	3.9 %	22.5 %	10/
ISK vinasse & oil (0324-005)	-	-	-	15 %	-	85 % ¹²	3.4%	-	11/
Corn vinasse & oil (0324-006)	-	-	-	15 %	-	85 % ¹²	4.8%	-	11/
IKA vinasse & oil (0324-007)	-	-	-	20 %	-	80 % ¹²	10.5%	-	11/
ISK lignin (0324-008)	100 % ¹²	-	-	-	-	-	-	-	-
ISK vinasse (0324-009)	-	-	-	-	-	100 % ¹²	-	-	-
Corn lignin (0324-010)	100 % ¹²	-	-	-	-	-	-	-	-
Corn vinasse (0324-011)	-	-	-	-	-	100 % ¹²	-	-	-
Thin ISK vinasse & lignin (0407-001a)	49 % ¹²	14	-	-	-	49 % ¹²	2.2 %	-	10/
Thin ISK vinasse & lignin with K (0407-001b)	38 % ¹²	14	-	-	2 %	38 % ¹²	3.6 %	-	10/
ISK vinasse, oil with K (0428-005a)	-	-	-	18 %	12%	70 % ¹²	7.9 %	-	11/
ISK vinasse + oil (0428-005c)	-	-	-	10 %	-	90 % ¹²	-	-	8/
ISK vinasse + oil (0428-005d)	-	-	-	5 %	-	95 % ¹²	-	-	7/
ISK vinasse, lignin and oil (0428-005e)	10 % ¹²	-	-	5 %	-	85 % ¹²	-	-	-
Corn vinasse, oil and K (0428-006)	-	-	-	18 %	?	80 % ¹²	12 %	-	11/
IKA vinasse and lignin (0428-007)	50 % ¹²	-	-	-	-	50 % ¹²	4.8 %	-	-

Lignin = ISK lignin; water = distilled water ; oil = heavy fuel oil

Figures 37 and 38 show mass loss during heating in N₂ and subsequently in O₂.

Results are normalized based on the mass after step 5 in the temperature routine

5 defined above. There is good reproducibility for TGA measurement of each individual sample. A noticeable feature is the way the mass loss slowly continues for IKA vinasse, even at temperatures above 800°C and in particular during the 15 min isotherm at 1100°C (step 7). This shows that IKA vinasse has fundamentally different properties than ISK vinasse and corn vinasse, since mass loss for ISK vinasse is

10 negligible above 900°C. It is, however, important to notice that mass loss is observed for all vinasse types above 550°C, which is the temperature usually used for ash determination.

Single particle burner experiments

The single particle burner is shown in **Figure 39**. The setup enables combustion of single particles/droplets at well-controlled conditions. For this experiment, small samples of 1 to 30 mg were prepared by mixing of the fuel components which were provided as described in Example 31. Variable parameters include temperature, gas velocity and gas composition (oxygen content). The setup comprises of a gas supply system (H₂, CH₄, O₂ and N₂), a metal flat flame hydrogen burner, a reactor tube (50 cm long cylindrical chamber with an inner diameter of 5 cm), a gas sampling and a conditioning system.

- Each experimental run starts with 2 hours of warm up to 1200°C and stabilization of the operating conditions (5% O₂) of the experimental setup. The samples (1-25 mg) placed on the plate sample holder are inserted into the reactor while shielded by a quartz tube. The tube is subsequently removed and the sample conversion/behavior is followed by a high speed camera. The first phase is the ignition delay (3A), followed by the pyrolysis phase with a steady flame. The duration of these phases are determined by the video camera recording.

Table 34 Sample compositions analysed in single particle burner experiments

	IKA		ISK		Corn stover		Fuel oil	Water	KAc
	Vinasse	Lignin	vinasse	lignin	vinasse				
	g	G	g	g	G	g	g	G	
150324_001	0	15.0	15.0	0	0	0	0	0	0
150324_002	0	0	0	15.0	15.0	0	0	0	0
150324_003	0	11.6	24.65	0	0	0	0	0	0
150324_004	0	0	0	10.5	19.5	0	0	0	0
150324_005	0	0	25.9	0	0	4.9	0	0	0
150324_005c	0	0	27.0	0	0	3.0	0	0	0
150324_005d	0	0	28.5	0	0	1.5	0	0	0
150324_006	0	0	0	0	25.5	4.5	0	0	0
150324_006a	0	0	0	0	17	3	0	5	

150324_007	24.0	0	0	0	0	6.0	0	0
150324_007a	0	3	25.5	0	0	1.5	0	0
150324_008	0	40.0	0	0	0	0	0	0
150324_009	0	0	40.0	0	0	0	0	0
150324_010	0	0	0	15.0	0	0	0	0
150324_011	0	0	0	0	15.0	0	0	0
150324_001_150407_01	0	4.65	4.65	0	0	0	1.60	0
150324_001_150407_02	0	0	0	4.55	4.55	0	1.66	0.80
150324_005_150407_01	0	0	8.91	0	0	1.69	0	0
150526_001	9.7	9.7*	0	0	0	0	0	0

* 9.7 g grinded IKA lignin, not ISK lignin(!)

IKA Vinasse as described in Example 31; ISK vinasse and lignin as described in Example 31; Corn stover lignin and vinasse as described in Example 31; Fuel oil= heavy fuel oil; Water= distilled water; KAc=potassium acetate

- 5 The main results from the single particle burner experiments are summarized in **Figures 40 and 41**. In general, short ignition delay (< 500 ms) is observed for samples containing oil, while samples with no oil have significantly longer ignition delay (see **Figure 40**). The only exception is sample 150526, containing a 50:50 mixture of IKA lignin and IKA vinasse. An explanation for this could be the
- 10 combination of relatively high homogeneity and high heating value.

Pyrolysis time seems to depend mainly on sample size, and not significantly on the actual chemical content or heating value (**Figure 41**).

- Samples with a high K swell during combustion to a volume several factors larger than the start volume. The reason may be that K-containing compounds form a semi-
- 15 liquid outer layer that partially traps gaseous compounds created or released during heating and combustion. The layer seems to be hollow and deteriorates during the pyrolysis phase. It is assumed to be easily collapsed by even weak physical disturbances. This K effect is observed for both IKA samples, where the vinasse has a “natural” high K content (due to pH adjustment with KOH) and in ISK samples that
- 20 have been “spiked” with potassium acetate.

Conclusions

A variety of different fuel configurations were tested in the single particle burner. The overall conclusion is that all fuel mixtures were combustible in the single particle burner. Different potassium concentrations were tested and no significant influence on the combustion properties were identified in high K content fuels in this test.

Ignition delay

Compared to the swift ignition of fuel oil (40 ± 70 ms), corn/straw vinasse samples with up to 35 wt. % lignin displayed a sluggish ignition (850 to 1800 ms) with a considerable standard deviation (up to 800 ms) indicating sample heterogeneity.

Addition of up to 15 wt. % fuel oil or 50 % lignin improve the ignition considerably (15 % oil = 30 ± 20 ms, 5 % oil = 270 ± 190 ms, 50 % lignin = 320 ± 320 ms), while potassium acetate addition (up to 12 % K dm.) only caused minor increases in ignition delays. The ignition delay of the investigated samples has been shown to be independent of sample mass/size, i.e. the observed range of ignition delays is therefore expected to be representative of smaller droplets as well.

Pyrolysis

The duration of the stable flame fueled by released pyrolysis gas was generally quite similar (app 1800-2800 ms at 5 mg) within the standard deviation (estimated to be 800 ms for fuel oil). Only exception was the somewhat shorter pyrolysis of pure vinasse samples. The pyrolysis of the samples investigated is clearly dependent of sample mass/size and the pyrolysis time in suspension fired boilers is therefore expected to be shorter.

Char burnout

Estimation of the duration of the burnout of the char remaining after pyrolysis has only been possible for a few swelling samples (i.e. samples high in potassium), in addition to willow wood blocks. Pure straw vinasse (IKA) show a very short char burnout (< 5 s), while the burnout of 50/50 % IKA vinasse and lignin are similar or slightly higher than the wood blocks (10 to 20 s for a 15 mg sample). The char oxidation of the samples investigated is clearly dependent of sample mass/size and conversion time in suspension fired boilers is therefore expected to be shorter.

Example 35 - Calculation of dry matter limits

Different heating values for different dry matters of the lignin rich, lignin depleted and lignin products are calculated and presented in **Table 35**. The calculated values are based on the sample data described in Example 32. The physical properties of the lignin depleted and lignin rich vinasse can be different from Example 32 and depends on the separation and drying equipment selected as well as the wished parameters for the optimal process configuration of the 2nd generation ethanol plant. In order to look into prescription of the fuel mixtures in a general the ash, lower heating value and K of lignin depleted and lignin rich vinasse at different dry matter are calculated.

Table 35 – physical data of fuel-related components used in fuel mixture calculations

<i>Wheat straw base case</i>	Dm	ash % of dm	LHV	K % of dm
			GJ/ton	
Lignin rich vinasse	30%	10%	3.6	1.5%
Lignin rich vinasse	65%	10%	7.8	1.5%
Lignin rich vinasse	100%	10%	18	1.5%
Lignin depleted vinasse	40%	20%	3.4	3.5%
Lignin depleted vinasse	65%	20%	6	3.5%
Lignin depleted vinasse	100%	20%	10	3.5%
Oil	99.5%	0%	40	0.0%
Lignin solid fuel	90%	10%	18	0.3%
Wood	90%	3%	17	0.02%
Coal	90%	11%	25	0.2%

dm, ash % of dm, LHV, and K % of dm are determined as described above. With respect to oil, 99.5% dm is to be understood as 99.5 pure oil, i.e. comprising not more than 0.5 impurities, such as water.

Lignin rich vinasse at 30% dm will typically possess a lower heating value of 3.6 GJ/ton or more, which makes this vinasse as a combustible compound though preferably mixed with higher value fuel. Lignin rich vinasse at 65% presents a fuel-related component with relatively high dry matter and the lower heating value is at the level of 8 GJ/ton, which is believed to be combustible. The 100% lignin-rich vinasse shows the energy content without water.

In the following Examples 36-41, different fuels and fuel mixtures are provided.

Example 36 Fuel comprising lignin-depleted vinasse and lignin

Fuel comprising essentially only lignin-depleted vinasse and lignin can e.g. be provided by mixing of lignin-depleted vinasse as described in Example 31 together

with lignin. Such fuels are presented in **Table 36**. Samples A and C have been produced and tested as described in Example 33. Samples A1 and C1 are further fuels comprising lignin-depleted vinasse and lignin. The presented fuel mixtures can be produced at different dry matter contents of the components. Depending on the dry matter contents of the fuel components and mixing ratio, different consistencies of the fuel are achieved: solid and liquid fuel.

Table 36 Fuel comprising lignin-depleted vinasse and lignin, A and C are tested samples, A1 and C1 are calculated fuel mixtures.

	Unit	A	A1	C	C1
Vinasse percentage, calculated	% of fuel mixture	50	60	75	70
Lignin percentage, calculated	% of fuel mixture	50	40	25	30
Vinasse dm	% of dm	65% or more	40% or more	65% or more	40% or more
Lignin dm	% of dm	0,9	0,9	90	90
Oil	%	0	0	0	0
Other fuel (co combustion)	Yes/No	no	no	yes	yes
Andet	%X	possible additive to make fuel thick (more solid consistence), so it is easy to pelletize or make briquette; It is belived that if there is more vinasse then the fuel will not be solid any more	possible additive to make fuel thick (more solid consistence), so it is easy to pelletize or make briquette; It is belived that if there is more vinasse then the fuel will not be solid any more	possible additive to make it thin (more liquid consistence) so it is possible to pump it	possible additive to make it thin (more liquid consistence) so it is possible to pump it
Fuel type	Solid/Liquid	Solid	Solid	Liquid (Co-combustion)	Liquid (Co-combustion)
LHV	GJ/ton	10 to 13	9 to 13	9	9
Ash content	% of dm	15,2	15,2	17	15
K content average	% of dm	1,7	1,7	2,5	2
K content max expected	% of dm	4,42	4,42	6,5	5,2
Flexibility, if K is higher than expected	Yes/No	Yes	Yes	Yes	Yes
Thermogravimetric analysis TGA	Yes/No	Ja	No	Ja	No
Boiler type	type	Grate boiler or low temperature Bubbling fluidized bed, circulating fluidized bed (LT BFB, CFB), LT CFB = Pyroneer	Grate boiler or low temperature Bubbling fluidized bed, circulating fluidized bed (LT BFB, CFB), LT CFB = Pyroneer	Co-combustion, fire ball (Co-combustion can be done in almost any boiler - the contribution of the vinasse fuel can vary)	Co-combustion, fire ball (Co-combustion can be done in almost any boiler: BFB, CFB - the contribution of the vinasse fuel can vary)

Example 37 Fuel comprising lignin-depleted vinasse and oil

Fuel comprising essentially only lignin-depleted vinasse and oil can e.g. be provided by mixing of lignin-depleted vinasse as described in Example 31 together with different oils. **Table** presents B, a sample which has been produced and tested as described in Example 33. B1 is a further fuel comprising lignin-depleted vinasse and oil. The presented fuel mixtures can be produced at different dry matter contents of the components. Depending on the dry matter contents of the fuel components and mixing ratio different qualities of the fuels can be achieved. [A2]

Table 37 Fuel comprising lignin-depleted vinasse and lignin, B is tested sample, B1 is calculated fuel mixtures

		B	B1
	Unit	Lignin depleted vinasse and oil	Lignin depleted vinasse and oil
Vinasse percentage, calculated	% of fuel mixture	85 to 90%	50 to 90%
Lignin percentage, calculated	% of fuel mixture	0 to 15%	0 to 15%
Vinasse dm	% of dm	65% or more	40% or more
Lignin dm	% of dm	-	-
Oil	%	5-15%	5-40%
Other fuel (co combustion)	Yes/No	yes	yes
Andet	%X	possible additive to make it thin (more liquid consistence) so it i spossible to pump it	possible additive to make it thin (more liquid consistence) so it i spossible to pump it
Fuel type	Solid/Liquid	Liquid	Liquid
LHV	GJ/ton	9 to 11	8 to 11
Ash content	% of dm	18-22	18-22
K content average	% of dm	3,4-6	3,4-6
K content max expected	% of dm	8,84	8,84
Flexibility, if K is higher than expected	Yes/No	No	No
Thermogravimetric analysis TGA	Yes/No	Ja	No
Boiler type	type	Adjusted oil boiler, Co- combustion, fire ball (Co- combustion can be dony in almost any boiler - the contribution of the vinasse fuel can vary)	Adjusted oil boiler, Co- combustion, fire ball (Co- combustion can be dony in almost any boiler - the contribution of the vinasse fuel can vary)

Example 38 - Fuel comprising lignin-depleted vinasse

Fuel comprising essentially lignin-depleted vinasse only can e.g. be provided by the method described in Example 31. Table 38 presents sample D, which has been produced and tested as described in Example 33. The presented fuel can be produced at different dry matter contents. Depending on the dry matter contents of the fuel components and mixing ratio different qualities of the fuels can be achieved. [A3]

Table 38 Fuel comprising lignin-depleted vinasse

		D
	Unit	Lignin depleted vinasse
Vinasse percentage, calculated	% of fuel mixture	100
Lignin percentage, calculated	% of fuel mixture	0
Vinasse dm	% of dm	40% or more
Lignin dm	% of dm	-
Oil	%	0
Other fuel (co combustion)	Yes/No	no
Andet	%X	possible additive to make it thin (more liquid consistence) so it i spossible to pump it
Fuel type	Solid/Liquid	Liquid (Co-combustion
LHV	GJ/ton	5,5-7
Ash content	% of dm	18-22
K content average	% of dm	4 to 6
K content max expected	% of dm	10,4
Flexibility, if K is higher than expected	Yes/No	
Thermogravimetric analysis TGA	Yes/No	Ja
Boiler type	type	Co-combustion, fire ball (Co-combustion can be done in almost any boiler: BFB, CFB - the contribution of the vinasse fuel can vary)

Example 39 - Fuel comprising lignin-depleted vinasse and wood

Fuel comprising lignin-depleted vinasse and wood can be provided e.g. by mixing of lignin-depleted vinasse as described in Example 31 together with wood. **Table** 5 presents E1 fuel mixture, the calculated fuels comprising lignin-depleted vinasse and other fuel. The presented fuel mixtures can be produced at different dry matter contents of the components.

Table 39 Fuel comprising lignin-depleted vinasse and wood, E1 is a calculated fuel mixture

		E1
	Unit	Lignin depleted vinasse and other fuel (wood pellet biomass or other)
Vinasse percentage, calculated	% of fuel mixture	0,01- 99,9
Lignin percentage, calculated	% of fuel mixture	0
Vinasse dm	% of dm	40% or more
Lignin dm	% of dm	-
Oil	%	-
Other fuel (co combustion)	Yes/No	0 - 99,9
Andet	%X	possible additive to make fuel thick (more solid consistence), so it is easy to pelletize or make briquette
Fuel type	Solid/Liquid	Solid
LHV	GJ/ton	depends on other fuel
Ash content	% of dm	depends on other fuel
K content average	% of dm	depends on other fuel
K content max expected	% of dm	depends on other fuel
Flexibility, if K is higher than expected	Yes/No	
Thermogravimetric analysis TGA	Yes/No	No
Boiler type	type	depending on the other fuel, possible in any type of solid fuelboiler : Grate boiler or low temperature Bubbling fluidized bed, circulating fluidized bed (LT BFB, CFB), LT CFB = Pyroneer

Example 40 - Fuel comprising lignin-rich vinasse and oil (Column F1)

Fuel comprising lignin-rich vinasse and oil can be provided e.g. by mixing of lignin-rich vinasse as described in Example 31 together with a suitable oil. **Table** shoes the calculated fuel comprising lignin-rich vinasse and oil (sample F1). The presented fuel mixtures can be produced at different dry matter contents of the components. Depending on the dry matter contents of the fuel components and mixing ratio, different qualities of the fuels can be achieved. [A4]

Table 40 Fuel comprising lignin-rich vinasse and oil, F1 is a calculated fuel mixture

		F1
	Unit	Lignin rich vinasse and oil
Vinasse percentage, calculated	% of fuel mixture	85 to 90%
Lignin percentage, calculated	% of fuel mixture	0 to 15%
Vinasse dm	% of dm	30% or more
Lignin dm	% of dm	-
Oil	%	5-15%
Other fuel (co combustion)	Yes/No	yes
Andet	%X	possible additive to make it thin (more liquid consistence) so it i spossible to pump it
Fuel type	Solid/Liquid	Liquid
LHV	GJ/ton	8 or more
Ash content	% of dm	18-22
K content average	% of dm	1
K content max expected	% of dm	3
Flexibility, if K is higher than expected	Yes/No	Yes
Thermogravimetric analysis TGA	Yes/No	No
Boiler type	type	Adjusted oil boiler, Co-combustion, fire ball (Co-combustion can be dony in almost any boiler - the contribution of the vinasse fuel can vary)

Example 41 - Fuel comprising lignin-rich vinasse (G1)

Fuel comprising essentially lignin-rich vinasse only can be provided by the method described in Example 31. **Table 41** presents a calculated fuel comprising lignin-rich vinasse (sample G1). Such a fuel can be produced at different dry matter contents. Depending on the dry matter contents of the fuel components and mixing ratio different qualities of the fuels can be achieved. [A5]

		G1
	Unit	Lignin rich vinasse
Vinasse percentage, calculated	% of fuel mixture	100
Lignin percentage, calculated	% of fuel mixture	
Vinasse dm	% of dm	65% or more
Lignin dm	% of dm	-
Oil	%	-
Other fuel (co combustion)	Yes/No	no
Andet	%X	possible additive to make fuel thick (more solid consistence), so it is easy to pelletize or make briquette
Fuel type	Solid/Liquid	Solid
LHV	GJ/ton	>7
Ash content	% of dm	10
K content average	% of dm	1,5
K content max expected	% of dm	3,9
Flexibility, if K is higher than expected	Yes/No	Yes
Thermogravimetric analysis TGA	Yes/No	No
Boiler type	type	Grate boiler or low temperature Bubbling fluidized bed, circulating fluidized bed (LT BFB, CFB), LT CFB = Pyroneer

Table 41 Fuel comprising lignin-rich vinasse, G1 is a calculated fuel mixture

References

- 5 Posarac, D. and Watkinson, A. "Mixing of a lignin-based slurry fuel," The Canadian Journal of Chemical Engineering (2000) 78:265
- Thammachote, N. "Combustion of lignin mixtures in a rotary lime kiln," Pulp & Paper Canada (1996) 97(9):51

CLAIMS

1. A fluid composition comprising a lignin component, an organic fraction in liquid state at 25°C, a vinasse, such as a vinasse provided according to any one of claims 221- 231 and optionally water and/or a further agent.

2. A fluid composition according to claim 1, wherein said lignin component is not lignin from paper and pulp production, such as Kraft lignin, wherein said Kraft lignin being provided from biomass by a process known in the art as Kraft process/method.

3. A fluid composition according to claim 1 or 2, wherein said lignin component has not been provided by a Kraft method or another method comprising an alkaline treatment, such as by addition of NaOH or another base to provide a pH of around 10 or higher, at or around pH 11 or higher, or at or around pH 12 or higher.

4. A fluid composition according to any one of the preceding claims, wherein said lignin component has not been esterified and/or subjected to an esterification step, such as disclosed in WO2015/094098.

5. A fluid composition according to any one of the preceding claims, wherein said lignin component has a Lignin Ion Exchange Capacity (LIEC) of 0.3 mol/kg dry matter (DM) or less, such as 0.25 mol/kg DM or less, such as 0.20 mol/kg DM or less, such as 0.15 mol/kg DM or less, or such as 0.10 mol/kg DM or less.

6. A fluid composition according to any one of the preceding claims, wherein said lignin component has a LIEC in the range of 0.05-0.30, 0.10-0.25, or 0.10-0.15 mol/kg DM.

7. A fluid composition according to any one of the preceding claims, wherein said lignin component is significantly less polar than Kraft lignin, such as assessed by LIEC measurement, such as having a LIEC at least 0.10, 0.11, 0.12, 0.13, 0.14, 0.15, 0.16, or 0.17 mol/kg DM lower than the LIEC of Kraft lignin.

8. A fluid composition according to any one of the preceding claims, wherein said lignin component is significantly less hygroscopic, such as binding at least 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 or 100 % (w/w) less water
5 when compared to Kraft lignin.
9. A fluid composition according to any one of the preceding claims, wherein said lignin component is swelling significantly less than Kraft lignin, such as swelling at least 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, 95 or 100 % less,
10 and optionally wherein said swelling is determined as change in particle size upon suspension in water or another suitable medium after 60 min.
10. A fluid composition according to any one of the preceding claims, wherein said fluid composition is significantly more stable and/or pumpable when compared to
15 a similar composition prepared with Kraft lignin.
11. A fluid composition according to claim 10, wherein the pumpability is defined as having a viscosity of 1 Pa.s or less, such as 0.9 Pa.s or less, such as 0.8 Pa.s or less, such as 0.7 Pa.s or less, such as 0.6 Pa.s or less, such as 0.5 Pa.s, such as
20 0.4 Pa.s or less, such as 0.3 Pa.s or less, such as 0.2 Pa.s or less, or such as 0.1 Pa.s or less at a shear rate of 100 s^{-1} , wherein said viscosity is being measured as average over a time period of 10 min.
12. A fluid composition according to any one of the preceding claims, wherein said
25 fluid composition possesses a significant short term, medium term, or long term stability and/or pumpability, wherein said short, medium, and long term are periods of time in the range of 1 - 60min, >1-24h, or >24h, respectively.
13. A fluid composition according to any one of the preceding claims, wherein said
30 fluid composition has an increased short term, medium term and/or long-term stability and/or pumpability, when compared to a similar composition prepared with Kraft lignin.

14. A fluid composition according to claim 12 or 13, wherein said short term time period is 1, 2, 5, 10, 15, 20, 30, 45, or 60 min.

5 15. A fluid composition according to claim 12 or 13, wherein said medium term time period is 90 min, 2h, 4h, 6h, 8h, 10h, 12h, 18h, 24h.

16. A fluid composition according to claim 12 or 13, wherein said long term time period is 25h, 30h, 40h, 2d, 3d, 4d, 5d, 6d, 1 week, 2 weeks, 3 weeks, 1 month, 2, months, 3 month, 4 months, 5 months, 6 months, or more than 6 month.

10 17. A fluid composition according to any one of claims 10-16, wherein said stability is defined in the sense that no more than 5.0, 4.0, 3.0, 2.0, 1.0 or 0.5 % (w/w) of any one of the fractions (e.g. water, liquid organic fraction, and/or lignin component) of said fluid composition will separate after said specified period of time.

15 18. A fluid composition according to any one of the preceding claims, wherein occasional or constant gentle stirring, agitation, and/or re-circulation but no high shear mixing is be required for maintaining said stability and/or pumpability.

20 19. A fluid composition according to any one of the preceding claims comprising two or more fractions, wherein (a) the first fraction is an organic fraction in liquid state at room temperature, said organic fraction comprising one or more organic compounds such as one or more fat, and/or one or more oil; and (b) the second fraction comprises one or more lignin component.

25 20. A fluid composition according to any one of the preceding claims comprising 5-60 % (w/w) lignin component, 0-40 % (w/w) organic fraction, 0-60 % (w/w) vinasse and/or water, and 0-1.0 % (w/w) further agent.

30 21. A fluid composition according to any one of the preceding claims, wherein the vinasse or water is comprised (i) in the first fraction, such as an oil- and/or fat-water-emulsion, as a homogenous solution of oil and/or fat and water; (ii) as a third, aqueous fraction; or (iii) as a combination of (i) and (ii).

22. A fluid composition according to any one of the preceding claims, wherein the Lignin Ion Exchange Capacity is around 0.40, 0.35, 0.30, 0.25, 0.20, 0.15, 0.10, mol/kg dry matter or less; in the range of 0.10-0.20, 0.20-0.30, 0.30-0.40 mol/kg dry matter; and/or in the range of 0.05-0.40, 0.10-0.30, or 0.10-0.20 mol/kg DM.

23. A fluid composition according to any one of the preceding claims, further comprising one or more further agent, such as an agent is selected from the group comprising or consisting of one or more dispersing agent(s), surfactant(s), hydrotropic agent(s), emulsifier(s), preserving agent(s), and any combination thereof.

24. A fluid composition according to any one of the preceding claims, wherein said one or more further agent is present in the range of 0.001% to 5% (w/w).

25. A fluid composition according to any one of the preceding claims, wherein the one or more fraction, liquid organic fraction, oil, lignin component, water, further agent, dispersing agent, surfactant, hydrotropic agent, emulsifier, preserving agent, and any combination thereof are in a state of being intermixed.

26. A fluid composition according to claim 25, wherein the state of being intermixed is selected from the group comprising or consisting of being intermixed as a solution; being intermixed as a suspension; being intermixed as an emulsion; being intermixed as a dispersion; being intermixed as a slurry; and any combination thereof.

27. A fluid composition according to any one of the preceding claims, wherein said lignin component comprises cellulose in an amount of 2,000 – 300,000 ppm, such as 3,000 – 180,000 ppm, e.g. 4,000 – 160,000 ppm, for example 5,000 – 140,000 ppm, such as 6,000 – 120,000 ppm, 7,000 – 100,000 ppm, for example 8,000 – 80,000 ppm, such as 9,000 – 70,000 ppm, e.g. 10,000 – 60,000 ppm, 12,000 – 50,000 ppm, such as 14,000 – 50,000 ppm, e.g. 16,000 – 40,000 ppm, 18,000 – 30,000 ppm, such as 20,000 – 28,000 ppm, for example 22,000 – 26,000 ppm (w/w) in relation to said fluid composition.

28. A fluid composition according to any one of the preceding claims, wherein said lignin component comprises hemicellulose in an amount of 2,000 – 200,000 ppm, such as 3,000 – 180,000 ppm, e.g. 4,000 – 160,000 ppm, for example 5,000 – 140,000 ppm, such as 6,000 – 120,000 ppm, 7,000 – 100,000 ppm, for example 8,000 – 80,000 ppm, such as 9,000 – 70,000 ppm, e.g. 10,000 – 60,000 ppm, 12,000 – 50,000 ppm, such as 14,000 – 50,000 ppm, e.g. 16,000 – 40,000 ppm, 18,000 – 30,000 ppm, such as 20,000 – 28,000 ppm, for example 22,000 – 26,000 ppm (w/w) in relation to said fluid composition.

29. A fluid composition according to any one of the preceding claims, wherein said lignin component comprises ash in an amount of 2,000 – 200,000 ppm, such as 3,000 – 180,000 ppm, e.g. 4,000 – 160,000 ppm, for example 5,000 – 140,000 ppm, such as 6,000 – 120,000 ppm, 7,000 – 100,000 ppm, for example 8,000 – 80,000 ppm, such as 9,000 – 70,000 ppm, e.g. 10,000 – 60,000 ppm, 12,000 – 50,000 ppm, such as 14,000 – 50,000 ppm, e.g. 16,000 – 40,000 ppm, 18,000 – 30,000 ppm, such as 20,000 – 28,000 ppm, for example 22,000 – 26,000 ppm (w/w) in relation to said fluid composition.

30. A fluid composition according to any one of claims 23 to 29, wherein said one or more dispersing agent is selected from the group comprising or consisting of non-ionic, anionic, cationic and amphoteric dispersing agent(s) and any combination and/or compatible mixture thereof.

31. A fluid composition according to any one of claims 23 to 30, wherein said dispersing agent is present in said fluid composition in an amount of 10- 50,000 ppm or 200 – 20,000 ppm, such as 300 – 18,000 ppm, e.g. 400 – 16,000 ppm, for example 500 – 14,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm, 1,200 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to said fluid composition.

32. A fluid composition according to any one of claims 23 to 31, wherein said one or more surfactant is selected from the group comprising or consisting of anionic, cationic, zwitterionic and nonionic surfactants, and any combination and/or compatible mixture thereof.

5 33. A fluid composition according to any one of claims 23 or 32, wherein said surfactant is present in said fluid composition in an amount of 10- 50,000 ppm or 200 – 20,000 ppm, such as 300 – 18,000 ppm, e.g. 400 – 16,000 ppm, for example 500 – 14,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for
10 example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm, 1,200 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to said fluid composition.

15 34. A fluid composition according to any one of claims 23 to 33, wherein said one or more hydrotrope is selected from the group comprising or consisting of: non-ionic, anionic, cationic and amphoteric hydrotropes and any combination and/or compatible mixtures thereof.

20 35. A fluid composition according to any one of claims 23 to 34, wherein said hydrotrope is present in said fluid composition in an amount of 10- 50,000 ppm or 200 – 40,000 ppm, such as 300 – 30,000 ppm, e.g. 400 – 20,000 ppm, for example 500 – 15,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm,
25 1,200 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to said fluid composition.

30 36. A fluid composition according to any one of claims 23 to 35, wherein said one or more emulsifier is selected from the group comprising or consisting of sodium phosphate(s), sodium stearyl lactylate cationic, lecithin, DATEM (diacetyl tartaric acid ester of monoglyceride), and any combination and/or compatible mixture thereof.

37. A fluid composition according to any one of claims 23 to 36, wherein said surfactant is present in said fluid composition in an amount of 10- 50,000 ppm or 200 – 20,000 ppm, such as 300 – 18,000 ppm, e.g. 400 – 16,000 ppm, for example 500 – 14,000 ppm, such as 600 – 12,000 ppm, 700 – 10,000 ppm, for example 800 – 8,000 ppm, such as 900 – 7,000 ppm, e.g. 1,000 - 6,000 ppm, 1,200 – 5,000 ppm, such as 1,400 – 5,000 ppm, e.g. 1,600 – 4,000 ppm, 1,800 – 3,000 ppm, such as 2,000 – 2,800 ppm, for example 2,200 – 2,600 ppm (w/w) in relation to said fluid composition.

38. A fluid composition according to any one of claims 23 to 37, wherein said preserving agent is selected from the group comprising or consisting of one or more carboxylate, benzoate, benzoic acid derivative such as parabene(s), aldehyde(s), thiazine(s), organic acid(s) and the like, and any combination thereof.

39. A fluid composition according to any one of claims 23 to 38, wherein said preserving agent is present in said fluid composition in an amount of 10- 50,000 ppm or 20 – 10,000 ppm, such as 30 – 8,000 ppm, e.g. 40 – 6,000 ppm, for example 50 – 5,000 ppm, such as 60 – 4,000 ppm, 70 – 3,000 ppm, for example 80 – 2,000 ppm, such as 90 – 1,500 ppm, e.g. 100 - 1,200 ppm, 120 – 1,000 ppm, such as 140 – 800 ppm, e.g. 160 – 600 ppm, 180 – 400 ppm, such as 200 – 300 ppm, for example 2,200 – 250 ppm (w/w) in relation to said fluid composition.

40. A fluid composition according to any one of the preceding claims, wherein the dry matter content of said lignin component in said fluid composition is 1.0 - 99% (w/w), 10 - 99 % (w/w) or 20 – 95 % (w/w), such as 21 – 94 % (w/w), e.g. 22 - 93 % (w/w), such as 23 – 92 % (w/w), such as 24 – 91 % (w/w), for example 25 – 90 % (w/w), such as 26 – 89 % (w/w), such as 27 – 88 % (w/w), for example 28 – 87 % (w/w), e.g. 29 – 86 % (w/w), such as 30 – 85 % (w/w), such as 31 – 84 % (w/w), such as 32 – 83 % (w/w), such as 33 – 82 % (w/w), for example 34 – 81 % (w/w), such as 35 – 80 % (w/w), e.g. 36 – 79 % (w/w), such as 37 – 78 % (w/w), e.g. 38 – 77 % (w/w), e.g. 39 – 76 % (w/w), such as 40 – 75 % (w/w), such as 41

– 74 % (w/w), such as 42 – 73 % (w/w), such as 43 – 72 % (w/w), for example 44 – 71 % (w/w), such as 45 – 70 % (w/w), e.g. 46 – 69 % (w/w), such as 47 – 68 % (w/w), e.g. 48 – 67 % (w/w), e.g. 49 – 66 % (w/w), such as 50 – 65 % (w/w), such as 51 – 64 % (w/w), such as 52 – 63 % (w/w), such as 53 – 62 % (w/w), for example 54 – 61 % (w/w), such as 55 – 60 % (w/w), e.g. 56 – 59 % (w/w), such as 57 – 58 % (w/w).

41. A fluid composition according to any one of the preceding claims, wherein lignin component comprises sulfur and the sulfur content of the dry matter content of said lignin component is 2.0 % (w/w) or less, such as 1.4 % (w/w) or less, such as 1.3 % (w/w) or less, for example 1.2 % (w/w) or less, such as 1.1 % (w/w) or less, e.g. 1.0 % (w/w) or less, such as 0.9 % (w/w) or less, for example 0.8 % (w/w) or less, such as 0.7 % (w/w) or less, e.g. 0.6 % (w/w) or less, e.g. 0.5 % (w/w) or less, such as 0.4 % (w/w) or less, for example 0.3 % (w/w) or less, such as 0.2 % (w/w) or less, or 0.1 % (w/w) or less, such as 0.09 % (w/w) or less, such as 0.08 % (w/w) or less, e.g. 0.07 % (w/w) or less, e.g. 0.06 % (w/w) or less, such as 0.05 % (w/w) or less, for example 0.04 % (w/w) or less, such as 0.03 % (w/w) or less, e.g. 0.02 % (w/w) or less, such as 0.01 % (w/w) or less.

42. A fluid composition according to any one of the preceding claims, wherein said lignin component in said fluid composition is having an average grain size of 1 - 2000 μm , 1-1500 μm , 1-1200 μm , 1-1000 μm , 1-800 μm , 1-600 μm , 1-500 μm , 1 - 450 μm , such as 1.5 - 430 μm , e.g. 2 - 420 μm , such as 3 - 410 μm , for example 4 - 400 μm , e.g. 5 - 390 μm , such as 6 - 380 μm , e.g. 7 - 370 μm , such as 8 - 360 μm , 9 - 350 μm , for example 10 - 340 μm , e.g. 12 - 330 μm , such as 14 - 320 μm , such as 16 - 310 μm , for example 18 - 300 μm , e.g. 20 - 290 μm , such as 22 - 280 μm , e.g. 25 - 270 μm , such as 30 - 260 μm , 35 - 250 μm , for example 40 - 240 μm , e.g. 45 - 230 μm , such as 50 - 220 μm , for example 60 - 210 μm , for example 70 - 200 μm , e.g. 80 - 190, for example 90 - 180 μm , e.g. 100 - 170 μm , such as 110 - 160 μm , 120 - 150 μm , for example 130 - 140 μm .

43. A fluid composition according to claim 42, wherein said average grain or particle size is determined before or after providing said fluid composition, and optionally,

wherein said grain or particle size being measured by laser diffraction spectroscopy, or e.g. by a Malvern Mastersizer.

44. A fluid composition according to any one of the preceding claims, wherein said
5 lignin component originates from a lignocellulosic biomass having been subjected to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of the hemicellulose present in said lignocellulosic biomass.

10 45. A fluid composition according to any one of the preceding claims, wherein said lignin component originates from a lignocellulosic biomass having been subject to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of the hemicellulose present in said lignocellulosic biomass; and optionally followed by a fermentation, such as an alcohol
15 fermentation.

46. A fluid composition according to claim 44 or 45, wherein said hydrolysis is an acid catalyzed hydrolysis, an enzymatic hydrolysis or a combination of acid/enzyme-catalyzed hydrolysis.

20 47. A fluid composition according to any one of the preceding claims, wherein said lignin component is having an average molecular weight (Da) of 1,000 or above, 1,500 or above, 2,000 or above, 2,500 or above, 3,000 or above, such as 3,500 or above, e.g. 4,000 or above, such as 5,000 or above, for example 5,500 or
25 above, such as 6,000 or above, e.g. 7,000 or above, for example 8,000 or above, such as 9,000 or above, for example 10,000 or above, such as 12,000 or above, e.g. 14,000 or above, for example 16,000 or above, e.g. 18,000 or above, e.g. 20,000 or above, such as 25,000 or above, e.g. 30,000 or above, such as 35,000 or above, for example 40,000 or above, such as 45,000 or above, e.g. 50,000 or
30 above, such as 55,000 or above, e.g. 60,000 or above, such as 65,000 or above, e.g. 70,000 or above, such as 75,000 or above, for example 80,000 or above, such as 85,000 or above, e.g. 90,000 or above, such as 95,000 or above, or 100,000 or above.

48. A fluid composition according to any one of the preceding claims, wherein said lignin component originates from a lignocellulosic biomass obtained from an annual or a perennial plant.

49. A fluid composition according to any one of the preceding claims, wherein said lignin component originates from a lignocellulosic biomass obtained, obtainable or derived from the group comprising or consisting of one or more of: cereal, wheat, wheat straw, rice, rice straw, corn, corn fiber, corn cobs, corn stover, hardwood bulk, softwood bulk, sugar cane, sweat sorghum, bagasse, nut shells, empty fruit bunches, grass, cotton seed hairs, barley, rye, oats, sorghum, brewer's spent grains, palm waste material, wood, soft lignocellulosic biomass, and any combination thereof.

50. A fluid composition according to any one of the preceding claims, wherein said lignin component comprises one or more impurities originating from its mode of production, such as enzyme residues, yeast residues, foam depressant(s), clean in place (CIP) compounds, salts and the like.

51. A fluid composition according to any one of the preceding claims, wherein said lignin component comprises impurity/impurities originating from compounds native for the lignocellulosic material, such as cellulose residues, hemicellulose residues, monomeric sugar compounds, dimeric sugar compounds, oligomeric sugar compounds, carbohydrate residues, wax residues, minerals, ash, silica (SiO₂), silica comprising compositions, salts, organic acids, and the like, and any combination thereof.

52. A fluid composition according to any one of the preceding claims, wherein the purity of said lignin component is 50 % (w/w) or more, such as 52 % (w/w) or more, for example 54 % (w/w) or more, such as 56 % (w/w) or more, e.g. 58 % (w/w) or more, such as 60 % (w/w) or more, such as 62 % (w/w) or more, for example 64 % (w/w) or more, such as 66 % (w/w) or more, e.g. 68 % (w/w) or more, such as 70 % (w/w) or more, such as 72 % (w/w) or more, for example 74

% (w/w) or more, such as 76 % (w/w) or more, e.g. 78 % (w/w) or more, such as 80 % (w/w) or more, such as 82 % (w/w) or more, for example 84 % (w/w) or more, such as 86 % (w/w) or more, e.g. 88 % (w/w) or more, such as 90 % (w/w) or more, such as 92 % (w/w) or more, for example 94 % (w/w) or more, such as 96 % (w/w) or more, e.g. 98 % (w/w) or more.

53. A fluid composition according to claim 48, wherein said purity is determined based on content of Klason lignin or acid insoluble lignin, and optionally, wherein the corresponding percentage constituting impurities may be any one or more impurity as defined in claims 50 or 51.

54. A fluid composition according to any one of the preceding claims, wherein the content of said organic fraction in said fluid composition is at least 2, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, 90, or 95 % (w/w) or more, such as 2 – 95 % (w/w), such as 4 – 78 % (w/w), e.g. 6 – 76 % (w/w), such as 8 – 74 % (w/w), e.g. 10 – 72 % (w/w), such as 12 – 70 % (w/w), e.g. 14 – 68 % (w/w), such as 16 – 66 % (w/w), for example 18 – 64 % (w/w), such as 20 – 62 % (w/w), e.g. 22 – 60 % (w/w), for example 24 – 58 % (w/w), such as 26 – 56 % (w/w), such as 28 – 54 % (w/w), such as 30 – 52 % (w/w), 32 – 50 % (w/w), e.g. 34 – 48 % (w/w), such as 36 – 46 % (w/w), such as 38 – 44 % (w/w), for example 40 – 42 % (w/w).

55. A fluid composition according to any one of the preceding claims, wherein said organic fraction comprises or consists essentially of an organic solvent, a distillate, and/or a residue from a hydrocarbon distillation.

56. A fluid composition according to claim 55, wherein said distillate is selected from the group comprising or consisting of one or more mineral oil, kerosene, diesel, No. 2 fuel oil, No. 3 fuel oil, No. 4 fuel oil, No. 5 fuel oil, No. 6 fuel oil, and No. 7 fuel oil, and any mixture(s) thereof.

57. A fluid composition according to any one of the preceding claims, wherein said one or more organic compound of said liquid organic fraction is of plant origin or animal origin.

58. A fluid composition according to claim 57, wherein said one or more organic compound of said liquid organic fraction is an oil of plant origin or a fat of animal origin.

5 59. A fluid composition according to any one of the preceding claims, wherein said one or more organic compound of said liquid organic fraction is an oil originating from pyrolysis of a biomass, such as a cellulosic or lignocellulosic material or wherein said oil is a pyrolysis oil originating from pyrolysis of a lignin component.

10 60. A fluid composition according to any one of the preceding claims, wherein said one or more organic compound of said liquid organic fraction is an oil originating from pyrolysis of a polymer, such as a synthetic plastic or synthetic elastomer.

15 61. A fluid composition according to any one of the preceding claims, wherein said one or more organic compound of said liquid organic fraction is selected from the group comprising or consisting of glycerol, biodiesel, synfuel, biomass to liquid (BTL) diesel, gas to liquid (GTL) diesel, coal to liquid (CTL) diesel, and any combination thereof.

20 62. A fluid composition according to any one of the preceding claims, wherein said one or more organic compound of said liquid organic fraction originates from treatment of a biomass with water and/or other polar liquid(s), such as ethanol or methanol.

25 63. A fluid composition according to any one of the preceding claims, wherein the said biomass treatment comprises treatment under supercritical conditions.

30 64. A fluid composition according to any one of the preceding claims, wherein said biomass which has been treated with water or other polar liquid(s) under supercritical conditions is selected from the group comprising or consisting of one or more lignocellulosic material, cellulose, lignin component, and any combination thereof.

65. A fluid composition according to any one of the preceding claims, wherein said liquid organic fraction or compound of said liquid organic fraction is in itself a mixture of two or more such organic substances, such as three or more such organic substances, e.g. four or more such organic substances, such as five or six or more of such organic substances.

66. A fluid composition according to any one of the preceding claims, wherein the sulfur content of said liquid organic fraction, and/or of the one or more organic compound and/or substance of said organic liquid fraction is 5.0 % (w/w) or less, such as 4.5 % (w/w) or less, for example 4.0 % (w/w) or less, such as 3.8 % (w/w) or less, e.g. 3.6 % (w/w) or less, for example 3.4 % (w/w) or less, e.g. 3.2 % (w/w) or less, such as 3.0 % (w/w) or less, for example 2.8 % (w/w) or less, e.g. 2.6 % (w/w) or less, for example 2.4 % (w/w) or less, e.g. 2.2 % (w/w) or less, such as 2.0 % (w/w) or less, for example 1.8 % (w/w) or less, such as 1.6 % (w/w) or less, for example 1.4 % (w/w) or less, e.g. 1.2 % (w/w) or less, such as 1.0 % (w/w) or less, for example 0.8 % (w/w) or less, such as 0.4 % (w/w) or less, such as 0.2 % (w/w) or less, for example 0.1 % (w/w) or less, such as 0.08 % (w/w) or less, e.g. 0.06 % (w/w) or less, such as 0.04 % (w/w) or less, e.g. 0.02 % (w/w) or less, for example 0.01 % (w/w) or less, such as 0.008 % (w/w) or less, e.g. 0.006 % (w/w) or less, such as 0.004 % (w/w) or less, e.g. 0.002 % (w/w) or less, such as 0.001 % (w/w), such as 800 ppm or less, e.g. 600 ppm or less, such as 400 ppm or less, e.g. 200 ppm or less, for example 100 ppm or less, such as 50 ppm (w/w) or less.

67. A fluid composition according to any one of the preceding claims, wherein said liquid organic fraction, organic compound or substance of said liquid organic fraction is immiscible with water.

68. A fluid composition according to any one of the preceding claims, wherein said fluid composition, organic fraction, one or more organic compound or substance at 25 °C is having a viscosity of 0.0005 – 10,000 CSt, such as 0.0010 – 9,000 CSt, e.g. 0.0050 – 8,000 CSt, for example 0.01 – 6,000 CSt, for example 0.05 – 4,000 CSt, such as 0.1 – 2,000 CSt, e.g. 0.5 – 1,000 CSt, such as 1.0 – 800 CSt,

e.g. 5.0 – 600 CSt, such as 10 – 400 CSt, for example 50 – 300 CSt, such as 100 – 200 CSt.

69. A fluid composition according to any one of the preceding claims, wherein said
5 fluid composition, organic fraction, one or more organic compound or substance,
wherein said organic substance at 50 °C is having a viscosity of 0.0004 – 2,000
CSt, such as 0.0010 – 1,500 CSt, e.g. 0.0050 – 1,000 CSt, for example 0.01 –
800 CSt, for example 0.05 – 600 CSt, such as 0.1 – 400 CSt, e.g. 0.5 – 200 CSt,
such as 1.0 – 100 CSt, e.g. 5.0 – 80 CSt, such as 10 – 70 CSt, for example 20 –
10 50 CSt, such as 30 – 40 CSt.

70. A fluid composition according to any one of the preceding claims, wherein said
fluid composition, organic fraction, one or more organic compound or substance,
wherein said organic substance at 75 °C is having a viscosity of 0.0002 – 200 CSt,
15 such as 0.0001 – 150 CSt, e.g. 0.001 – 100 CSt, for example 0.005 – 80 CSt,
such as 0.01 – 60 CSt, e.g. 0.05 – 40 CSt, such as 0.05 – 20 CSt, for example
0.1 – 10 CSt, such as 0.5 – 5 CSt, for example 1.0 – 3 CSt.

71. A fluid composition according to any one of the preceding claims, wherein the
20 content of said water in said fluid composition is less than 90, 85, 80, 75, 70, 65,
60, 55, 50, 45, 40, 35, 30, 25, 20, 15, 12, 10, 8, 7, 6, 5, 4, 3, 2, 1, 0.5 % (w/w)
such as in the range of 2 – 80 % (w/w), such as 4 – 78 % (w/w), e.g. 6 – 76 %
(w/w), such as 8 – 74 % (w/w), e.g. 10 – 72 % (w/w), such as 12 – 70 % (w/w),
e.g. 14 – 68 % (w/w), such as 16 – 66 % (w/w), for example 18 – 64 % (w/w), such
25 as 20 – 62 % (w/w), e.g. 22 – 60 % (w/w), for example 24 – 58 % (w/w), such as
26 – 56 % (w/w), such as 28 – 54 % (w/w), such as 30 – 52 % (w/w), 32 – 50 %
(w/w), e.g. 34 – 48 % (w/w), such as 36 – 46 % (w/w), such as 38 – 44 % (w/w),
for example 40 – 42 % (w/w).

30 72. A fluid composition according to any one of the preceding claims, wherein the
ratio lignin component : water is selected from the range of 0.4 – 8.0, such as 0.5
– 7.9, e.g. 0.6 – 7.8, such as 0.7 – 7.6, for example 0.8 – 7.5, for example 0.9 –
7.4, such as 1.0 – 7.3, for example 1.1 – 7.2, e.g. 1.2 – 7.1, such as 1.3 – 7.0, for

example 1.4 – 6.9, such as 1.5 – 6.8, such as 1.6 – 6.7, such as 1.7 – 6.6, for example 1.8 – 6.5, for example 1.9 – 6.4, such as 2.0 – 6.3, for example 2.1 – 6.2, e.g. 2.2 – 6.1, such as 2.3 – 6.0, for example 2.4 – 5.9, such as 2.5 – 5.8, such as 2.6 – 5.7, such as for example 2.8 – 5.5, for example 2.9 – 5.4, such as 3.0 – 5.3, for example 3.1 – 5.2, e.g. 3.2 – 5.1, such as 3.3 – 5.0, for example 3.4 – 4.9, such as 3.5 – 4.8, such as 3.6 – 4.7, such as 3.7 – 4.6, for example 3.8 – 4.5, for example 3.9 – 4.4, such as 4.0 – 4.3, for example 4.1 – 4.2, all ratios being based on dry matter content of said lignin component.

- 10 73. A fluid composition according to any one of the preceding claims, wherein said fluid composition at 25, 50 or 75 °C is having a viscosity of 20 – 10,000 CSt, such as 50 – 8,000 CSt, for example 100 – 6,000 CSt, such as 200 – 4,000 CSt, such as 400 – 2,000 CSt, e.g. 500 – 1,000 CSt, such as 600 – 800 CSt.
- 15 74. A fluid composition according to any one of the preceding claims, wherein said fluid composition at 25, 50 or 75 °C is having a viscosity of 5 – 2,000 CSt, such as 10 – 1,000 CSt, for example 20 – 800 CSt, such as 50 – 600 CSt, e.g. 100 – 400 CSt, such as 200 – 300 CSt.
- 20 75. A fluid composition according to any one of the preceding claims, wherein said fluid composition at 25, 50 or 75 °C is having a viscosity of 2 -200 CSt, such as 5 – 150 CSt, e.g. 10 – 120 CSt, such as 20 – 100 CSt, for example 30 – 80 CSt, such as 40 - 60 CSt.
- 25 76. A fluid composition according to any one of the preceding claims, wherein said fluid composition is having a lower heating value of 4 – 37 MJ/kg, such as 5 – 36 MJ/kg., for example 6 – 35 MJ/kg, such as 7 – 34 MJ/kg, for example 8 - 33 MJ/kg, e.g. 9 – 32 MJ/kg, such as 10 – 31 MJ/kg, for example 11 - 30 MJ/kg, such as 12 – 29 MJ/kg, e.g. 13 – 28 MJ/kg, such as 14 – 27 MJ/kg, such as 15 – 26 MJ/kg., for example 16 – 25 MJ/kg, such as 17 – 24 MJ/kg, for example 18 - 23 MJ/kg, e.g. 19 – 22 MJ/kg, such as 20 – 21 MJ/kg.
- 30

77. A fluid composition according to any one of the preceding claims, wherein said fluid composition is stable and/or pumpable for 2 weeks or more, such as 3 weeks or more, e.g. 4 weeks or more, such as 6 weeks or more, for example 7 weeks or more, such as 8 weeks or more, such as 2 months or more, e.g. 3 months or more, for example 4 months or more, such as 5 months or more, or 6 months or more; in the sense that no more than 5.0, 4.0, 3.0, 2.0, 1.0 or 0.5 % (w/w) of any one of the fractions (e.g. water and or lignin component fraction) of said fluid composition will separate after said specified period of time.

78. A fluid composition according to any one of the preceding claims, wherein gentle stirring, agitation, and/or re-circulation is required for maintaining said stability and/or pumpability.

79. A fluid composition according to any one of the preceding claims, wherein the sulfur content of said fluid composition is 3.0 % (w/w) or less, for example 2.8 % (w/w) or less, e.g. 2.6 % (w/w) or less, for example 2.4 % (w/w) or less, e.g. 2.2 % (w/w) or less, such as 2.0 % (w/w) or less, for example 1.8 % (w/w) or less, such as 1.6 % (w/w) or less, for example 1.4 % (w/w) or less, e.g. 1.2 % (w/w) or less, such as 1.0 % (w/w) or less, for example 0.8 % (w/w) or less, such as 0.4 % (w/w) or less, such as 0.2 % (w/w) or less, for example 0.1 % (w/w) or less, such as 0.08 % (w/w) or less, e.g. 0.06 % (w/w) or less, such as 0.04 % (w/w) or less.

80. A process for the manufacture of a fluid composition according to any one of the preceding claims, or claims 148-155, said process comprising the steps of:

- i. providing a fraction, preferably a solid fraction comprising a lignin component;
- ii. providing an organic compound to make up at least part of said liquid organic fraction;
- iii. providing a vinasse, such as a vinasse according to any one of claims 221-231, and/or a vinasse obtained or obtainable according to any one of claims 201-220;

- iv. intermixing the fraction provided in step (i) with the organic compound and/or liquid organic fraction provided in step (ii), and/or the vinasse provided in step (iii).

- 5 81. A process according to claim 80, wherein said lignin component originates from a lignocellulosic biomass which has been subjected to a hydrothermal pretreatment followed by a hydrolysis of at least part of the cellulose and at least part of said hemicellulose present in said lignocellulosic biomass, optionally followed by fermentation and/or hydrolysis.
- 10 82. A process according to claim 80 or 81, wherein said lignin component is obtained by pressing a fibrous fraction obtained after subjecting said lignocellulosic biomass to said hydrothermal pretreatment followed by said hydrolysis.
- 15 83. A process according to claim 82, wherein said pressing of said fibrous fraction is preceded by rinsing and/or washing of said fibrous fraction.
84. A process according to claim 82 or 83, wherein said lignin component is obtained by mechanically comminuting said pressed fibrous fraction to a desired extent.
- 20 85. A process according to any one of the claims 80-84, wherein said lignin component is having characteristics as defined in any of the preceding claims.
86. A process according to any one of the claims 80-85, wherein said organic substance of said liquid fraction is having characteristics as defined in any of the preceding claims.
- 25 87. A process according to any one of the claims 80-86, further comprising admixing of an amount of water.
- 30 88. A process according to one of the claims 80-87, further comprising admixing of one or more further agent.

89. A process according to claim 88, wherein the further agent is selected from the group comprising or consisting of one or more dispersing agent(s), surfactant(s), hydrotropic agent(s), emulsifier(s), preserving agent(s), and any combination thereof.

90. A process according to any one of the claims 80-89, wherein said intermixing is performed using one or more mixing device(s), such as a mechanical stirrer, high shear mixer, and/or a pump.

91. A process according to any of the claims 80-90, comprising the step of separately intermixing an amount of water and optionally one or more further agent(s) such as a dispersing agent and with (a) said lignin component, (b) said organic compound of said liquid organic fraction, and/or (c) said liquid organic fraction, and optionally wherein said separately mixed mixtures are mixed and stirred.

92. A process for treatment of a lignocellulosic biomass, said process comprising:

a) subjecting said lignocellulosic biomass for hydrothermal pretreatment resulting in a hydrothermally pretreated lignocellulosic biomass;

b) subjecting at least part of said hydrothermally pretreated lignocellulosic biomass obtained in step (a) to a hydrolysis resulting in a liquid fraction comprising soluble carbohydrates, and a fiber fraction comprising a lignin component;

c) optionally subjecting at least part of the liquid fraction obtained in step (b) to a fermentation in order to ferment at least part of said soluble carbohydrates to a fermentation product, such as ethanol, methane or butanol, thereby obtaining a fermentation broth;

d) optionally isolating at least part of said fermentation product from the fermentation broth obtained in step (c) e.g. by distillation;

e) isolating at least part of the lignin component from one or more of: the fiber fraction obtained in step (b); the fermentation broth obtained in step (c); or after isolation of at least a part of the fermentation product in step (d);

f) converting at least part of the lignin component obtained in step (e) to a fluid composition by admixing said lignin component with a liquid organic fraction comprising an organic compound or substance, and/or a vinasse according to any

one of claims 221-231, and/or a vinasse provided according to any one of claims 201-220;.

93. A process according to claim 92, wherein the fluid composition obtained in step (f) is a fluid composition according to any one of the preceding claims.

94. A process according to claim 92 or 93, wherein the vinasse is provided by removal of water from a whole stillage (lignin-rich vinasse) and/or from a thin stillage (lignin-depleted vinasse), and optionally, wherein said lignin-rich vinasse and/or lignin-depleted vinasse are provided (i) from the fiber fraction obtained in step (b); (ii) the fermentation broth obtained in step (c); and/or (iii) after isolation of at least a part of the fermentation product in step (d); said vinasse being provided either before (lignin-rich vinasse) or after removal of the lignin component (lignin-depleted vinasse).

95. A process according to any one of the claims 92 to 94, wherein said at least part of said lignin fraction is isolated from the fiber fraction obtained in step (b) or from said fermentation broth obtained in step (c).

96. A process according to any one of the claims 92 to 95, wherein said lignin component is obtained in step (e) by removing an associated liquid phase by using one or more separation device(s), such as a hydraulic press, a vacuum filtration unit, a belt filter, a rotary filter or a centrifuge decanter.

97. A process according to any one of the claims 92 to 96, wherein said lignin component obtained in step (e) is dried to a residual water content at 110 °C of 2 – 20 % (w/w), such as 4 – 18 % (w/w), for example 6 – 16 % (w/w), such as 8 – 14 % (w/w), e.g. 10 – 12 % (w/w).

98. A process according to any one of the claims 92 to 97, wherein said hydrothermal pretreatment of said lignocellulosic biomass is performed at a temperature of 150 – 260 °C, such as 160 – 250 °C, e.g. 170 – 240 °C, such as 180 – 230 °C, for example 190 – 220 °C, such as 200 – 210 °C.

99. A process according to any of the claims 92 to 98, wherein said hydrothermal pretreatment of said lignocellulosic biomass is performed in a period of residence time of 2 – 120 min., such as 5 – 110 min., e.g. 10 – 100 min., for example 15 – 90 min., such as 20 - 80 min., such as 25 – 70 min., e.g. 30 – 60 min, such as 35 – 50 min, such as 40 – 45 min.

100. A process according to any one of the claims 92 to 99, wherein said hydrothermal pretreatment of said lignocellulosic biomass is performed by subjecting said lignocellulosic biomass to a log severity, $\log(R_o)$ of 2.5 or more, such as a $\log(R_o)$ of 2.6 or more, e.g. a $\log(R_o)$ of 2.7 or more, such as a $\log(R_o)$ of 2.8 or more, for example a $\log(R_o)$ of 2.9 or more, such as a $\log(R_o)$ of 3.0 or more, such as a $\log(R_o)$ of 3.1 or more, for example a $\log(R_o)$ of 3.2 or more, e.g. a $\log(R_o)$ of 3.3 or more, such as a $\log(R_o)$ of 3.4 or more, such as a $\log(R_o)$ of 3.5 or more; such as a $\log(R_o)$ of 3.6 or more; for example such as a $\log(R_o)$ of 3.7 or more, e.g. a $\log(R_o)$ of 3.8 or more, for example a $\log(R_o)$ of 3.9 or more, for example a $\log(R_o)$ of 4.0 or more, such as a $\log(R_o)$ of 4.1 or more, or a $\log(R_o)$ of 4.2 or more; wherein the log severity is defined as: $\log(R_o) = (\text{residence time}) \times (\exp[\text{Temperature} - 100/14.75])$.

101. A process according to any one of the claims 92 to 100, wherein said hydrolysis is an acid catalyzed hydrolysis and/or enzymatic hydrolysis.

102. A process according to claim 101, wherein said hydrolysis is performed by one or more cellulases.

103. A process according to claim 102, wherein said one or more cellulases are selected from the group comprising exo-glucanases, endo-glucanases, hemicellulases and beta-glucosidases.

104. A process according to any one of the claims 92 to 103 wherein said hydrolysis is performed for a period of time of 1 – 200 hours, such as 5 – 190 hours, such as 10 – 185 hours, e.g. 15 – 180 hours, for example 20 – 175 hours, such as 25 – 170 hours, such as 30 – 165 hours, e.g. 35 – 160 hours, for

example 40 – 155 hours, such as 45 – 150 hours, such as 50 – 145 hours, e.g. 55 – 140 hours, for example 60 – 135 hours, such as 65 – 130 hours, such as 70 – 125 hours, e.g. 75 – 120 hours, for example 80 – 115 hours, such as 85 – 110 hours, such as 90 – 105 hours, e.g. 95 – 100 hours.

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105. A process according to any one of the claims 92 to 104, wherein said step (b) and step (c) are performed as a separate hydrolysis and fermentation step (SHF), and wherein said hydrolysis is performed at a temperature of 30 – 72 °C, such as 32 – 70 °C, e.g. 34 – 68 °C, for example 36 – 66 °C, such as 38 – 64 °C, e.g. 40 – 62 °C, 42 – 60 °C, e.g. 44 – 58 °C, for example 46 – 56 °C, such as 48 – 54 °C, e.g. 50 – 52 °C.

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106. A process according to claim 105, wherein said hydrolysis is performed in a period of time of 70 – 125 hours, e.g. 75 – 120 hours, for example 80 – 115 hours, such as 85 – 110 hours, such as 90 – 105 hours, e.g. 95 – 100 hours.

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107. A process according to any one of the claims 92 to 106, wherein said step (b) and step (c) are performed as a simultaneous saccharification and fermentation step (SSF), and wherein said hydrolysis is performed at a temperature of 30 – 72 °C, such as 32 – 70 °C, e.g. 34 – 68 °C, for example 36 – 66 °C, such as 38 – 64 °C, e.g. 40 – 62 °C, 42 – 60 °C, e.g. 44 – 58 °C, for example 46 – 56 °C, such as 48 – 54 °C, e.g. 50 – 52 °C.

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108. A process according to claim 107, wherein said hydrolysis is performed in a period of time of 1 – 12 hours, such as 2 – 11 hours, for example 3 – 10 hours, such as 4 – 9 hours, e.g. 5 – 8 hours, such as 6 – 7 hours.

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109. A process according to any one of the claims 92 to 108, wherein said step (b) and step (c) are performed as a simultaneous saccharification and fermentation step (SSF), and wherein said fermentation is performed at a temperature of 25 – 40 °C, such as 26 – 39 °C, e.g. 27 – 38 °C, for example 28 – 37 °C, e.g. 29 – 36 °C, for example 30 – 35 °C, such as 31 – 34 °C or 32 – 33 °C.

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110. A process according to claims 92 to 109 wherein said fermentation is performed in a period of time of 100 – 200 hours, such as 105 – 190 hours, such as 110 – 185 hours, e.g. 115 – 180 hours, for example 120 – 175 hours, such as 125 – 170 hours, such as 130 – 165 hours, e.g. 135 – 160 hours, for example 140 – 155 hours, such as 145 – 150 hours.

111. A process according to any one of the claims 92 to 110, wherein said lignin fraction obtained in step e) is converted to a fluid composition by admixing said lignin fraction with an organic substance, said organic substance constituting a liquid fraction.

112. A process according to any one of the claims 92 to 111, wherein said lignin fraction obtained in step e) is converted to a fluid composition by admixing said lignin fraction with an organic substance and with vinasse, such as a vinasse according to any one of claims 92-111, 221-231, and/or provided according to any one of claims 201-220 and optionally water, said organic substance constituting a liquid fraction.

113. A process according to any of the claims 92 to 112, wherein said lignin fraction obtained in step e) is converted to a fluid composition by admixing said lignin fraction with an organic substance; a vinasse according to any one of claims 92-112, 221-231, and/or provided according to any one of claims 201-220; and optionally with water and/or and with a dispersing agent.

114. Use of a fluid composition according to any of claims 1-79, 148-155 or a fluid composition provided by a process according to any one of claims 80-113 as a fuel.

115. Use according to claim 114, wherein the fluid composition is used as a fuel for a household burner.

116. Use according to claim 114, wherein the fluid composition is used as a fuel for a boiler in a district heat plant or in a combined heat and power (CHP) plant.

117. Use according to claim 115 or 116, wherein the fluid composition is used as a fuel for producing steam or other thermal energy products in an industry or factory using such steam or other thermal energy products to power its power consuming facilities.

118. Use according to any one of claims 114, 116, or 117, wherein the fluid composition is used as a fuel in a boiler in a power plant.

119. Use according to claim any one of claims 114, 116-118, wherein the fluid composition is used as a fuel in a start-up situation in a boiler in a power plant.

120. Use of lignin or a lignin component for a fluid composition according to any one of claims 1 to 113, 148-155, and optionally, wherein said lignin or a lignin component is dry, i.e. having a residual water content of or around 20 % (w/w) or below, such as of or around 15 % (w/w), such as of or around 10 % (w/w), such as of or around 5 % (w/w), such as of or around 2.5 % (w/w), or lower.

121. Use according to claim 120, wherein said solid lignin component originates from a lignocellulosic biomass which has been subjected to a hydrothermal pretreatment followed by a hydrolysis.

122. Use according to claim 120 or 121, wherein said lignin component originates from a lignocellulosic biomass which has been subjected to a hydrothermal pretreatment followed by fermentation and/or distillation.

123. Use according to claim 121 or 122, wherein said hydrolysis is an acid catalyzed hydrolysis.

124. Use according to claim 121 or 122, wherein said hydrolysis is an enzymatic hydrolysis.

125. Use according to claim 121 or 122, wherein said hydrolysis comprises acid and enzymatic hydrolysis.

126. Use according to any one of claims 119-125, wherein said fluid composition comprises a solid fraction and a liquid fraction.

127. Use according to claim 126, wherein said solid fraction and said liquid fraction are present in a state of being intermixed; said solid fraction comprises said lignin component; and said liquid fraction comprises an organic substance.

128. Use according to any of the claims 119-127, wherein said fluid composition is a fluid composition according to any of the claims 1 – 113, 148-155.

129. Use of a fluid composition according to any of the claims 1 – 113, 148-155 for chemical processing of a lignin component and/or providing a conversion product thereof.

130. Use according to claim 129, wherein said chemical processing involves catalytic processing of said lignin component or a conversion product thereof.

131. Use according to claim 129 or 130, wherein said chemical processing involves non-catalytic processing of said lignin component or a conversion product thereof.

132. Use according to any one of claims 129 to 131, wherein said chemical processing involves acid and/or base reactions of said lignin component or a conversion product thereof.

133. Use according to any one of claims 129 to 132, wherein said chemical processing involves an oxidation reaction of said lignin component or a conversion product thereof.

134. Use according to any one of claims 129 to 133, wherein said chemical processing involves a reduction reaction of said lignin component or a conversion product thereof.

135. Use according to any one of claims 129 to 134, wherein said chemical processing involves a hydrolysis reaction of said lignin component or a conversion product thereof.

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136. Use according to any one of claims 129 to 135, wherein said chemical processing involves a pyrolysis of said lignin component or a conversion product thereof.

10 137. Use according to any one of claims 129 to 136, wherein said chemical processing involves a hydrothermal conversion of said lignin component or a conversion product thereof.

15 138. Use according to any one of claims 129 to 137, wherein said chemical processing involves a supercritical fluid conversion of said lignin component or a conversion product thereof, such as a conversion involving water or methanol or ethanol at supercritical conditions.

20 139. Use according to any one of claims 129 to 138, wherein said chemical processing involves hydrogenation of said lignin component or a conversion product thereof.

25 140. Use according to any one of claims 129 to 139, wherein said chemical processing involves hydrodesulfurization of said lignin component or a conversion product thereof.

30 141. Use according to any one of claims 129 to 140, wherein said chemical processing involves hydrodenitrogenation of said lignin component or a conversion product thereof.

142. Use according to any one of claims 129 to 141, wherein said chemical processing involves hydrodeoxygenation of said lignin component or a conversion product thereof.

143. Use according to any one of claims 129 to 142, wherein said chemical processing involves hydrocracking of said lignin component or a conversion product thereof.

5 144. Use according to any one of claims 129 to 143, wherein said chemical processing involves hydrodenitrification of said lignin component or a conversion product thereof.

10 145. Use according to any one of claims 129 to 144 wherein said chemical processing involves cracking of said lignin component of said lignin component or a conversion product thereof.

146. Use according to claim 145, wherein said cracking is a technical and/or catalytical cracking of said lignin component or a conversion product thereof.

15 147. Use according to any one of claim 114-146, in a system according to any one of claims 260-272.

20 148. A fluid composition according to any one of claims 1-113, comprising, containing, consisting, or consisting essentially of:

- 25
- i. "L" (Lignin component) 5-60 % (w/w)
 - ii. "O" (Liquid organic fraction) 0-60 % (w/w)
 - iii. "W" (Vinasse, e.g. according to any one of claims 221-231) 0.1-95 % (w/w), and optionally
 - iv. "A" (Further agent) 0-5.0 or 0-1.0 % (w/w); and optionally water.

30 149. A fluid composition according to claim 148, wherein the "L-O-W" expressed in the individual % (w/w) is, or is around:
5-0-95, 5-5-90, 5-10-85, 5-15-80, 5-20-75, 5-25-70, 5-30-65, 5-35-60, 10-0-90, 10-5-85, 10-10-80, 10-15-75, 10-20-70, 10-25-65, 10-30-60, 15-0-85, 15-5-80, 15-10-75, 15-15-70, 15-20-65, 15-25-60, 15-30-55, 20-0-80, 20-5-75, 20-10-70, 20-15-65, 20-20-60, 20-25-55, 20-30-50, 25-0-75, 25-5-70, 25-10-65, 25-15-60, 25-20-55, 25-25-50, 25-30-45, 30-0-70, 30-5-65, 30-10-60, 30-15-55, 30-20-50, 30-

25-45, 30-30-40, 35-0-65, 35-5-60, 35-10-55, 35-15-50, 35-20-45, 35-25-40, 35-30-35, 40-0-60, 40-5-55, 40-10-50, 40-15-45, 40-20-40, 40-25-35, 40-30-30, 45-0-55, 45-5-50, 45-10-45, 45-15-40, 45-20-35, 45-25-30, 45-30-25, 50-0-50, 50-5-45, 50-10-40, 50-15-35, 50-20-30, 50-25-25, 50-30-20, 55-0-45, 55-5-40, 55-10-35, 55-15-30, 55-20-25, 55-25-20, 55-30-15, 60-0-40, 60-5-35, 60-10-30, 60-15-25, 60-20-20, 60-25-15, 60-30-10, 10-35-55, 15-35-50, 20-35-45, 25-35-40, 30-35-35, 35-35-30, 40-35-25, 45-35-20, 50-35-15, 55-35-10, 60-35-5, 5-40-55, 10-40-50, 15-40-45, 20-40-40, 25-40-35, 30-40-30, 35-40-25, 40-40-20, 45-40-15, 50-40-10, 55-40-5, 59.9-40-0.1, 5-45-50, 10-45-45, 15-45-40, 20-45-35, 25-45-30, 30-45-25, 35-45-20, 40-45-15, 45-45-10, 50-45-5, 54.9-45-0.1, 5-50-45, 10-50-40, 15-50-35, 20-50-30, 25-50-25, 30-50-20, 35-50-15, 40-50-10, 45-50-5, 49.9-50-0.1, 5-55-40, 10-55-35, 15-55-30, 20-55-25, 25-55-20, 30-55-15, 35-55-10, 40-55-5, 44.9-55-0.1, 5-60-35, 10-60-30, 15-60-25, 20-60-20, 25-60-15, 30-60-10, 35-60-5, or 39.9-60-0.1.

150. A fluid composition according to claim 148 or 149, wherein the lignin component is, or is around 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w).

151. A fluid composition according to any one of claims 148-150, wherein the liquid organic fraction is, or is around 0, 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w).

152. A fluid composition according to any one of claims 148-151, wherein the water content is, or is around 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w); and/or wherein the vinasse content is, or is around 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, or 60 % (w/w).

153. A fluid composition according to any one of claims 149-152, wherein "around" means +/- 1, 2, or 2.5% (w/w) based on the total composition.

154. A fluid composition according to any one of claims 148-153, wherein the further agent is or is around 0, 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.40, 0.45, 0.50, 0.55, 0.60, 0.65, 0.70, 0.75, 0.8, 0.85, 0.90, 0.95, or 1.0 % (w/w).

5 155. A fluid composition according to claim 154, wherein "around" means +/- 0.1, 0.2, or 0.25% (w/w) based on the total composition.

156. A process according to any one of claims 80-113 comprising the fluid composition according to any one of claims 148-155.

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157. A use according to any one of claims 114-147 comprising the fluid composition according to any one of claims 148-155.

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201. A method for providing a vinasse, said vinasse being lignin-rich and/or lignin-depleted vinasse, said method comprising the steps of:

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- (i) pretreatment of lignocellulosic biomass;
- (ii) hydrolysis of at least a portion of the pretreated lignocellulosic biomass from step (i) to provide one or more products;
- (iii) fermentation of the one or more products from step (ii) to provide a fermentation product;
- (iv) separation/removal of the one or more products from step (ii), and/or the fermentation product from step (iii) to provide a whole stillage;
- (v) solid/liquid separation, whereby a lignin-rich fraction ("lignin") and an aqueous thin stillage are provided;
- (vi) removal of water from the lignin-rich fraction; and
- (vii) removal of water from the whole stillage of step (iv), or from the thin stillage of step (v), to provide said lignin-rich vinasse or said lignin-depleted vinasse;

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wherein any one of steps (iii), (v) and/or step (vi) are optional; and wherein the lignin-rich vinasse has a dry matter content (dm) of at least 30% (w/w), and the lignin-depleted vinasse has a dm of at least 40% (w/w).

202. A method according to claim 201, wherein the fermentation product is an alcohol, such as EtOH.

203. A method according to claim 201 or 202, wherein the fermentation is a fermentation with yeast, optionally a yeast fermenting C5 and C6 sugars.

5 204. A method according to any one of the preceding claims, wherein the separation of the fermentation product comprises distillation.

205. A method according to any one of the preceding claims, wherein the lignocellulosic biomass is obtained, obtainable or derived from an annual or a perennial plant, such as a cereal, wheat, wheat straw, rice, rice straw, corn,
10 corn fiber, corn cobs, corn stover, hardwood bulk, softwood bulk, sugar cane, sweat sorghum, bagasse, nut shells, empty fruit bunches, grass, cotton seed hairs, barley, rye, oats, sorghum, brewer's spent grains, palm waste material, wood, soft lignocellulosic biomass, and any combination thereof.

206. A method according to any one of the preceding claims, wherein the
15 pretreatment comprises addition of an acid and/or a base, such as H₂SO₄ and/or ammonia (NH₄OH).

207. A method according to any one of claims 201 - 205, wherein the pretreatment is an autohydrolysis process without addition of an acid and/or a base.

20 208. A method according to any one of the preceding claims, wherein the pretreatment is a single stage process.

209. A method according to any one of the preceding claims, wherein the pretreatment is a two- or more stage process.

210. A method according to any one of the preceding claims, said process
25 comprising a "C5 bypass", wherein a usually C5-sugar rich liquid fraction is provided, such as through a liquid/solid separation step, e.g. by pressing, after and/or during pretreatment, which can be conducted as a single stage, two- or more-stage pretreatment process, such as disclosed in PCT/DK2013/050256.

211. A method according to any one of the preceding claims, wherein said
30 process comprising a "whole slurry process", wherein pretreated biomass is used directly in a subsequent hydrolysis step, such as an enzymatic hydrolysis and/or fermentation, such as e.g. disclosed in PCT/DK2014/050030, and

optionally, wherein the ratio by weight of undissolved to dissolved solids at the start of enzymatic hydrolysis is less than 3.1 or 2.2:1.

212. A method according to any one of the preceding claims, wherein said hydrolysis step (ii) is (a) an enzymatic hydrolysis comprising addition of one or more cellulase and/or xylanase, optionally comprising one or more pH adjustment step(s) prior, during and/or after said enzymatic hydrolysis; or (b) the hydrolysis is an acid- or base-catalyzed process, usually not comprising the addition of enzymes.

213. A method according to any one of the preceding claims, wherein at least 50-98% of the carbohydrates of the lignocellulosic biomass are converted to mono- or disaccharides during said enzymatic hydrolysis.

214. A method according to any one of the preceding claims, wherein at least 50 - 98 of the carbohydrates of the lignocellulosic biomass are converted to said fermentation product.

215. A method according to any one of the preceding claims, wherein the solid/liquid separation step (v) comprises removal of at least 90% (w/w) of the undissolved solids from the whole stillage from step (iv), e.g. by pressing.

216. A method according to any one of the preceding claims, wherein water is removed from the lignin from step (v), e.g. by evaporation, yielding lignin with a dm of at least 50, 60, 70, 80, 90, 92, 94, 95, 96, 97, 98, 99 or 100 % (w/w).

217. A method according to any one of the preceding claims, wherein at least 60% of water is removed from the whole stillage from step (vii) e.g. by consecutive evaporation.

218. A method according to any one of the preceding claims, wherein at least 85% of water is removed from the thin stillage of step (vii), e.g. by consecutive evaporation.

219. A method according to any one of the preceding claims, further comprising combusting at least a fraction of said lignin-rich vinasse and or lignin-depleted vinasse.

220. A method according to claim 219, wherein said combusting comprises the use of a conventional boiler, such as grate boiler, bubbling fluidized bed (BFB), low temperature bubbling fluidized bed (LTBFB), circulating fluidized

bed (CFB), low temperature circulating fluidized bed (LT CFB - e.g. as disclosed in any one of WO 1999/032583, WO 2014/012556, or WO 2015/007285), or oil boiler.

221. A vinasse provided, defined or characterized according to any one of the preceding claims.

222. A vinasse according to claim 221, said vinasse being a lignin-rich vinasse.

223. A lignin-rich vinasse according to claim 222, having a LHV of at least 3.4 or more, 3.6 or more, 3.8 or more, 4.0 or more, 4.2 GJ/t or more, 4.5 GJ/t or more, 5.0 GJ/t or more, 6.0 GJ/t or more, 7.0 GJ/t or more, 8.0 GJ/t or more, 10.0 GJ/t or more, 12.0 GJ/t or more, 14.0 GJ/t or more, 16.0 GJ/t or more, or 18.0 GJ/t or more.

224. A lignin-rich vinasse according to claim 222 or 223, having a dm of at least 30% or more, 35% or more, 40% or more, 45% or more, 50% or more 55% or more, 60% or more 65% or more, 70% or more 75% or more, 80% or more 85% or more, 90% or more, 95% or more, or 100%.

225. A lignin-rich vinasse according to any one of claims 222 - 224, wherein the lignin-rich vinasse is liquid, optionally comprising one or more further agents, such as one or more liquefying agent(s), in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

226. A lignin-rich vinasse according to any one of claims 222 - 225, wherein the lignin-rich vinasse is solid, optionally comprising one or more further agents, such as one or more solidifying agent(s) in an active concentration, in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

227. A vinasse according to claim 221, said vinasse being a lignin-depleted vinasse.

228. A lignin-depleted vinasse according to claim 227, having a LHV of at least 3.4 GJ/t or more, 3.6 GJ/t or more, 3.8 GJ/t or more, 4.0 GJ/t or more, 4.2 GJ/t or more, 4.5 GJ/t or more, 5.0 GJ/t or more, 6.0 GJ/t or more, 7.0 GJ/t or more, 8.0 GJ/t or more, 9.0 GJ/t or more, or 10GJ/t or more.

229. A lignin-depleted vinasse according to claim 227 or 228, having a dm of at least 30% or more, 35% or more, 40% or more, 45% or more, 50% or more 55% or more, 60% or more 65% or more, 70% or more 75% or more, 80% or more 85% or more, 90% or more, 95% or more, or 100%.

5 230. A lignin-depleted vinasse according to any one of claims 227 - 229, wherein the lignin-depleted vinasse is liquid, optionally comprising one or more further agents, such as one or more liquefying agent(s), in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

10 231. A lignin-depleted vinasse according to any one of claims 227-230, wherein the lignin-depleted vinasse is solid, optionally comprising one or more further agents, such as one or more solidifying agent(s), in an active concentration, such as 0.001-10%, 0.01-5%, 0.1-1%, or more than 10%.

15 232. A fuel comprising 0.01-99.99, 0.1-99.99, 1-99.99, 10-99.99, 20-99.99, 30-99.99, 40-99.99, 50-99.99, 60-99.99, 70-99.99, 80-99.99, or 90-99.99% of a lignin-rich and/or lignin-depleted vinasse according to any one of claims 201 – 232, and optionally, wherein (a) said fuel is or comprises 0.01-99.99, 0.1-99.99, 1-99.99, 10-99.99, 20-99.99, 30-99.99, 40-99.99, 50-99.99, 60-99.99, 70-99.99, 80-99.99, or 90-99.99% a fluid according to any one of claims 1-70, 148-155 and/or a fluid provided according to any one of claims 80-113, 148-20 155; and/or (b) said fuel comprises a lignin or lignin-component according to any one of the preceding claims.

233. A fuel according to claim 232, wherein said fuel is a liquid, such as a pumpable composition; or a solid fuel, such as a non-pumpable fuel.

25 234. A fuel according to claim 232 or 233, wherein said lignin-rich and/or lignin-depleted vinasse has a lower heating value of 3.4 GJ/ton or more.

235. A fuel according to any one of claims 232 - 234, wherein said lignin-rich and/or lignin-depleted vinasse has a dry matter content of at least 30%, 40% or more than 40%.

30 236. A fuel according to any one of claims 232 - 235, further comprising one or more organic fractions, such as one or more oil, fat, or organic solvent, including any combination thereof.

237. A fuel according to any one of claims 232 - 236, further comprising a lignin-rich component, such as lignin provided according to any one of the preceding claims.

238. A fuel according to any one of claims 232 - 237, wherein said fuel comprises 0.01-100% (w/w) lignin-depleted and/or lignin-rich vinasse, 0-99.9 % (w/w) lignin-rich component; and optionally 0-99.9% (w/w) of other organic fraction, and optionally 0-20% (w/w) of one or more further agent(s).

239. A fuel according to any one of claims 232 - 238, wherein said lignin-depleted and/or lignin-rich vinasse comprises 0-70% (w/w) water, 30-100% (w/w) other matter both organic and inorganic.

240. A fuel according to any one of claims 232 - 239, wherein said fuel comprises 50-99.9% (w/w) lignin-depleted and/or lignin-rich vinasse, 0.1-50% (w/w) lignin-rich component.

241. A fuel according to any one of claims 232 - 240, wherein said fuel comprises 1-100% (w/w) lignin-depleted and/or lignin-rich vinasse with a dm of 30% or more, 0-99 % (w/w) organic fraction, 0-99% (w/w) lignin-rich component; and optionally 0-20% (w/w) of a further agent.

242. A fuel according to any one of claims 232 - 241, comprising up to around 50% lignin-depleted vinasse with a dm of around 65% or more, and around 50% lignin or more with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 10-13 GJ/t, ignition time of > 500 ms, low ash swelling, and suitable for grate boiler, BFB, LTBFB, CFB, LTCFB.

243. A fuel according to any one of claims 232 - 422, comprising not more than around 60% lignin-depleted vinasse with a dm of around 40% or more, and up to 99.9% lignin with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 9-13 GJ/t or more, ignition time of > 500 ms, low ash swelling, and suitable for grate boiler, BFB, LTBFB, CFB, LTCFB.

244. A fuel according to any one of claims 232 - 243, comprising at least 95% lignin-depleted vinasse with a dm of around 40% or more, and up to 5% oil; and optionally, wherein said fuel has one or more of the following features:

liquid, a LHV of around 9-11 GJ/t, ignition time of < 500 ms, and suitable for combustion e.g. in an adjusted oil boiler, co-combustion such as fireball co-combustion.

245. A fuel according to any one of claims 232 - 244, comprising at least 87% lignin-depleted vinasse with a dm of around 65% or more, and up to 13% oil; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 8-11 GJ/t or more, ignition time of < 500 ms, and suitable for combustion e.g. in an adjusted oil boiler, co-combustion such as fireball co-combustion.

246. A fuel according to any one of claims 232 - 245, comprising at least 75% lignin-depleted vinasse with a dm of around 65% or more, and up to 25% lignin with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 9 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fireball co-combustion.

247. A fuel according to any one of claims 232 - 246, comprising at least 70% lignin-depleted vinasse with a dm of around 40% or more, and up to 30% lignin with a dm of around 90% or more; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 9 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fire ball co-combustion, BFB or CFB.

248. A fuel according to any one of claims 232 - 247, comprising 90% or more, or around 100% lignin-depleted vinasse with a dm of around 65%, or 65-100%; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 5-6 GJ/t or more, and ignition time of > 500 ms, suitable for co-combustion e.g. fire ball combustion, BFB or CFB.

249. A fuel according to any one of claims 232 - 248, comprising 80% or more lignin-depleted vinasse with a dm of around 65% or more, and up to 20% wood or coal%; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 8 GJ/t or more, and ignition time of < or > 500 ms, suitable for combustion in a conventional solid fuel boiler, e.g. grate boiler, BFB, LTBFB, CFB or LTCFB.

250. A fuel according to any one of claims 232 - 249, comprising around 88% or more lignin-rich vinasse with a dm of around 30% or more, and up to 12% oil; and optionally, wherein said fuel has one or more of the following features: liquid, a LHV of around 8 GJ/t or more, and ignition time of < 500 ms, suitable for combustion in an adjusted oil boiler, fire ball combustion, and/or co-combustion.

251. A fuel according to any one of claims 232 - 250, comprising 90% or more, or around 100% lignin-rich vinasse with a dm of around 65% or 65-100%; and optionally, wherein said fuel has one or more of the following features: solid, a LHV of around 7 GJ/t or more, and ignition time of > 500 ms, suitable for combustion in a conventional solid fuel boiler, e.g. grate boiler, BFB, LTBFB, CFB or LTCFB

252. A fuel according to any one of claims 232 - 251, wherein said fuel is liquid fuel and comprises one or more of: diesel oil, bunker oil, petroleum, petrol, kerosene, paraffin, organic solvent, including any combination thereof.

253. A fuel according to any one of claims 232 - 252, wherein said fuel is a solid fuel, and comprises one or more of: lignin, lignin-rich component, coal (such as lignite (brown coal), flame coal, gas flame coal, gas coal, fat coal, forge coal, nonbaking coal, or anthracite), wood, (e.g. wood chips or wood pellets), including any combination thereof.

254. A fuel according to any one of claims 232 - 253, wherein said fuel is suitable for co-combustion, such as fireball co-combustion; and/or wherein said fuel is suitable for combustion in a conventional boiler, such as grate boiler, bubbling fluidized (BFB), low temperature bubbling fluidized bed (LTBFB), circulating fluidized bed (CFB), circulating low temperature fluidized bed (LT CFB), oil boiler, or fireball co-combustion.

255. A fuel according to any one of claims 232-254, said fuel comprising lignin or a lignin component obtained or obtainable in step (e) according to any one of claims 92-113.

256. Use of a lignin-rich- and/or lignin-depleted vinasse according to any one of claims 201 - 231 as a fuel, such as a fuel according to any one of claims 232 - 255.

257. Use according to claim 256, wherein said use comprises fluidized bed technology, such as circulating fluidized bed (CFB), bubbling fluidized bed (BFB), low temperature fluidized bed technology, such as low temperature circulating fluidized bed (LTCFB – e.g. as disclosed in any one of WO
5 1999/032583, WO 2014/012556, or WO 2015/007285) and low temperature bubbling fluidized bed LTBFB.

258. Use according to claim 256, wherein said use comprises boiler technology, such as grate boiler or oil boiler.

259. Use according to claim 258, wherein a conventional boiler can be used,
10 or wherein a conventional boiler has to be adapted, modified and/or adjusted.

260. A system comprising means for providing a vinasse according to any one of the preceding claims and/or means for combusting a vinasse-comprising fuel, such as a fuel according to any one of claims .

261. A system according to claim 260, said system comprising a boiler and/or
15 fluidized bed means for burning and/or gasifying a vinasse and/or vinasse-comprising fuel.

262. A system according to claim 261, wherein said boiler and/or fluidized bed means is a boiler and/or fluidized bed means according to any one of claims
57 - 59.

263. A system according to any one of claims 260 - 262, wherein said
20 vinasse-comprising fuel is a fuel according to any one of claims 232 - 255.

264. A system according to any one of claims 260 - 263, said system comprising means for providing energy.

265. A system according to any one of claims 260 - 264, said system
25 comprising a combined heat and power plant (CHP).

266. A system according to any one of claims 260 - 265, said system providing electricity, heat, steam and/or hot water to a biomass processing plant.

267. A system according to any one of claims 260 - 266, said system
30 comprising means for providing 1st and/or 2nd generation bioethanol.

268. A system according to any one of claims 260 - 267, said system comprising means for pre-treatment, hydrolysis, removal of water, and

combustion means, such as a system disclosed herein in e.g. Figures 48a-53a and/or Figures 48b-53b, optionally including the hereto corresponding figure legends and embodiments.

269. A system according to any one of claims 260 - 268, wherein essentially
5 all lignin provided is burned, such as burned as a solid lignin biofuel, as lignin-comprising fuel, and/or as lignin-rich vinasse.

270. A system according to any one of claims 260 - 268, said system providing a surplus of lignin, such as a lignin biofuel.

271. A system according to any one of claims 260 - 270, said system not
10 comprising means for anaerobic digestion, such as means for methan/biogas production from stillage, such as whole or thin stillage.

272. A system according to any one of claims 260 - 271, said system comprising anaerobic digestion means for treatment and/or purification of condensates, such as condensates from one or more water removal steps
15 from one or more of: lignin, the lignin-rich fraction provided in step (vi) according to any one of claims 201-220, stillage, vinasse, the lignin-depleted and/or lignin-rich vinasse provided in step (vii) according to any one of claims 201-220, and/or condensates from a process according to any one of claims 92-113.

273. A system according to any one of claims 260 - 271, said system not
20 comprising anaerobic digestion means for treatment and/or purification of condensates, such as condensates from one or more water removal steps from one or more of: lignin, the lignin-rich fraction provided in step (vi) according to any one of claims 201-220, stillage, vinasse, the lignin-depleted
25 and/or lignin-rich vinasse provided in step (vii) according to any one of claims 201-220, and/or condensates from a process according to any one of claims 92-113.

274. A system according to any one of claims 260-273, said system comprising means for performing one or more process steps of a process
30 according to any one of claims 80-113, or means for performing one or more process steps of a method according to any one of claims 201-220.

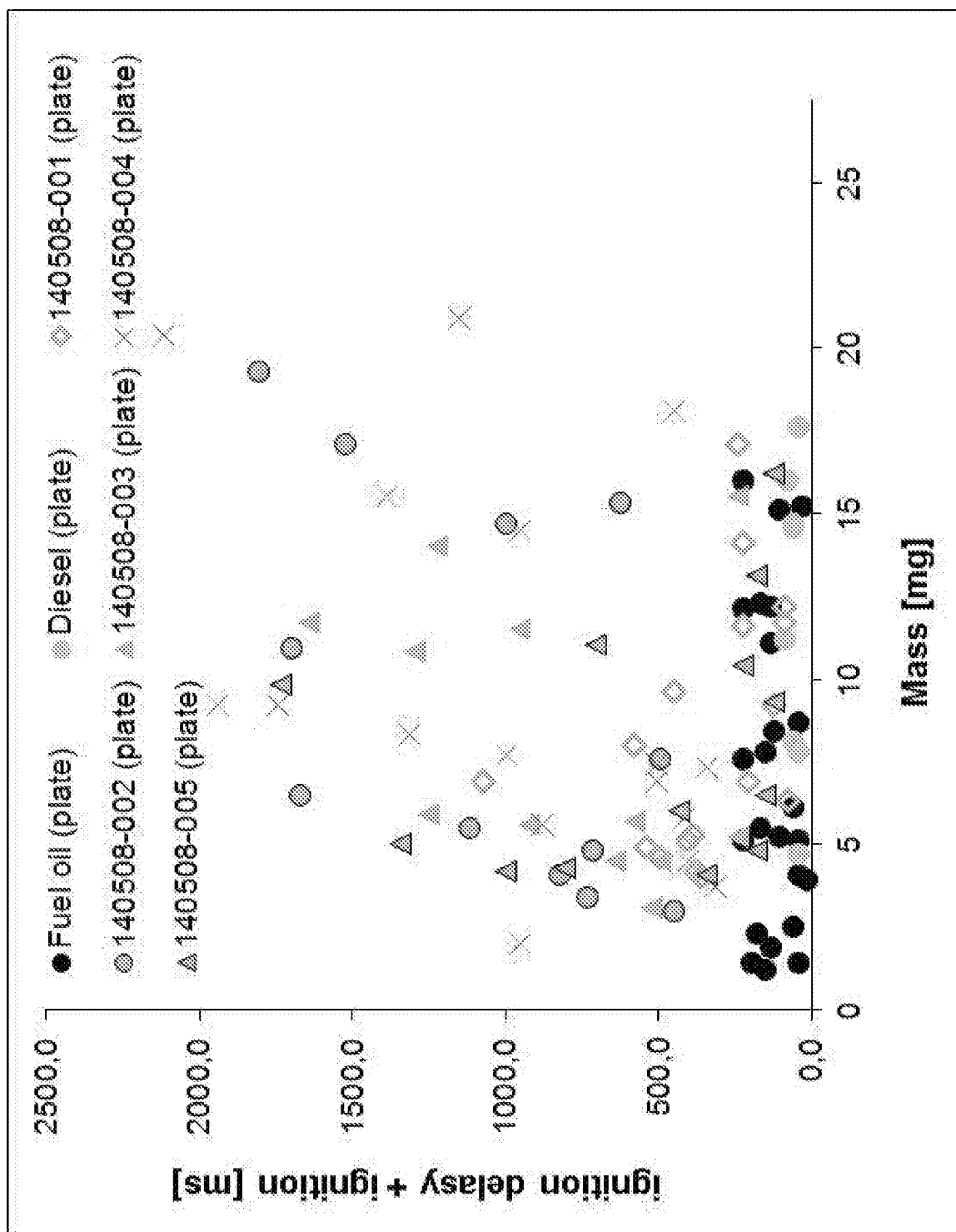


Figure 1a

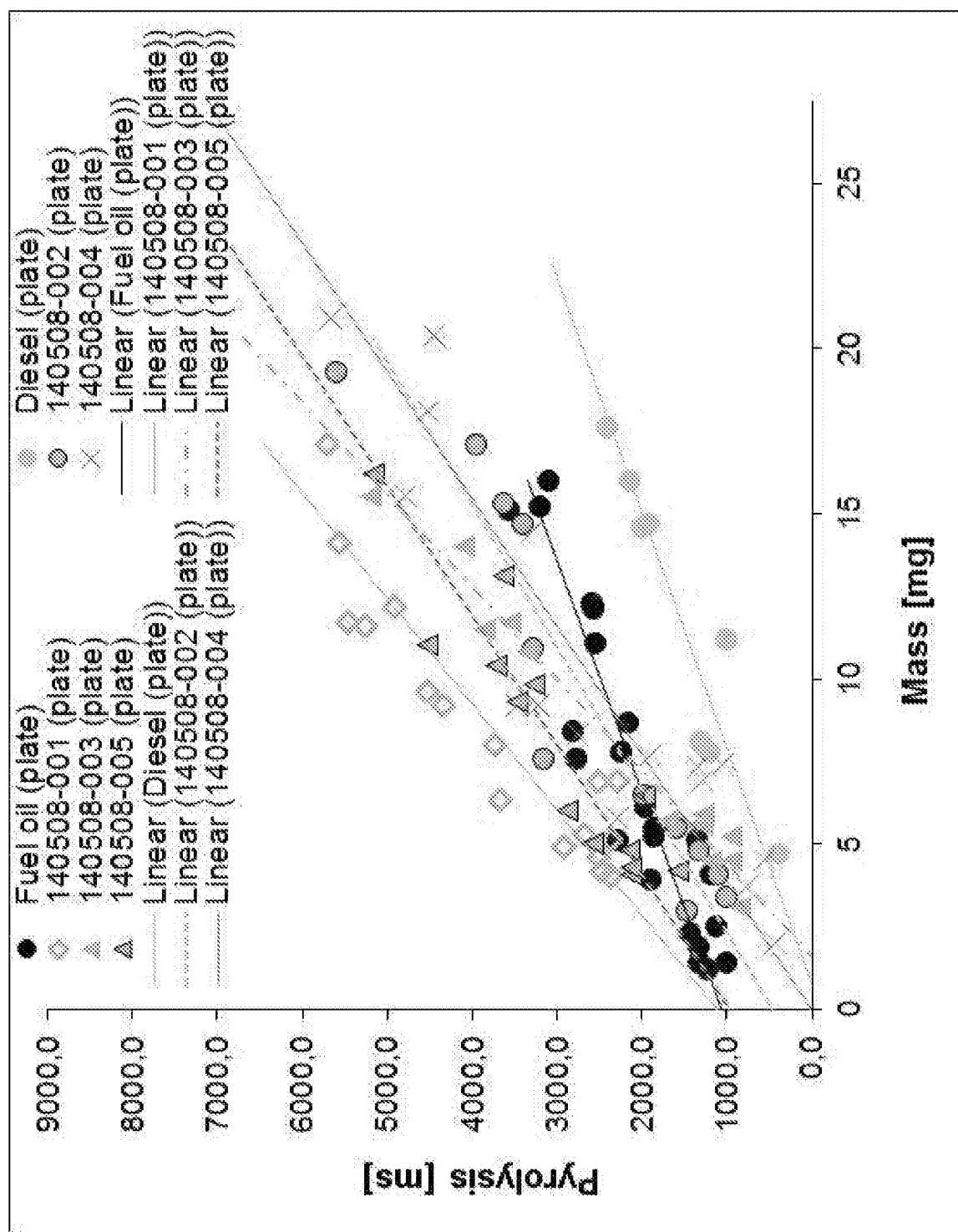


Figure 1b

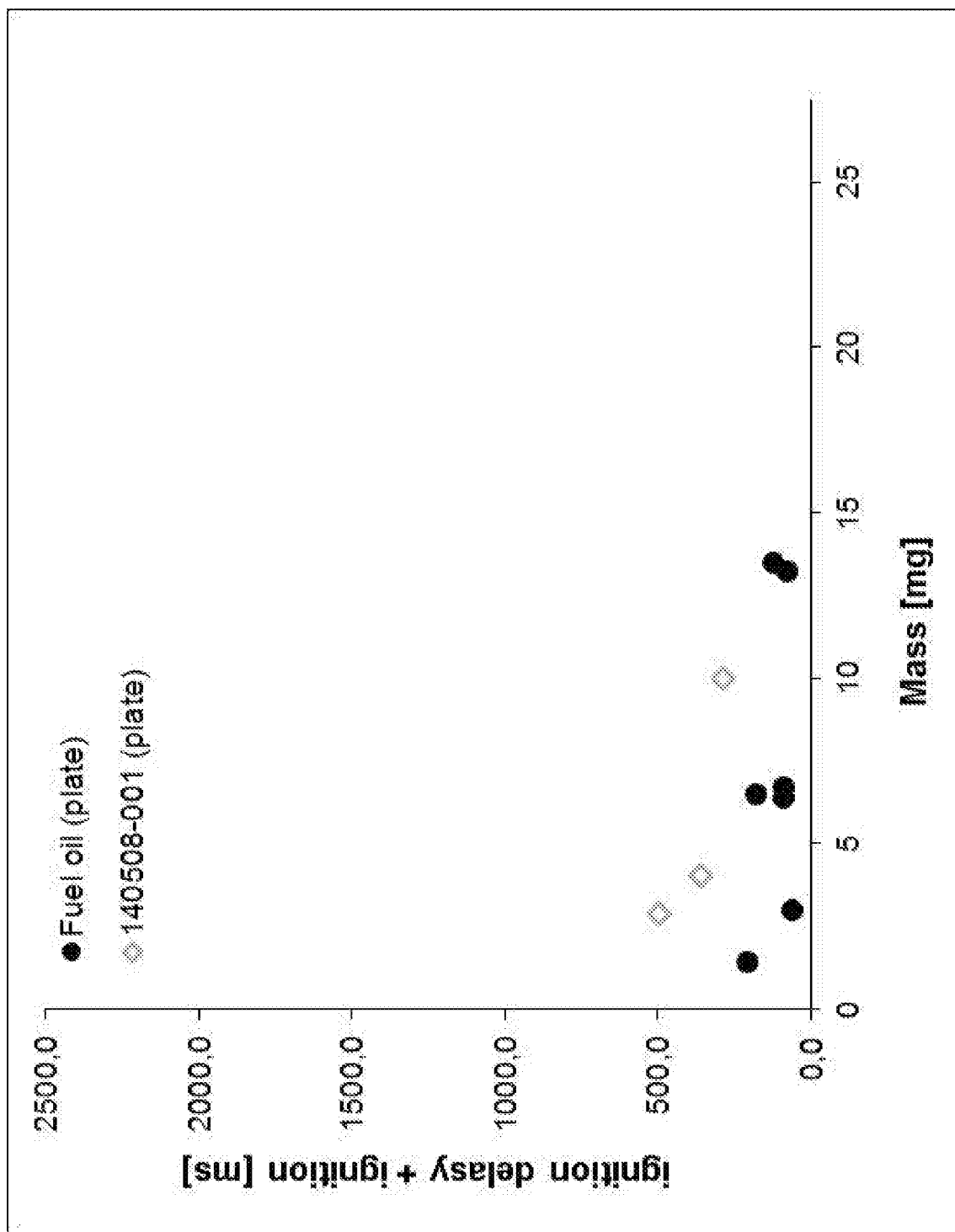


Figure 1c

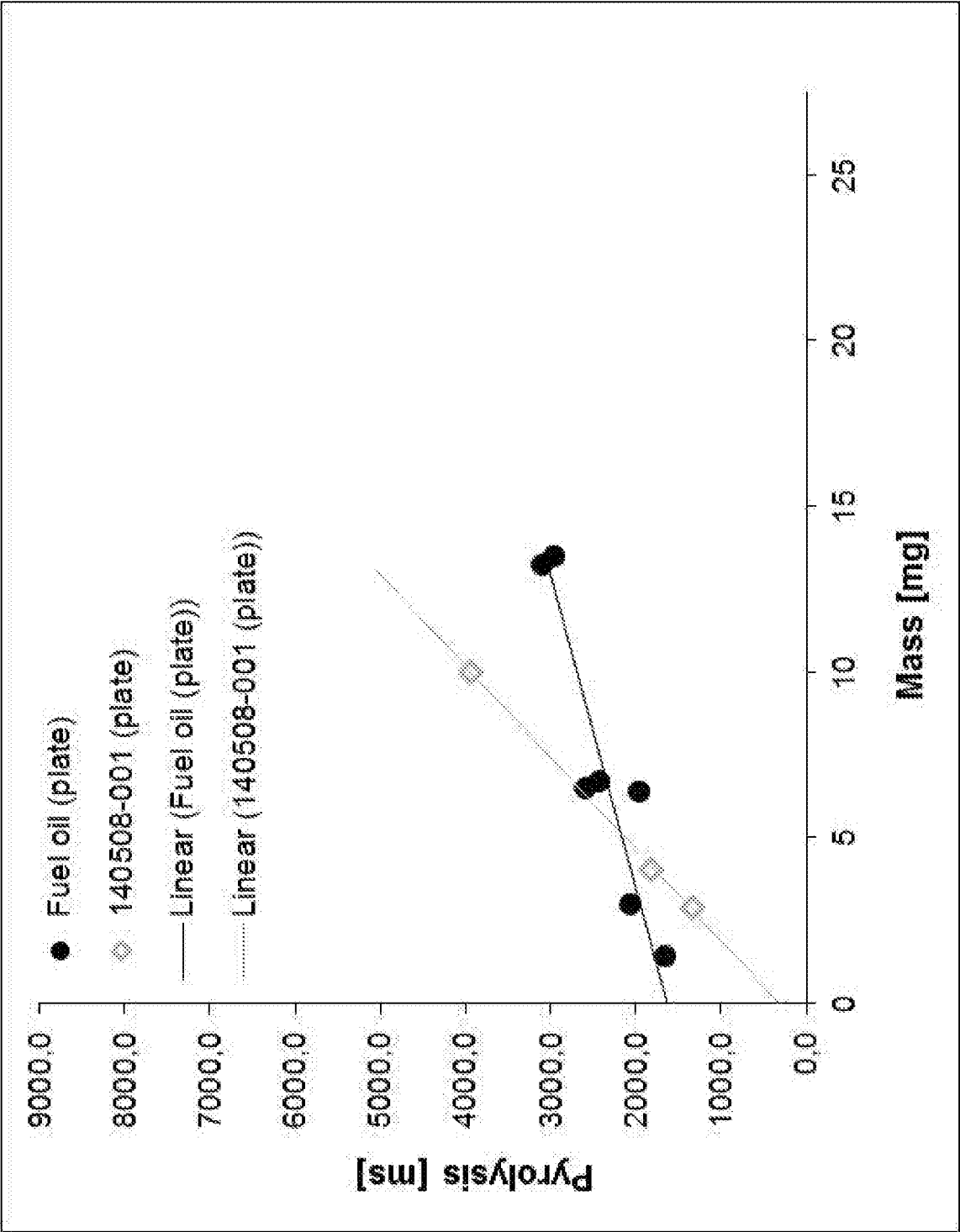


Figure 1d

c)

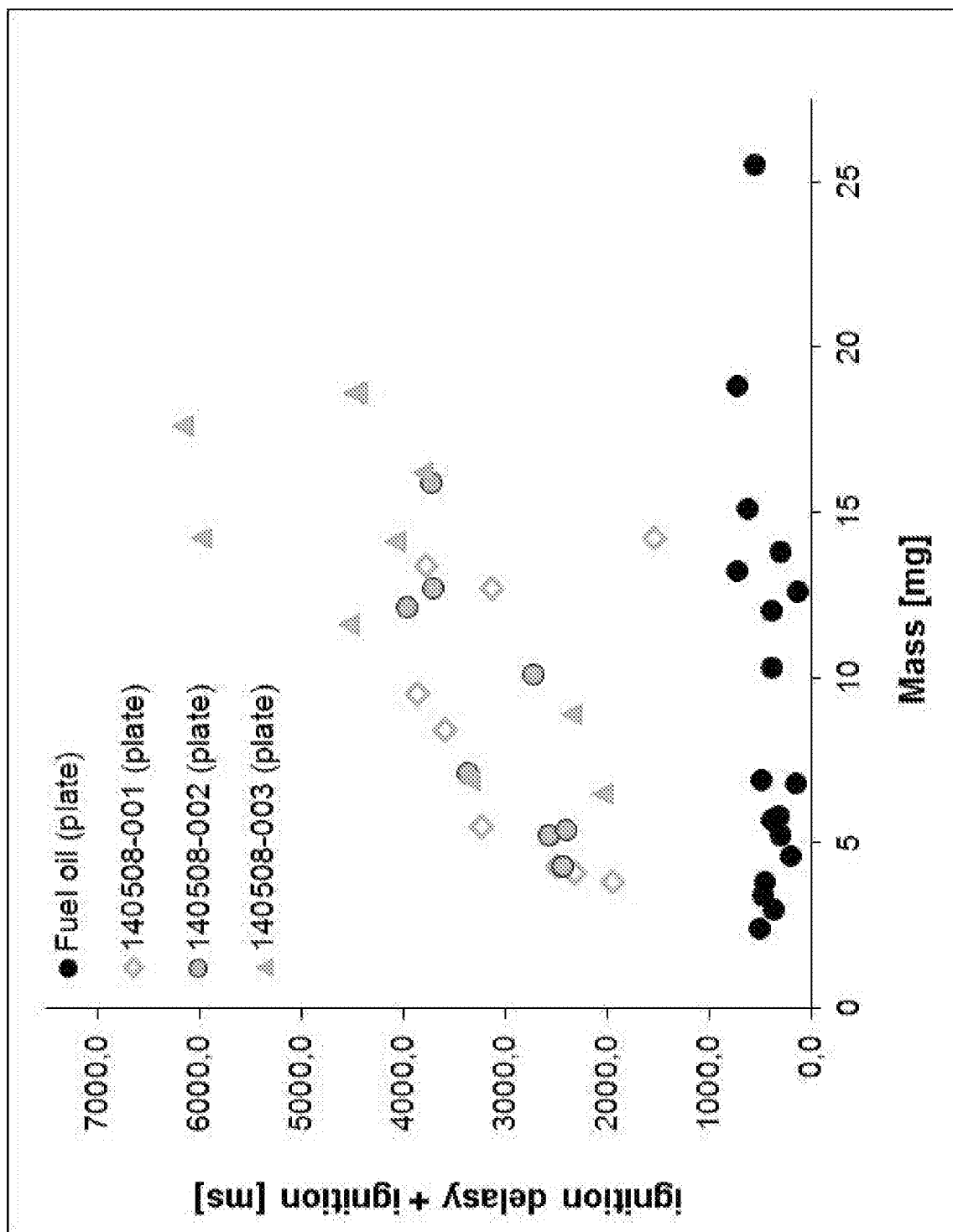


Figure 1e

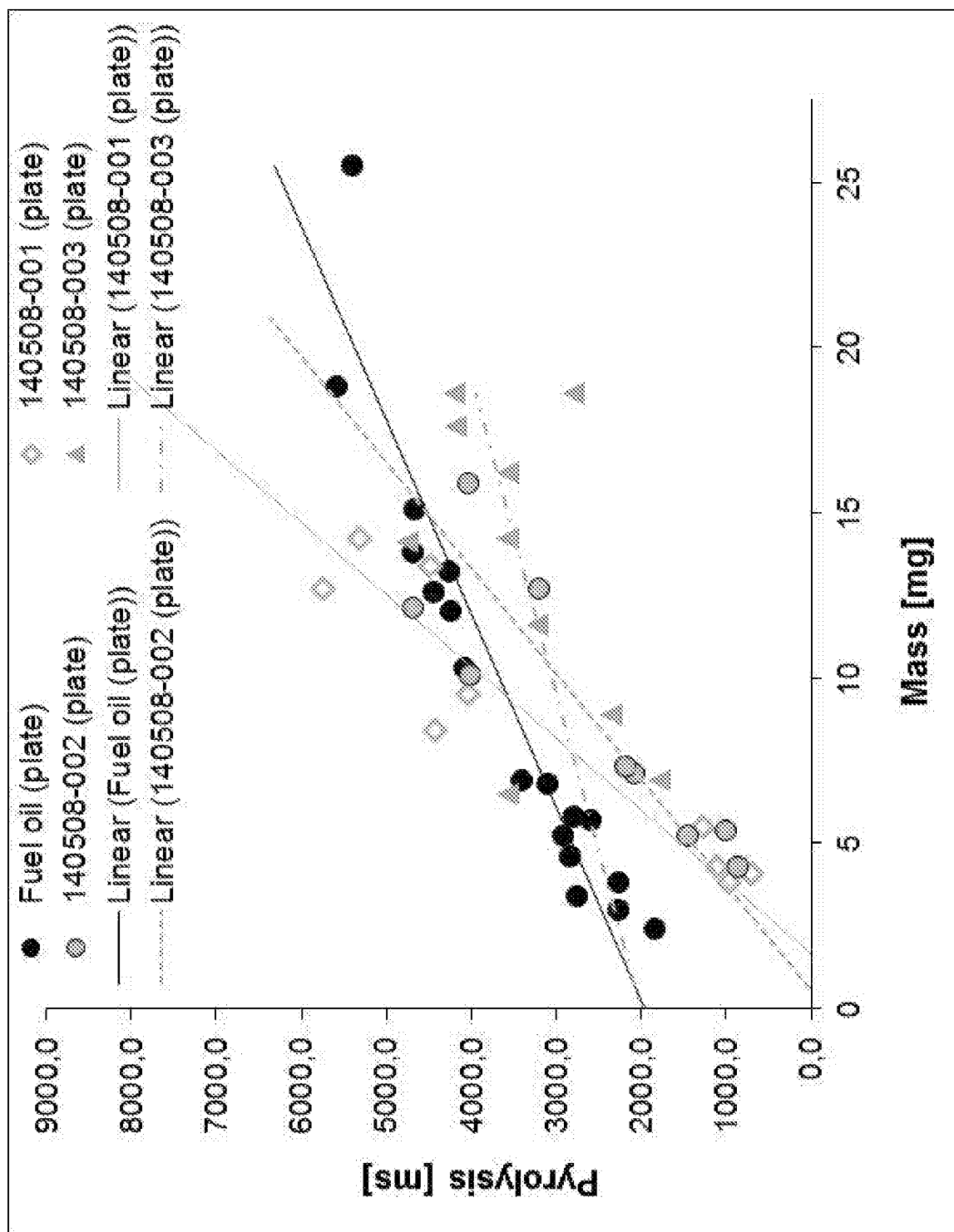


Figure 1f

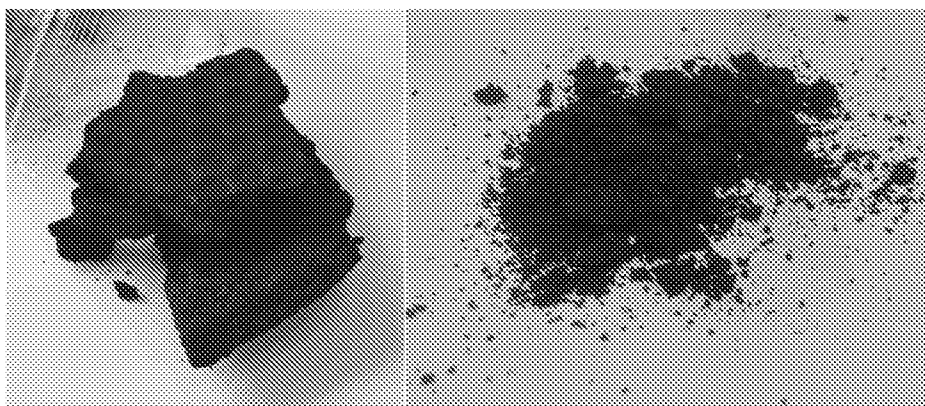


Figure 2

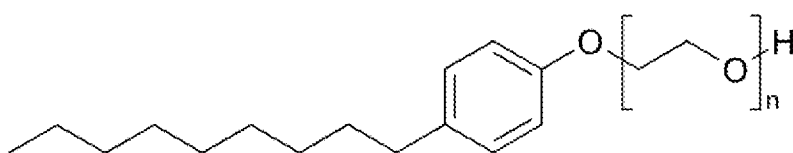


Figure 3

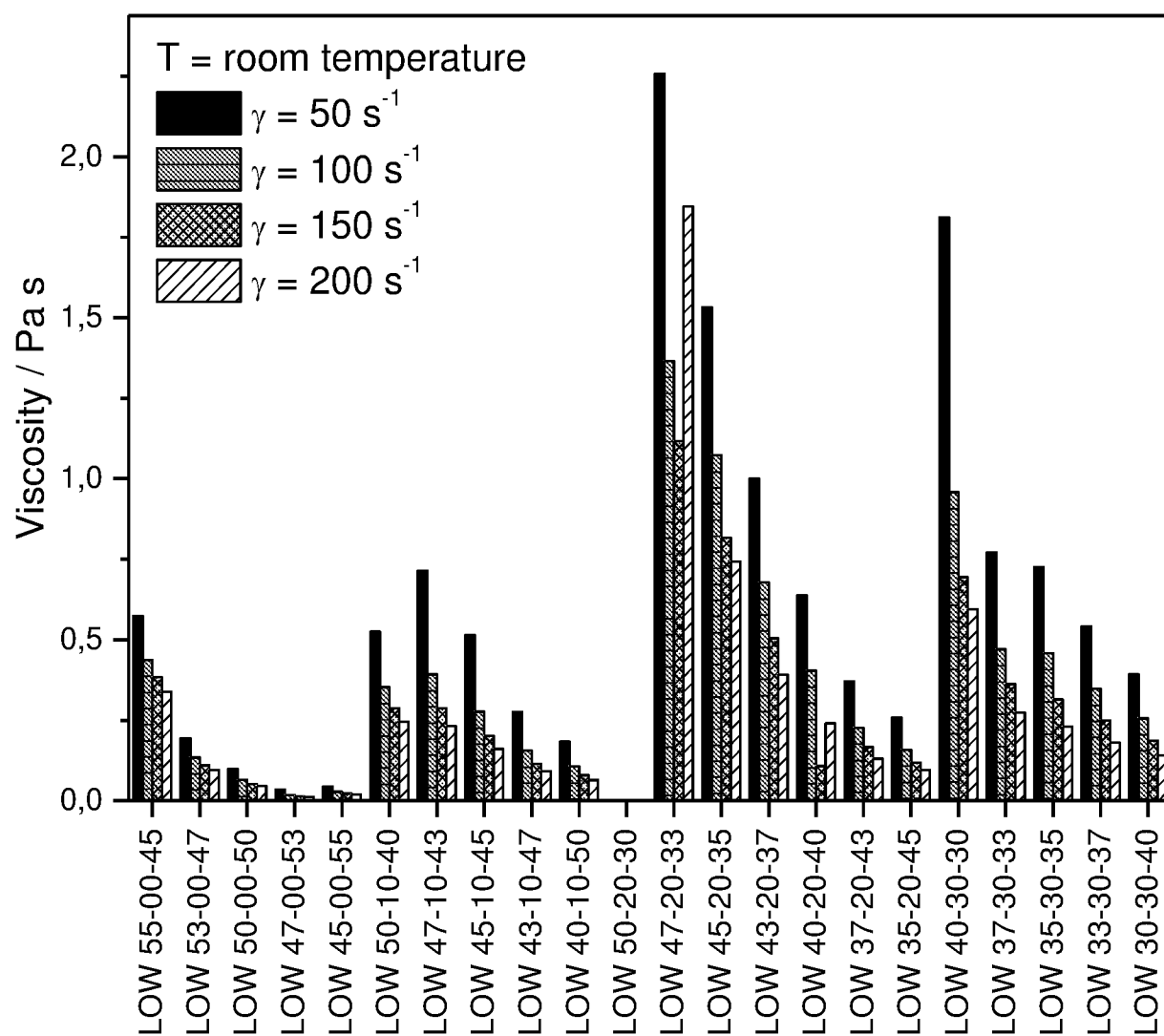


Figure 4

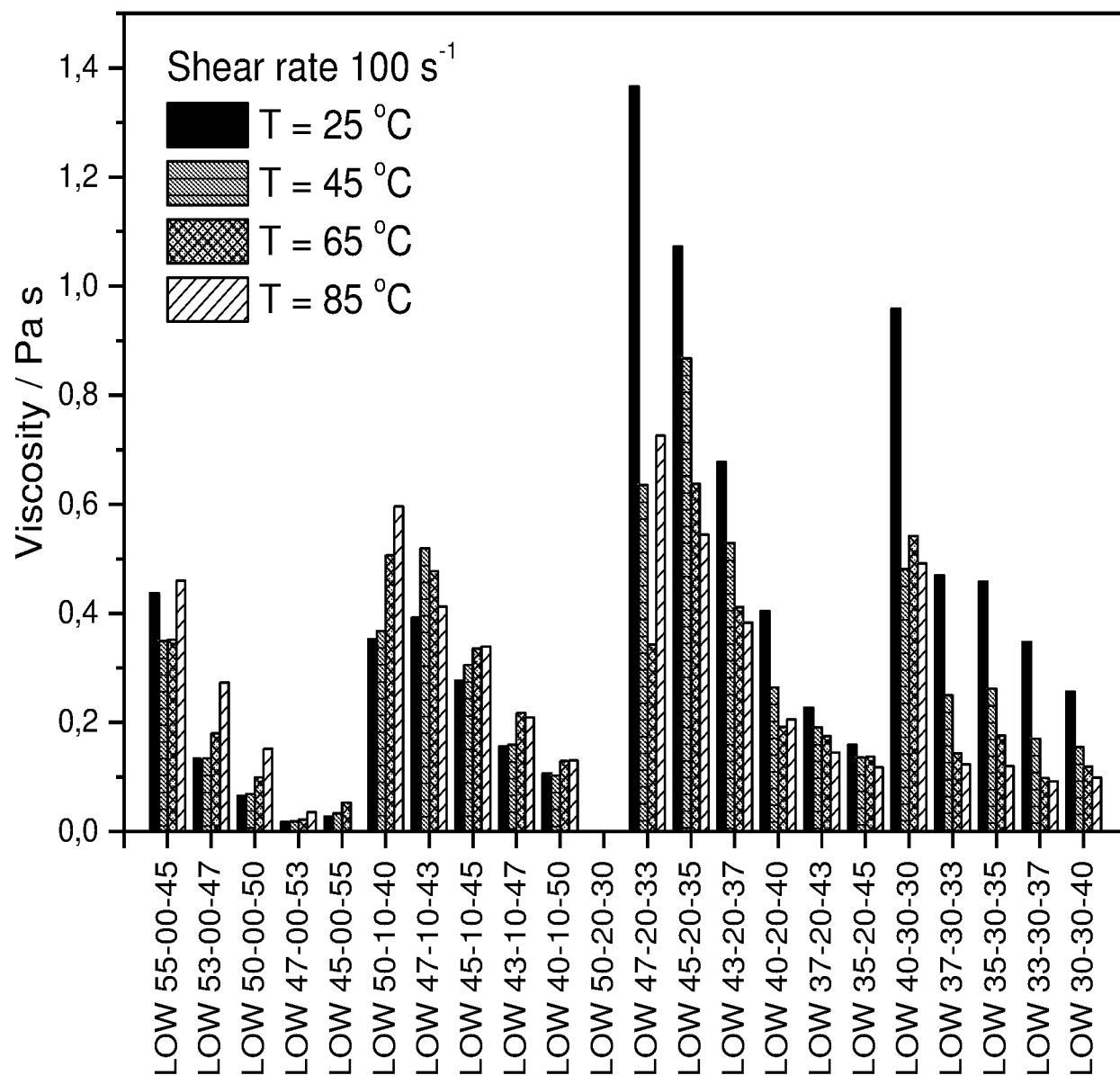


Figure 5

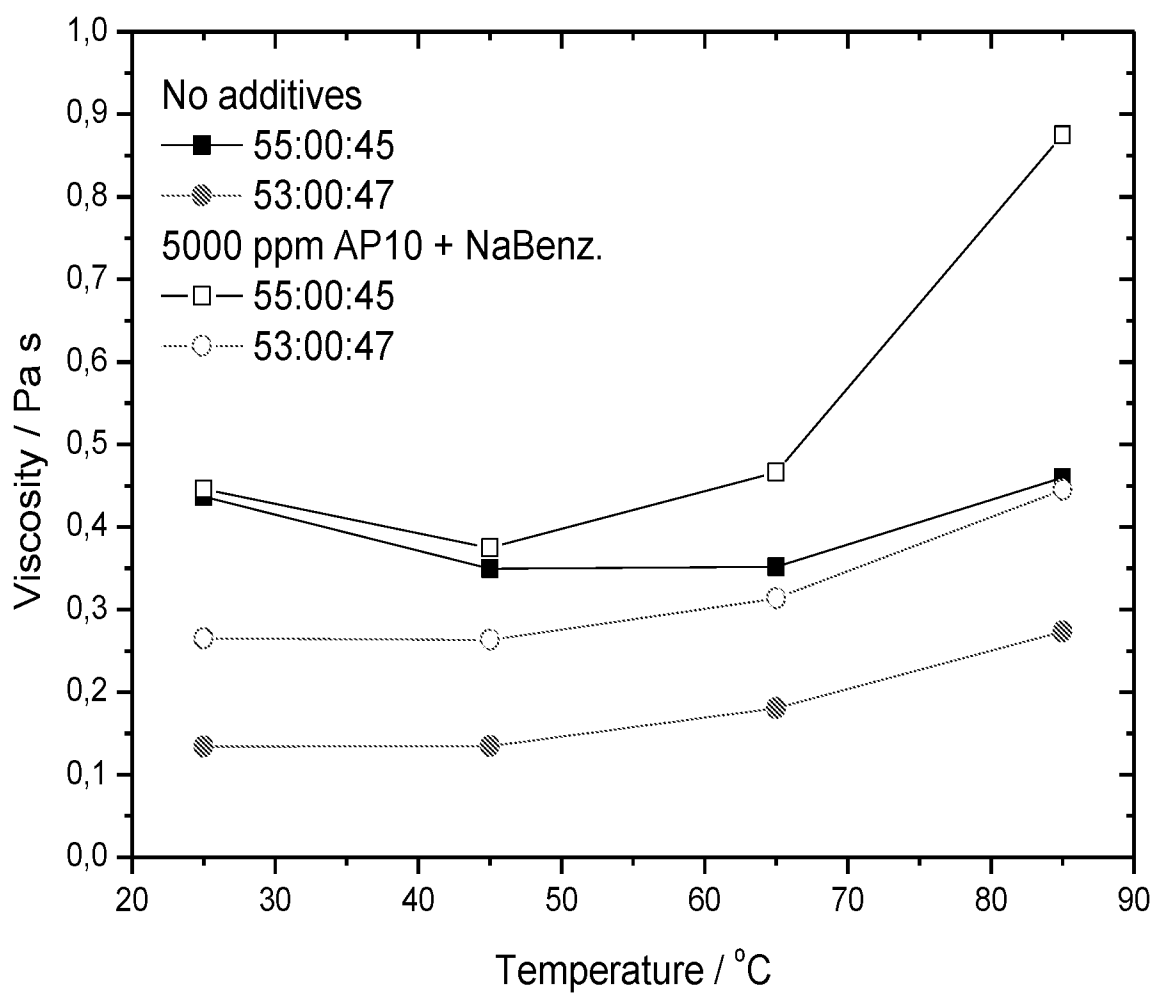


Figure 6a

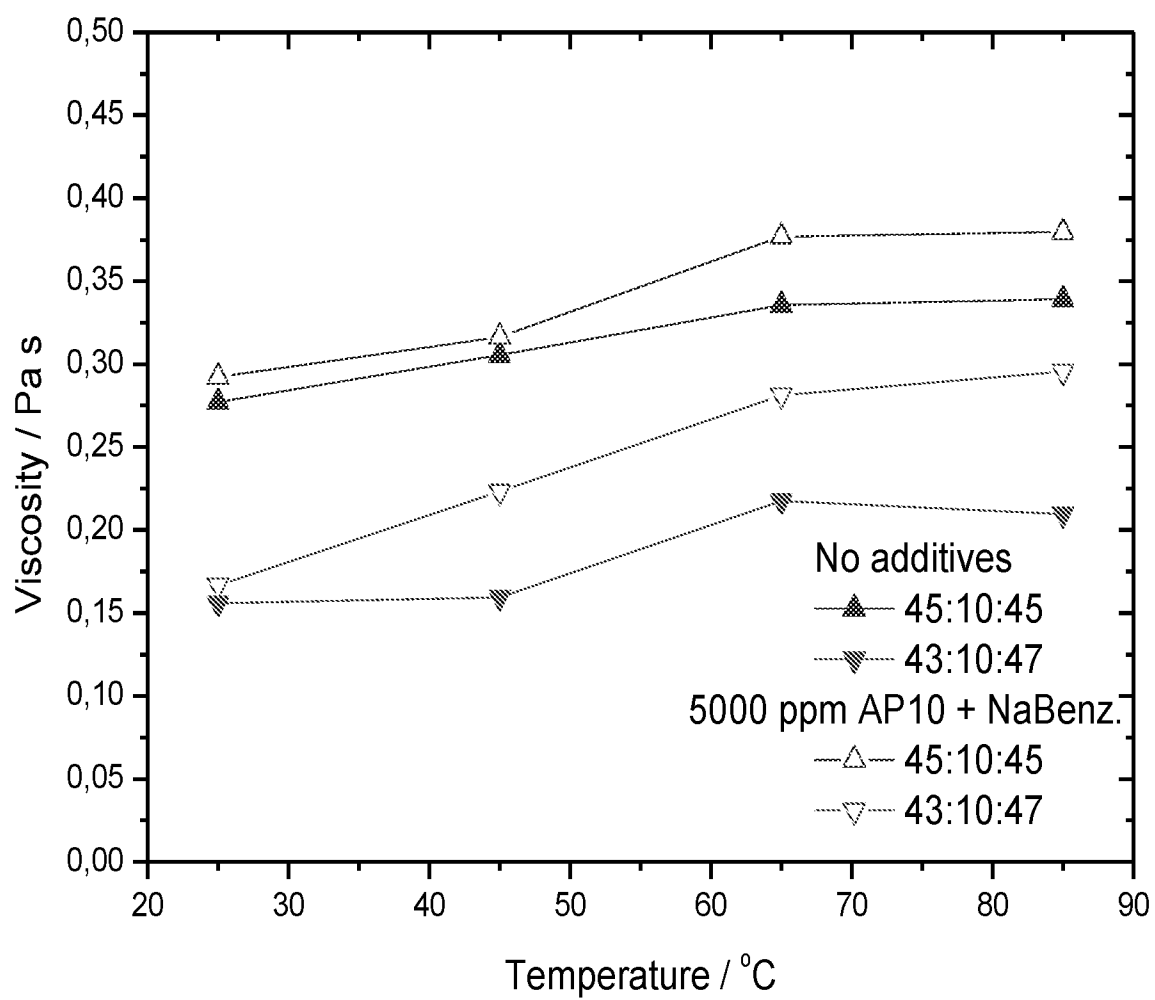


Figure 6b

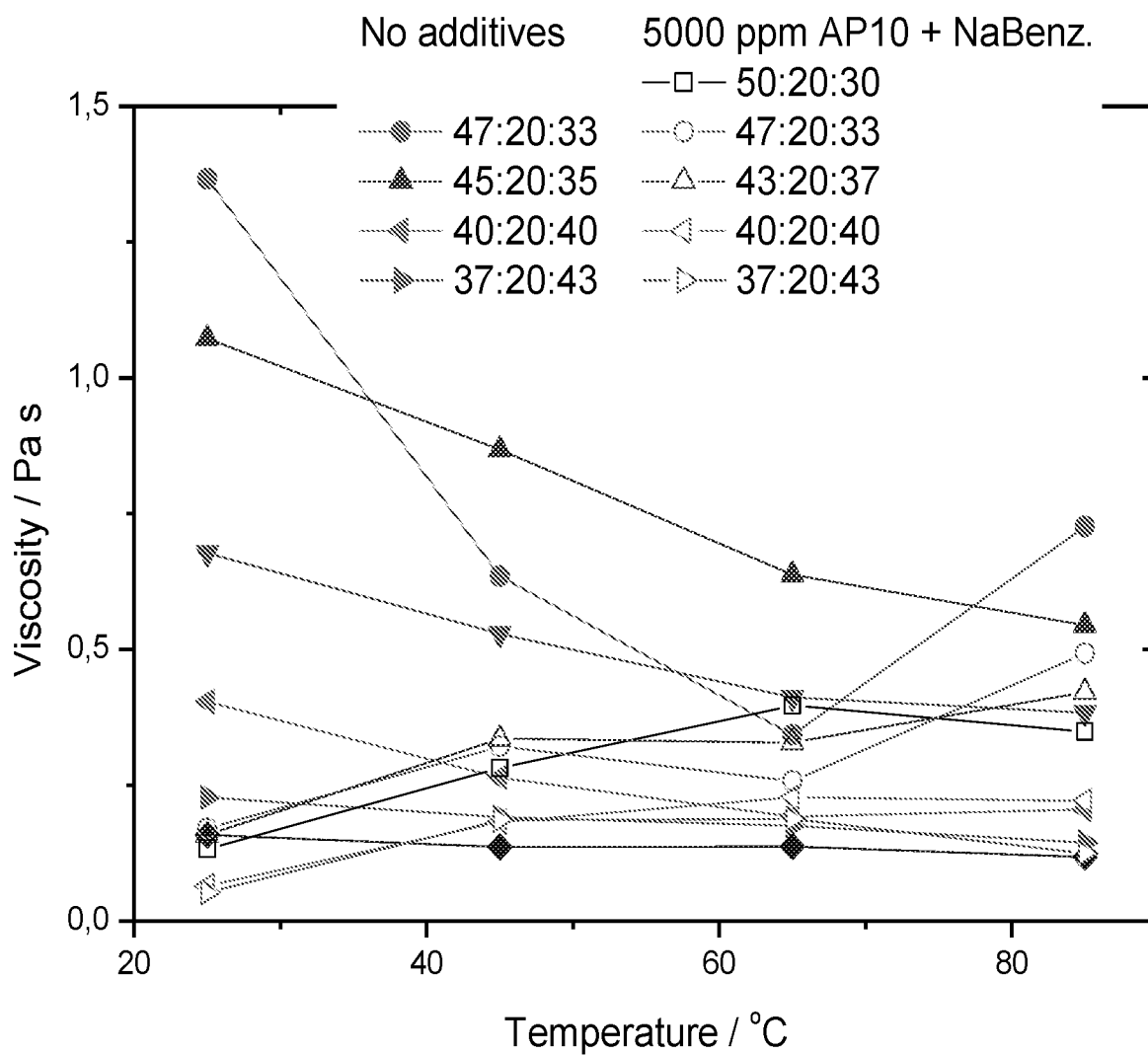


Figure 6c

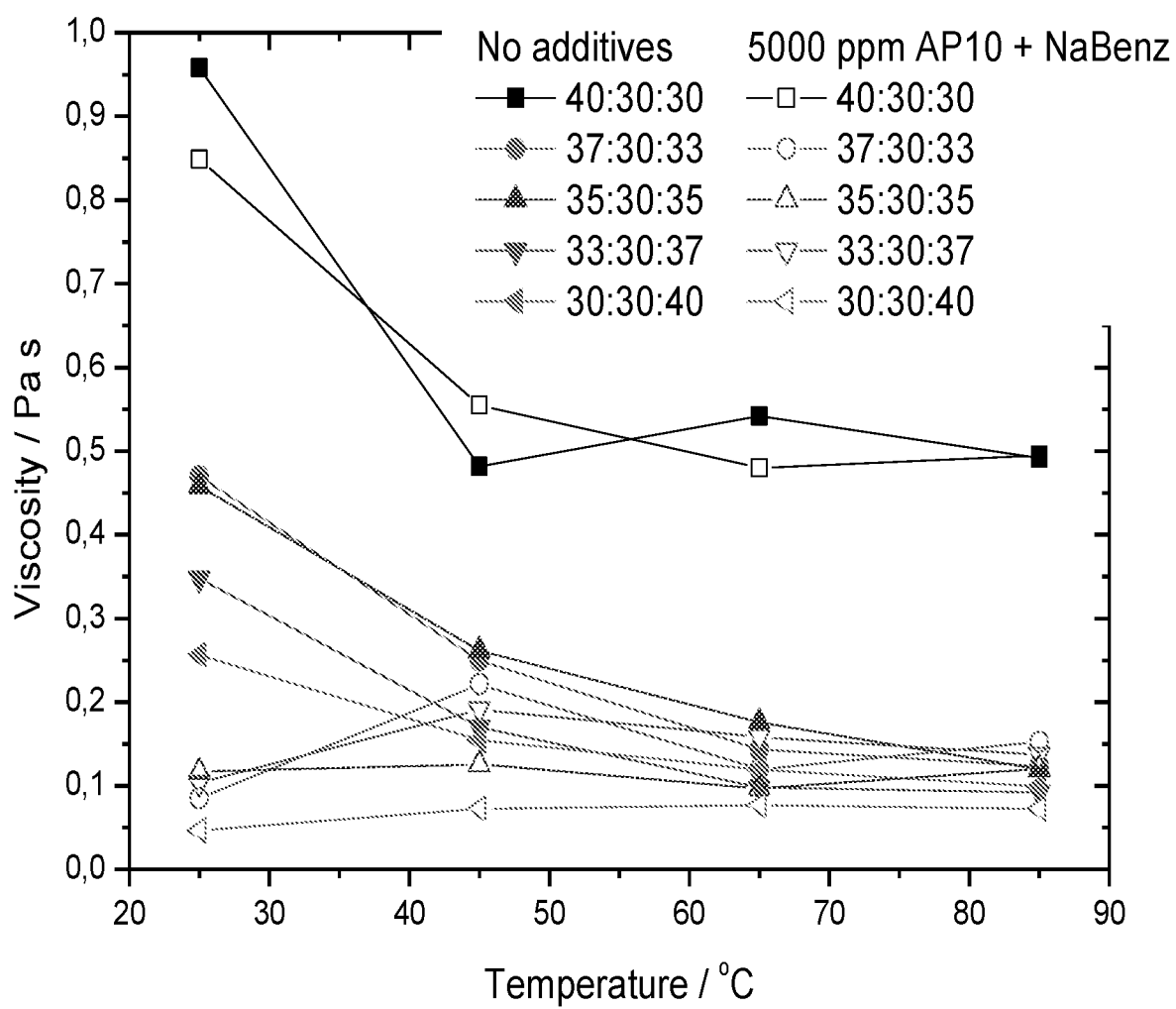


Figure 6d

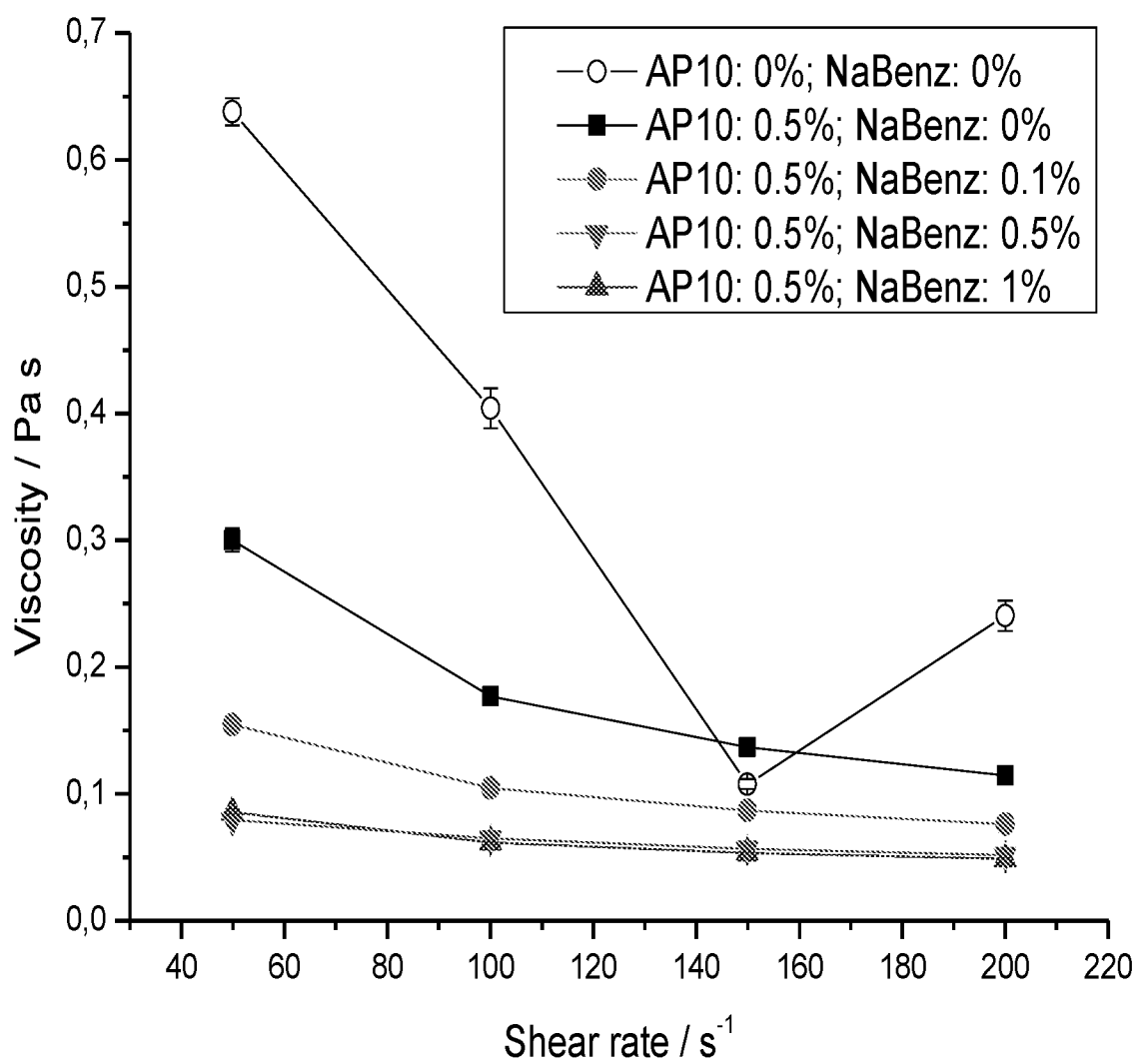


Figure 7a

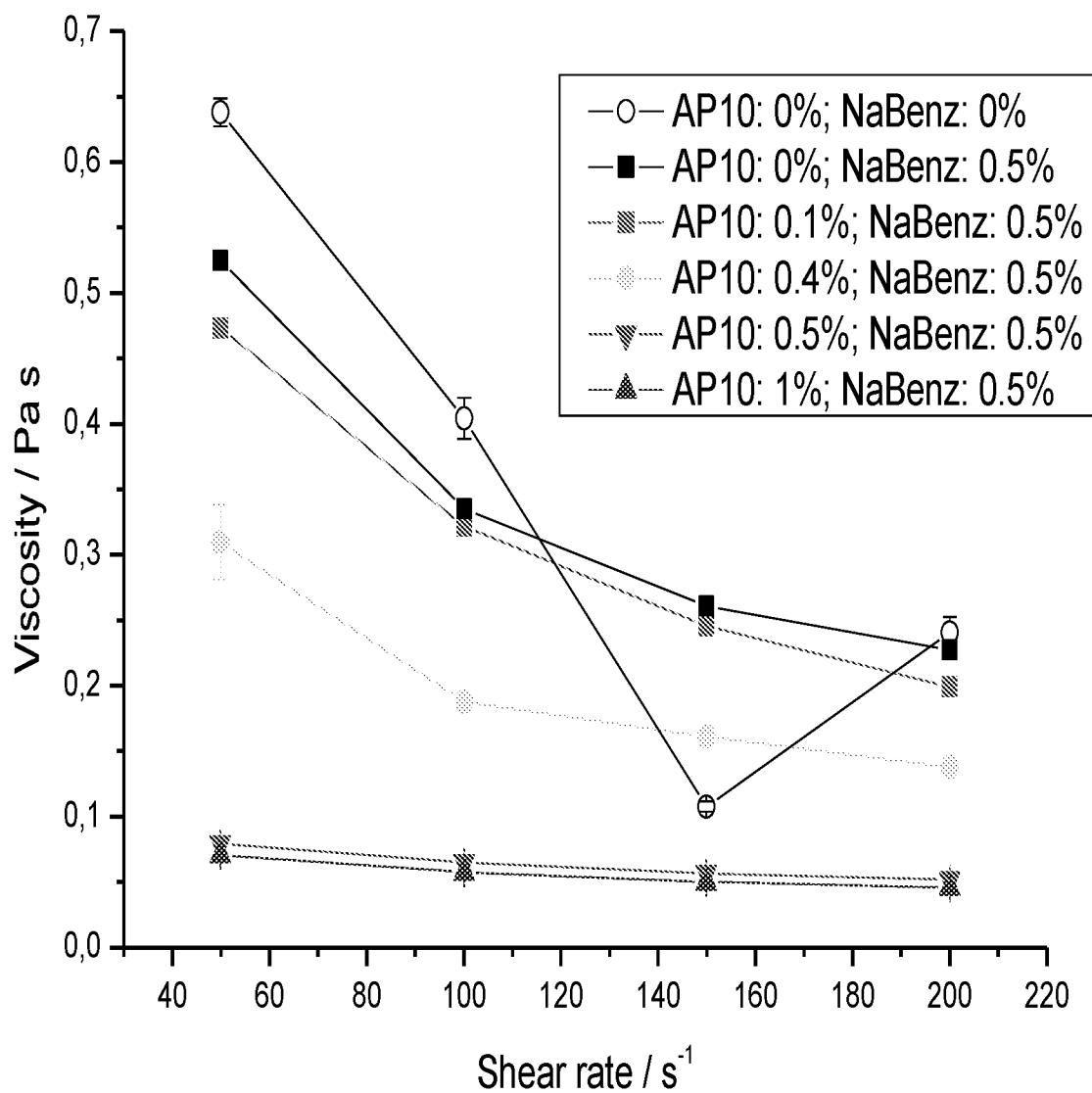


Figure 7b

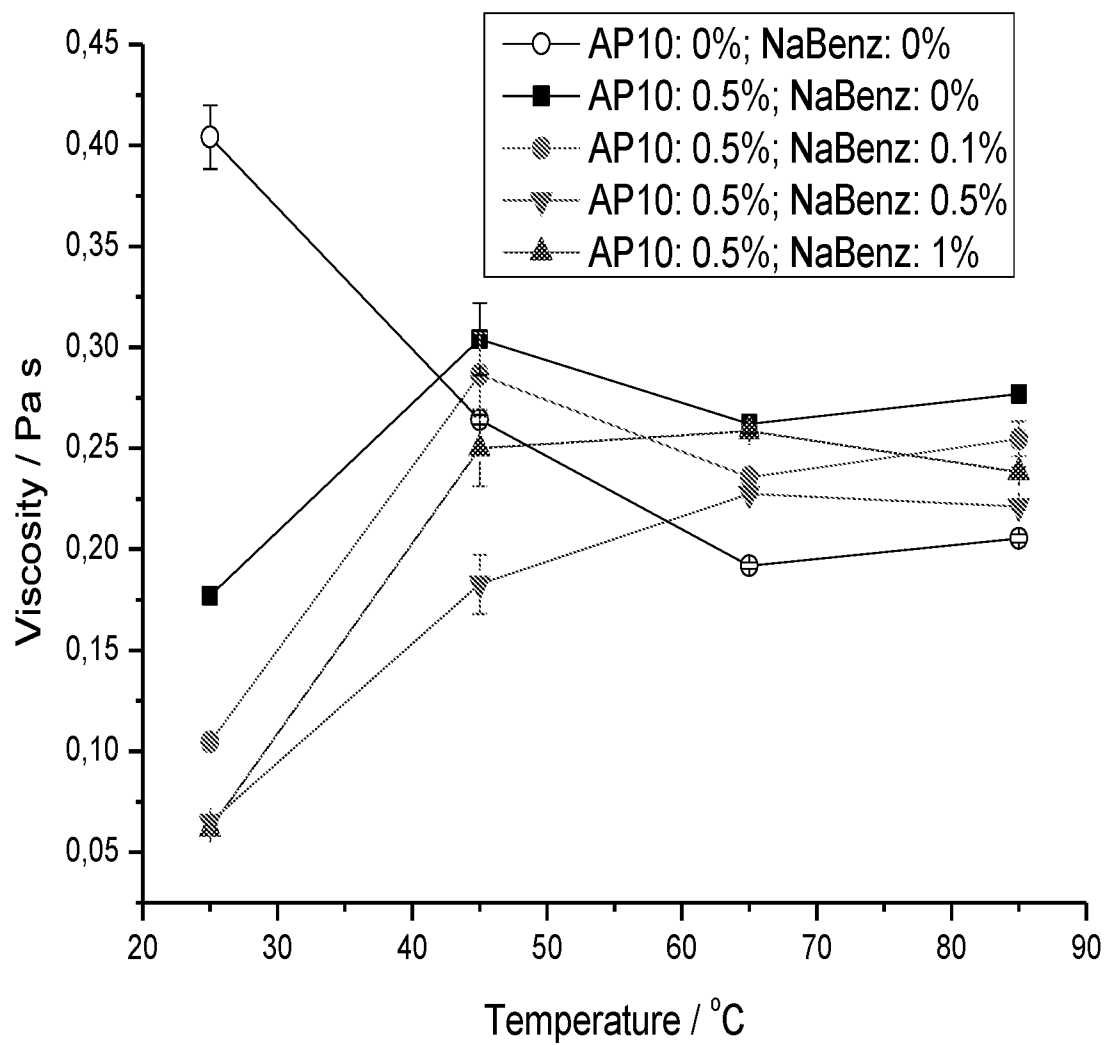


Figure 8a

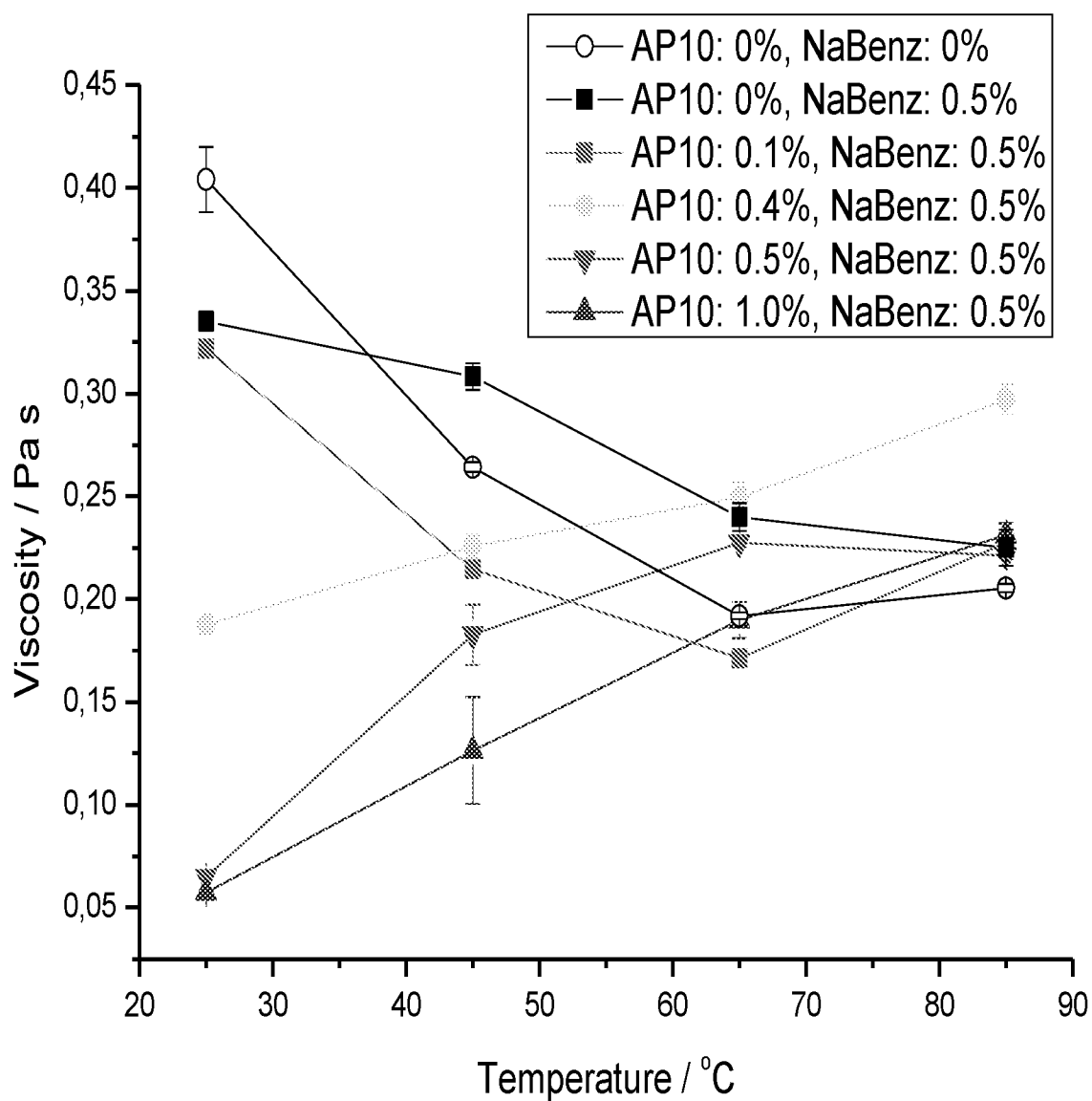


Figure 8b

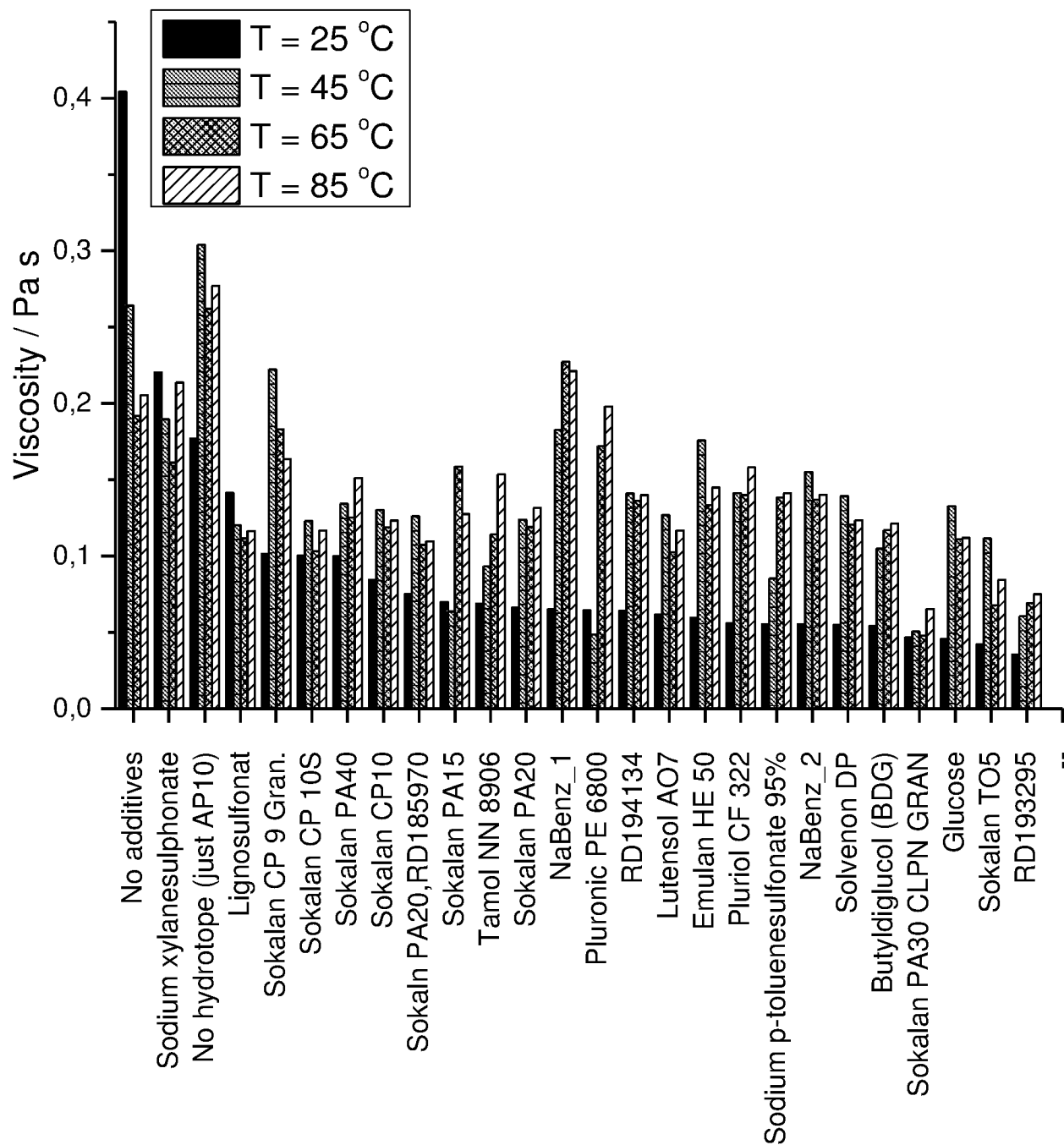


Figure 9

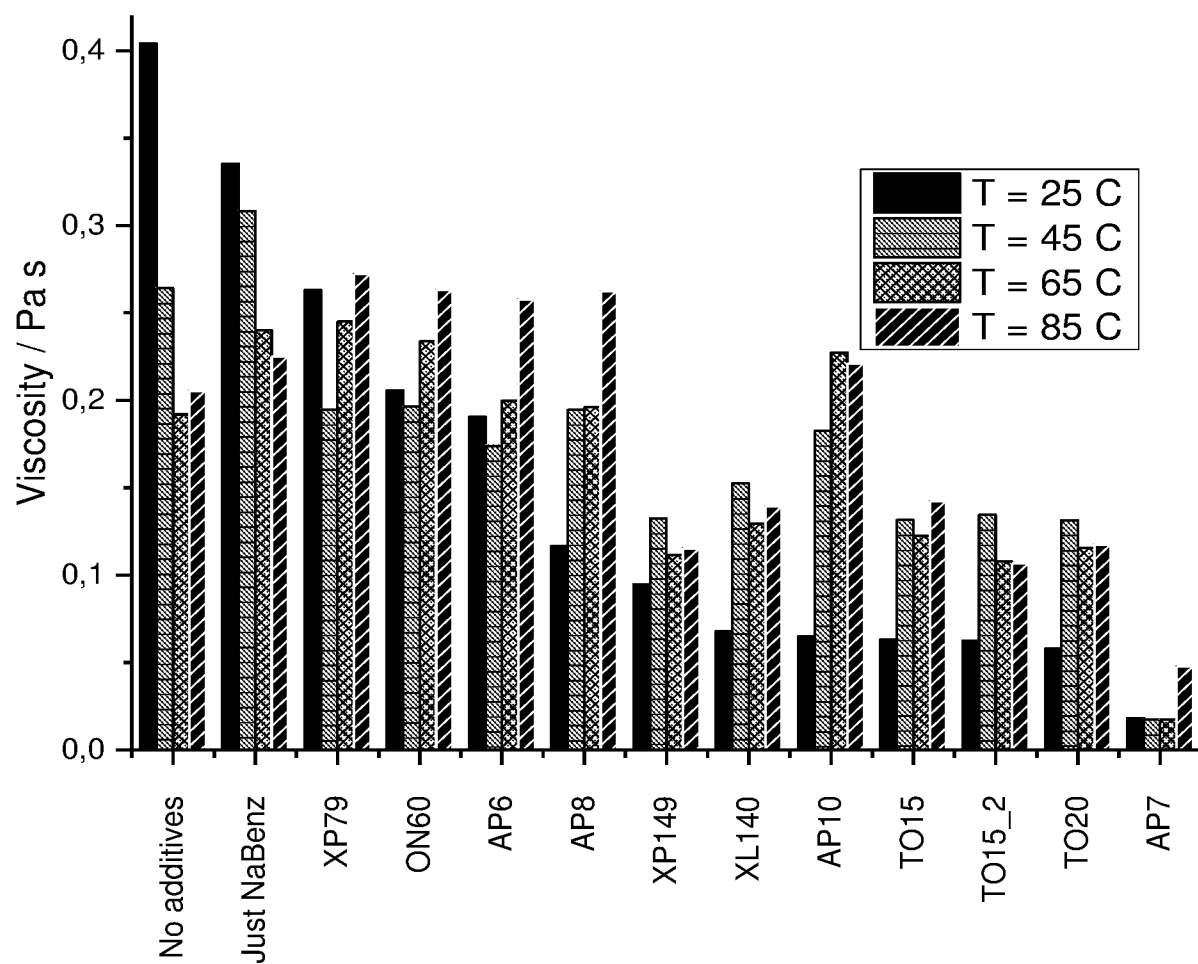


Figure 10

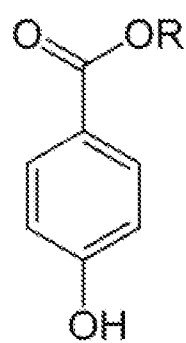


Figure 11

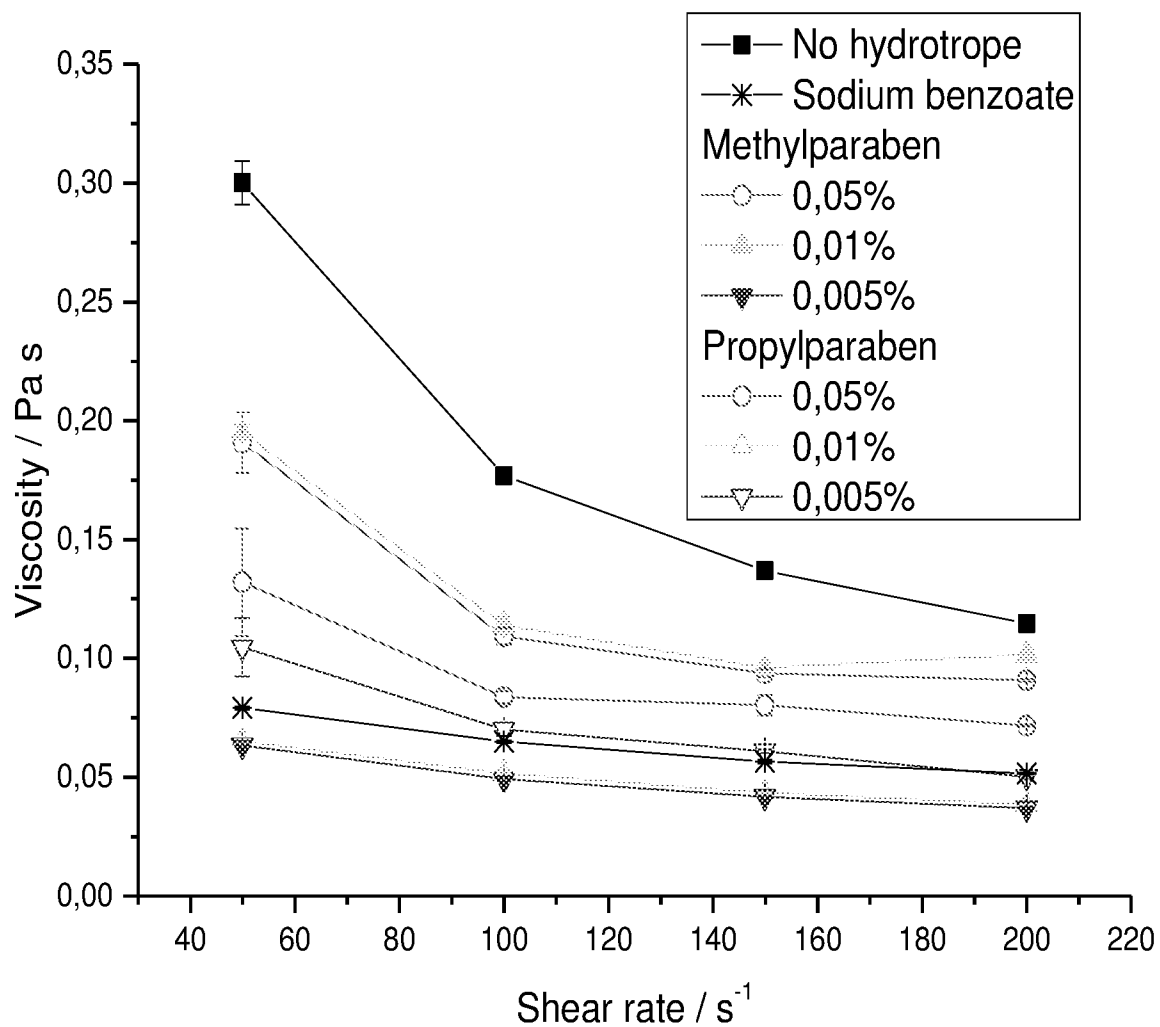


Figure 12a

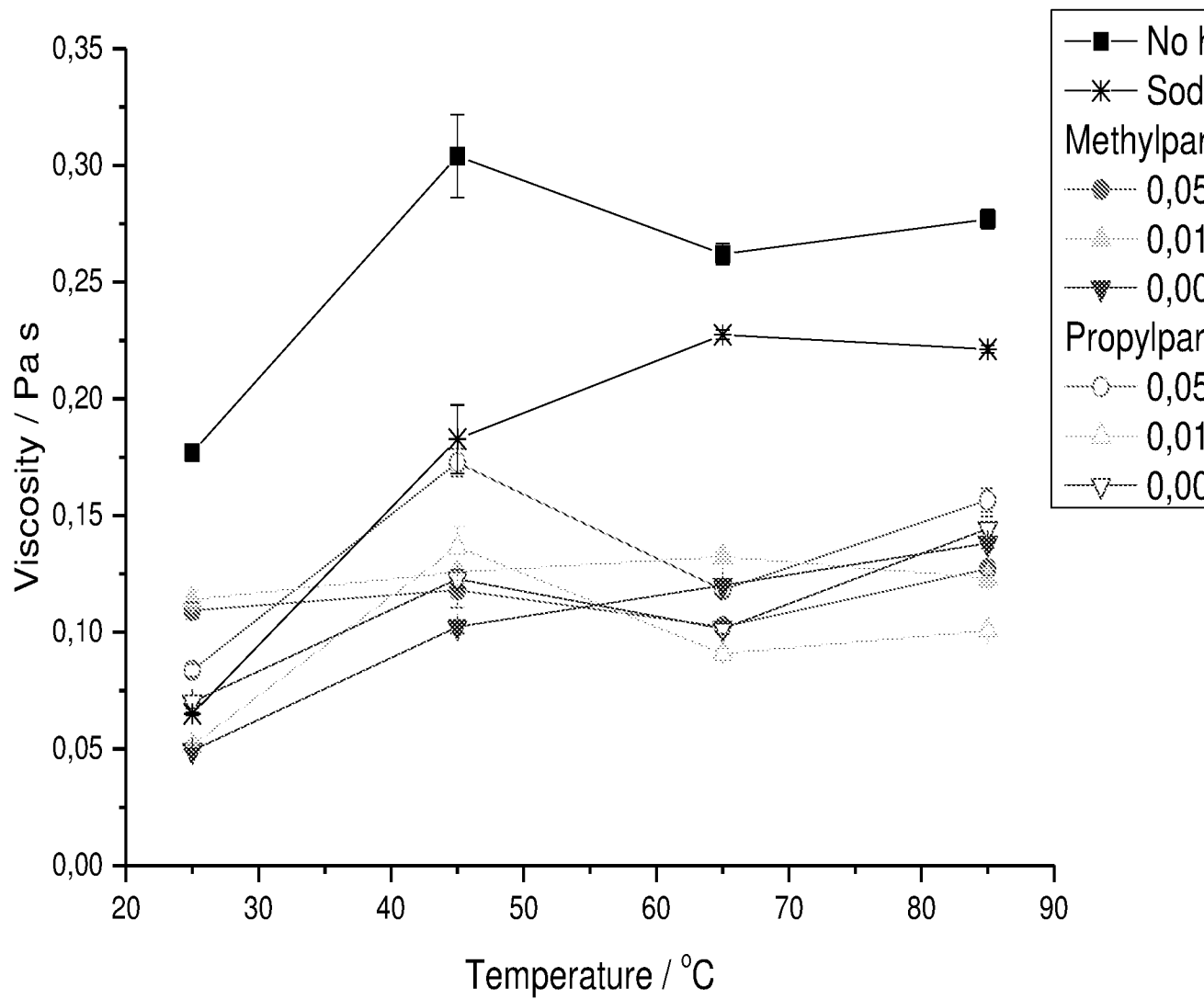


Figure 12b

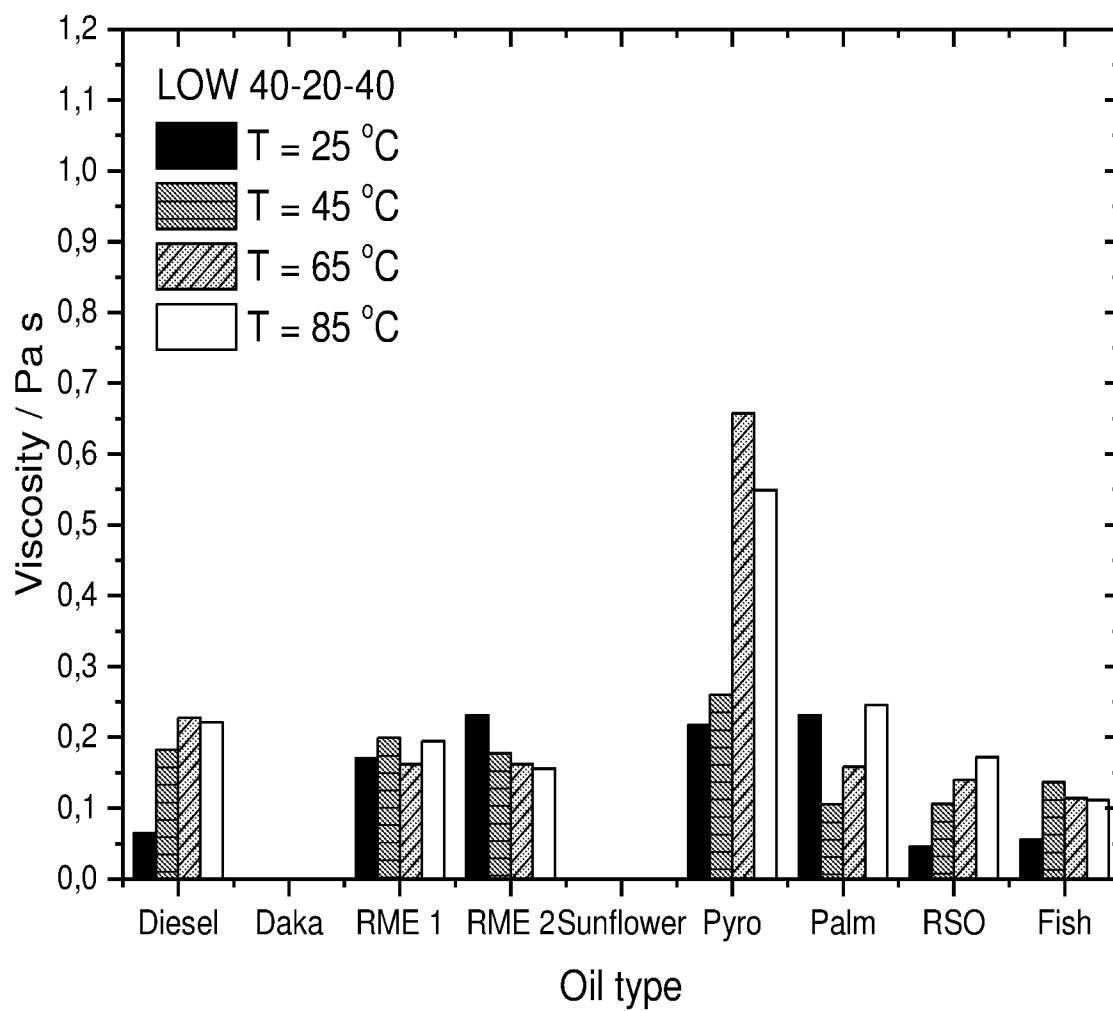


Figure 13a

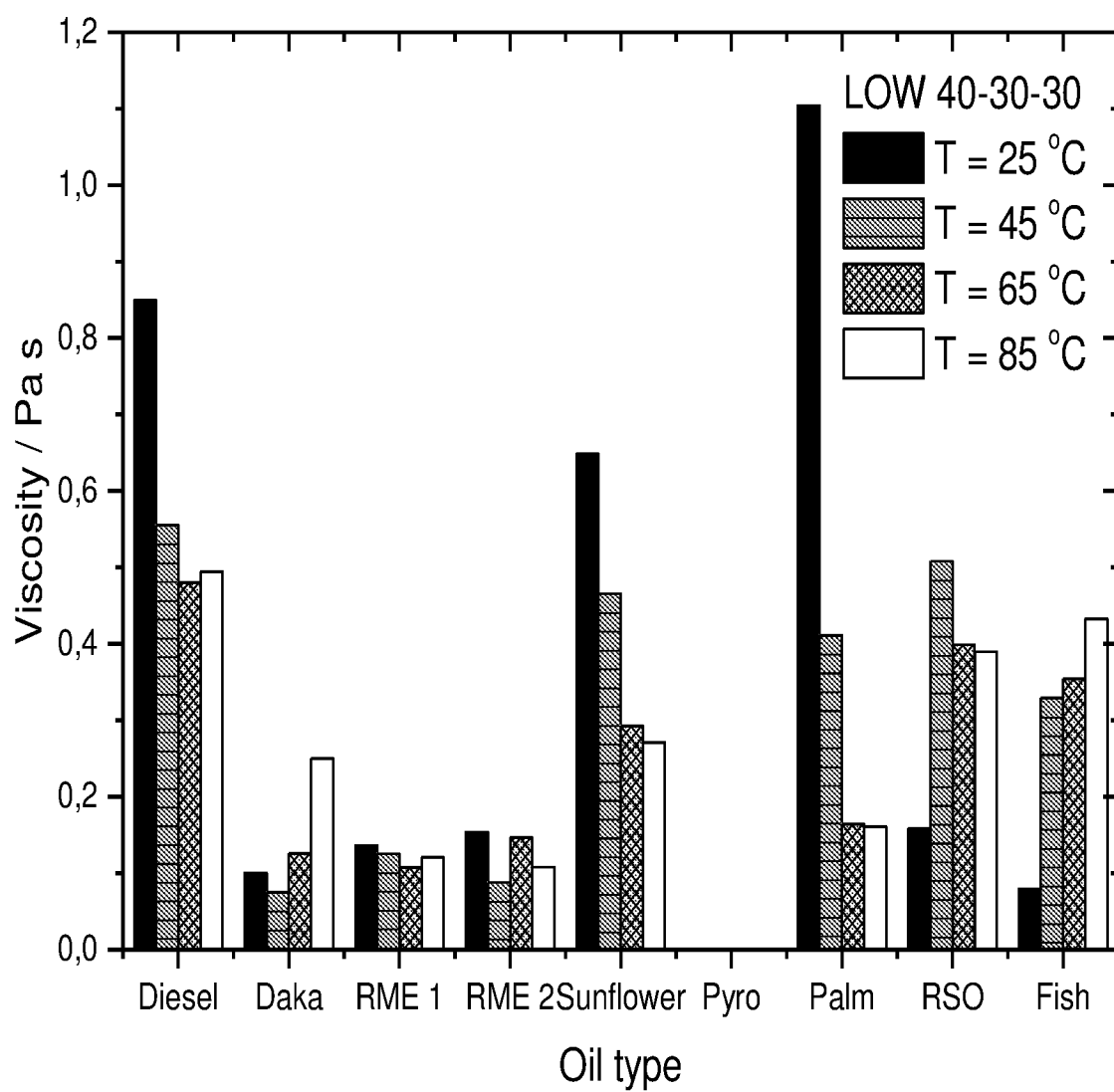


Figure 13b

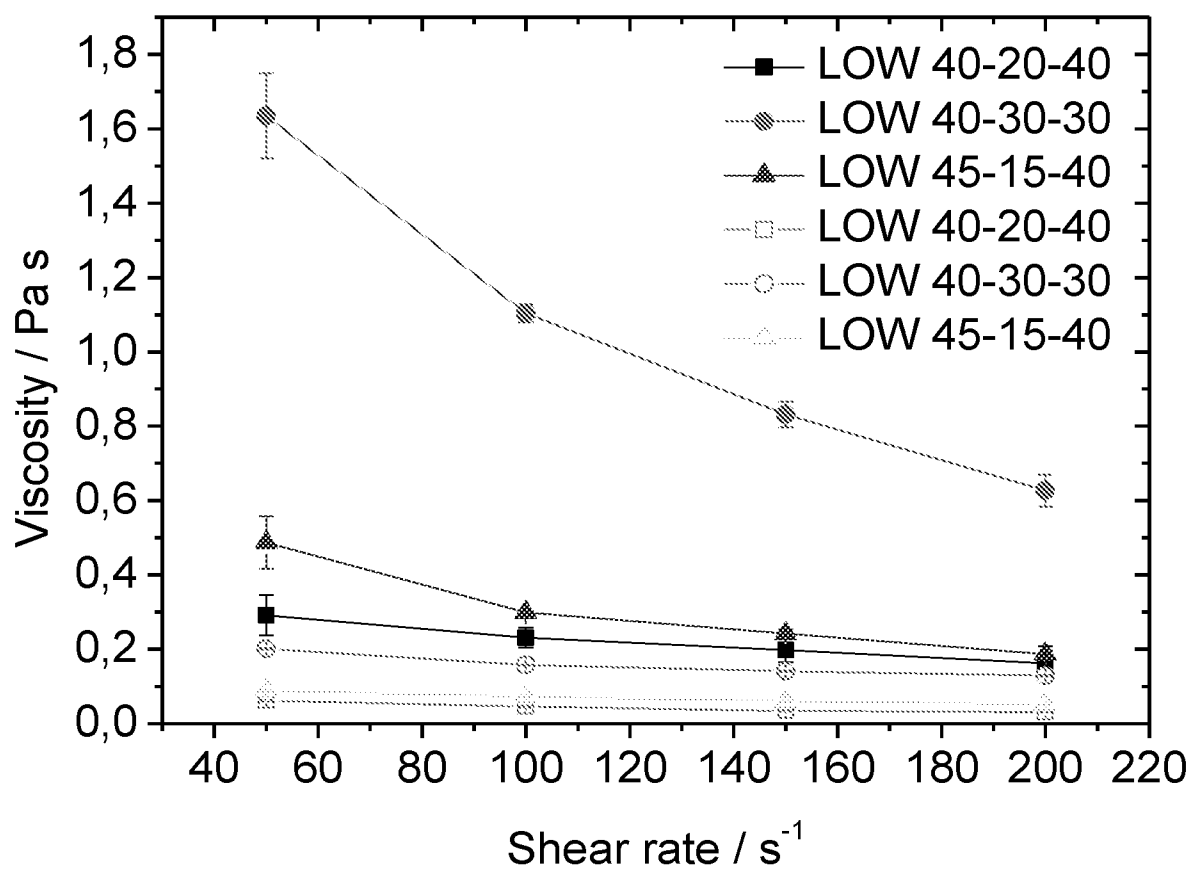


Figure 14a

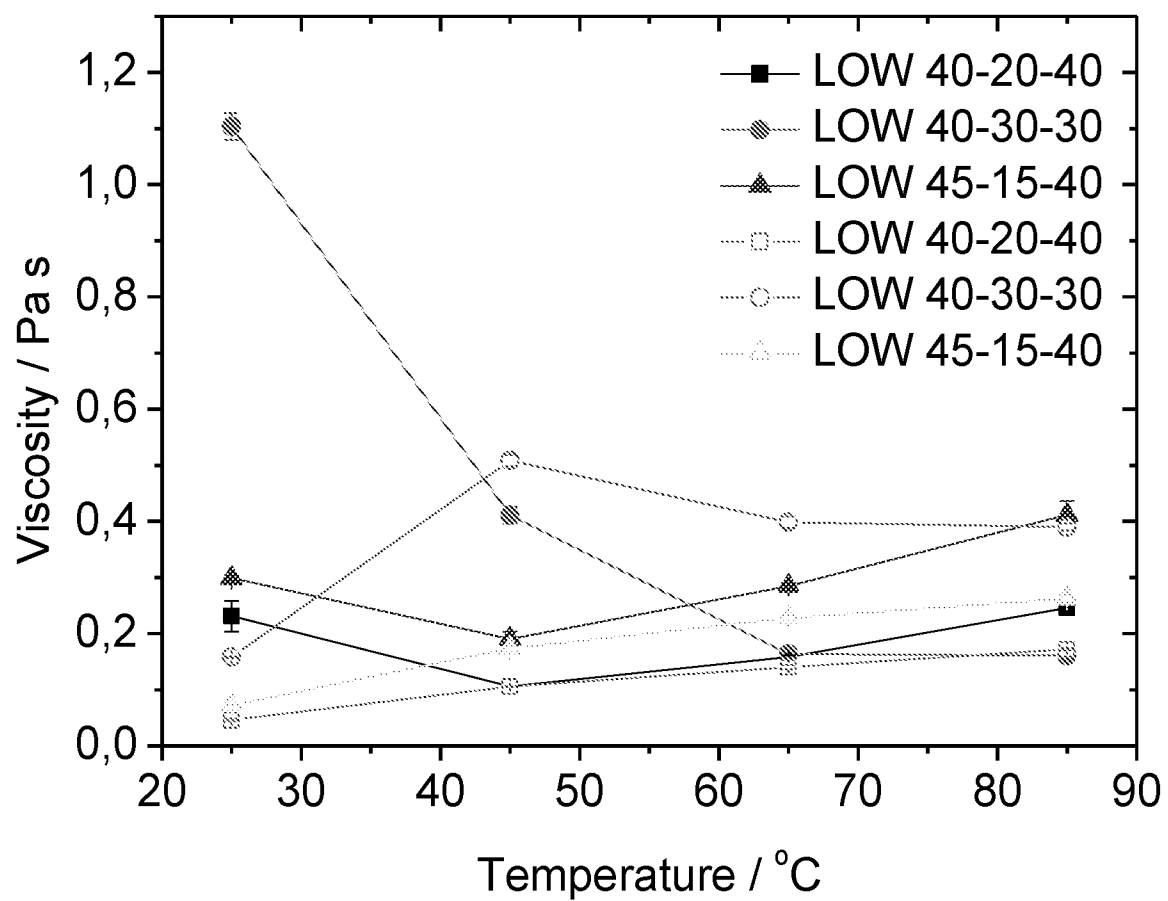


Figure 14b

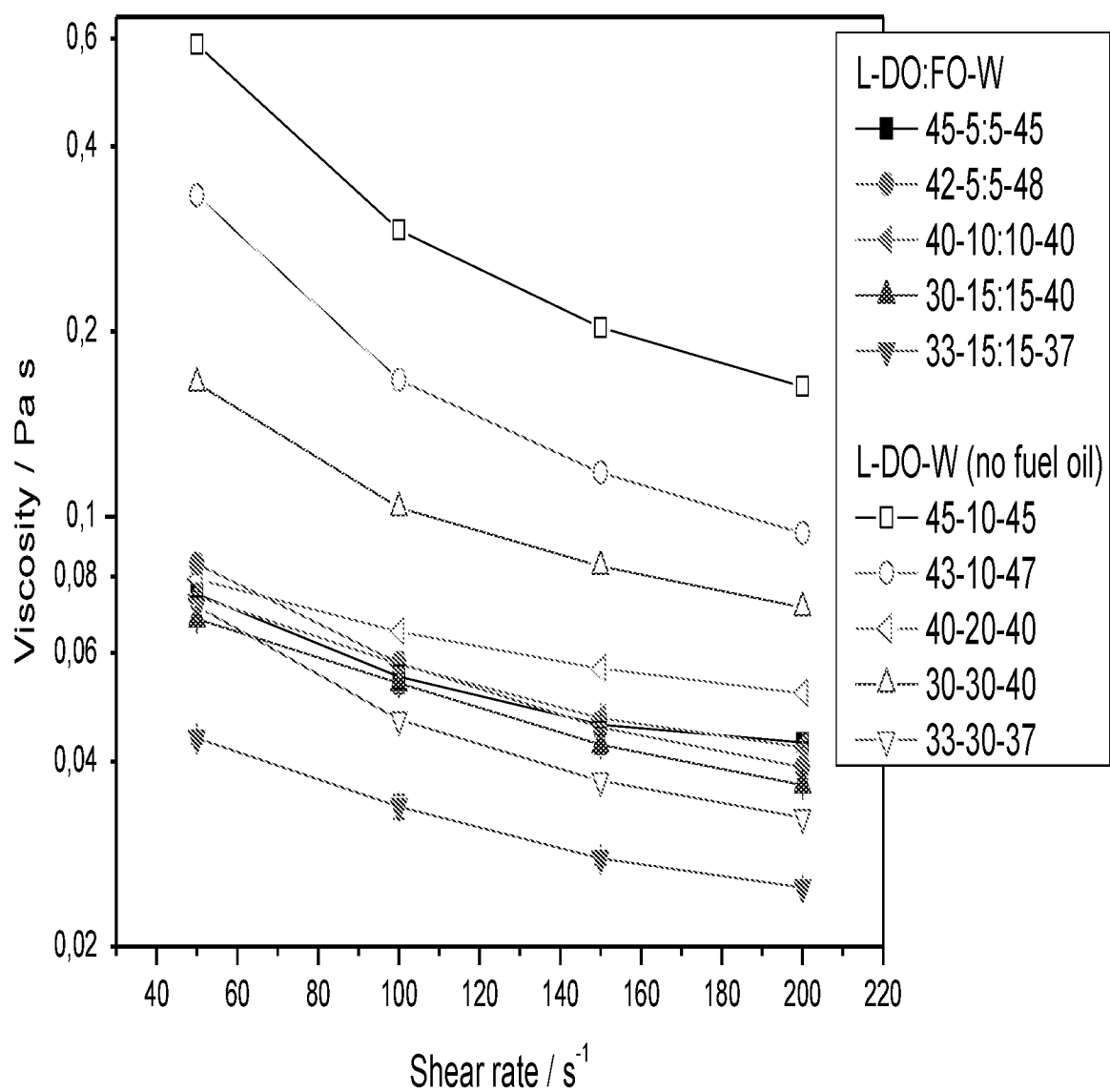


Figure 15

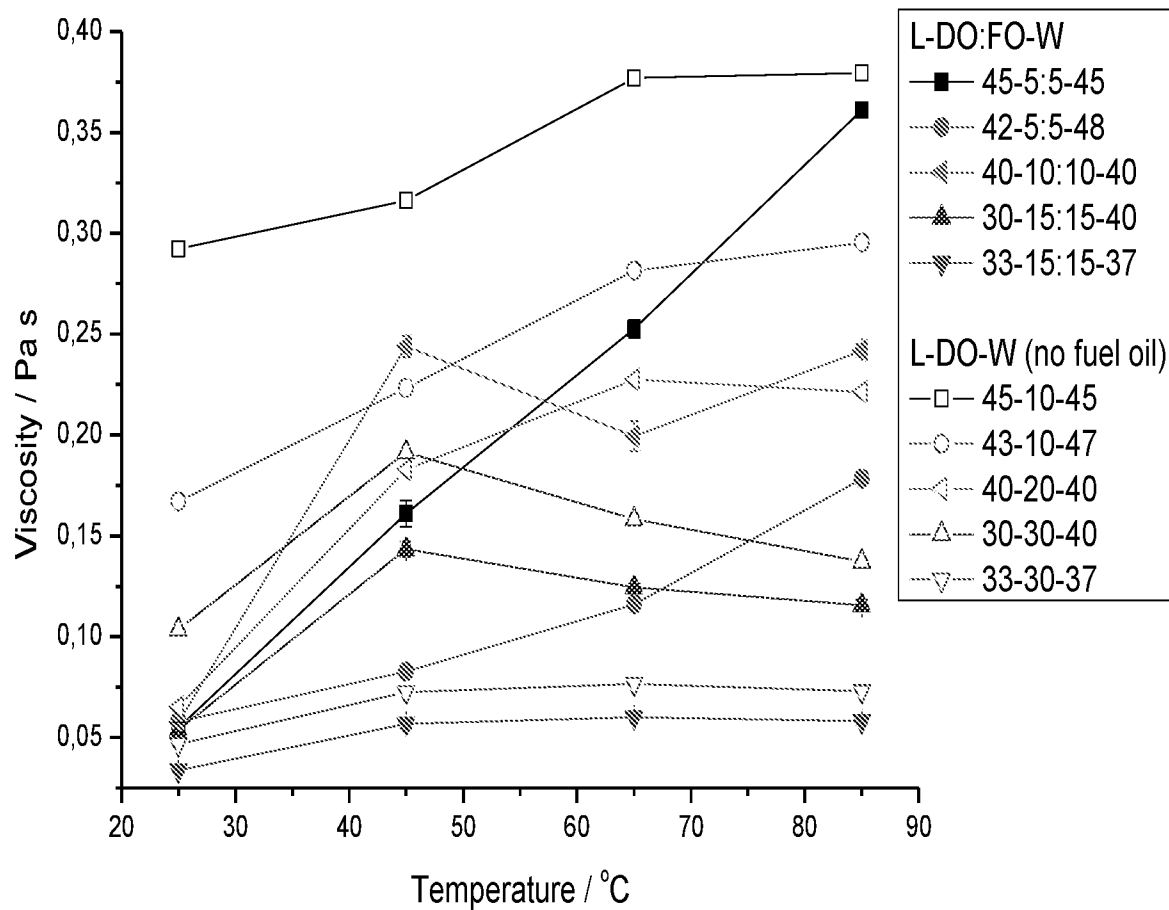


Figure 16

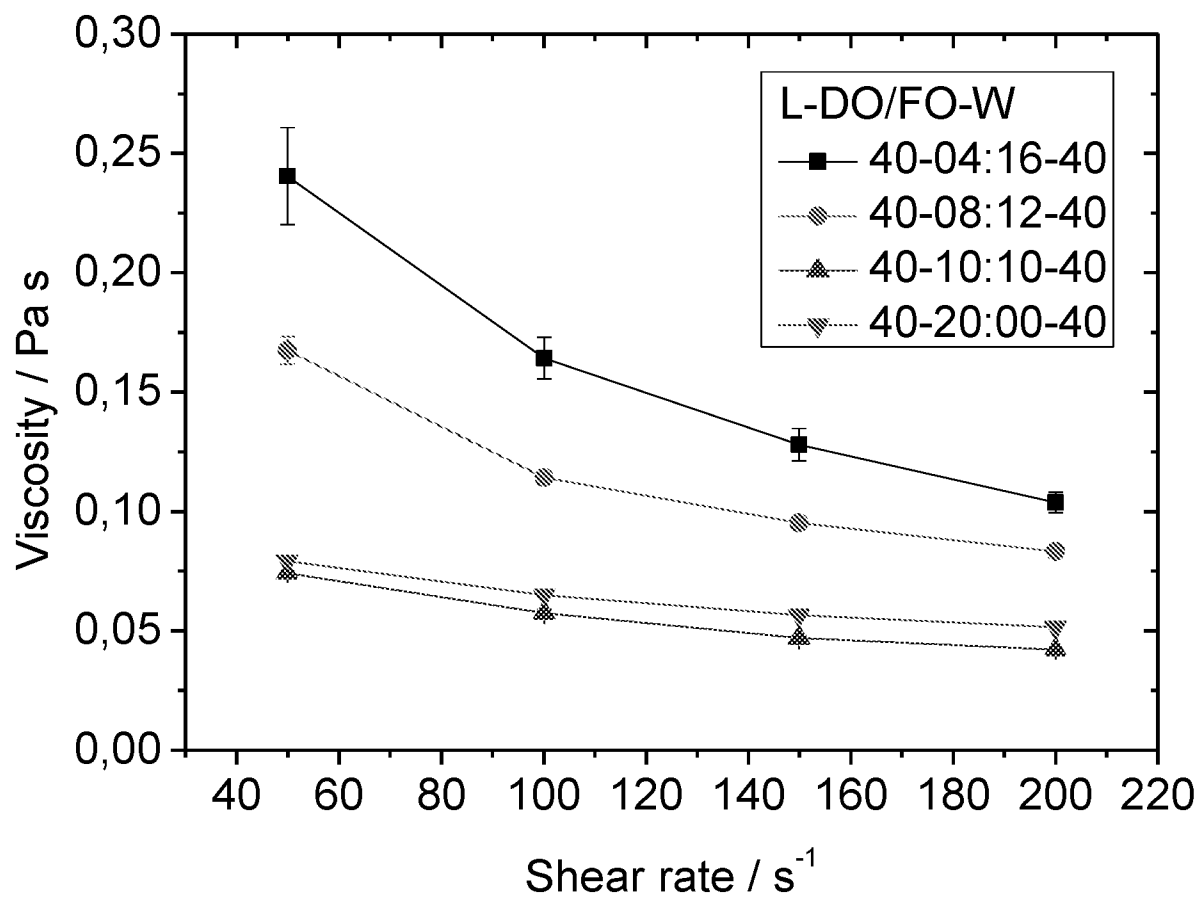


Figure 17a

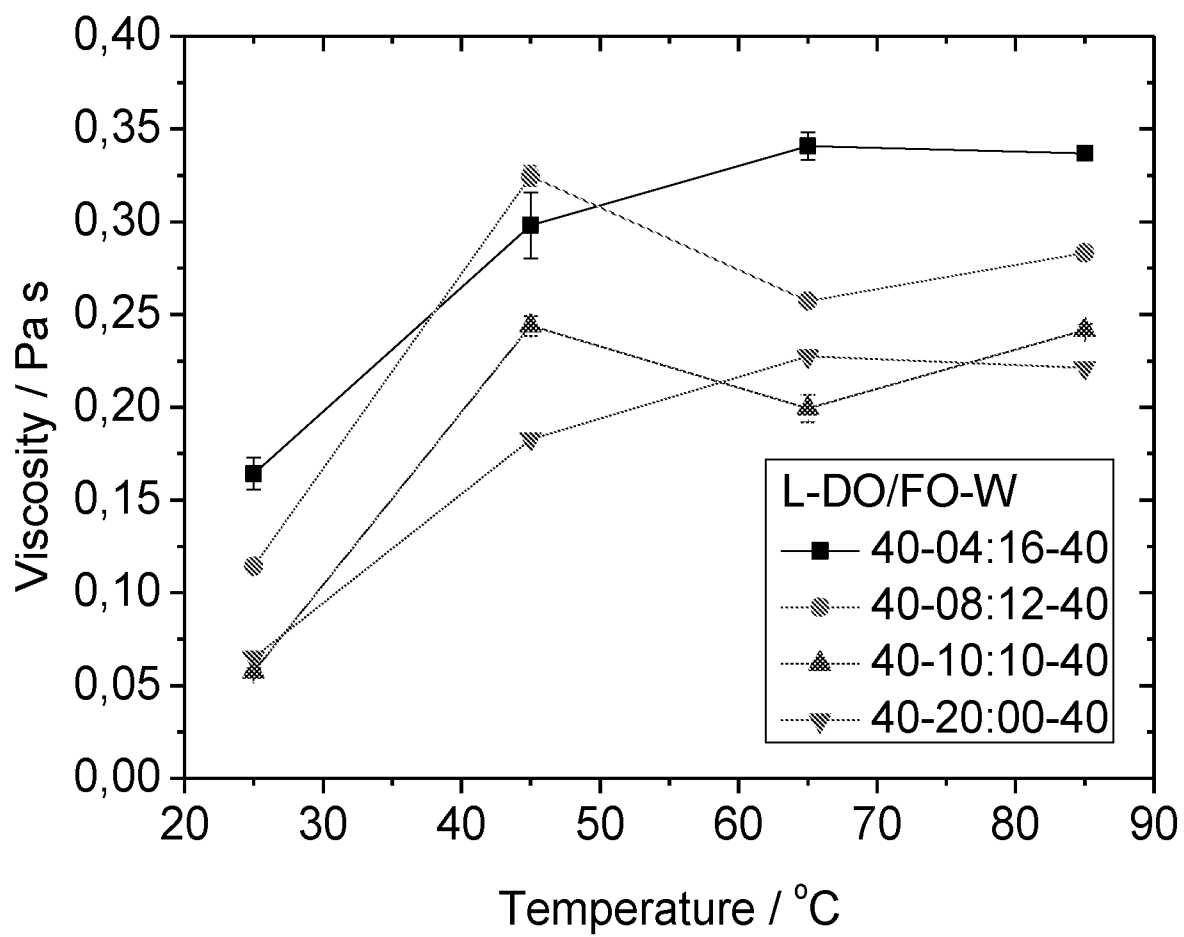


Figure 17b

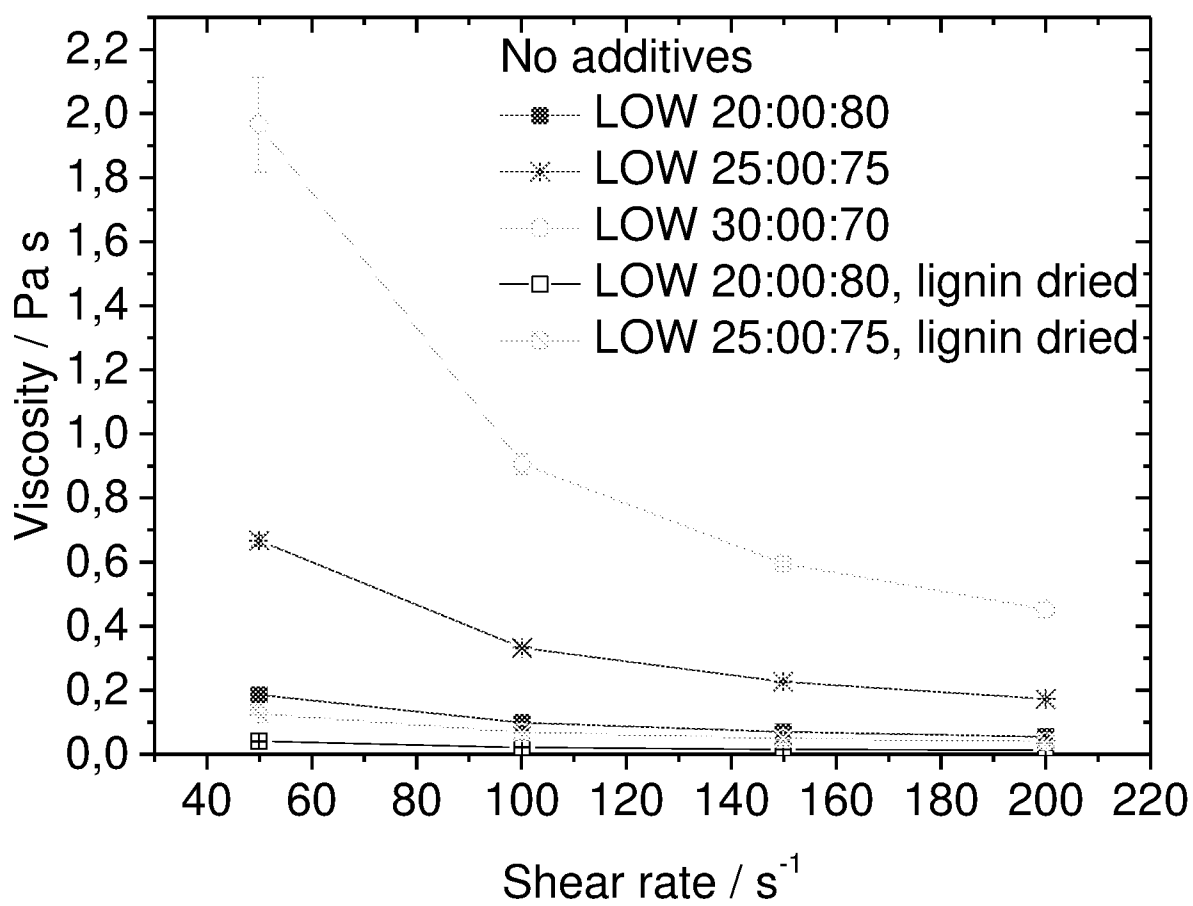


Figure 18a

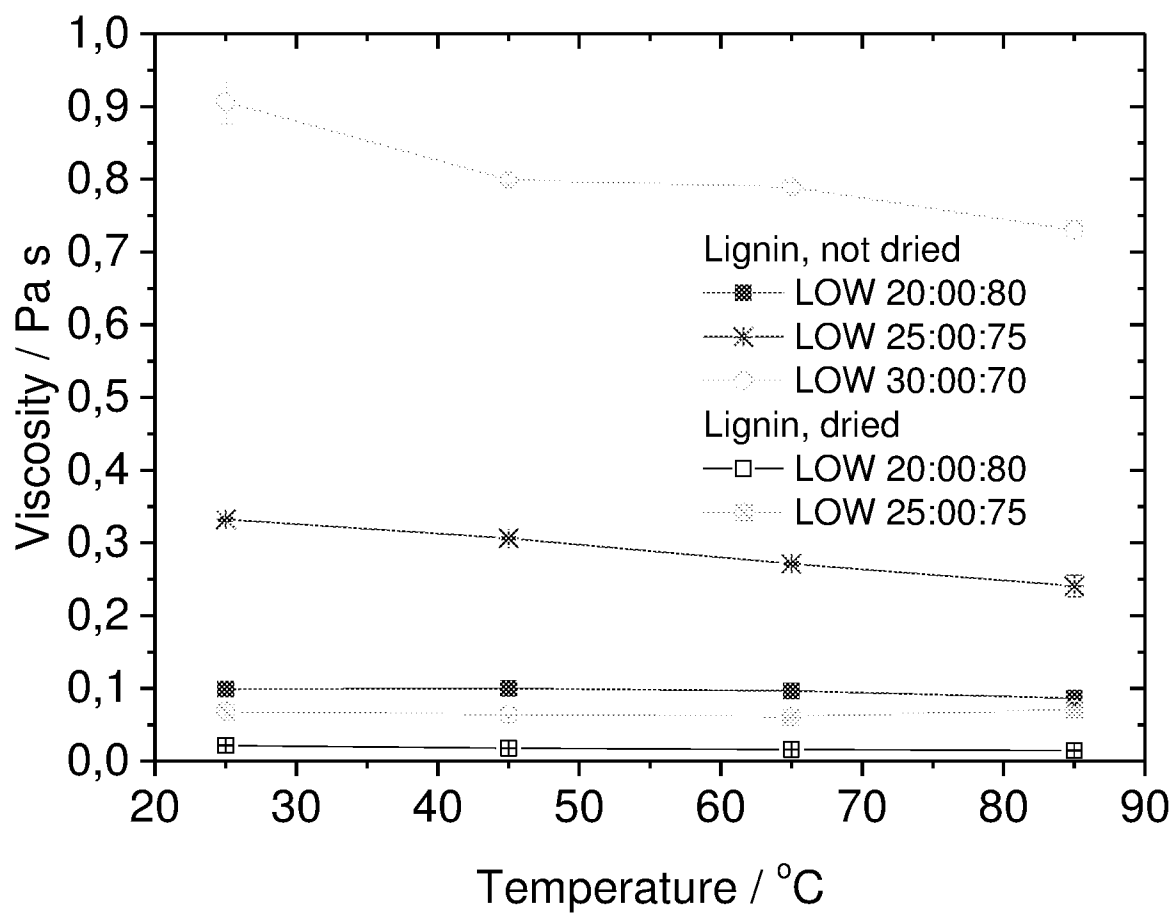


Figure 18b

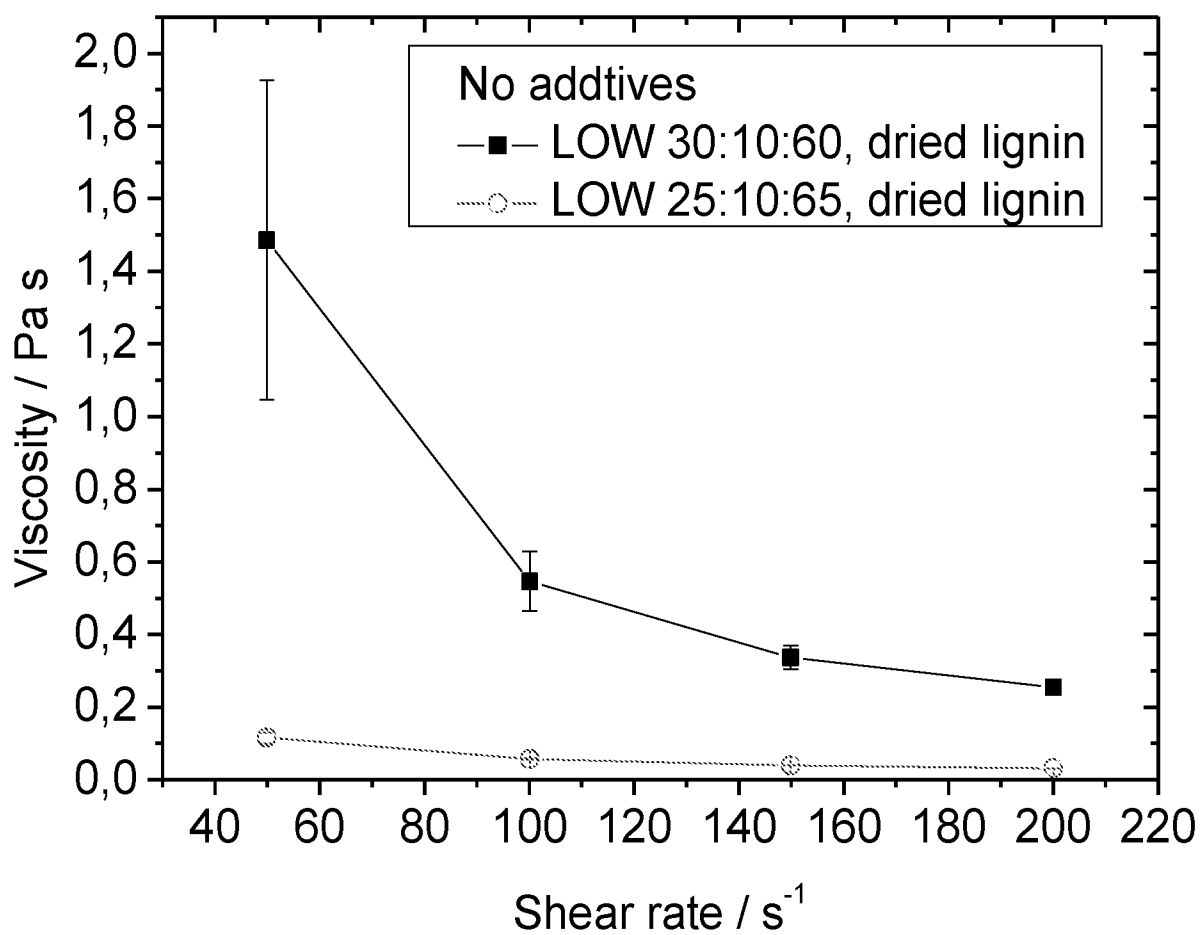


Figure 19a

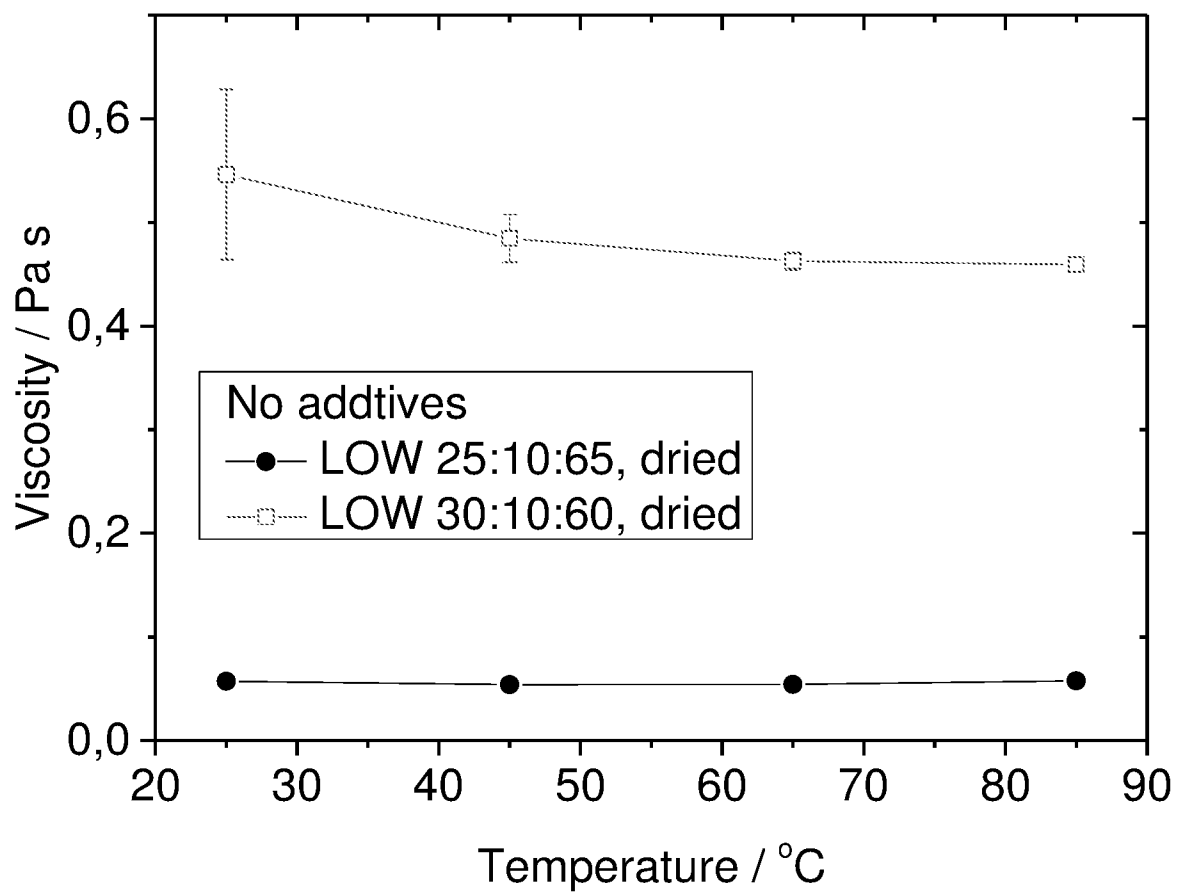


Figure 19b

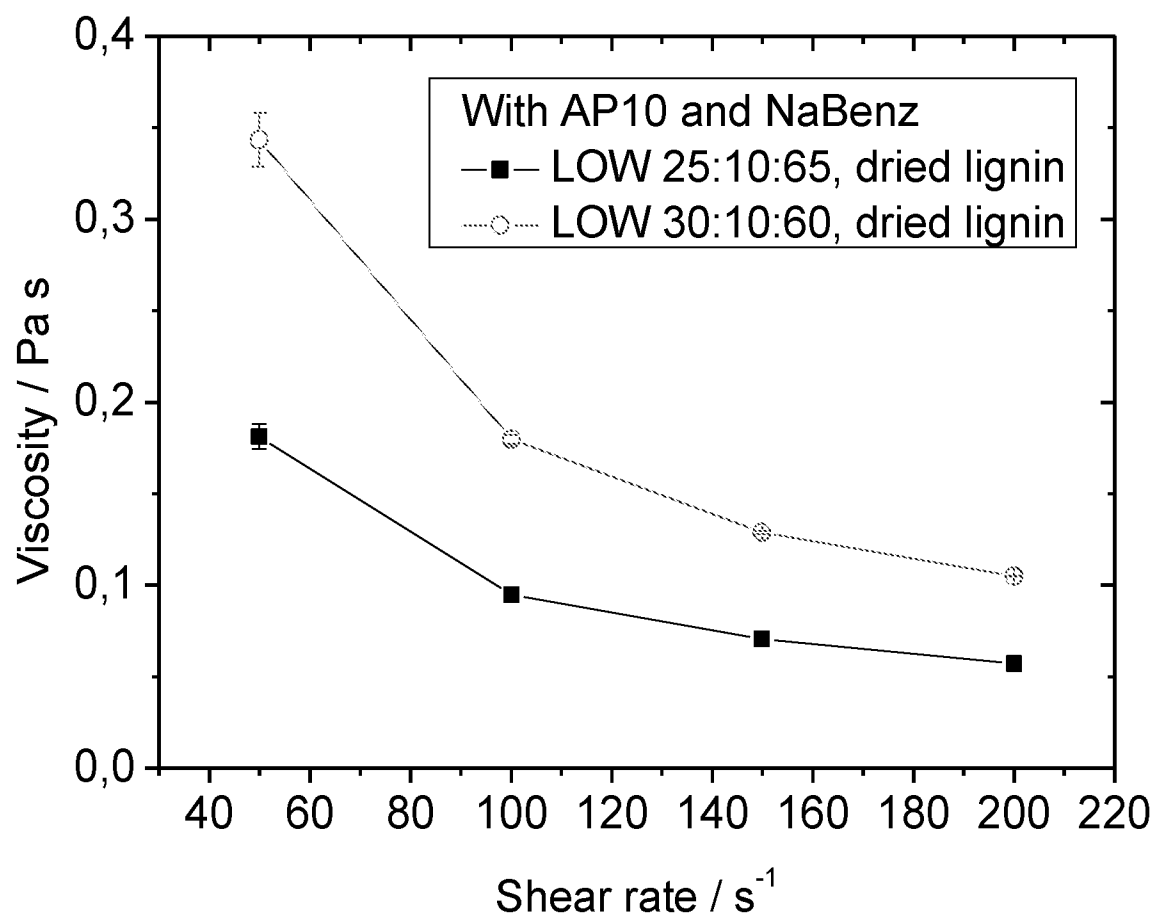


Figure 20a

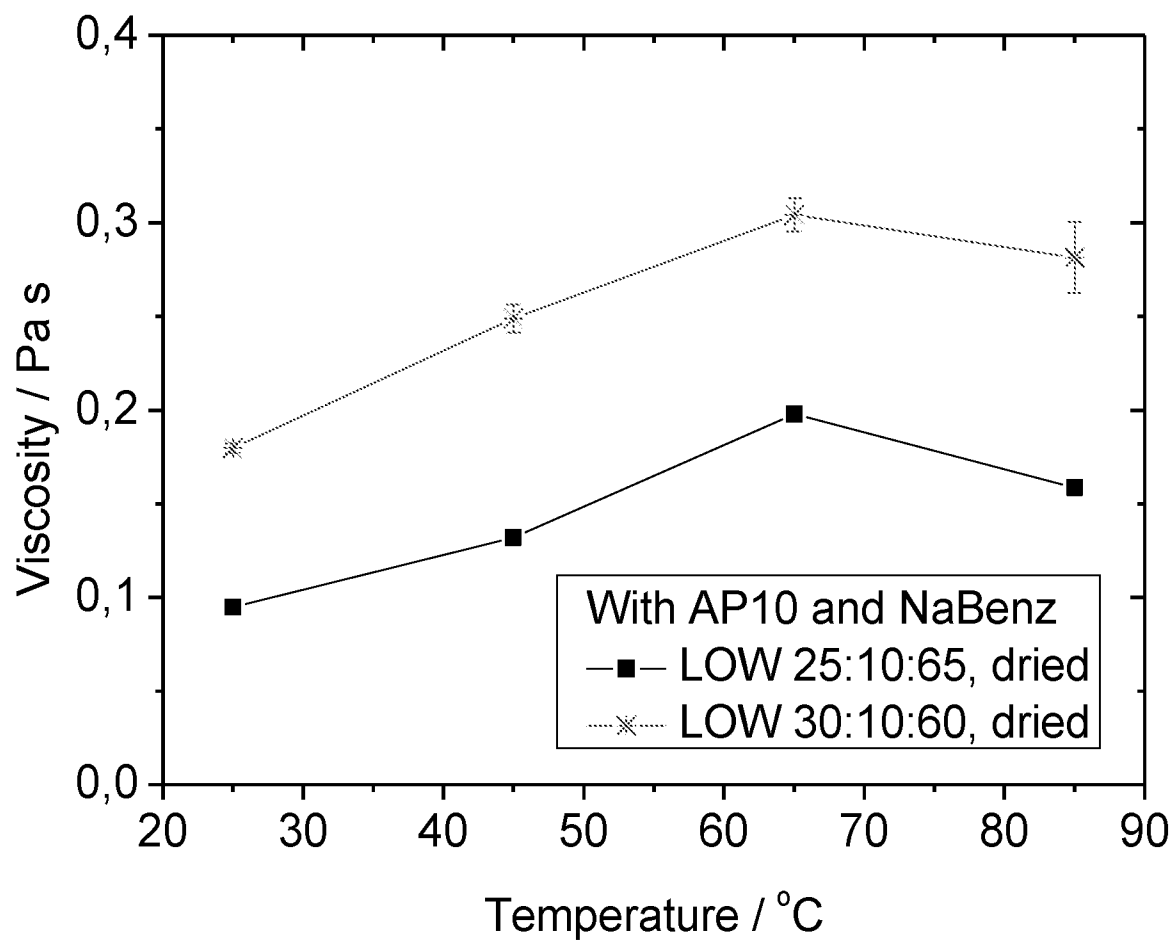


Figure 20b

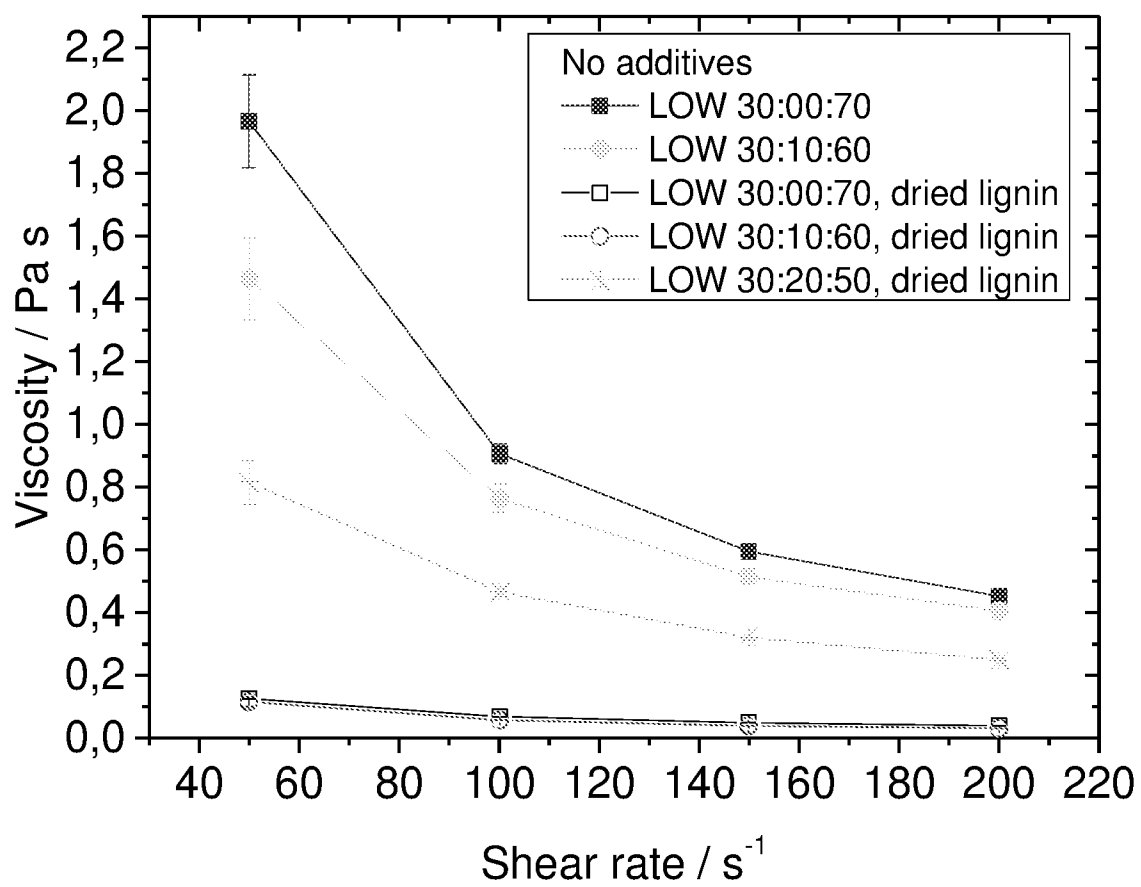


Figure 21a

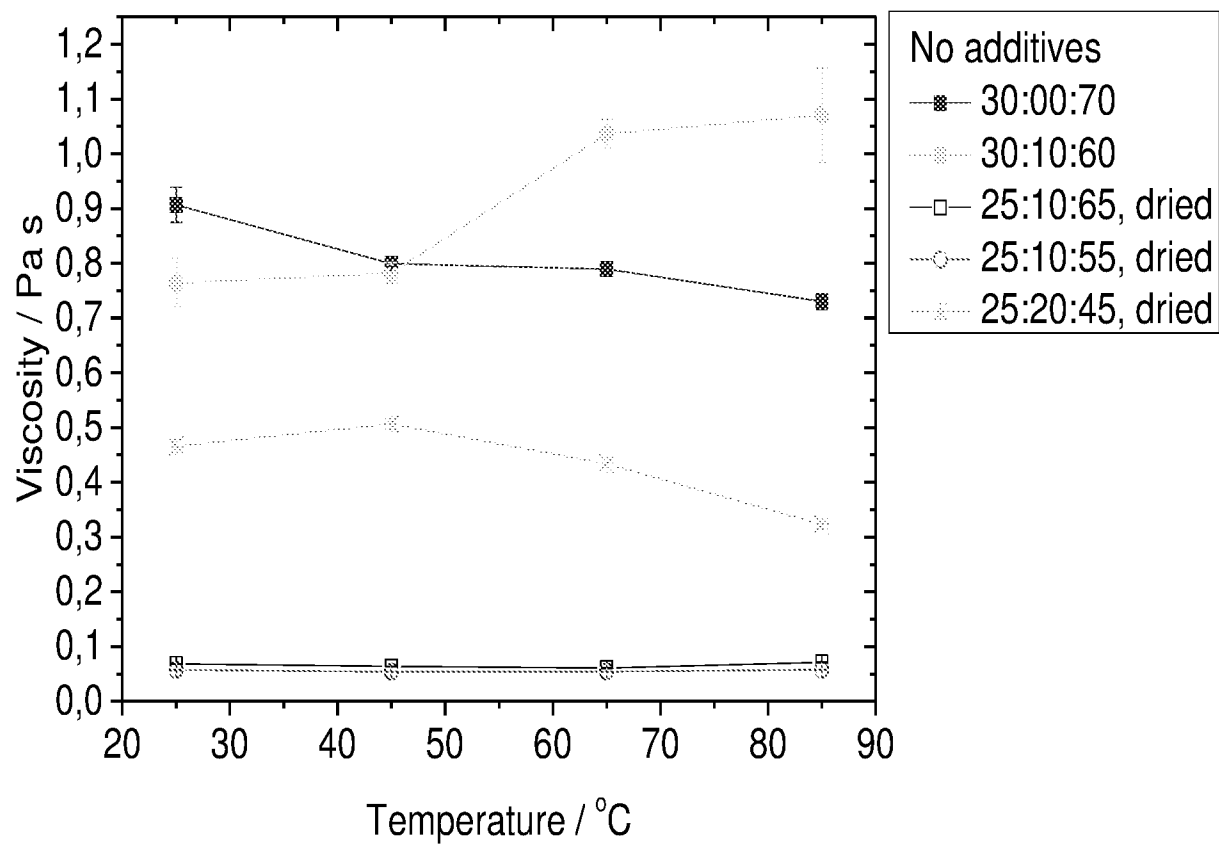


Figure 21b

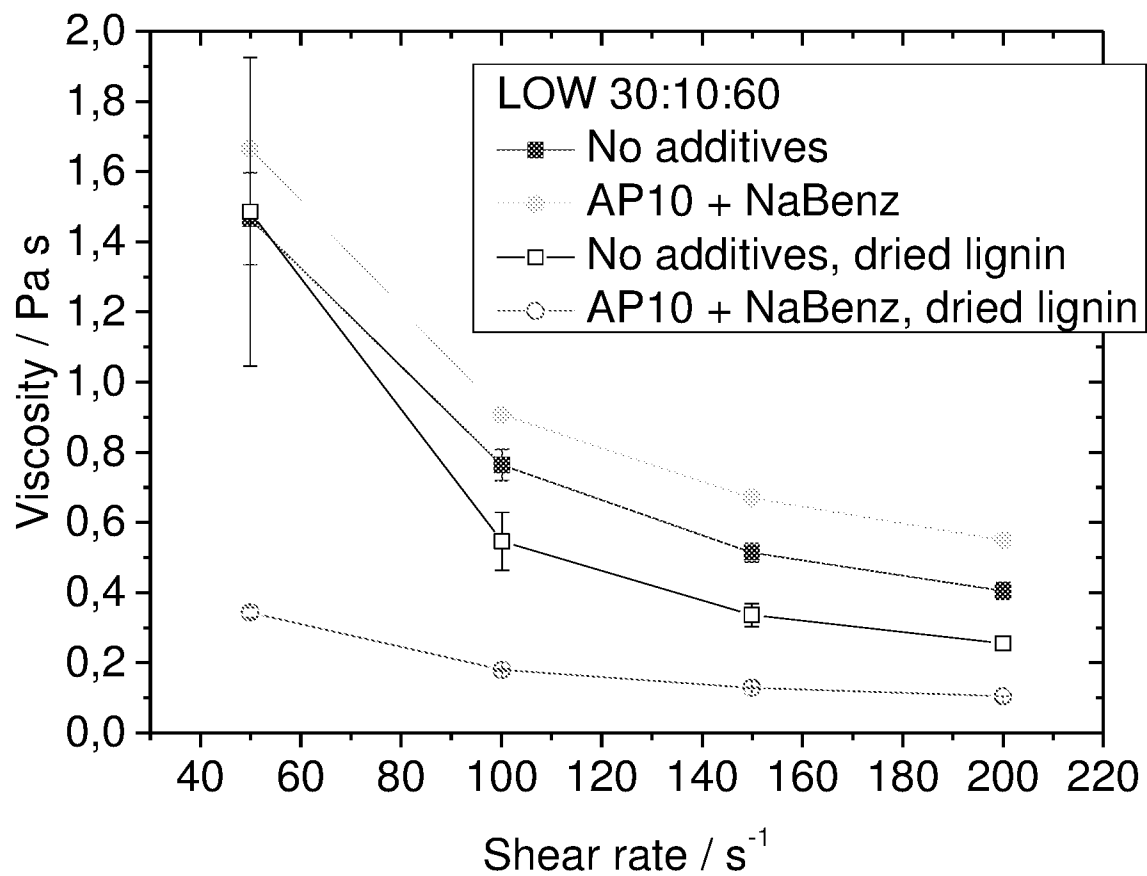


Figure 22a

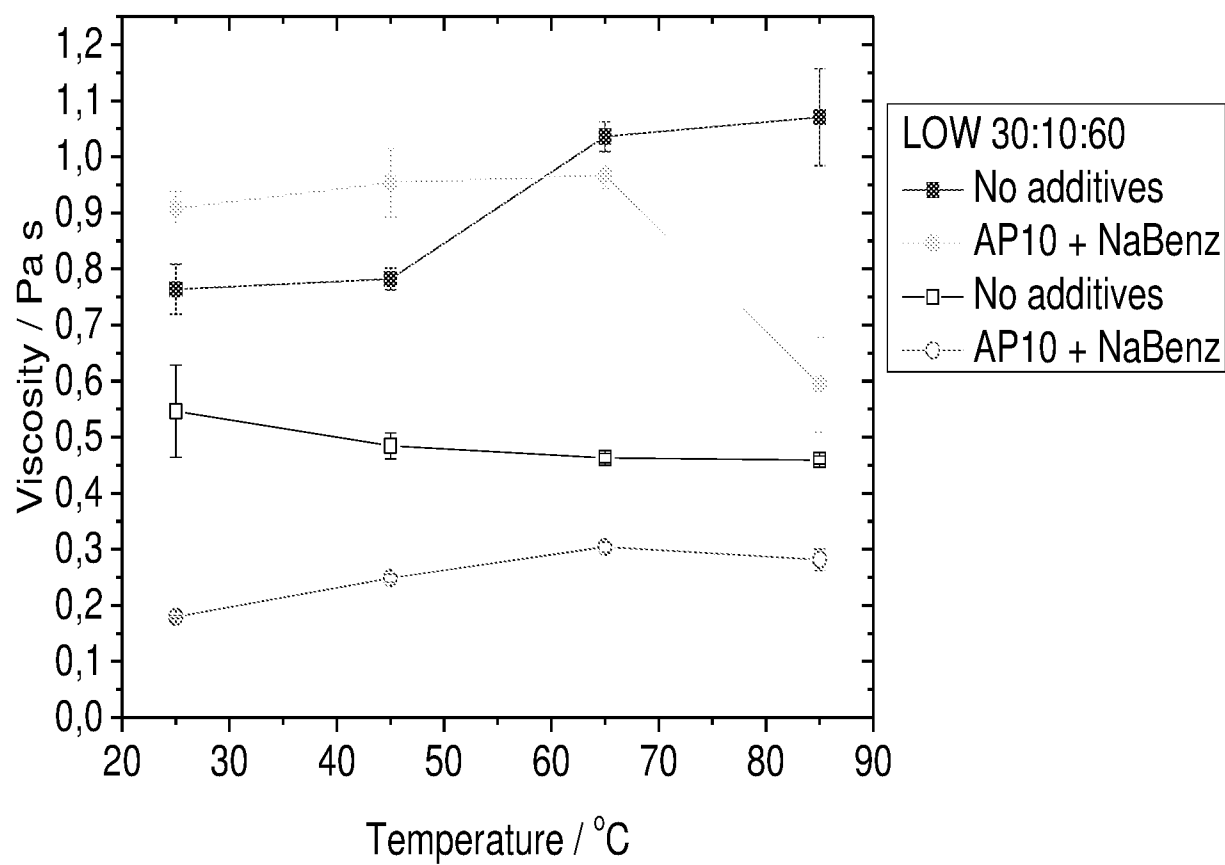


Figure 22b

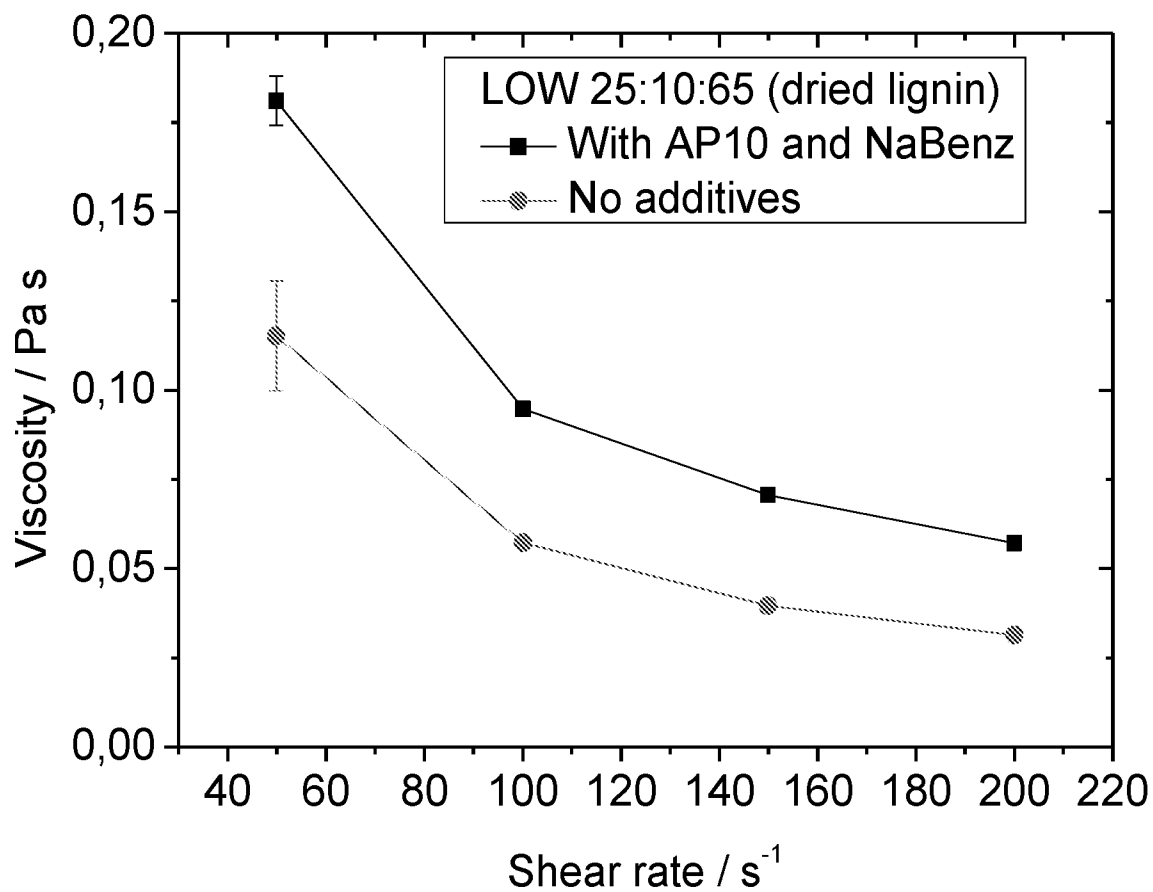


Figure 22c

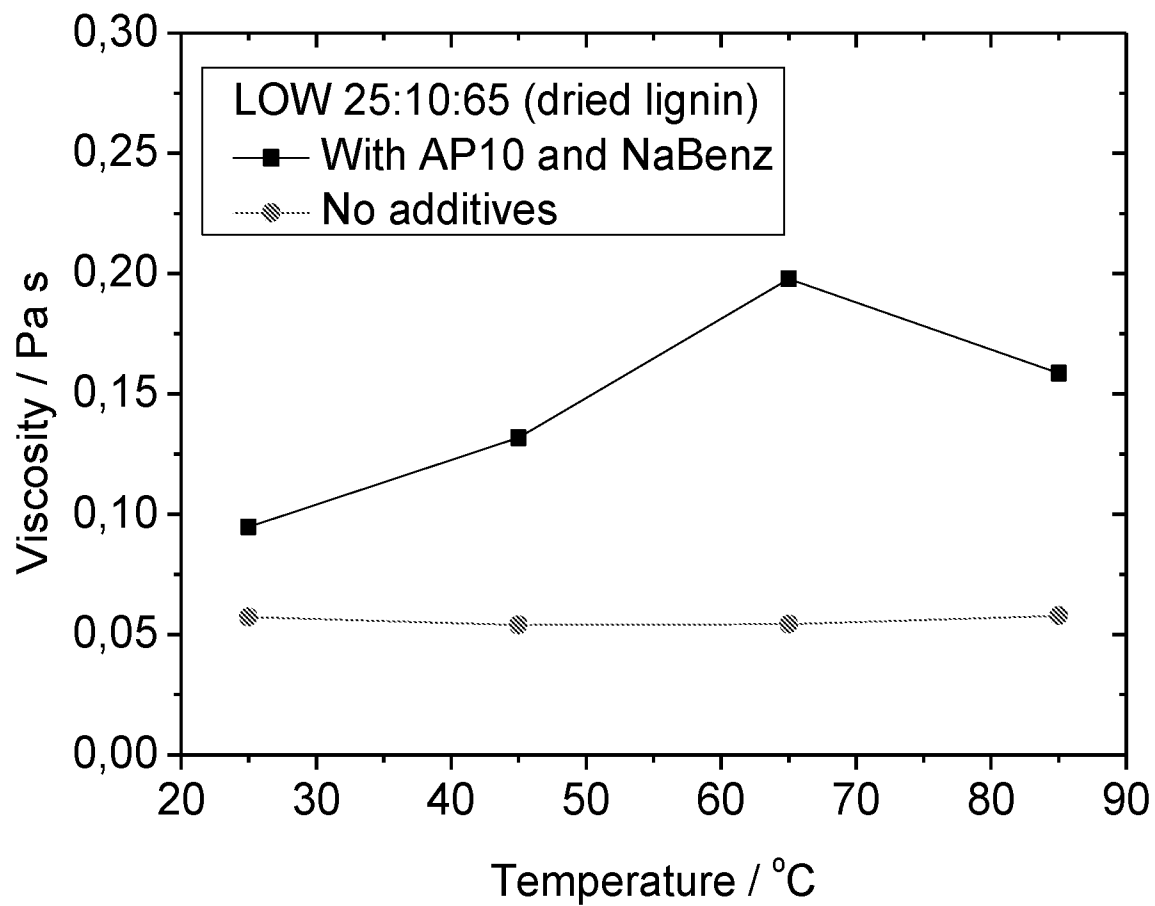


Figure 22d

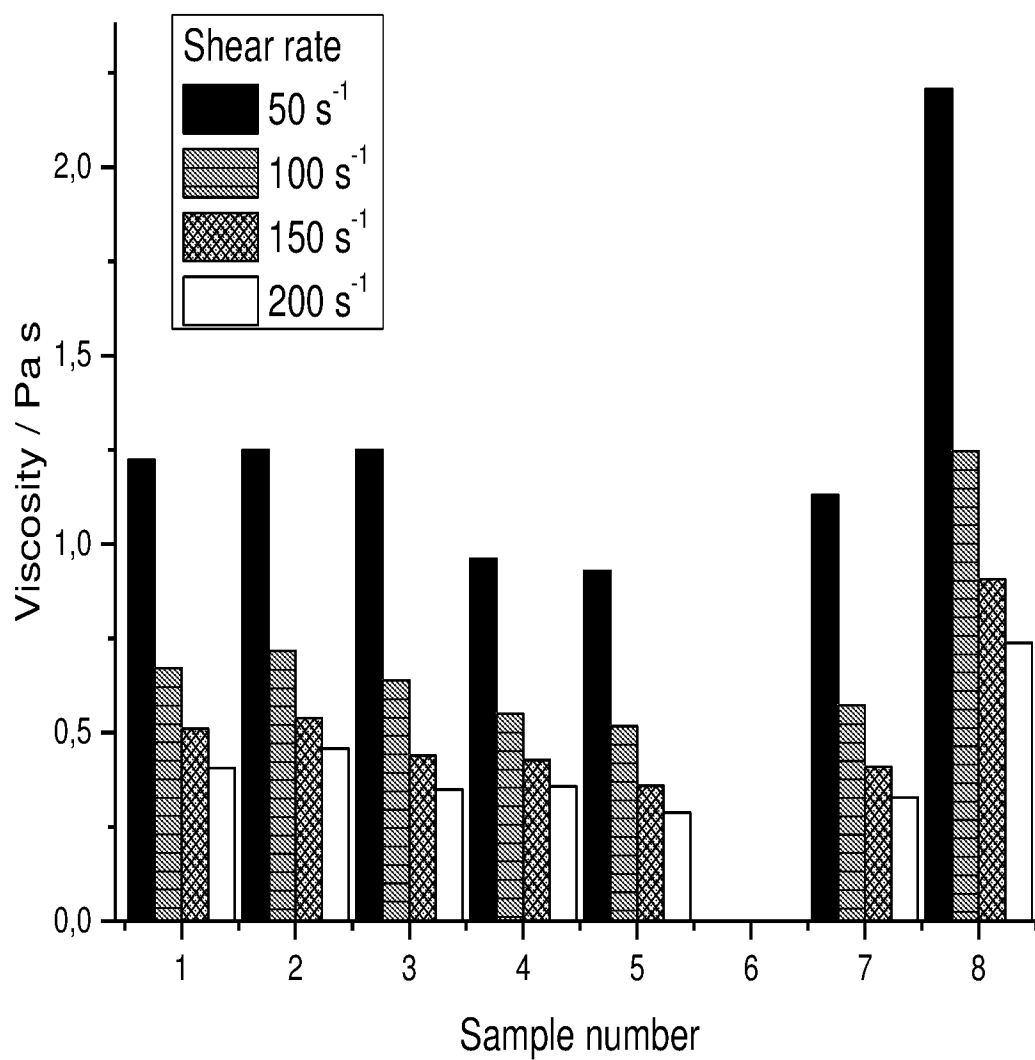


Figure 23

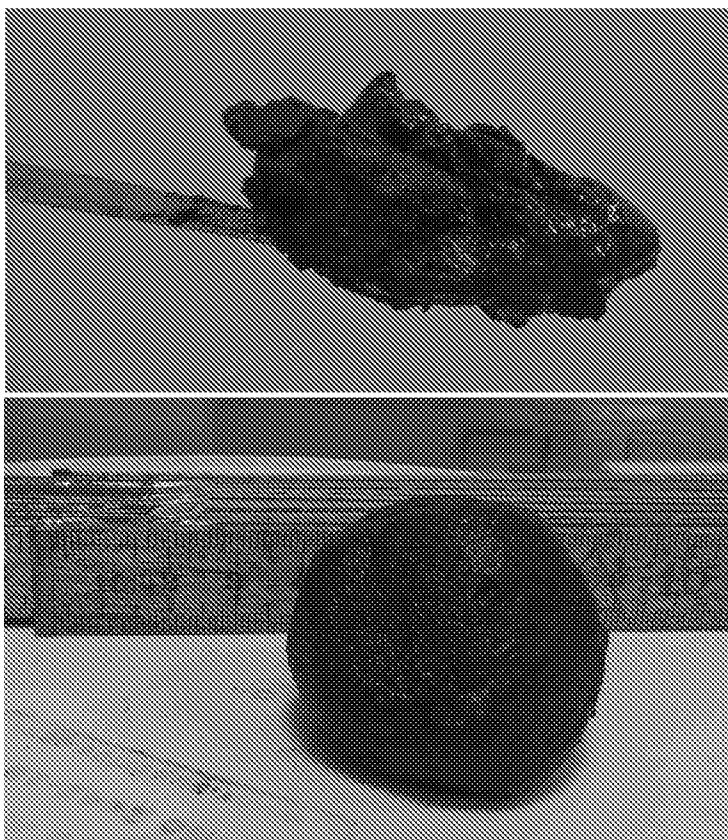


Figure 24

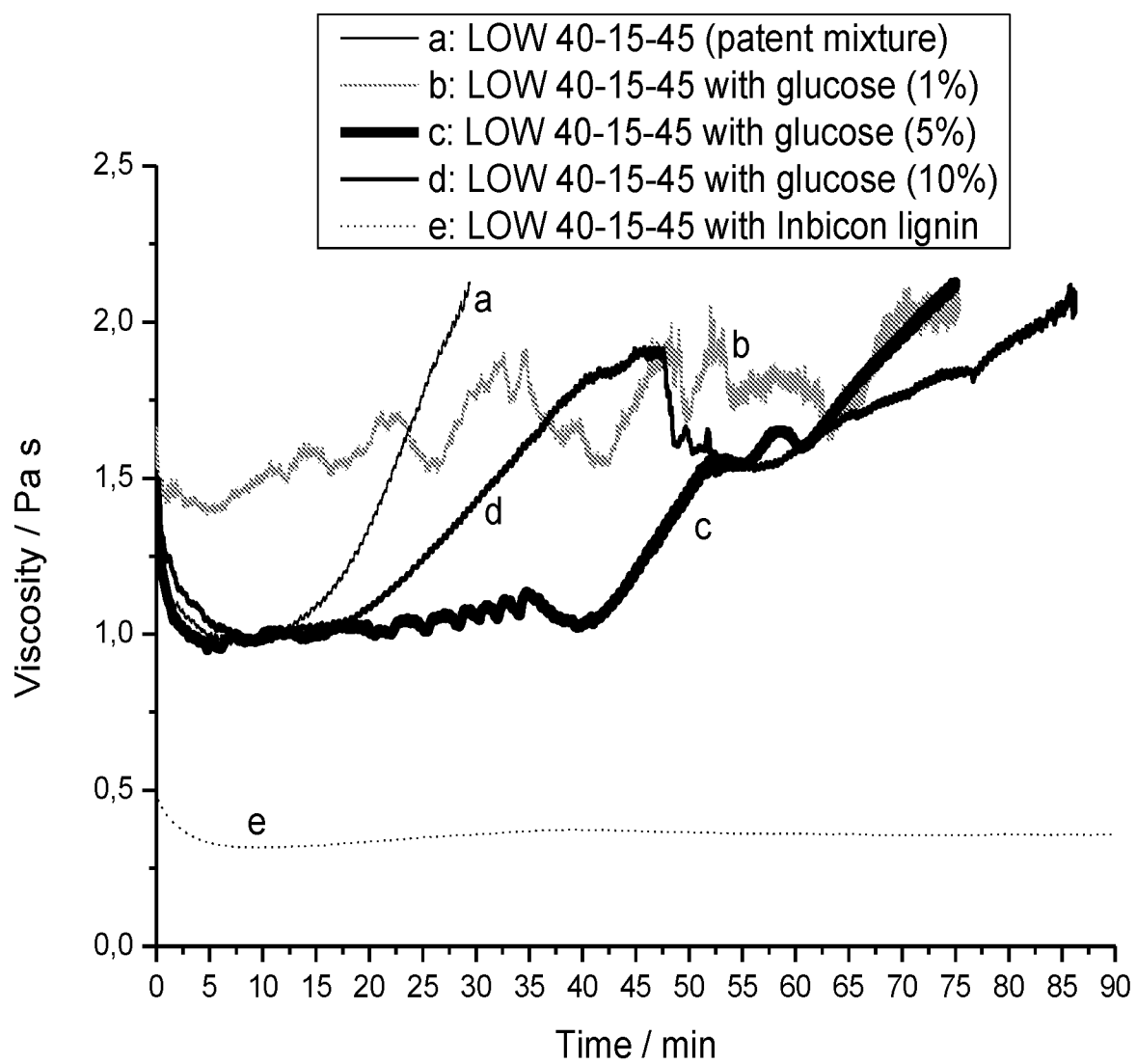


Figure 25

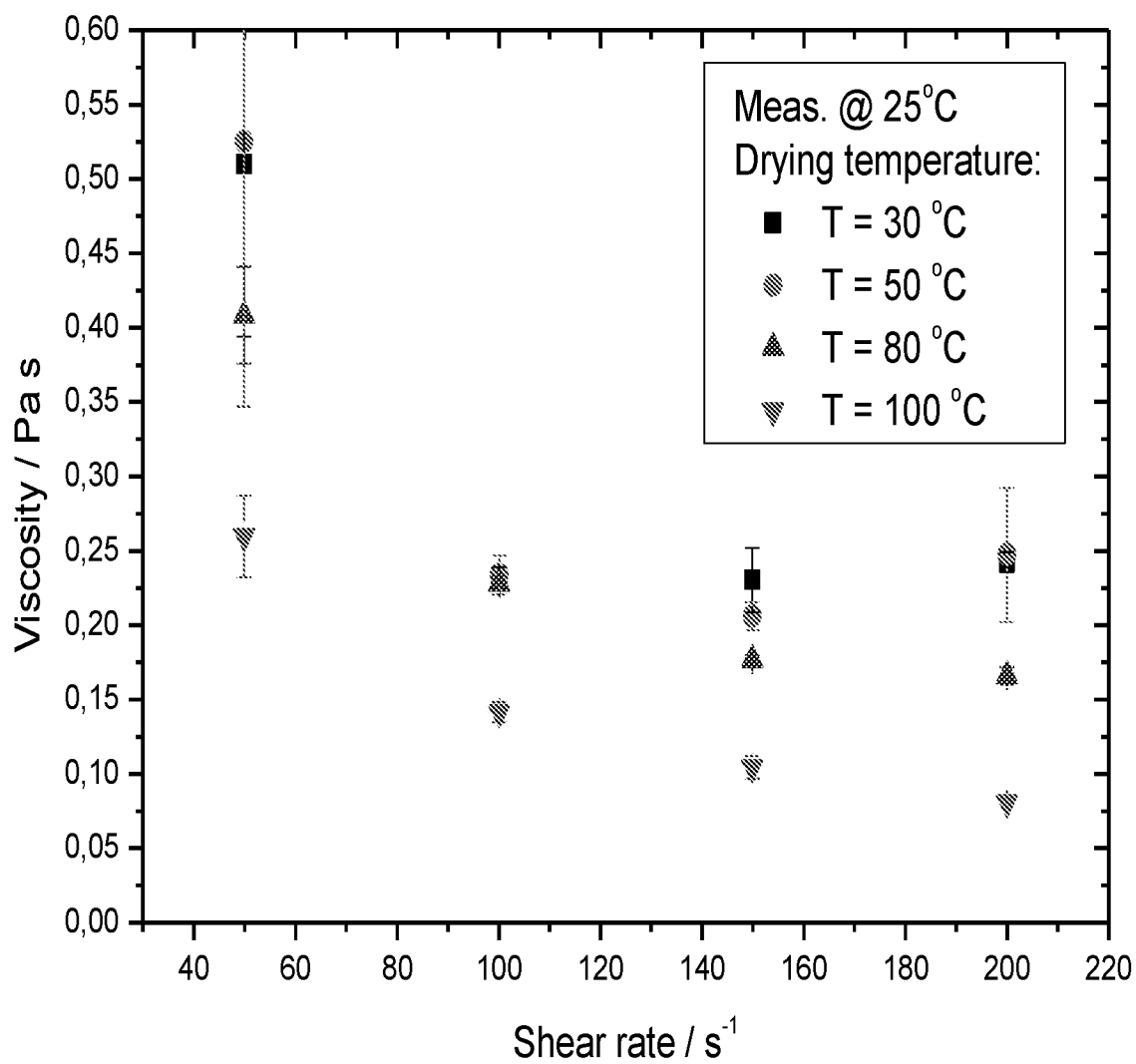


Figure 26a

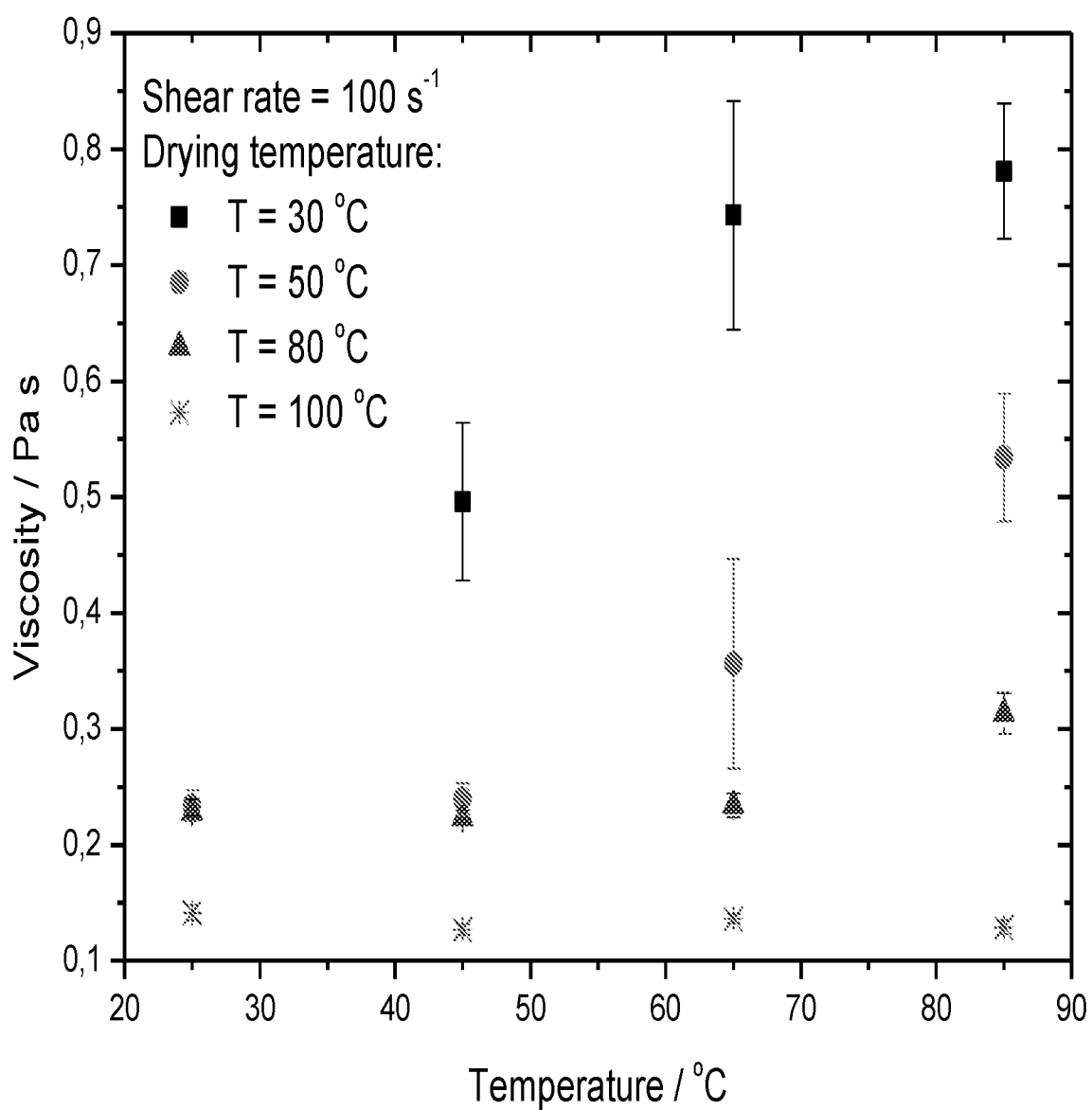


Figure 26b

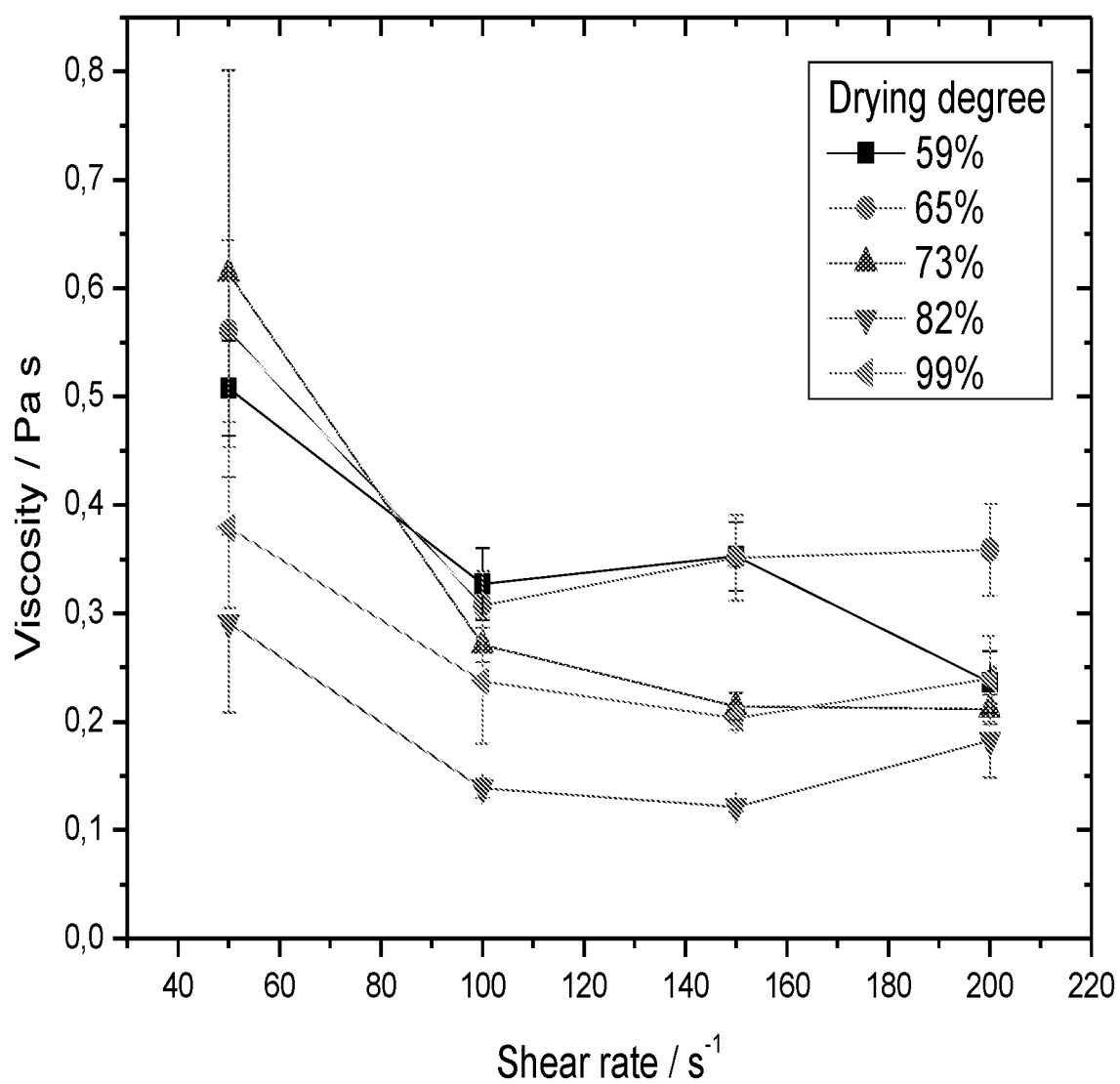


Figure 27a

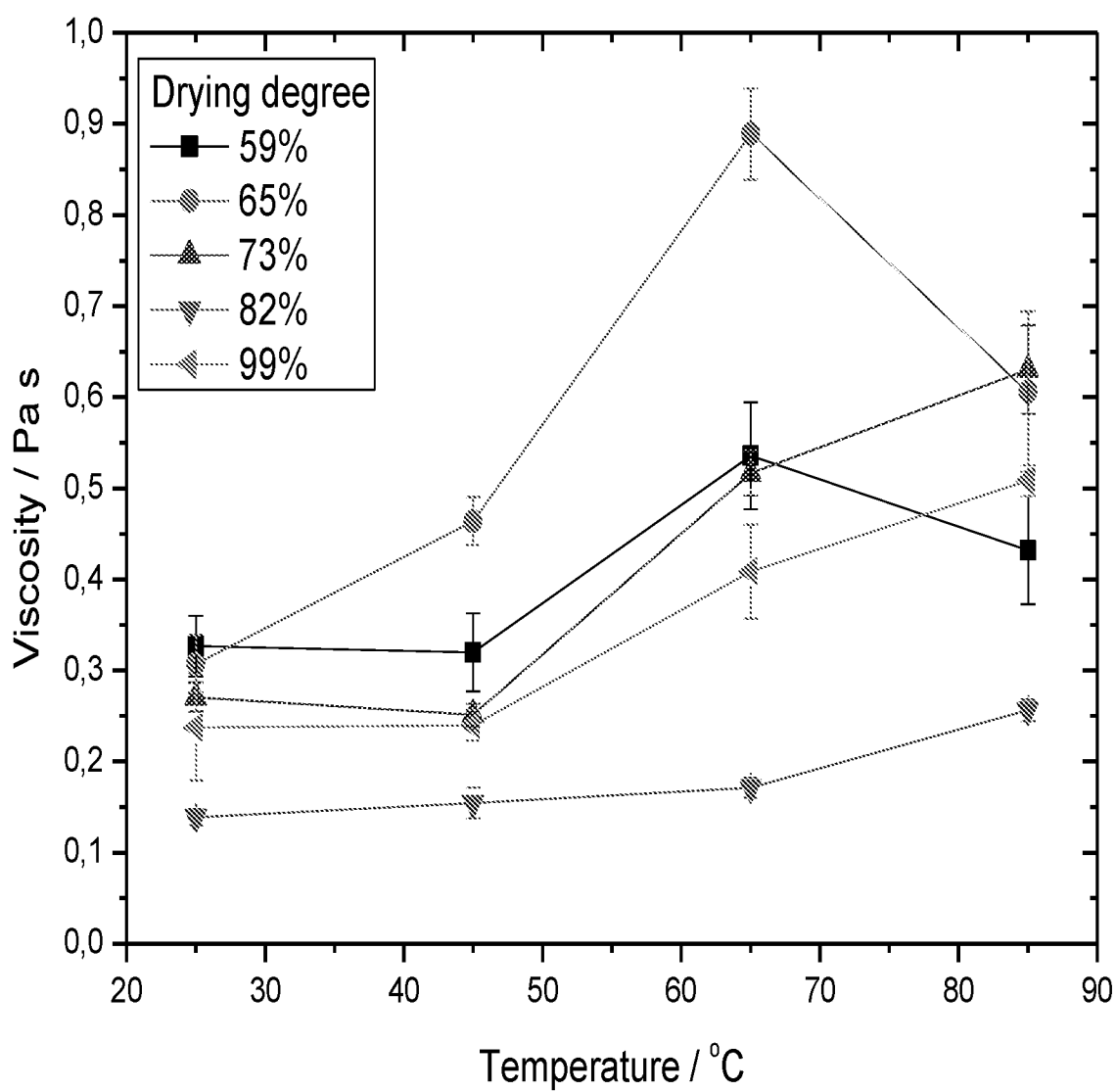


Figure 27b

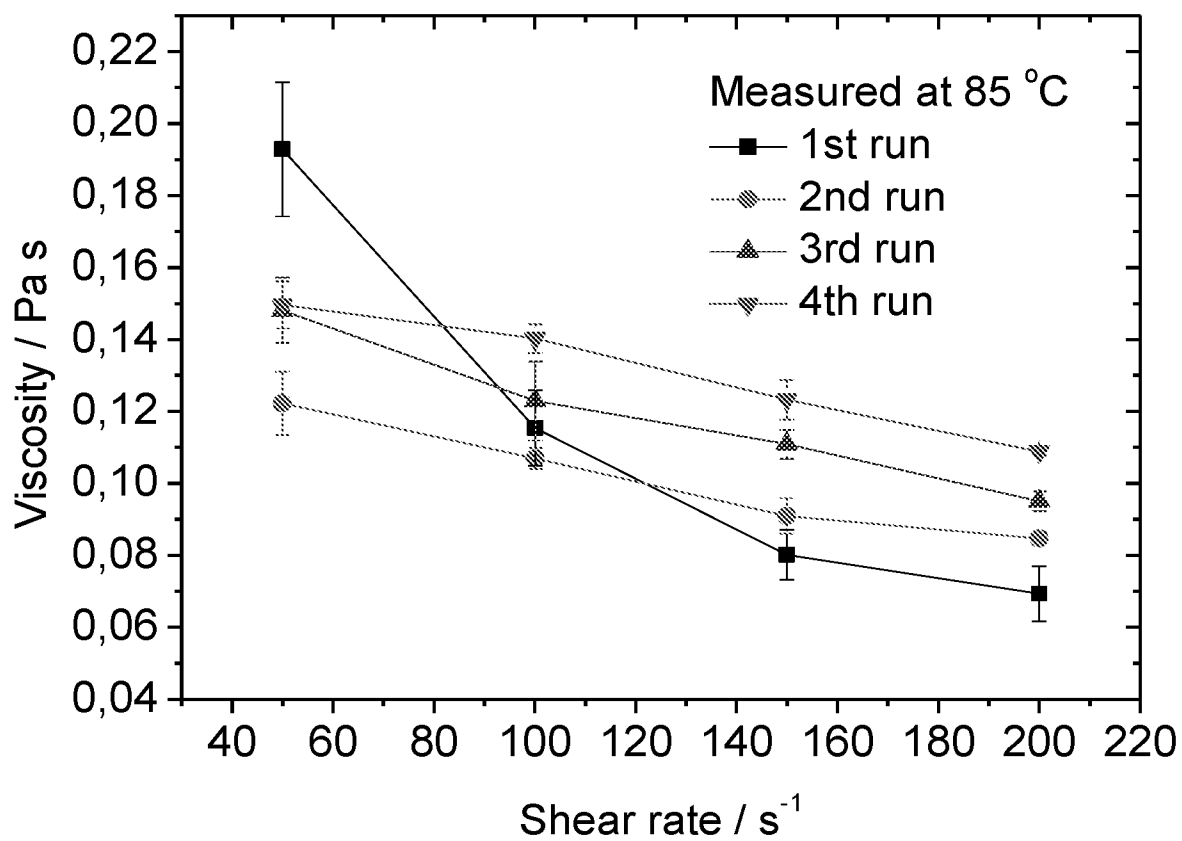


Figure 28

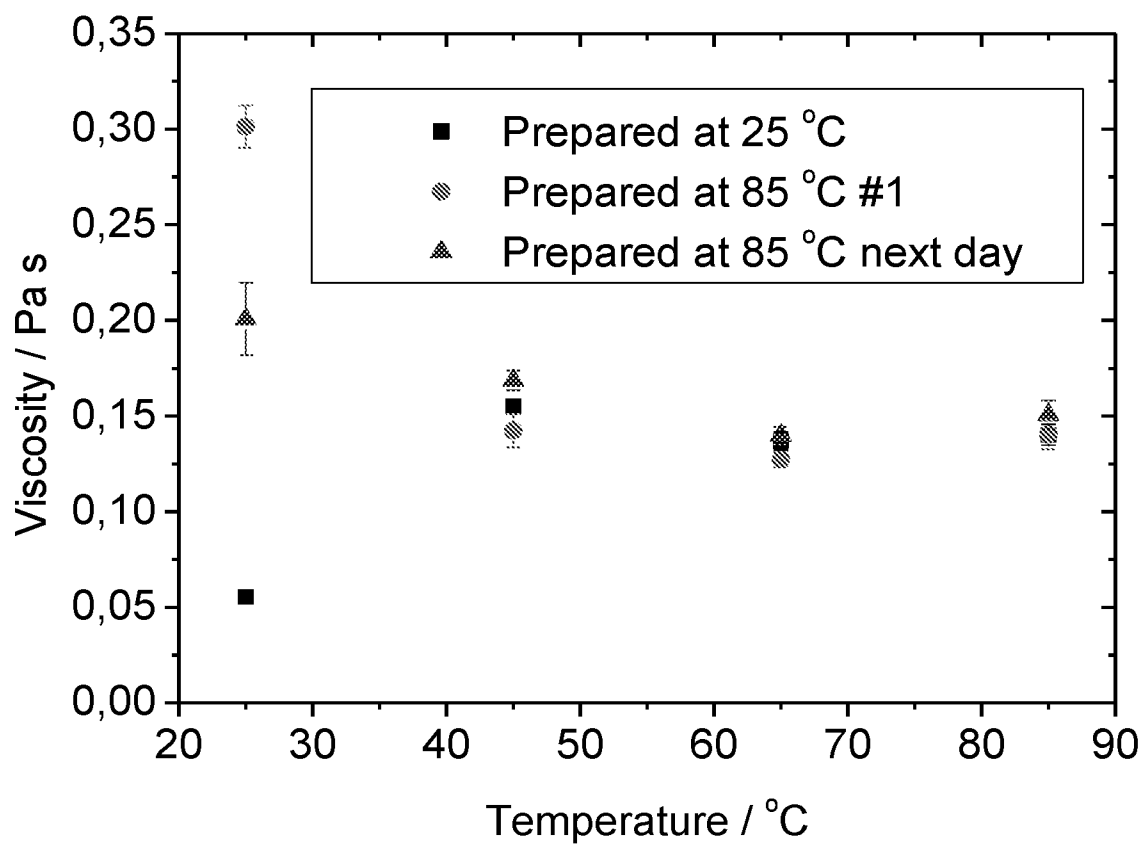


Figure 29

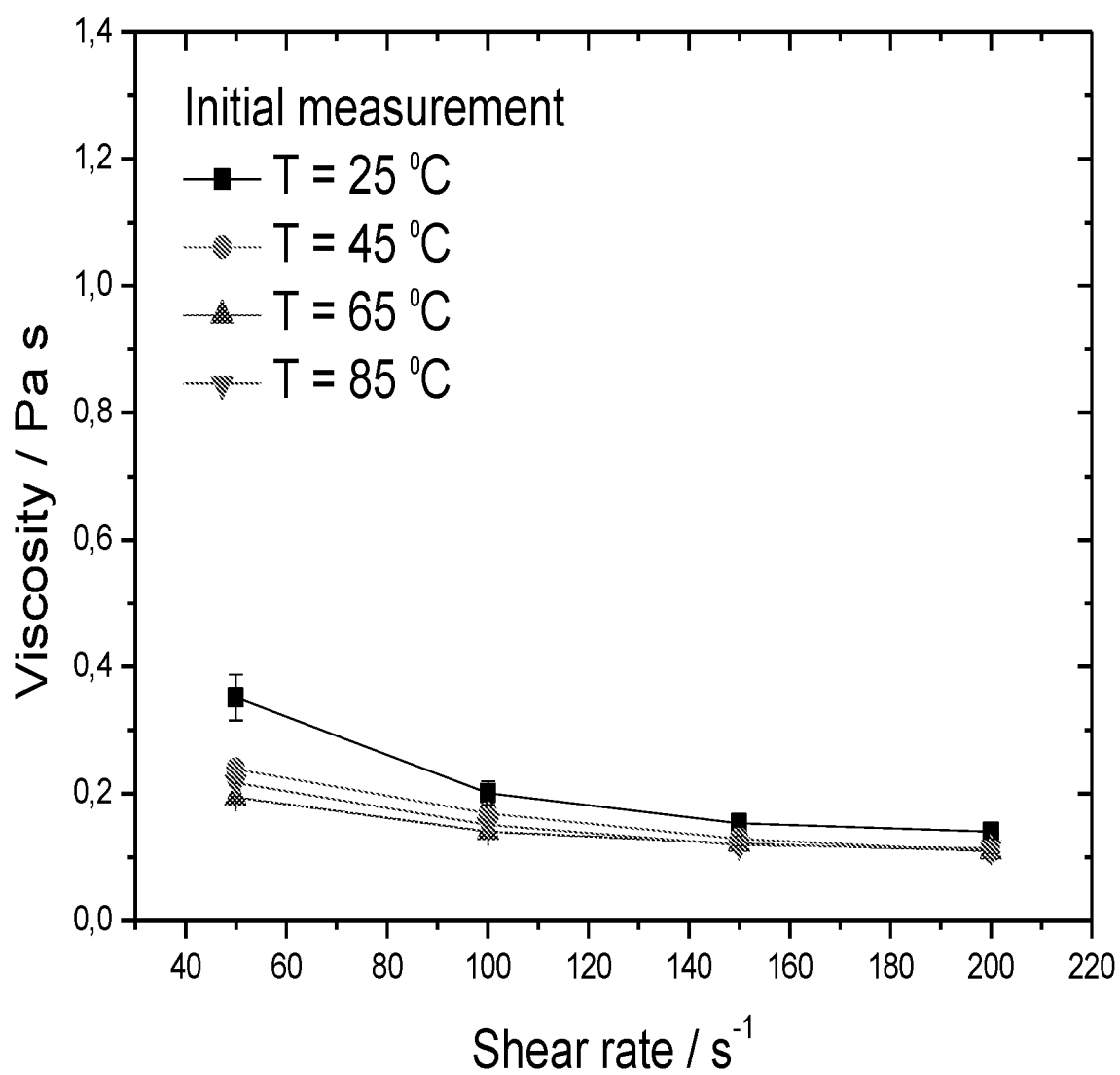


Figure 30a

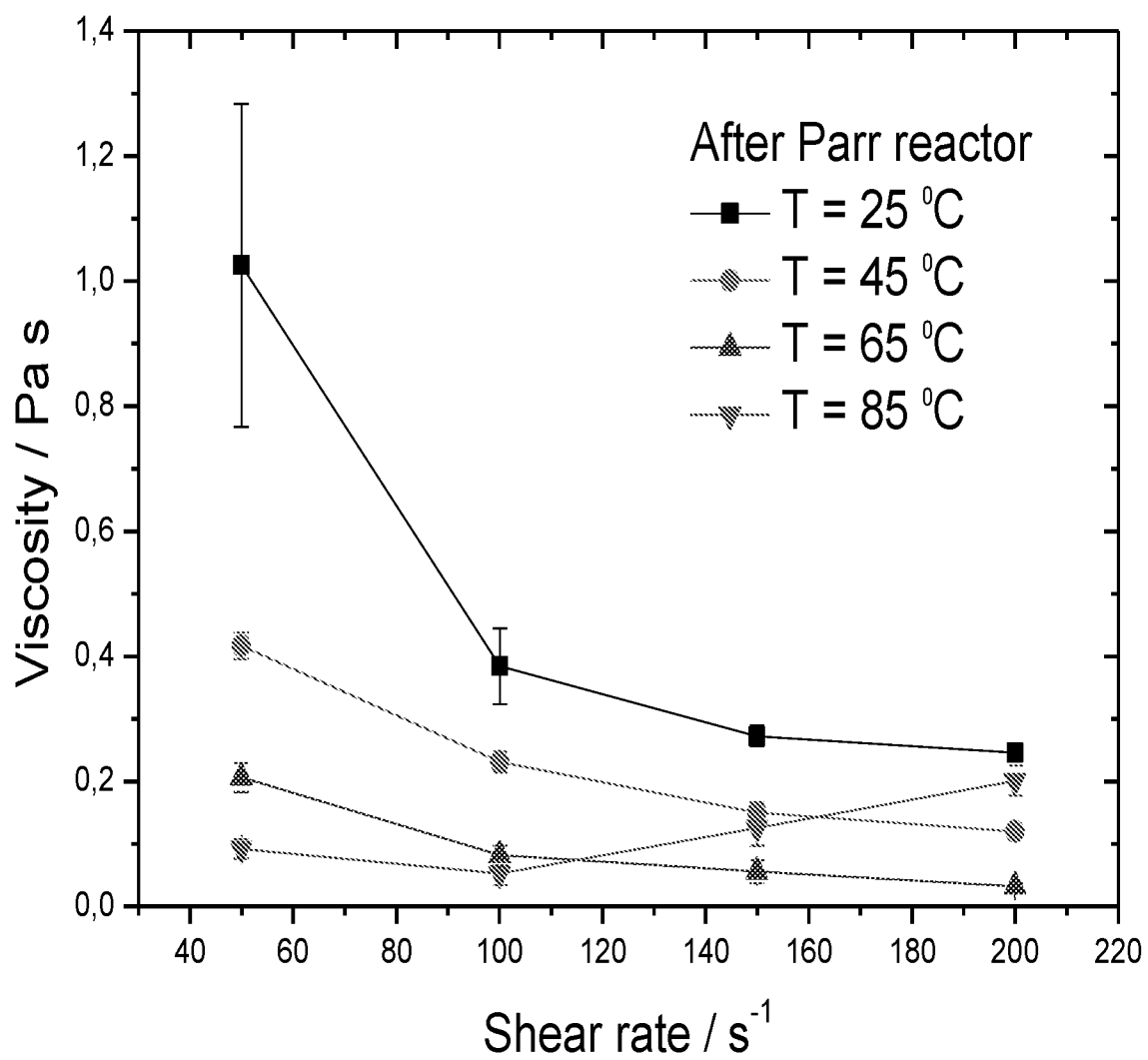


Figure 30b

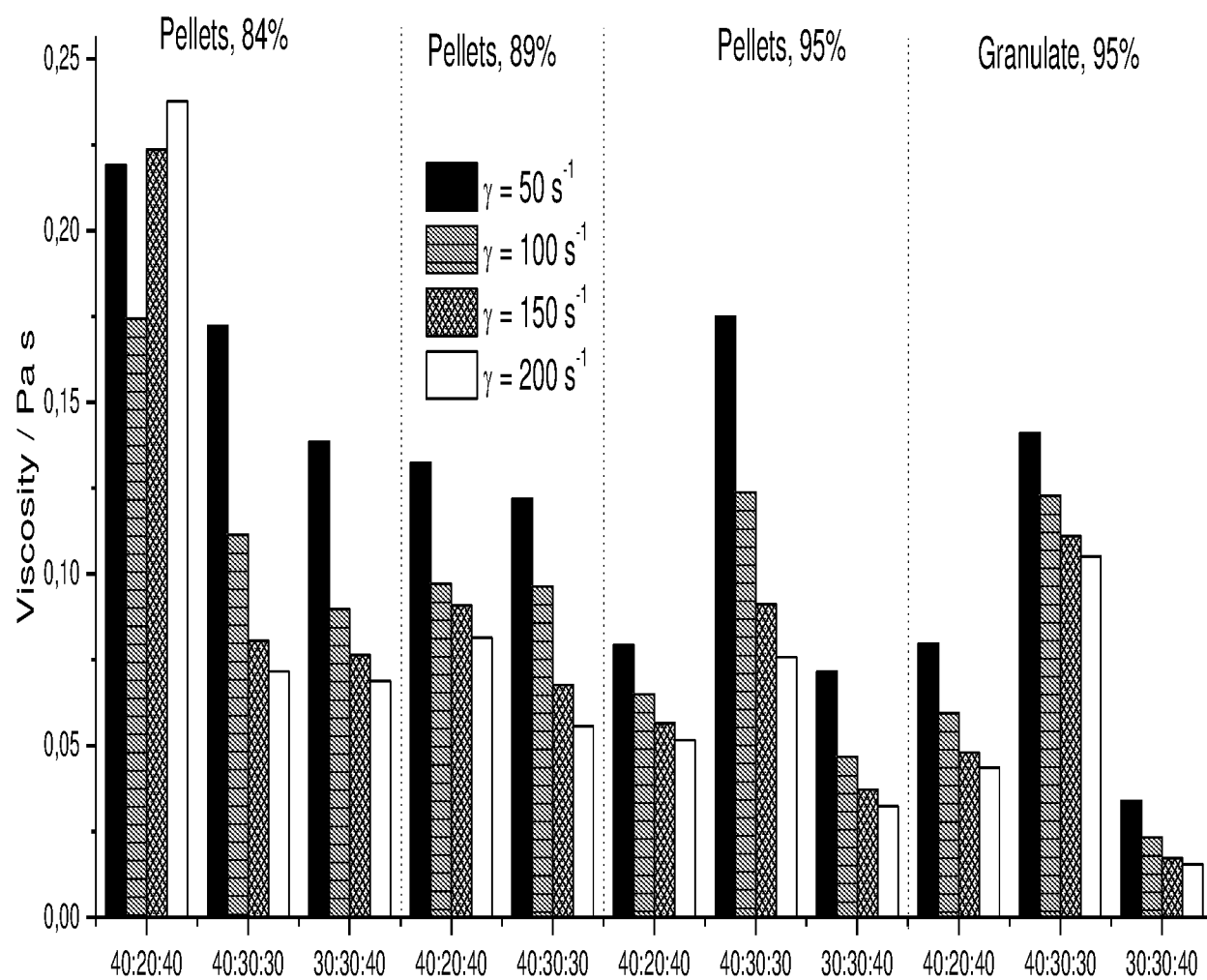


Figure 31a

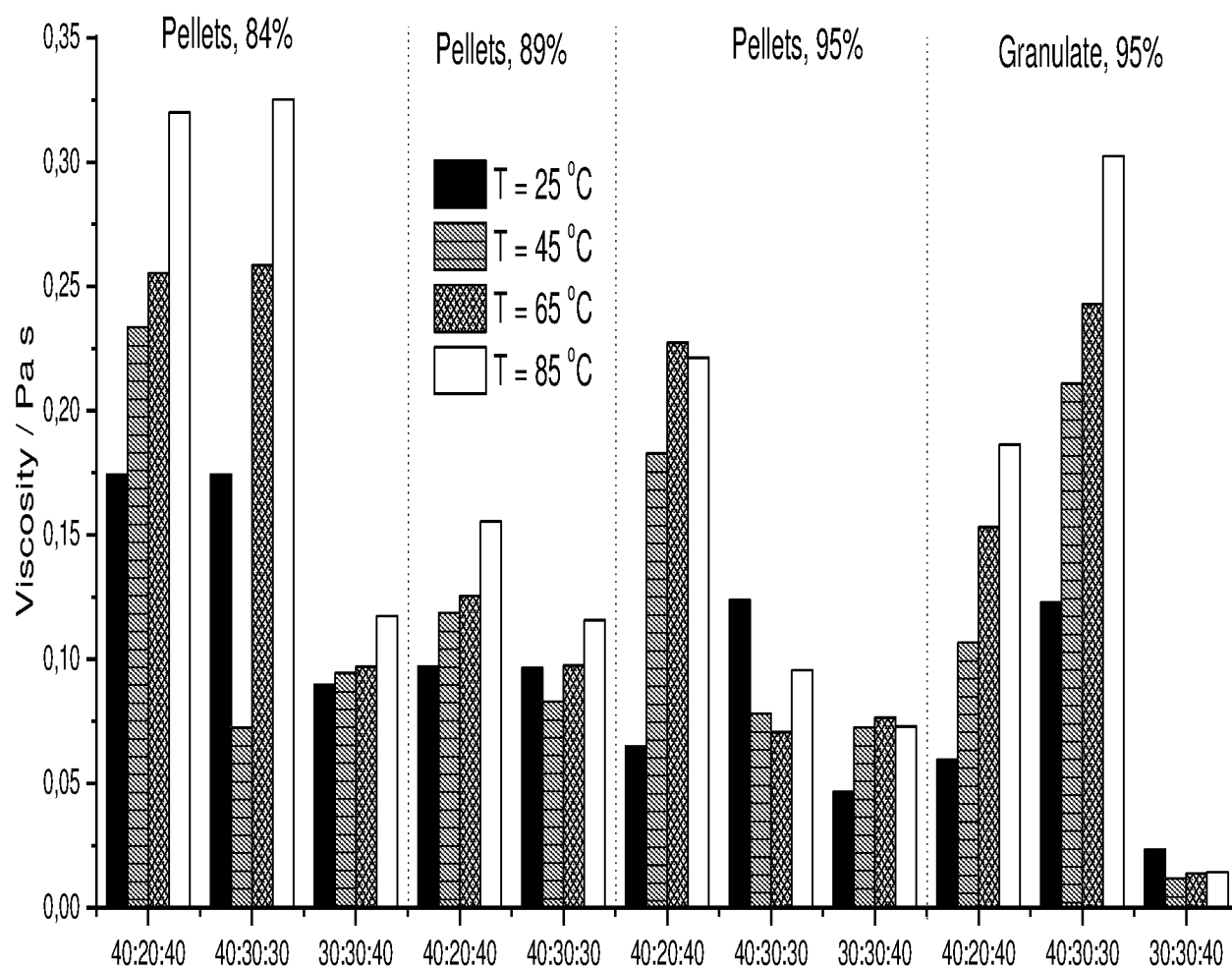


Figure 31b

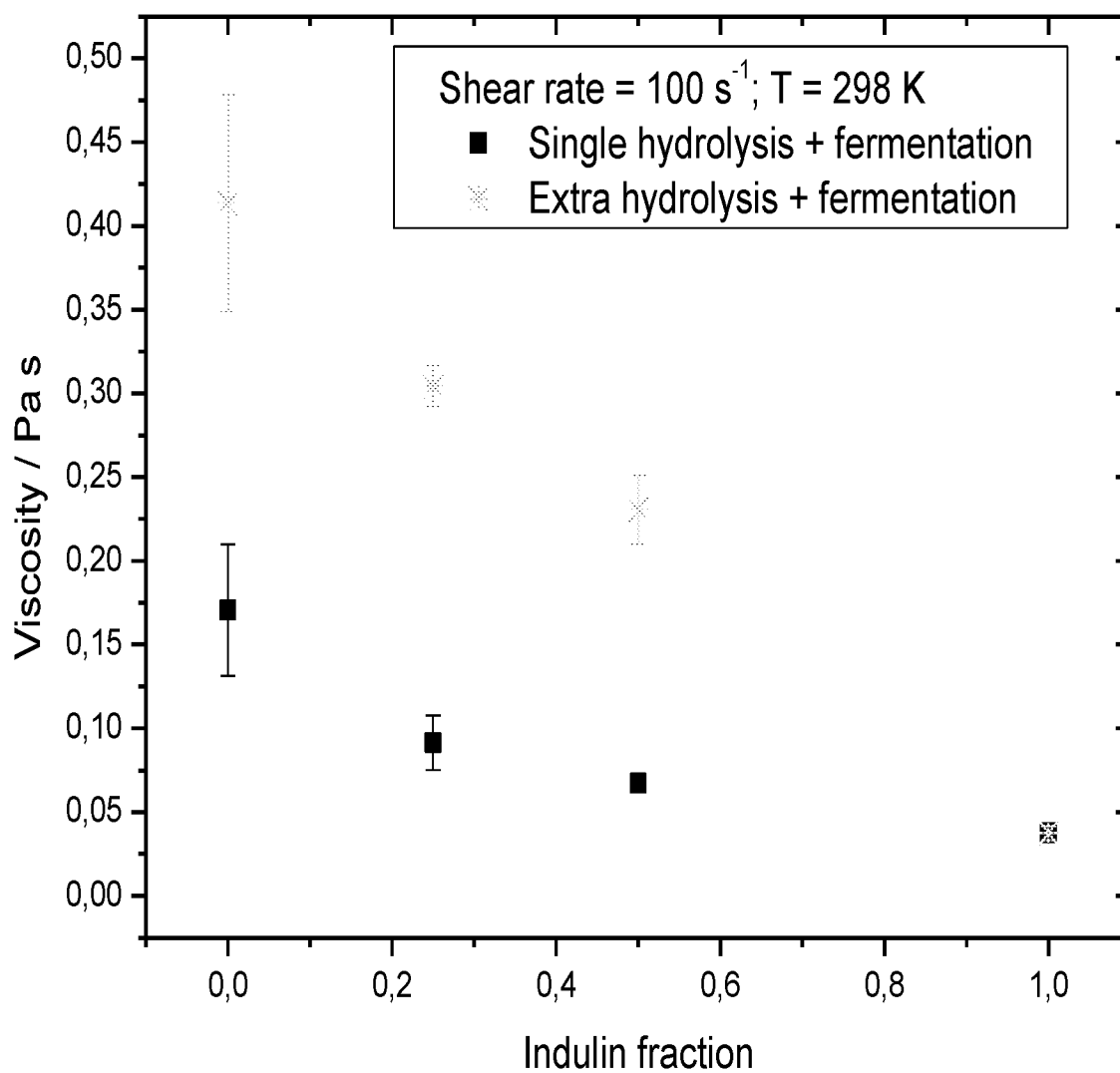


Figure 32

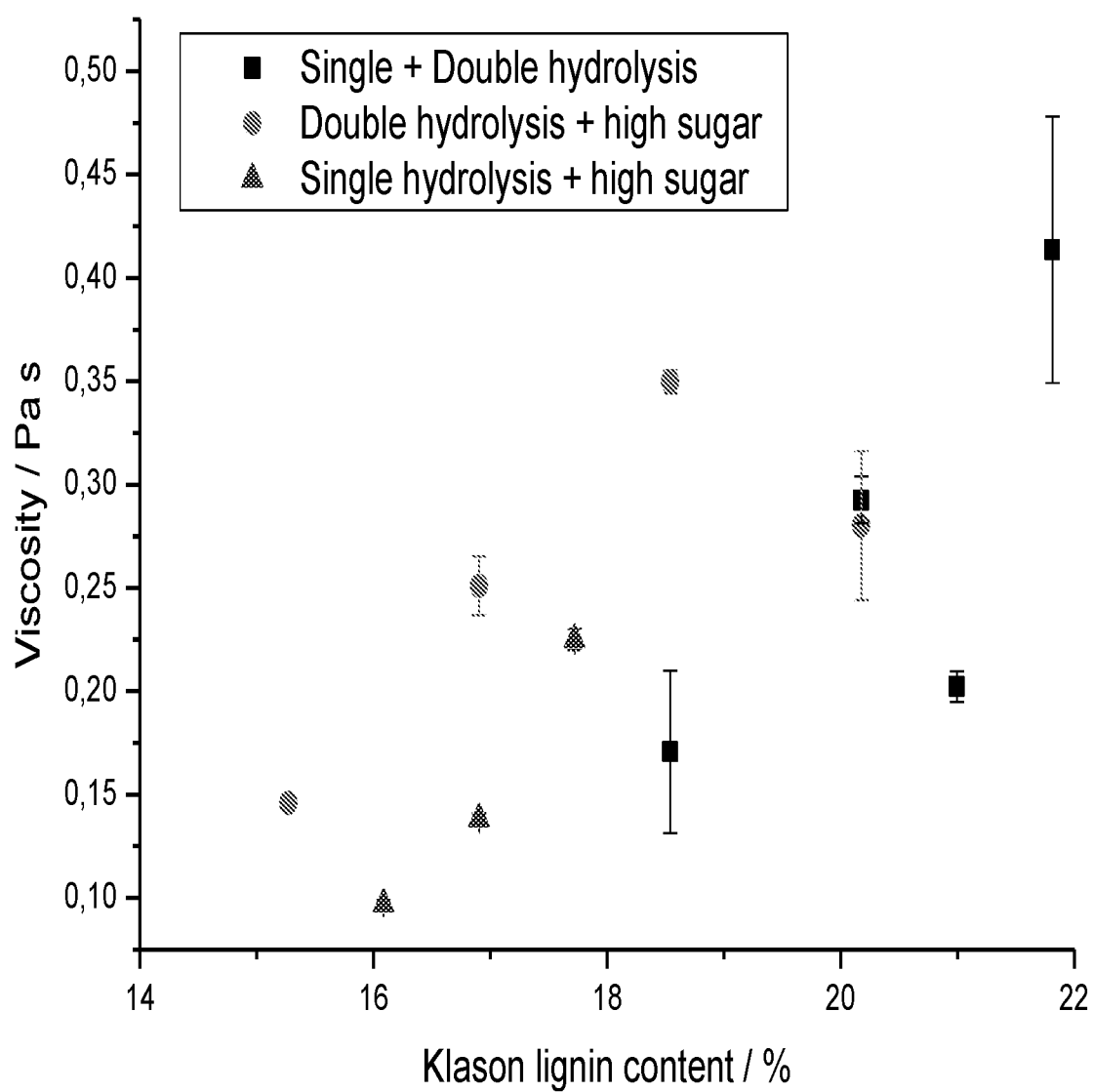


Figure 33a

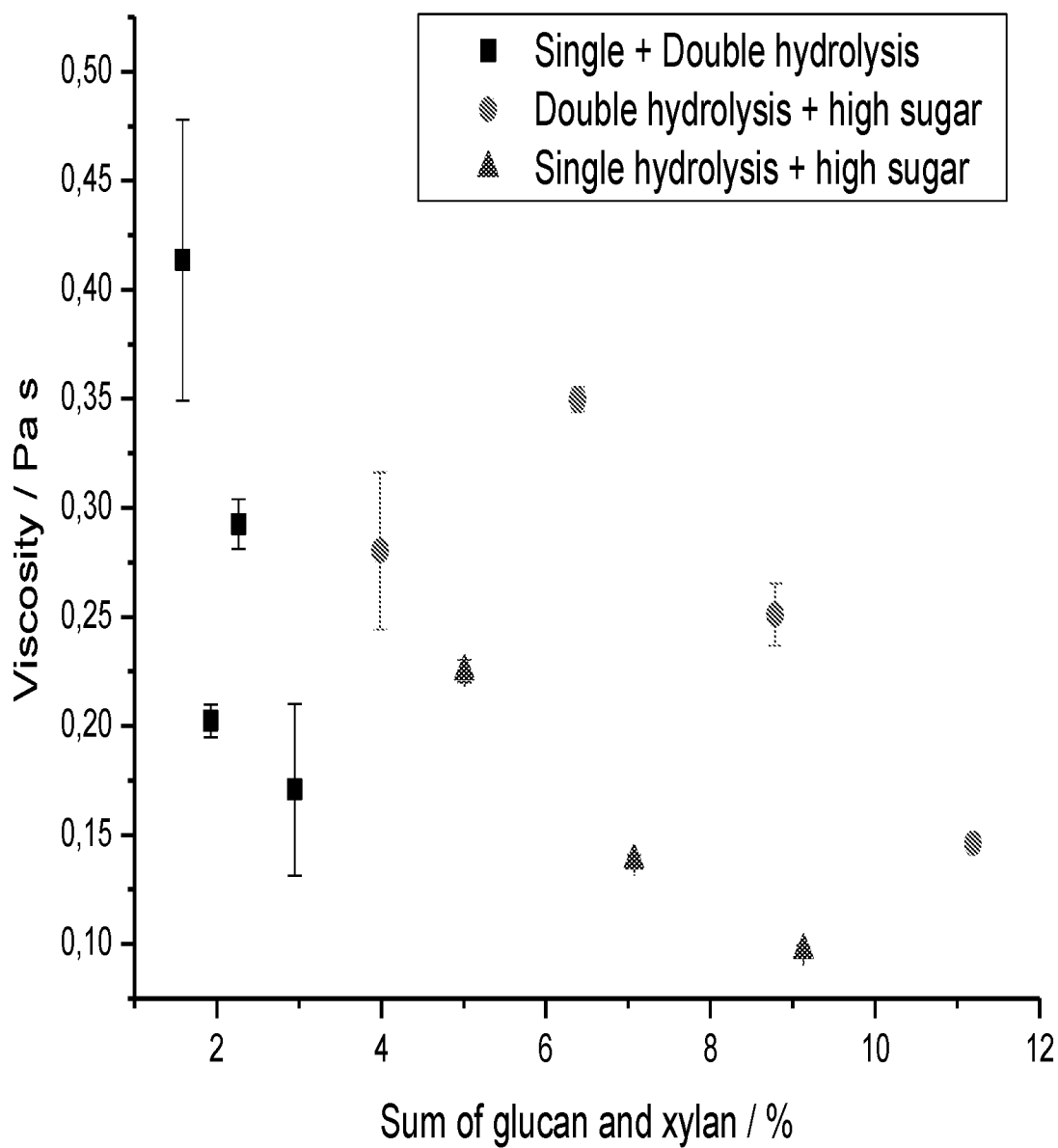


Figure 33b

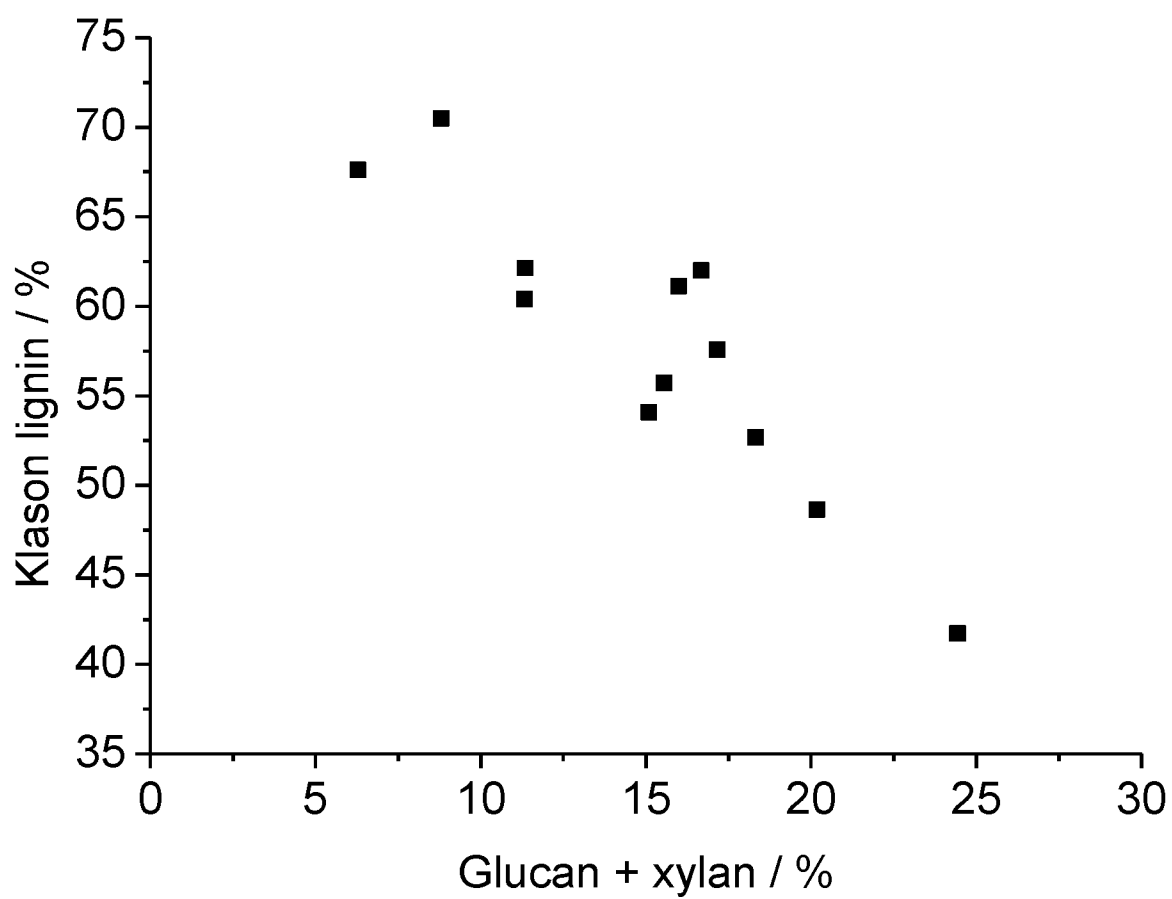


Figure 34

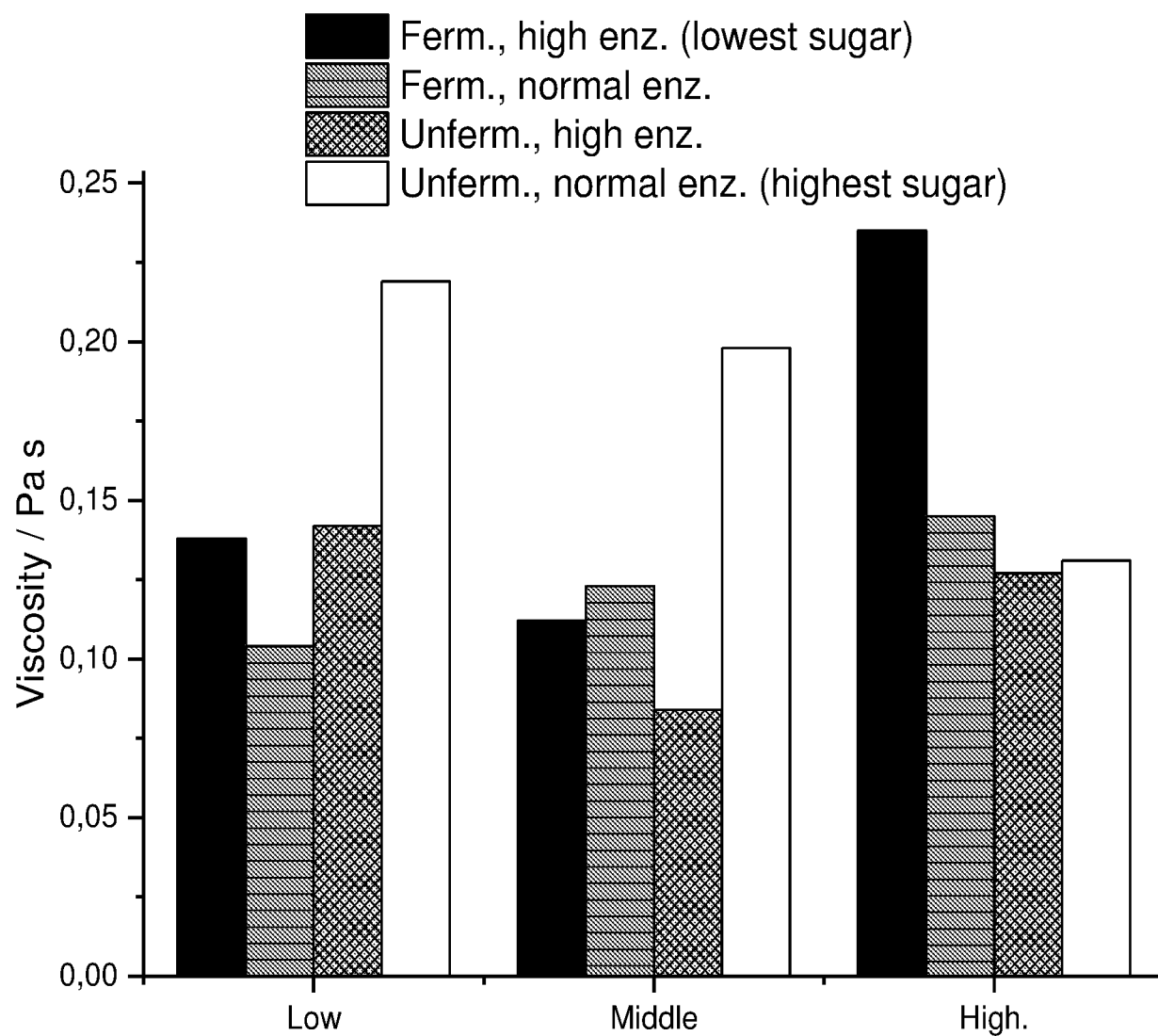
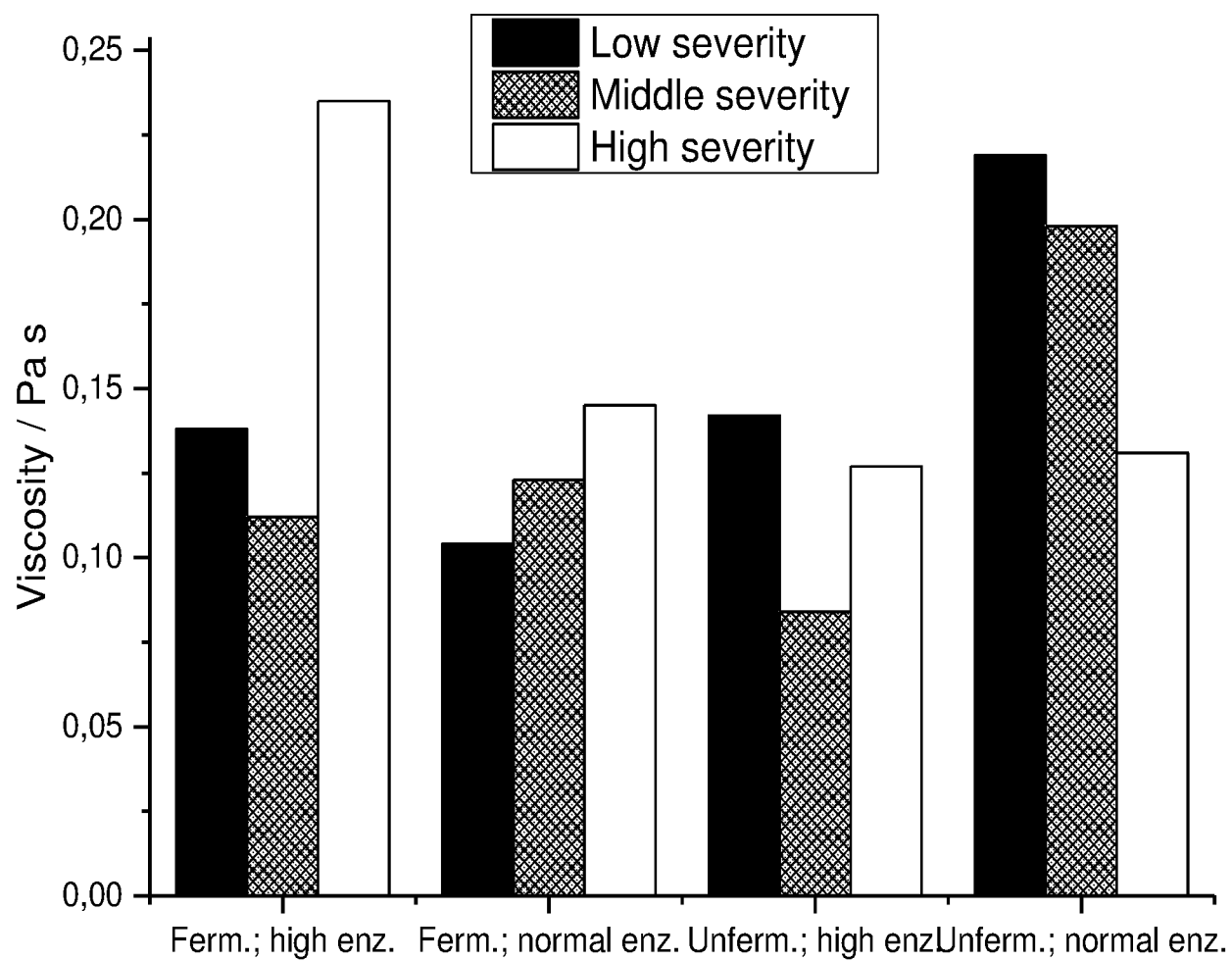


Figure 35a

**Figure 35b**

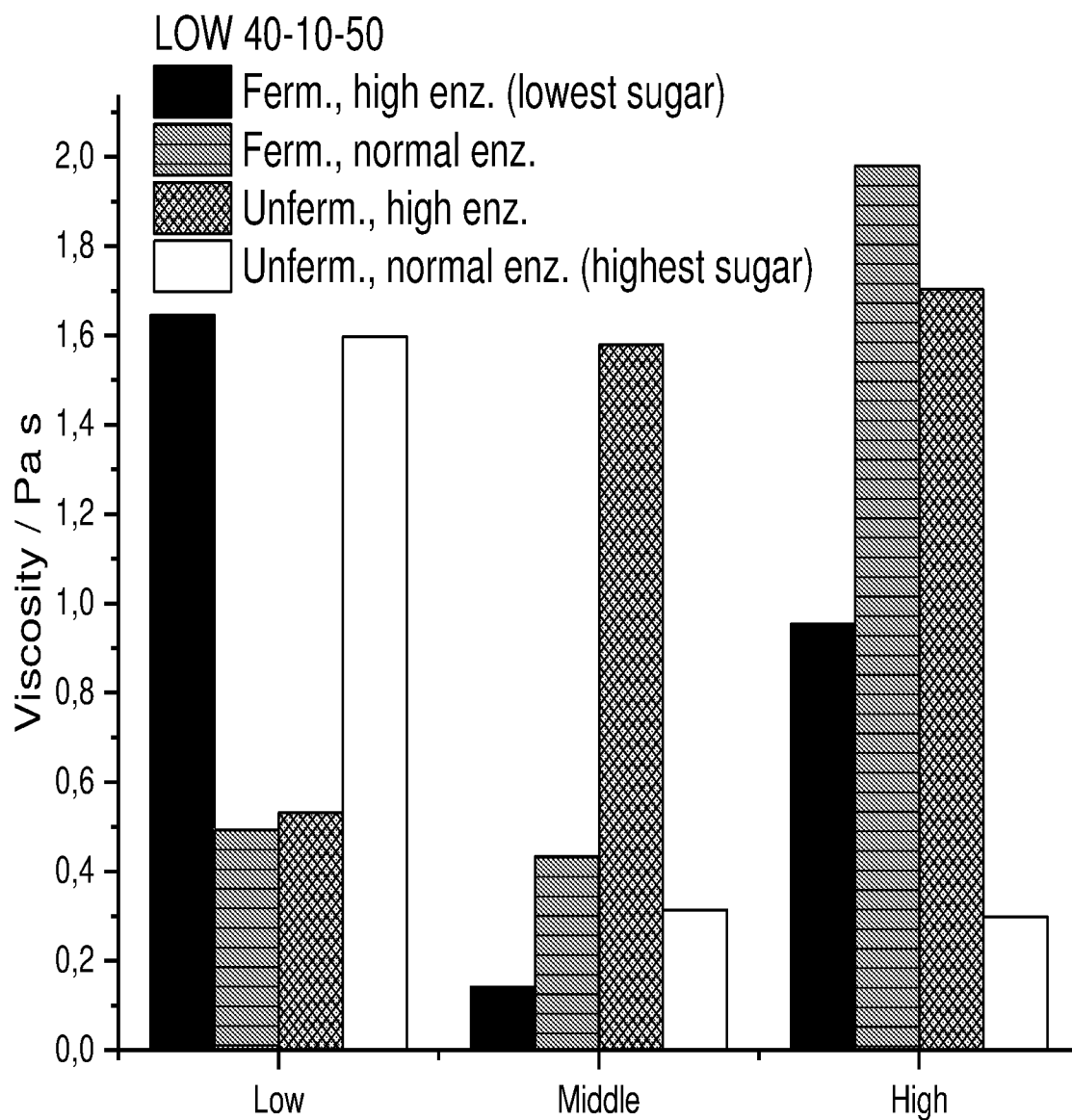


Figure 35c

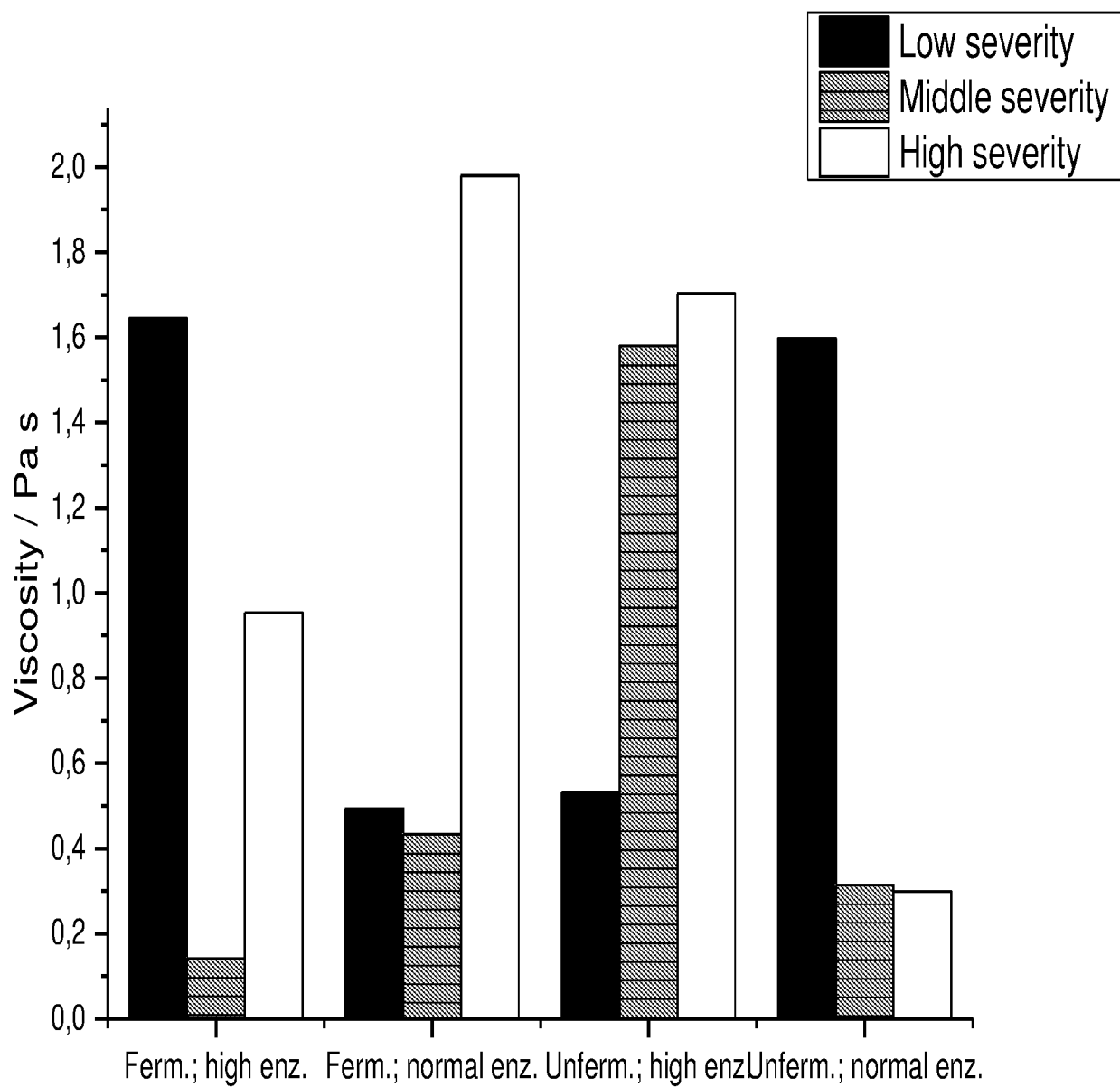


Figure 35d

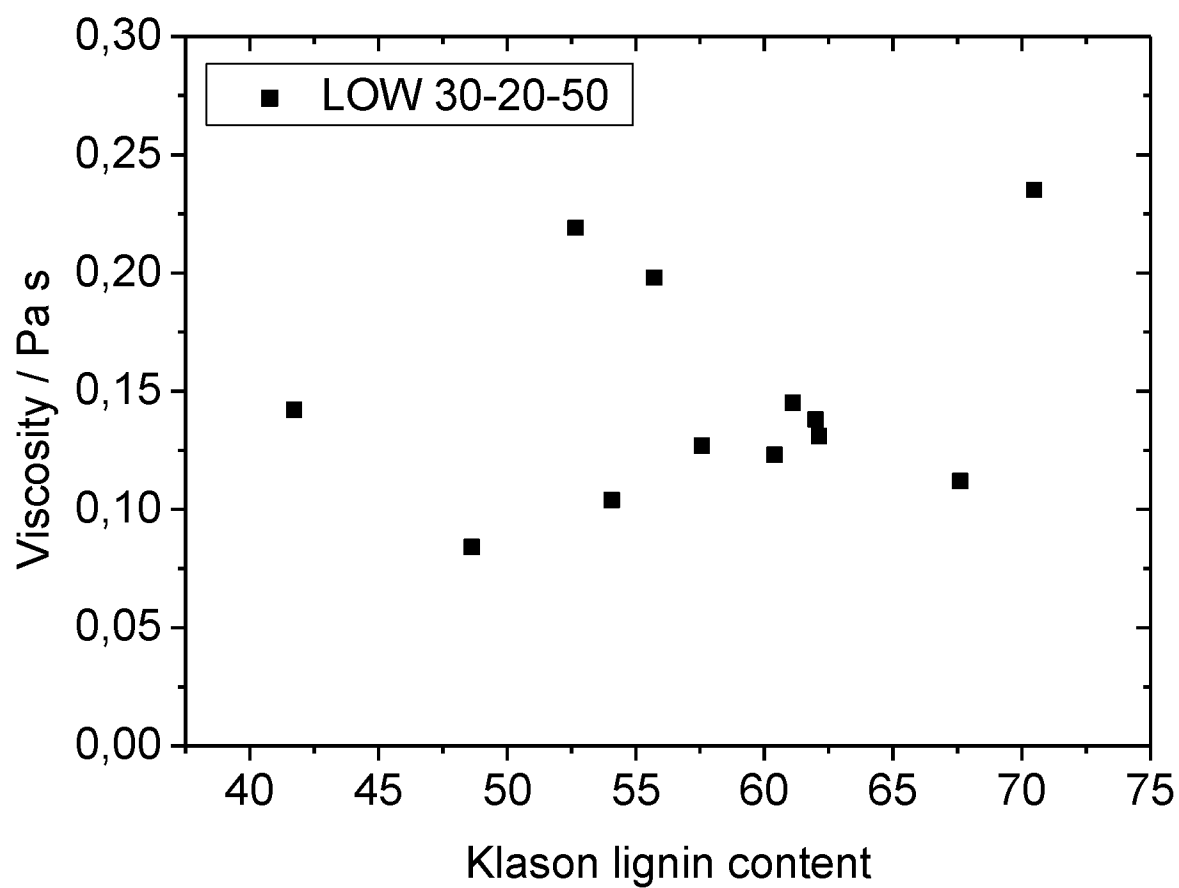


Figure 36a

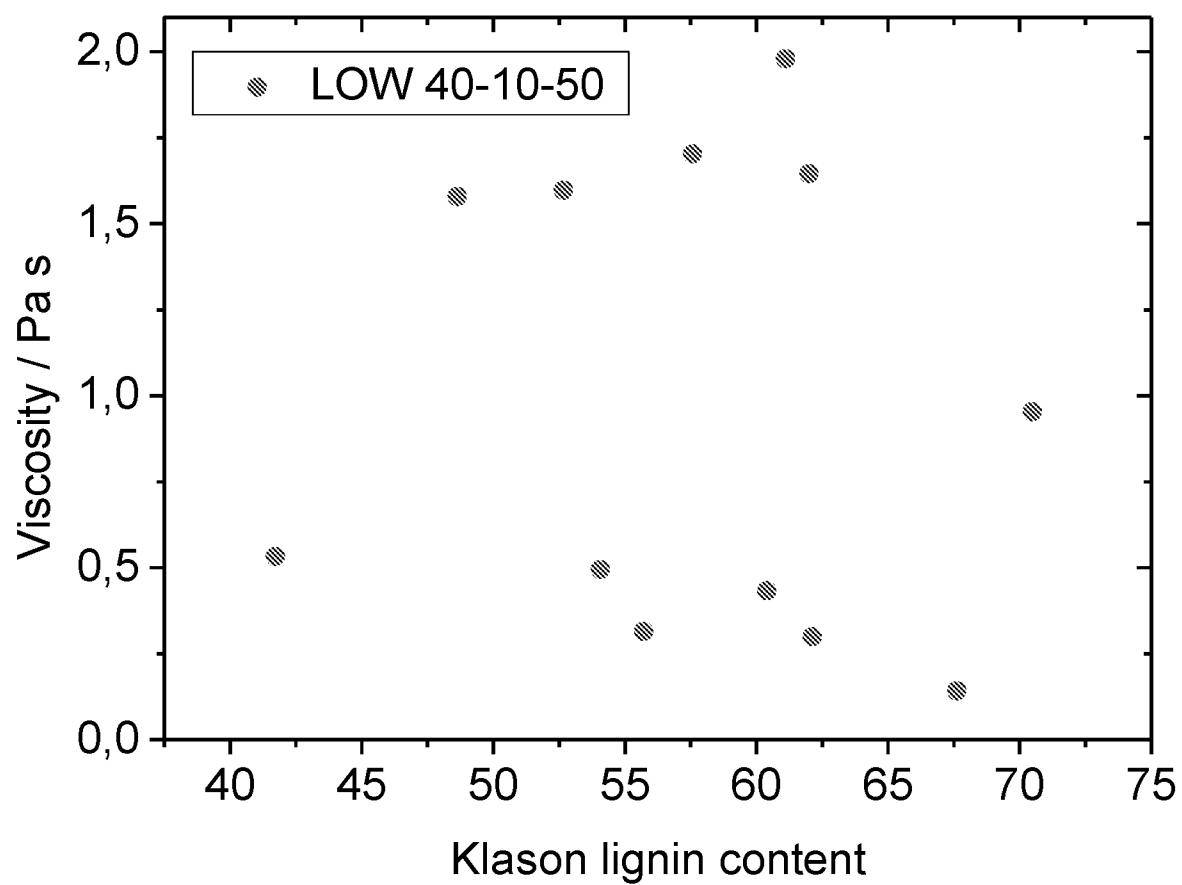


Figure 36b

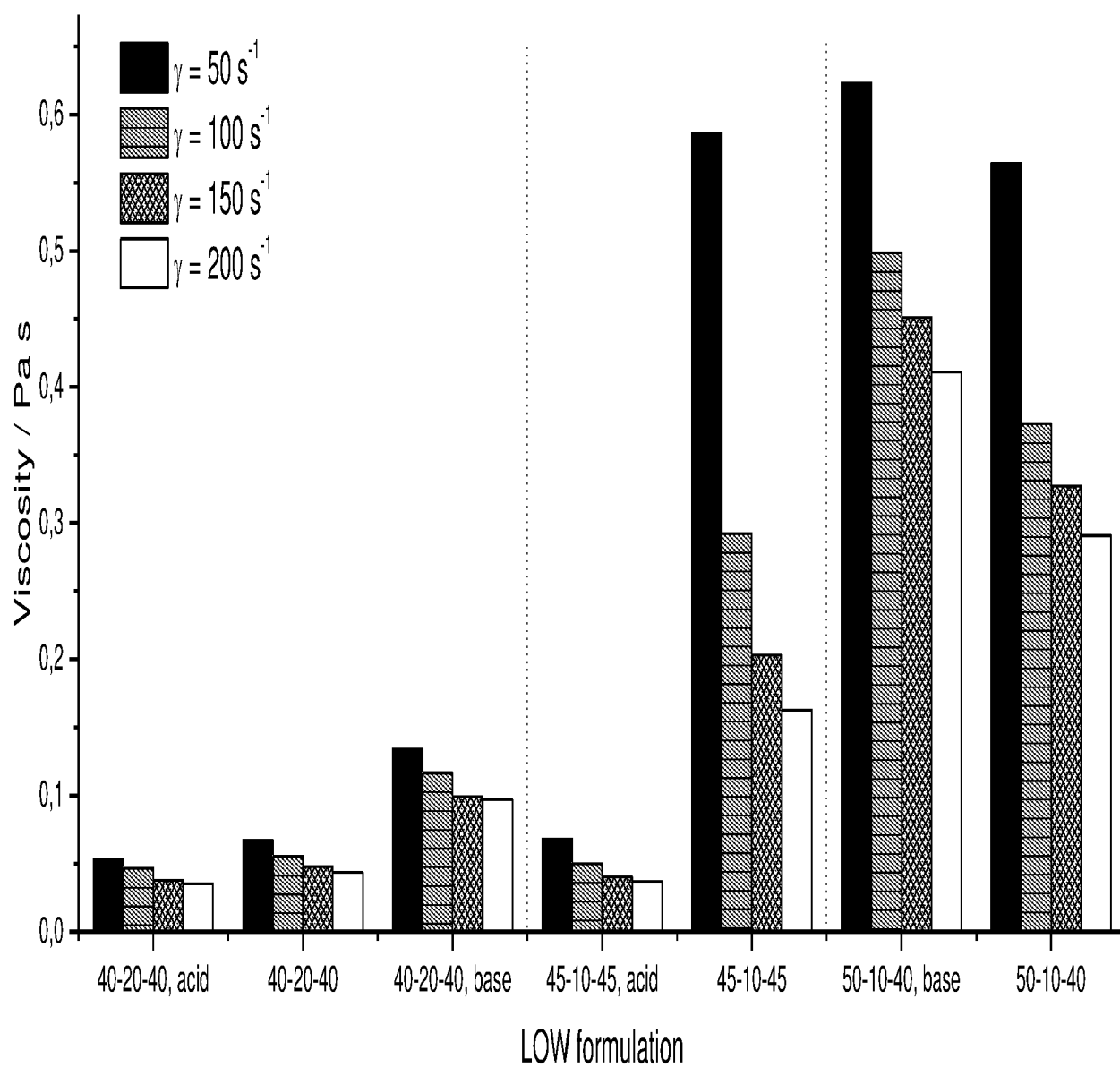


Figure 37

A)

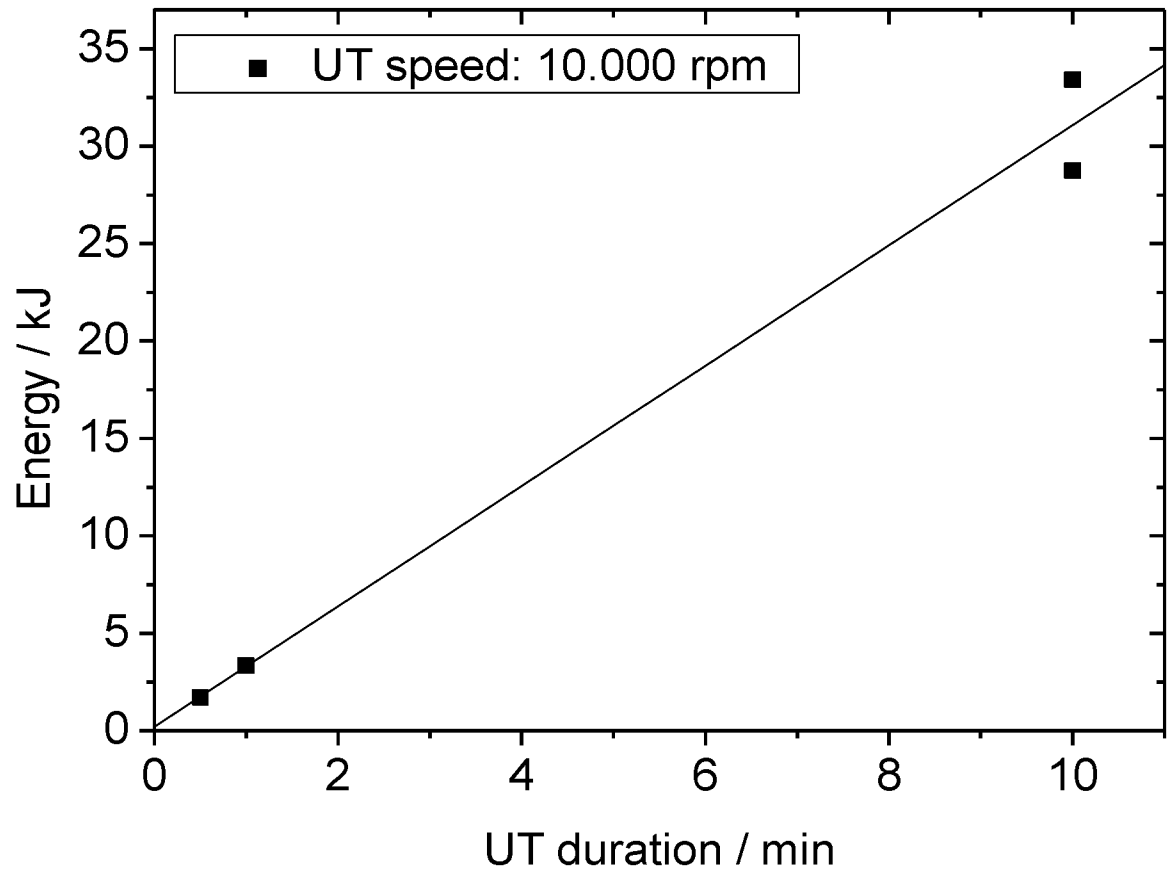


Figure 38a

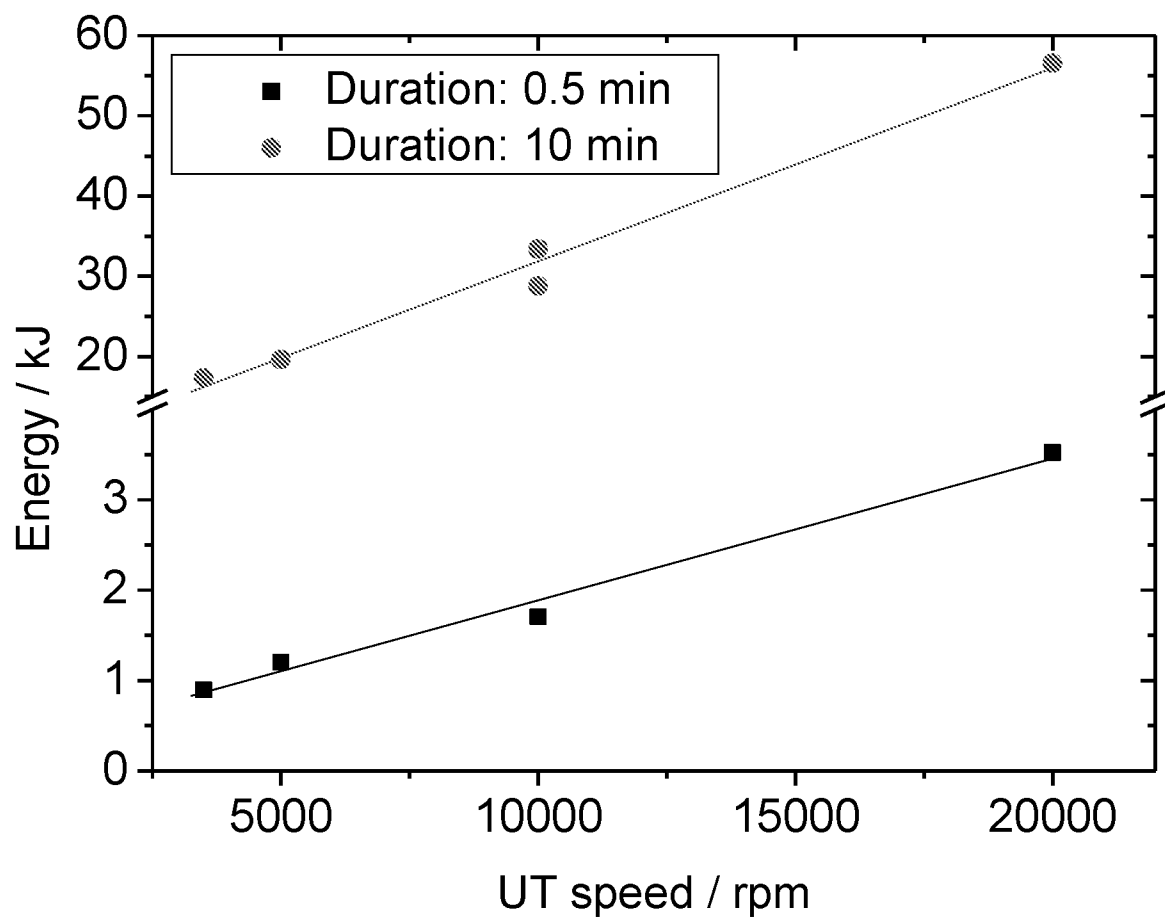


Figure 38b

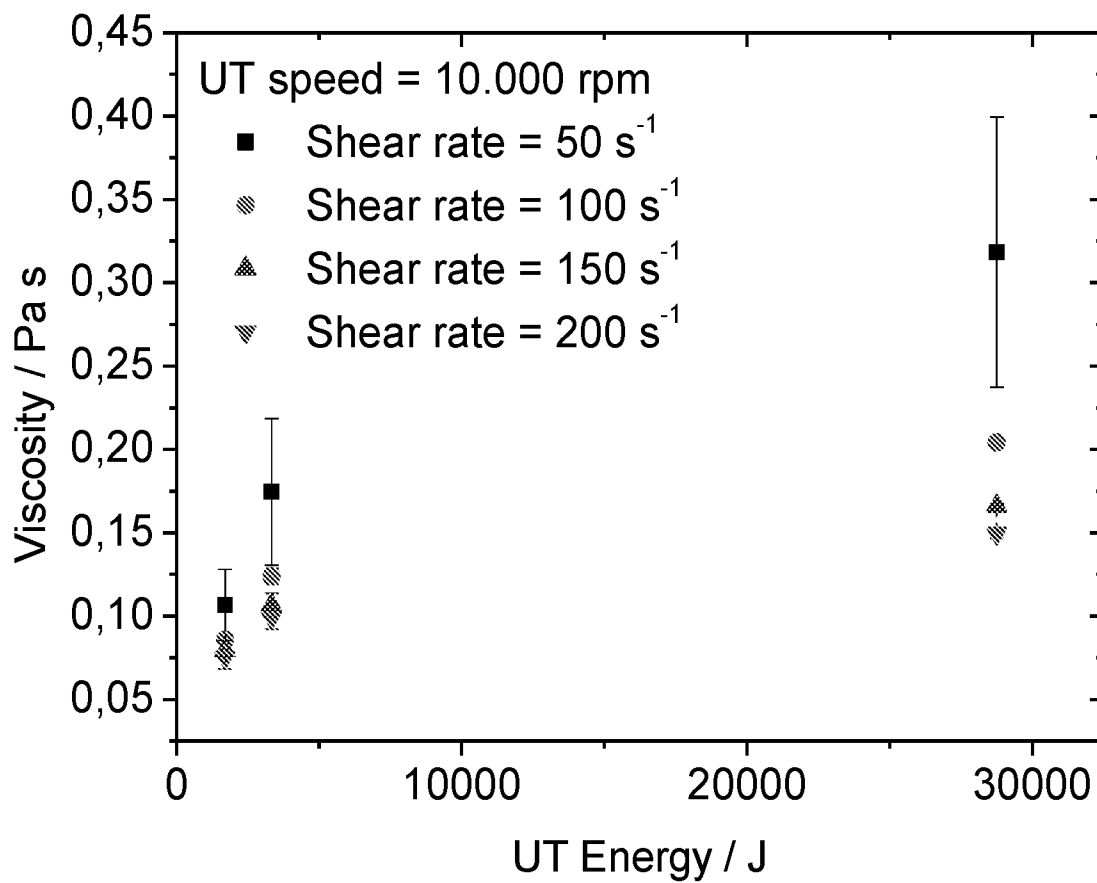


Figure 39

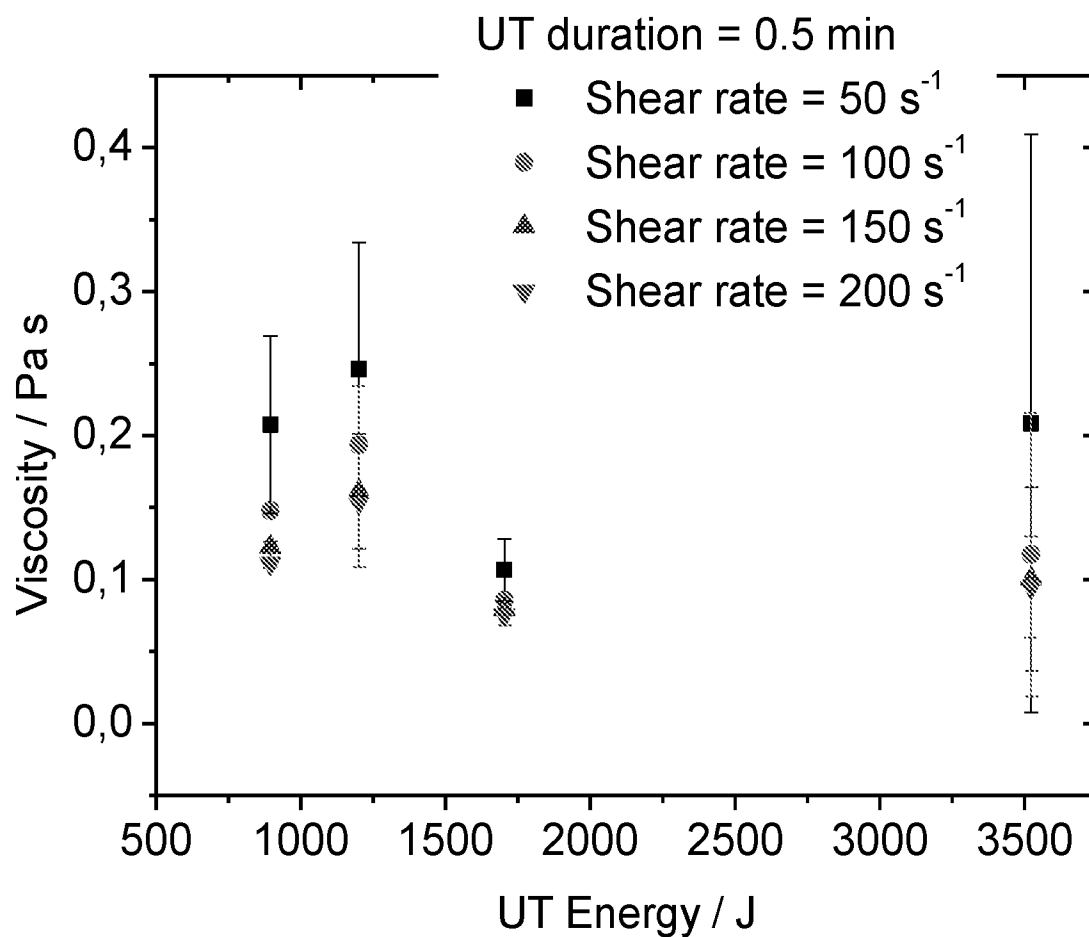


Figure 40a

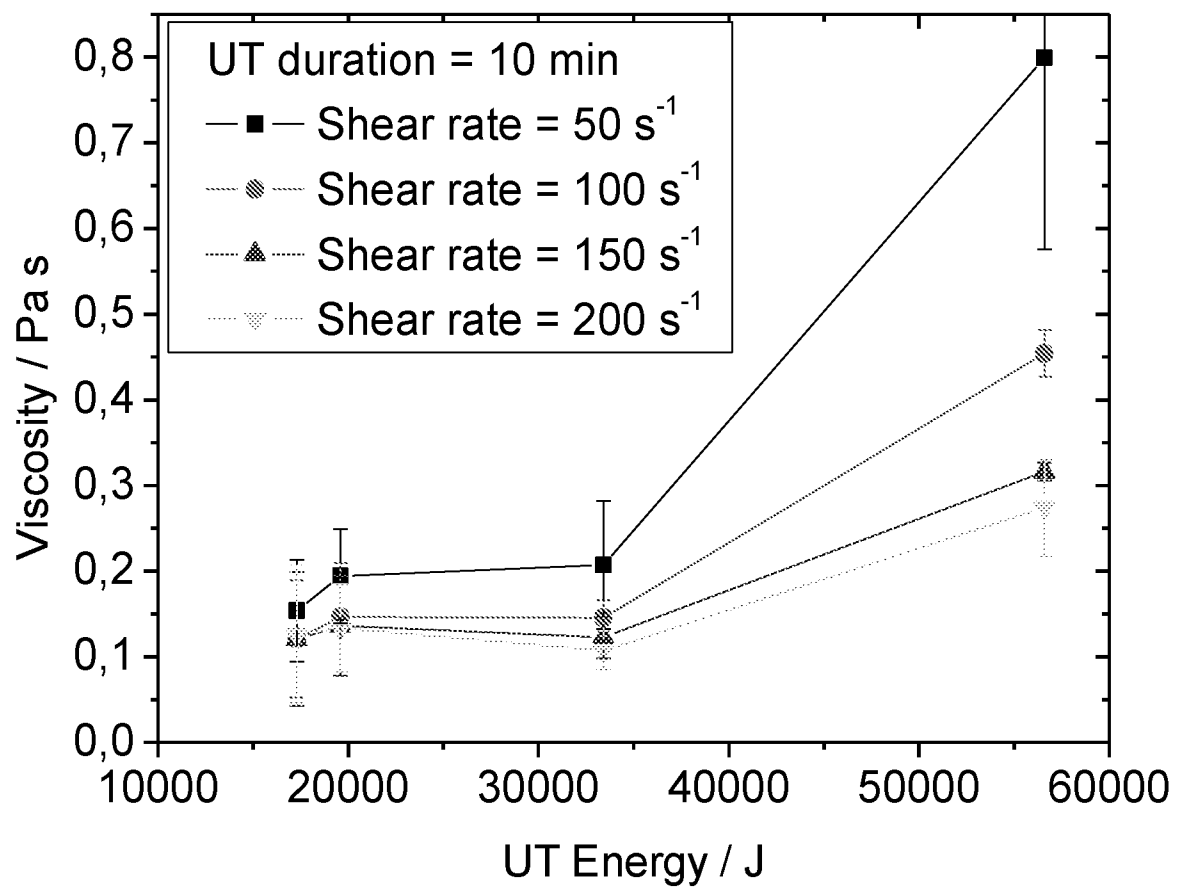


Figure 40b

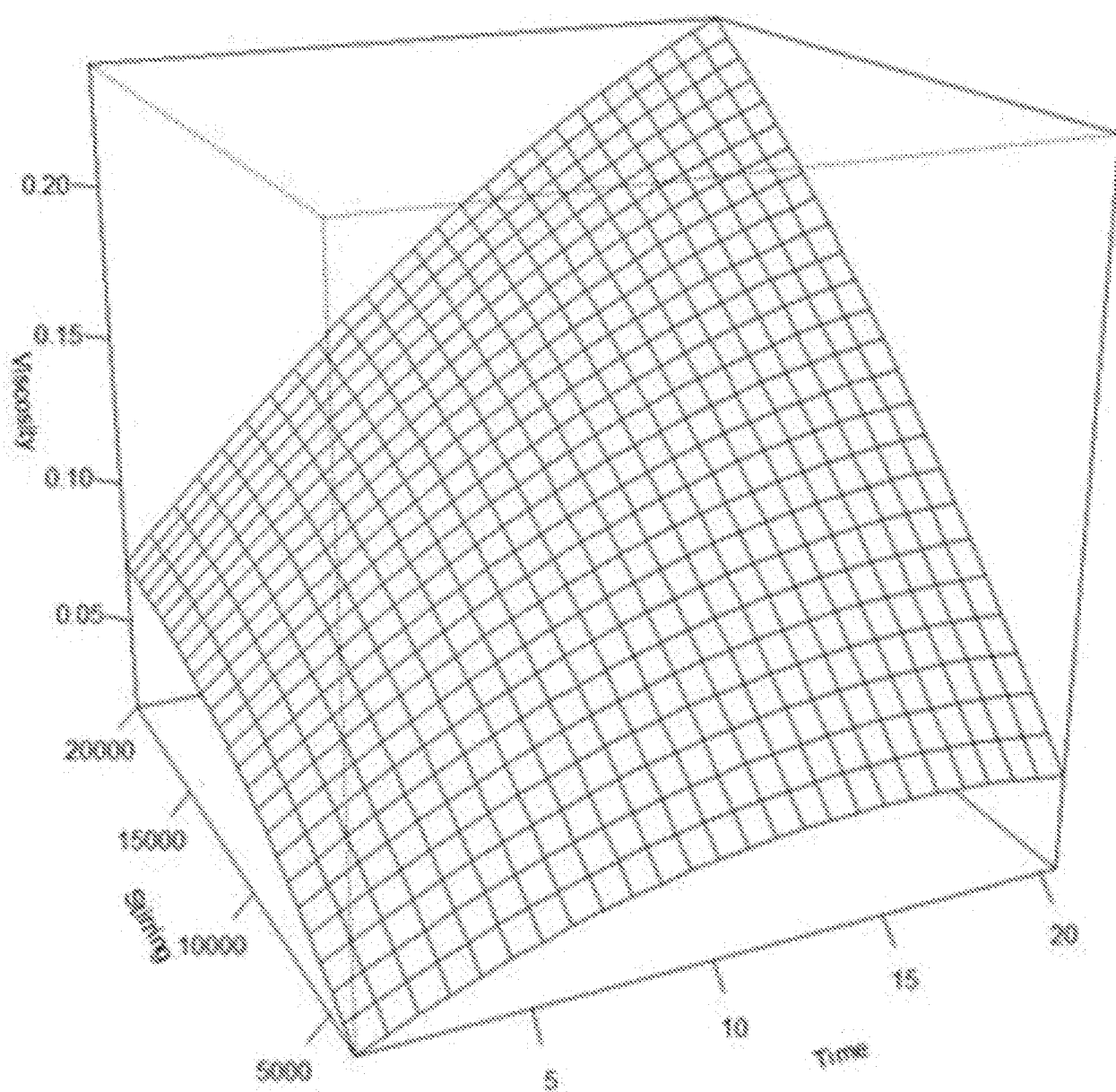


Figure 41

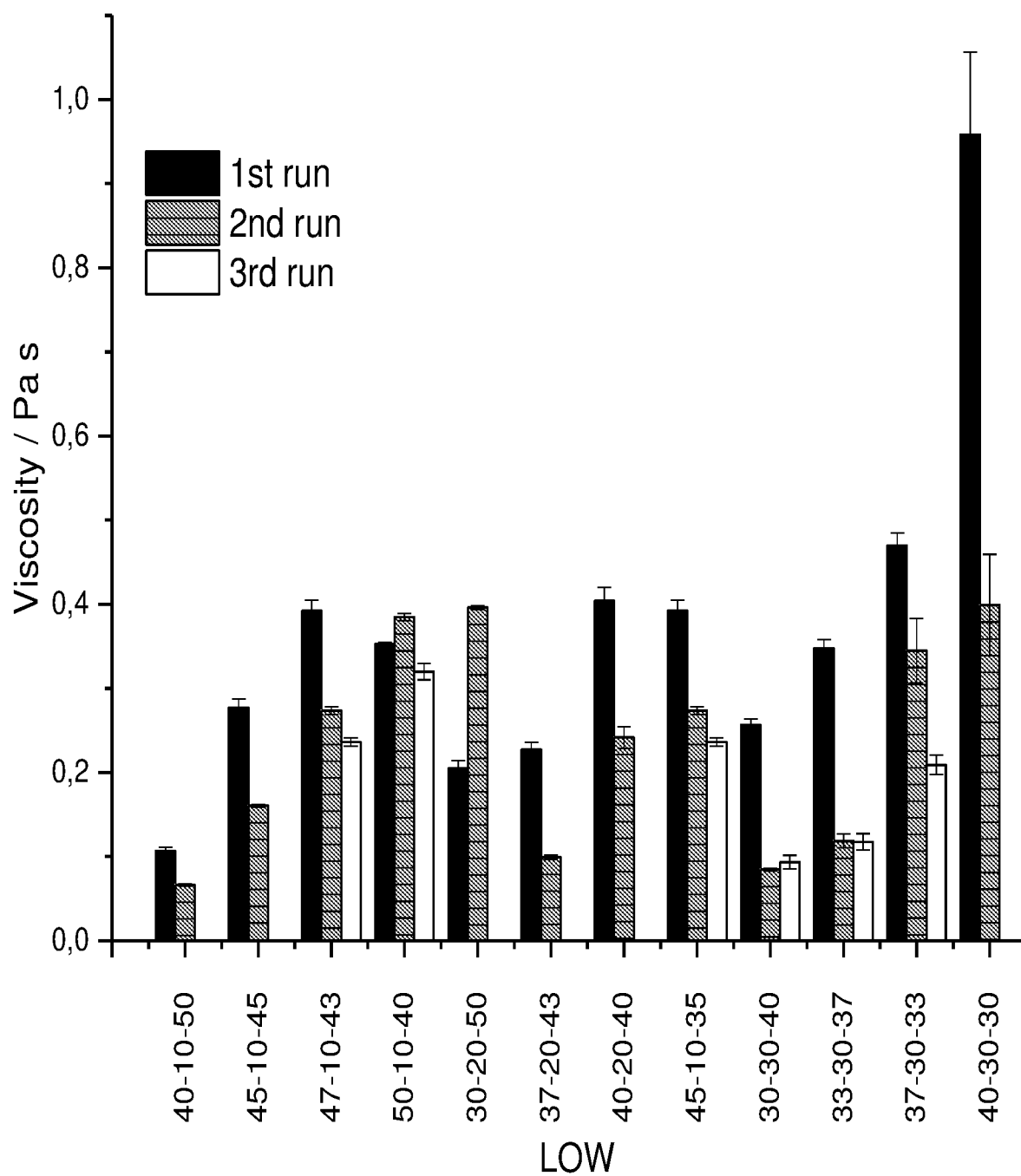


Figure 42a

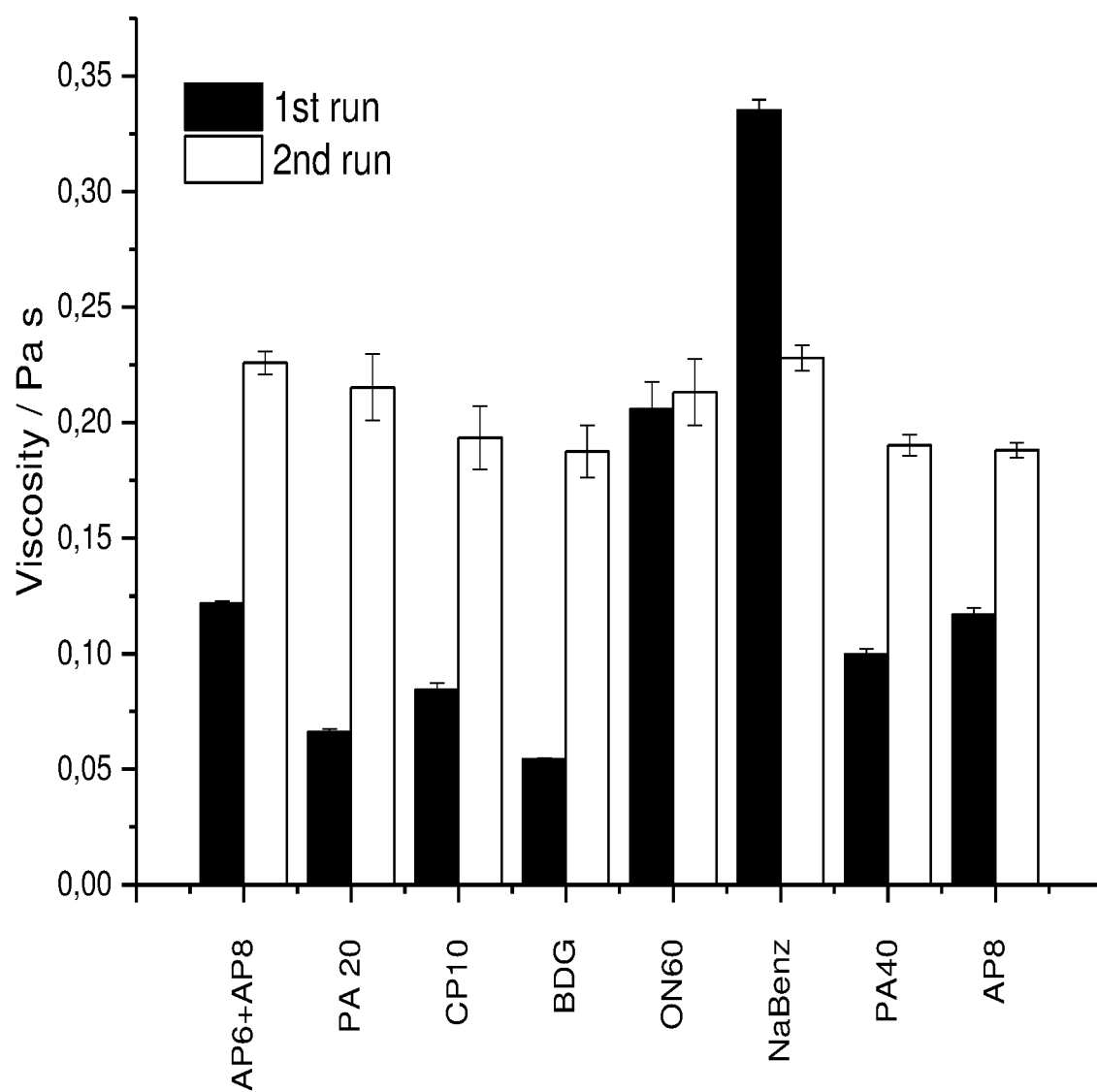


Figure 42b

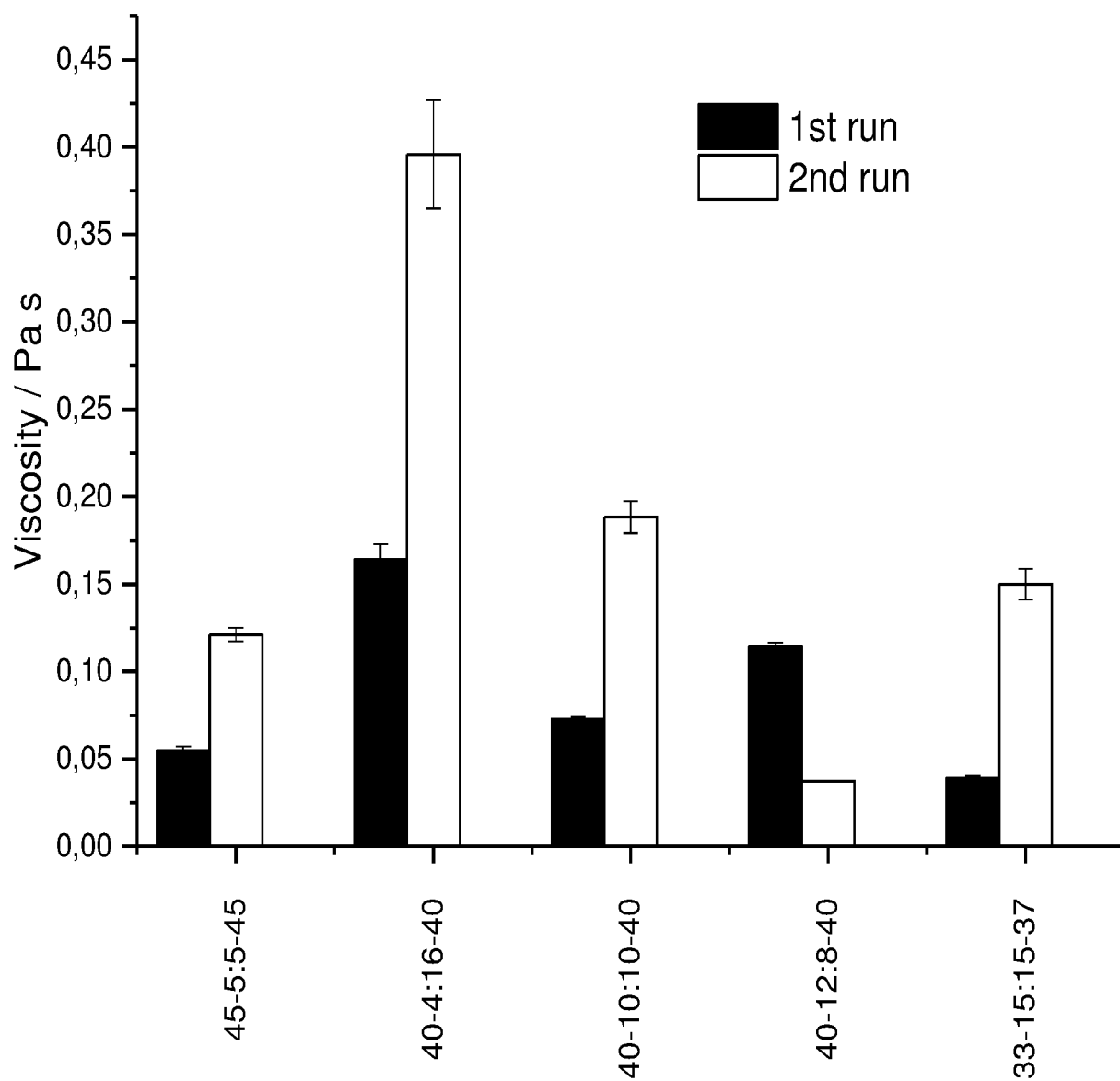


Figure 42c

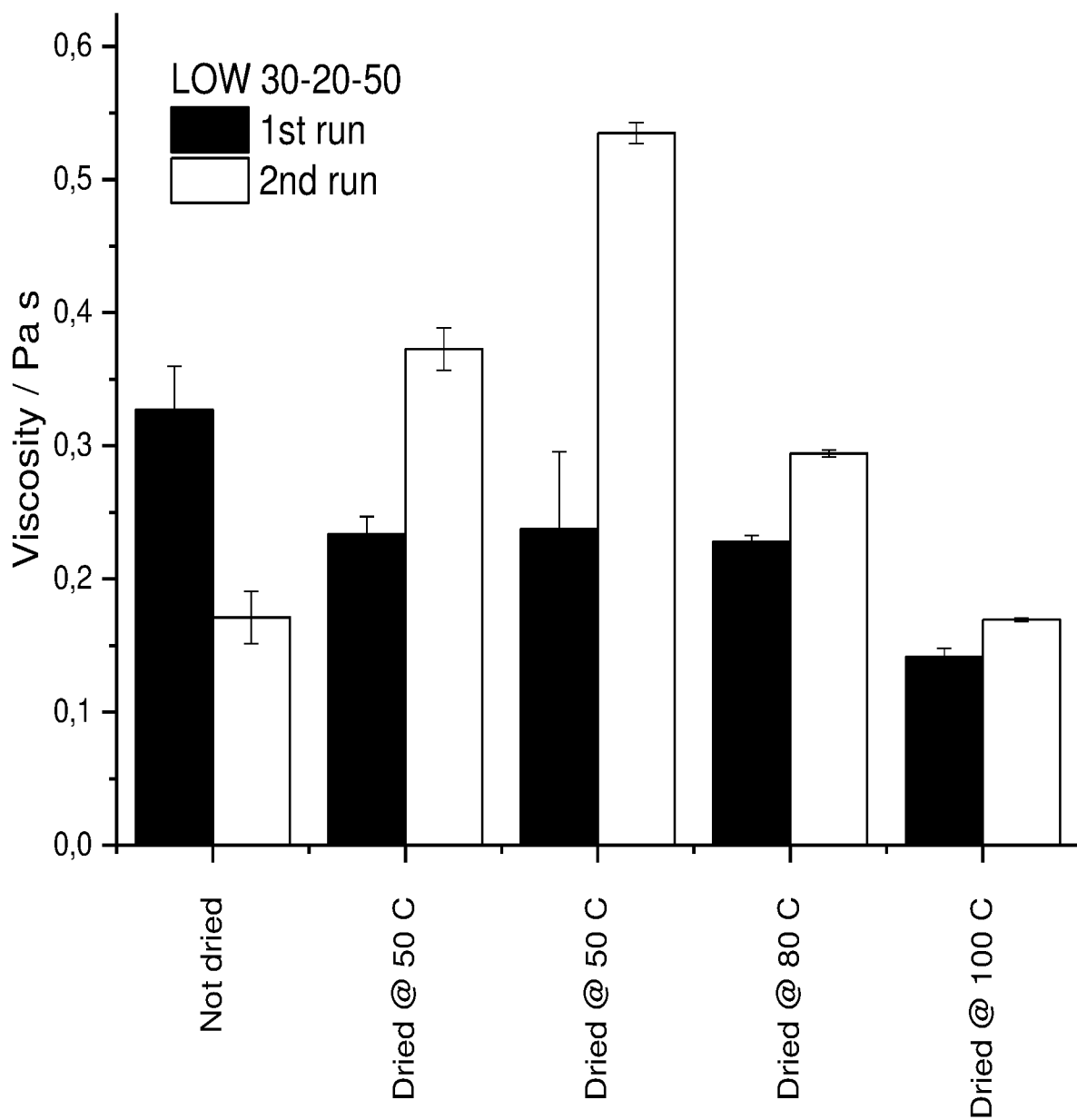


Figure 42d

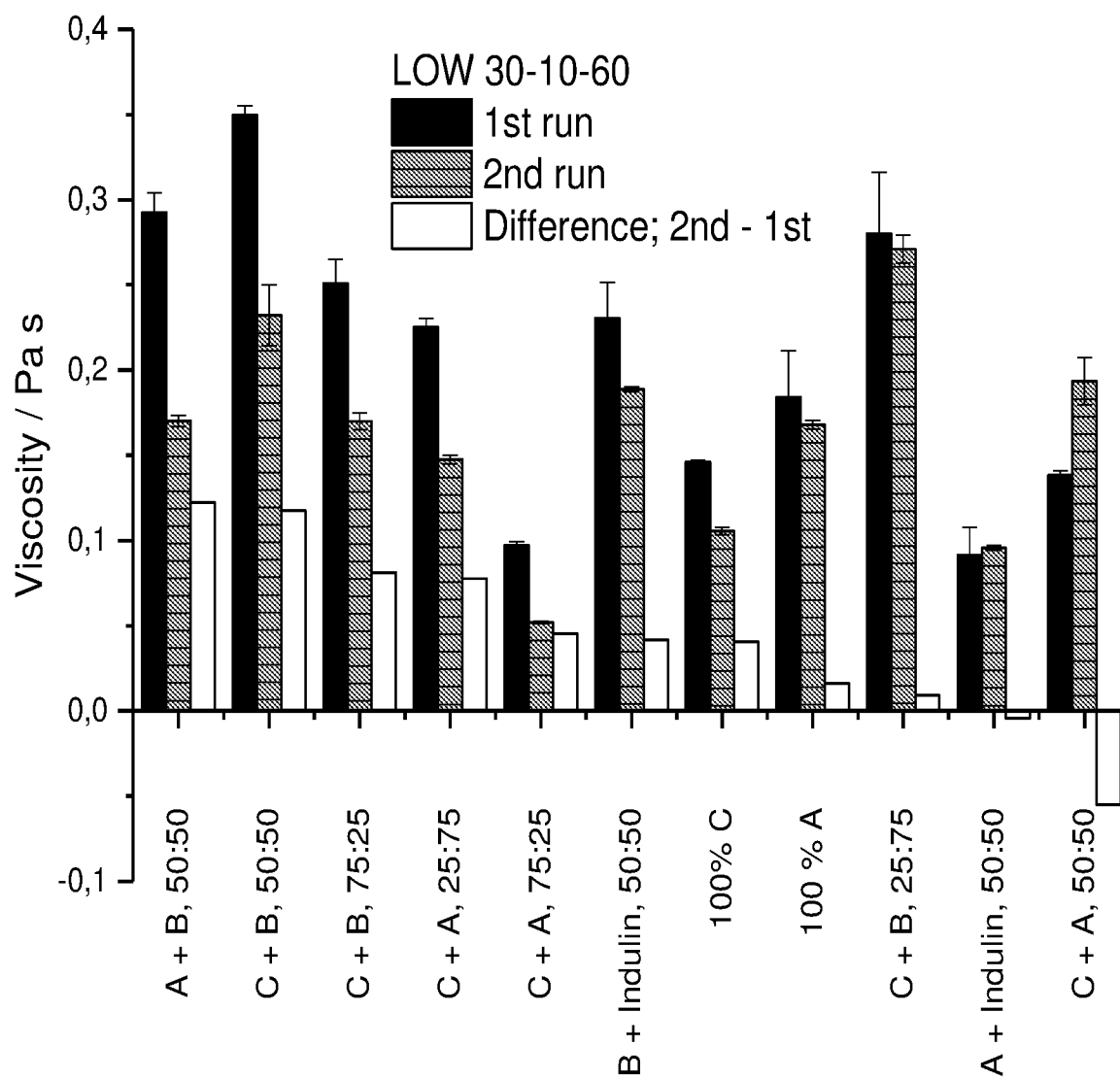


Figure 42e

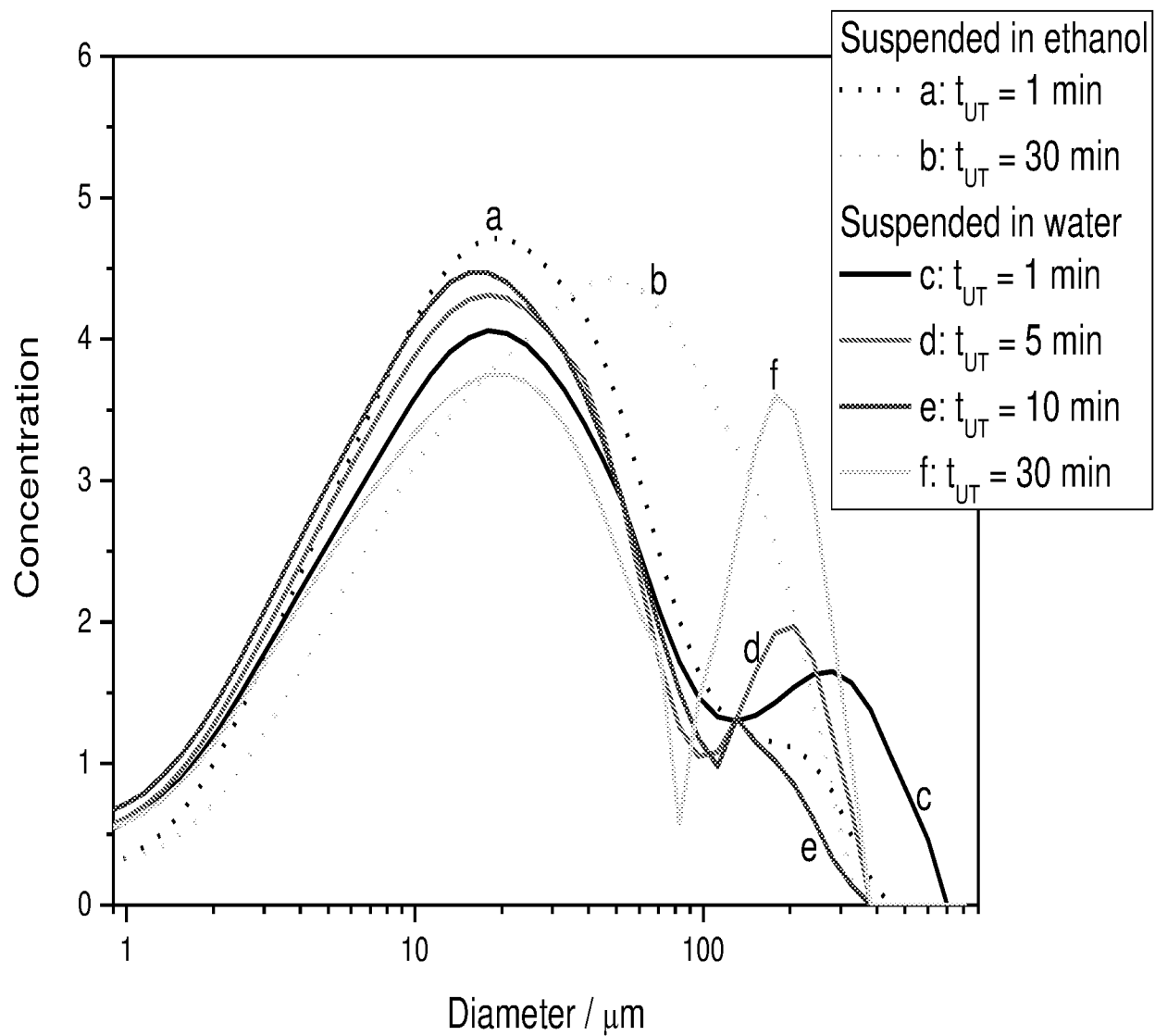
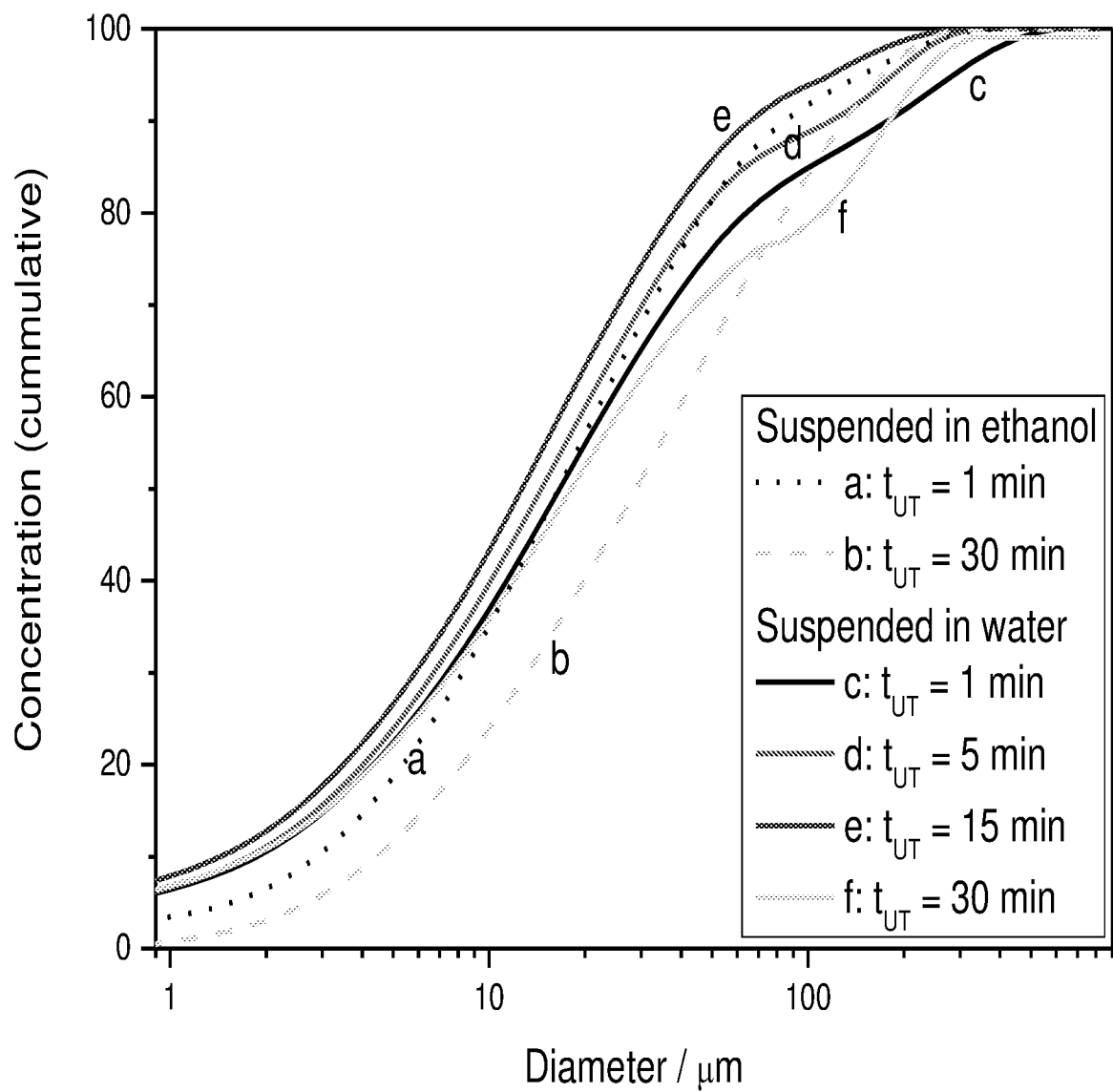


Figure 43a

**Figure 43b**

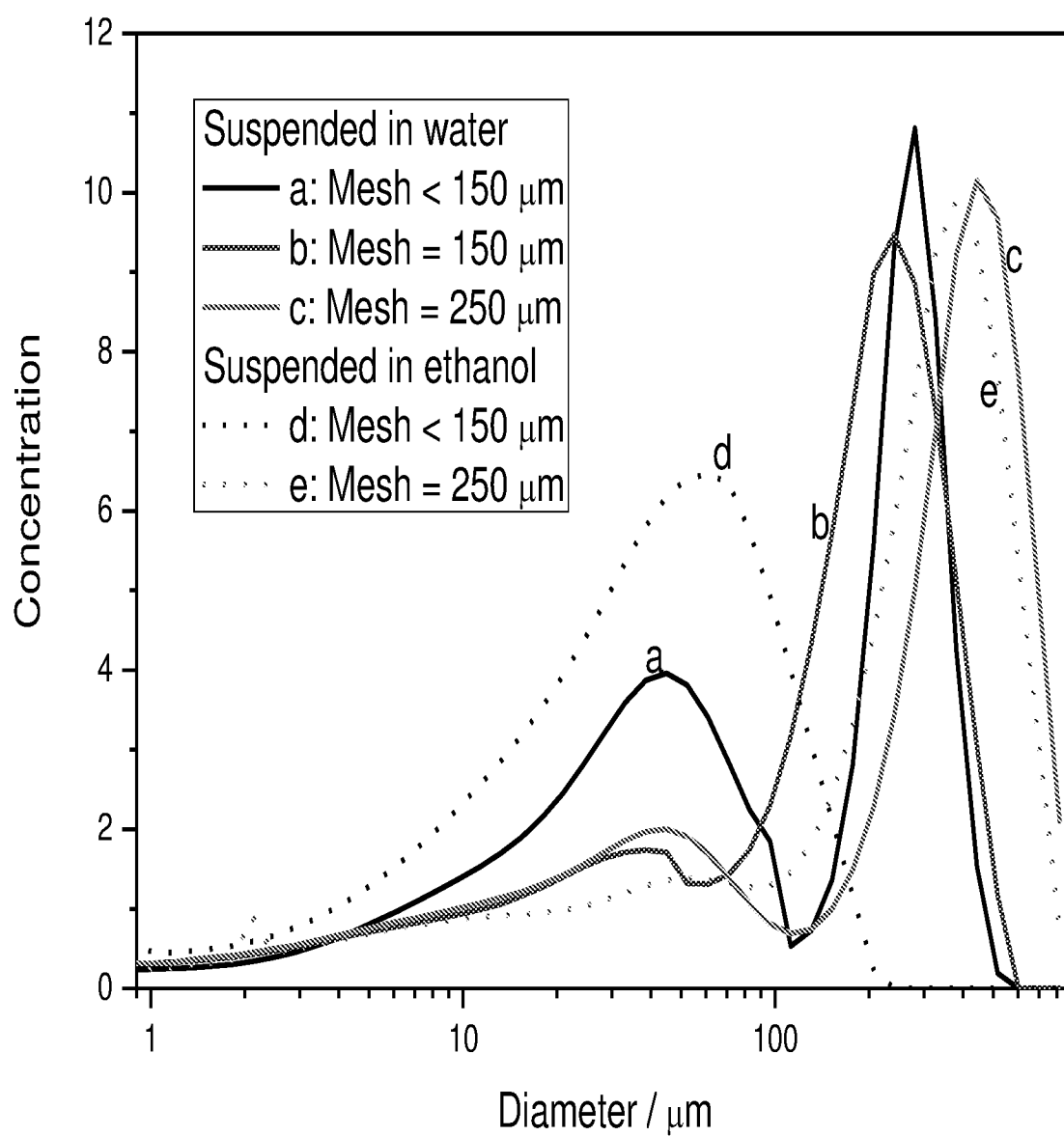


Figure 44a

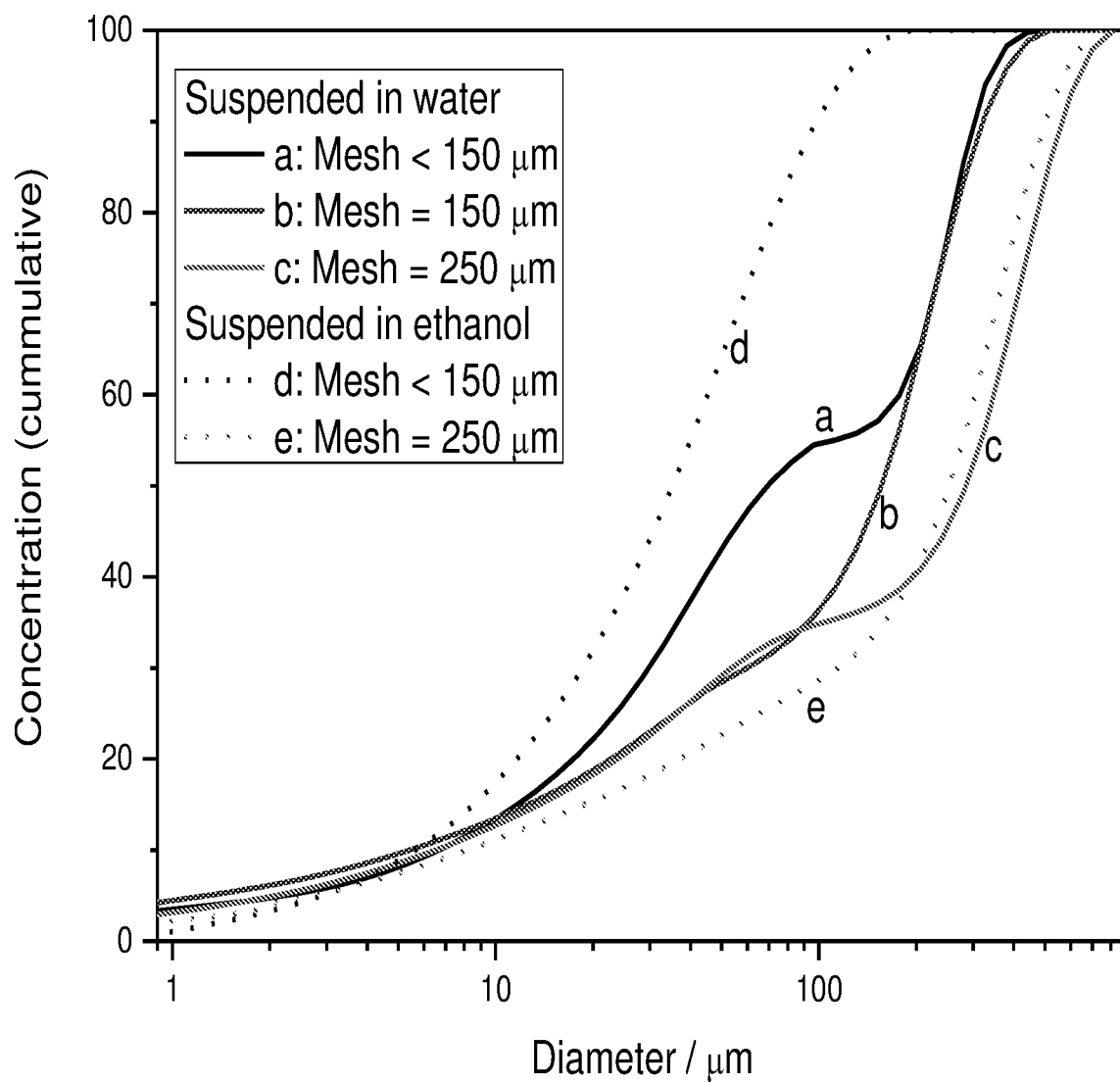


Figure 44b

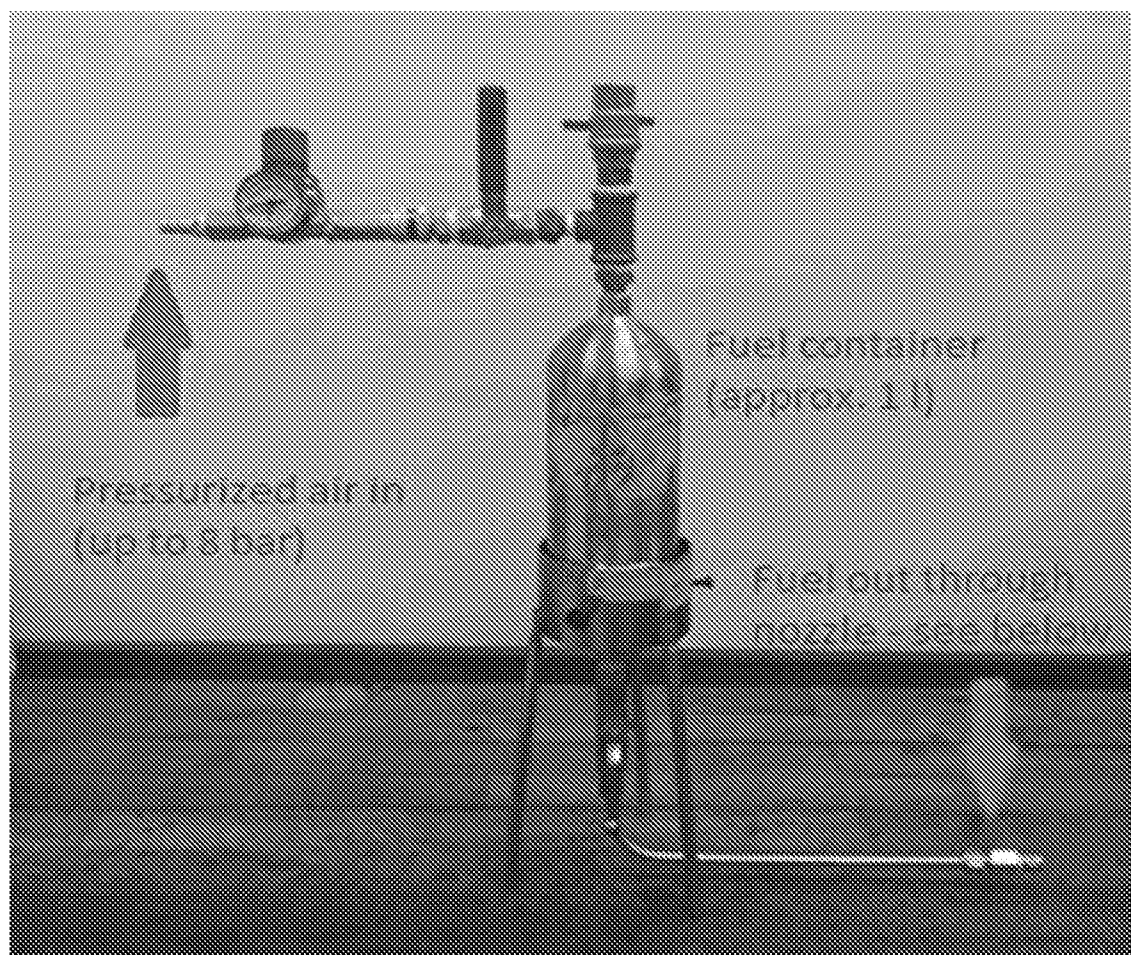
**Figure 45**



Figure 46

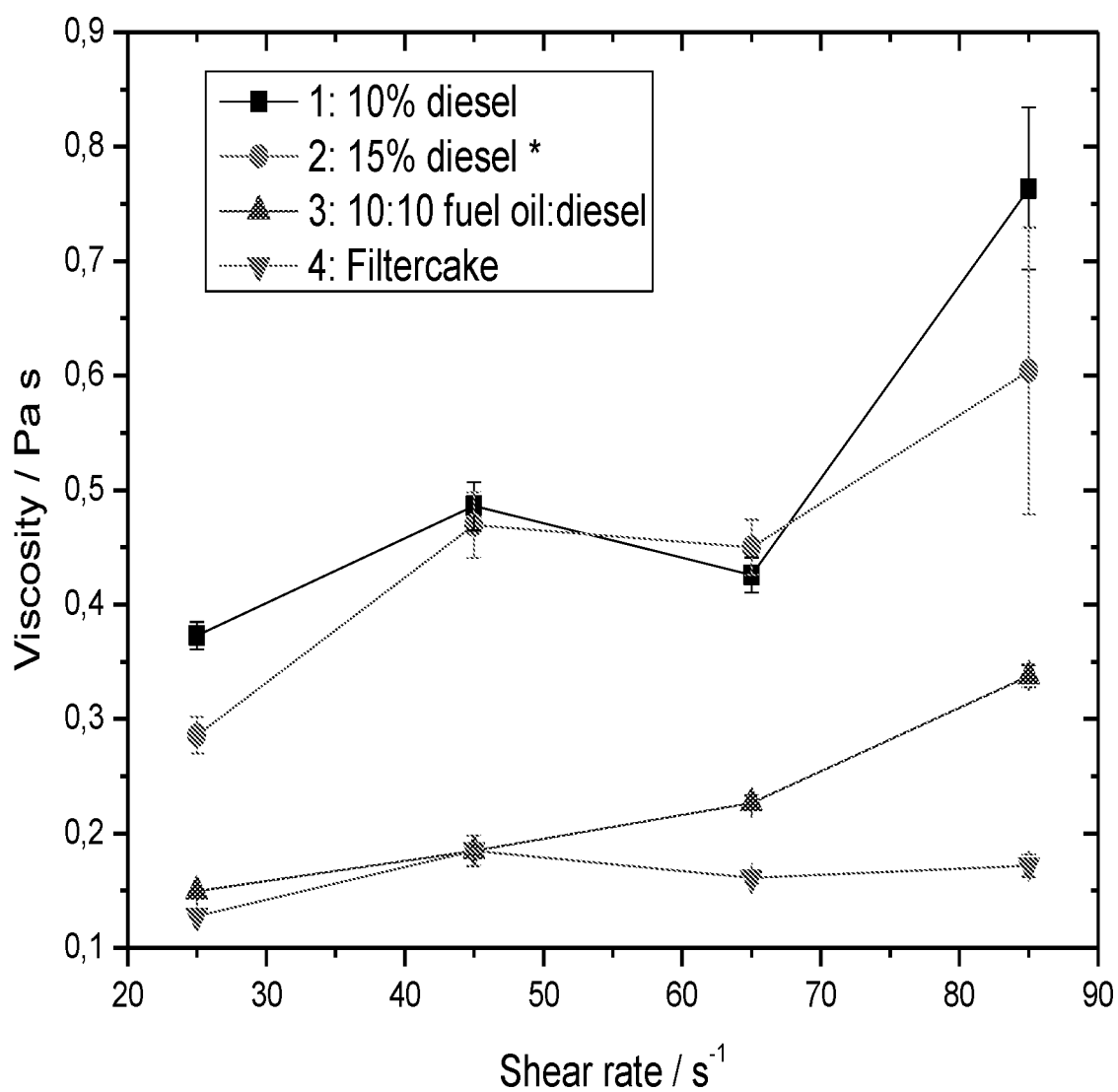


Figure 47

FIGURES

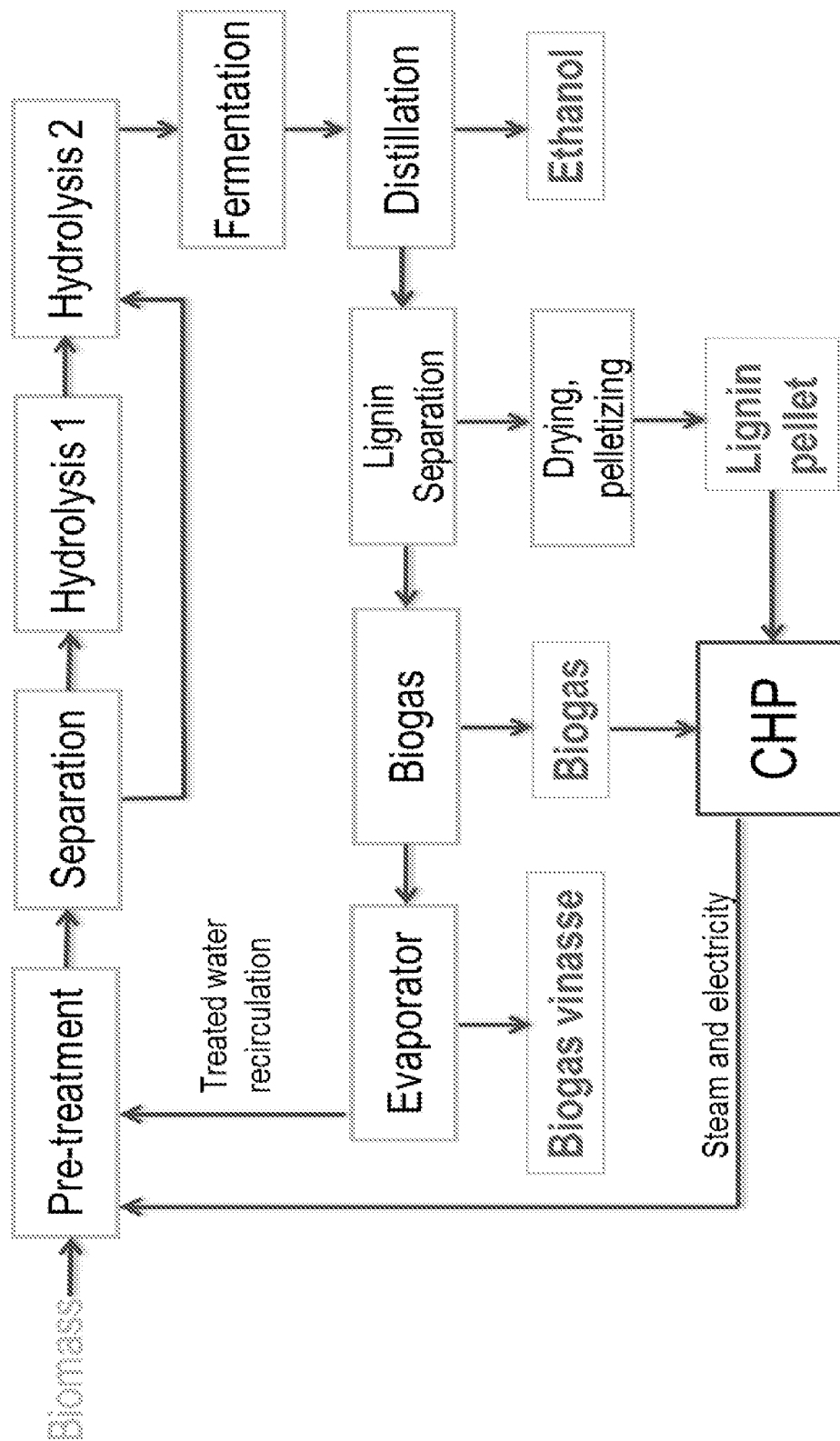


Figure 48a

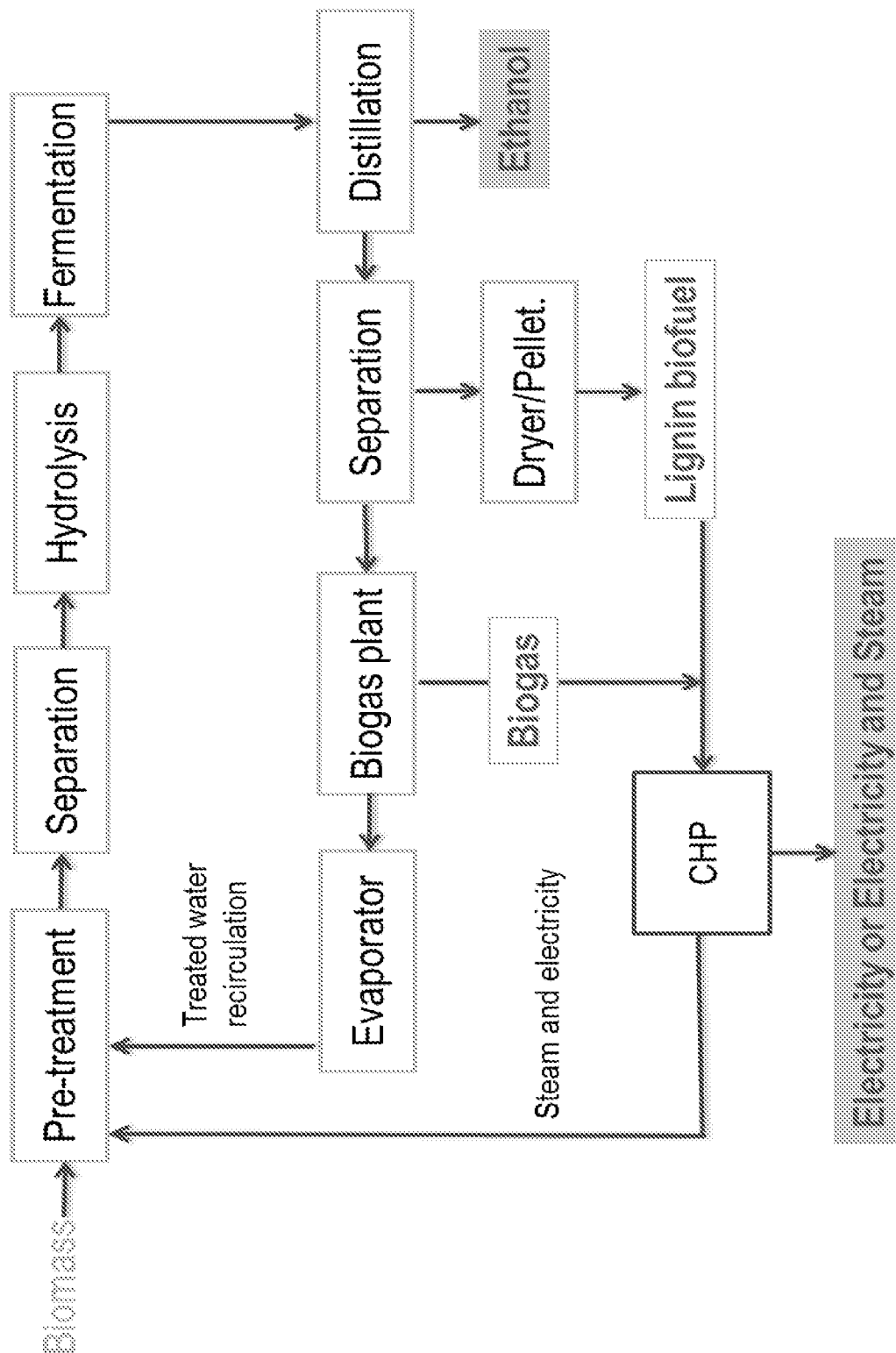


Figure 48b

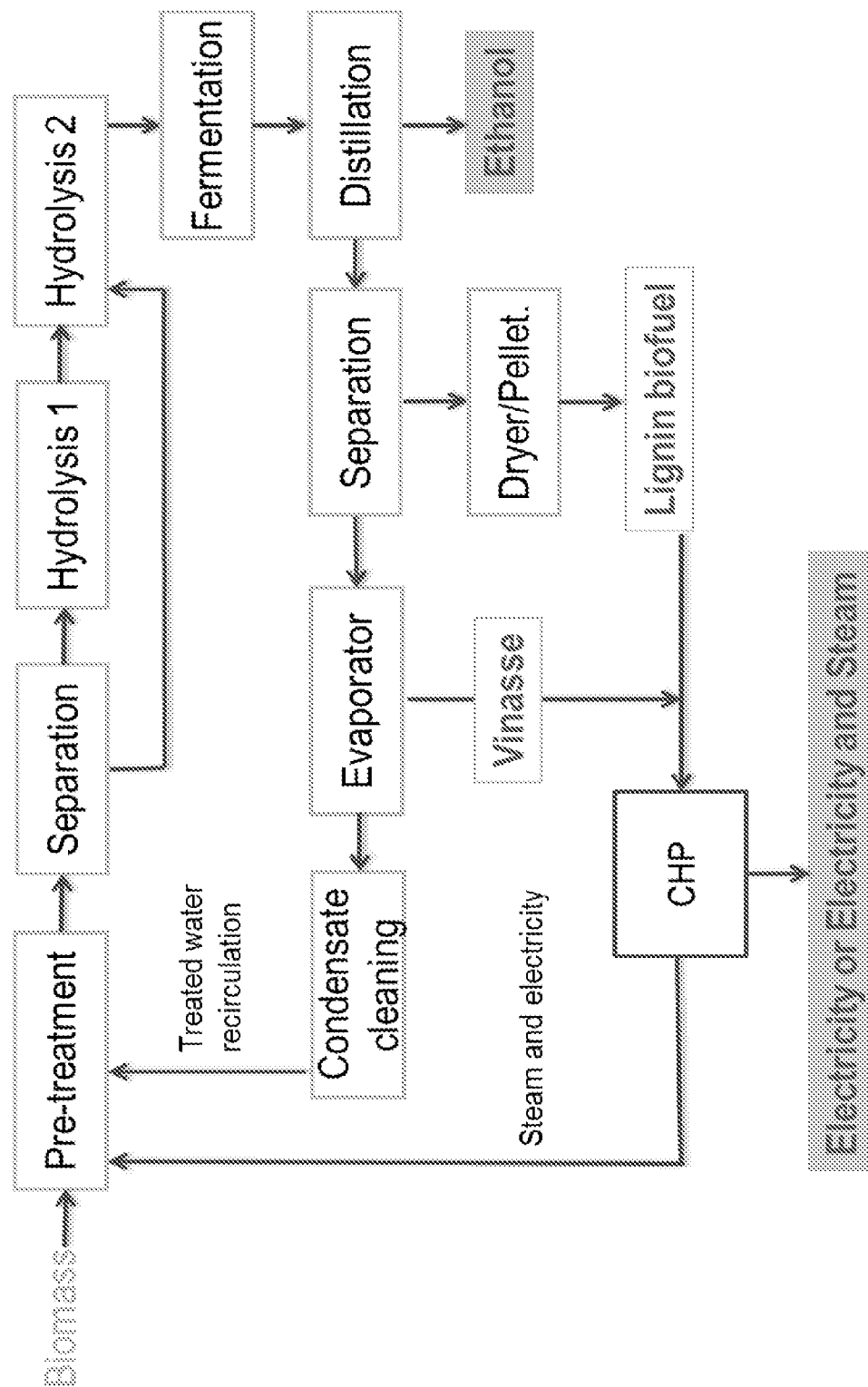


Figure 49a

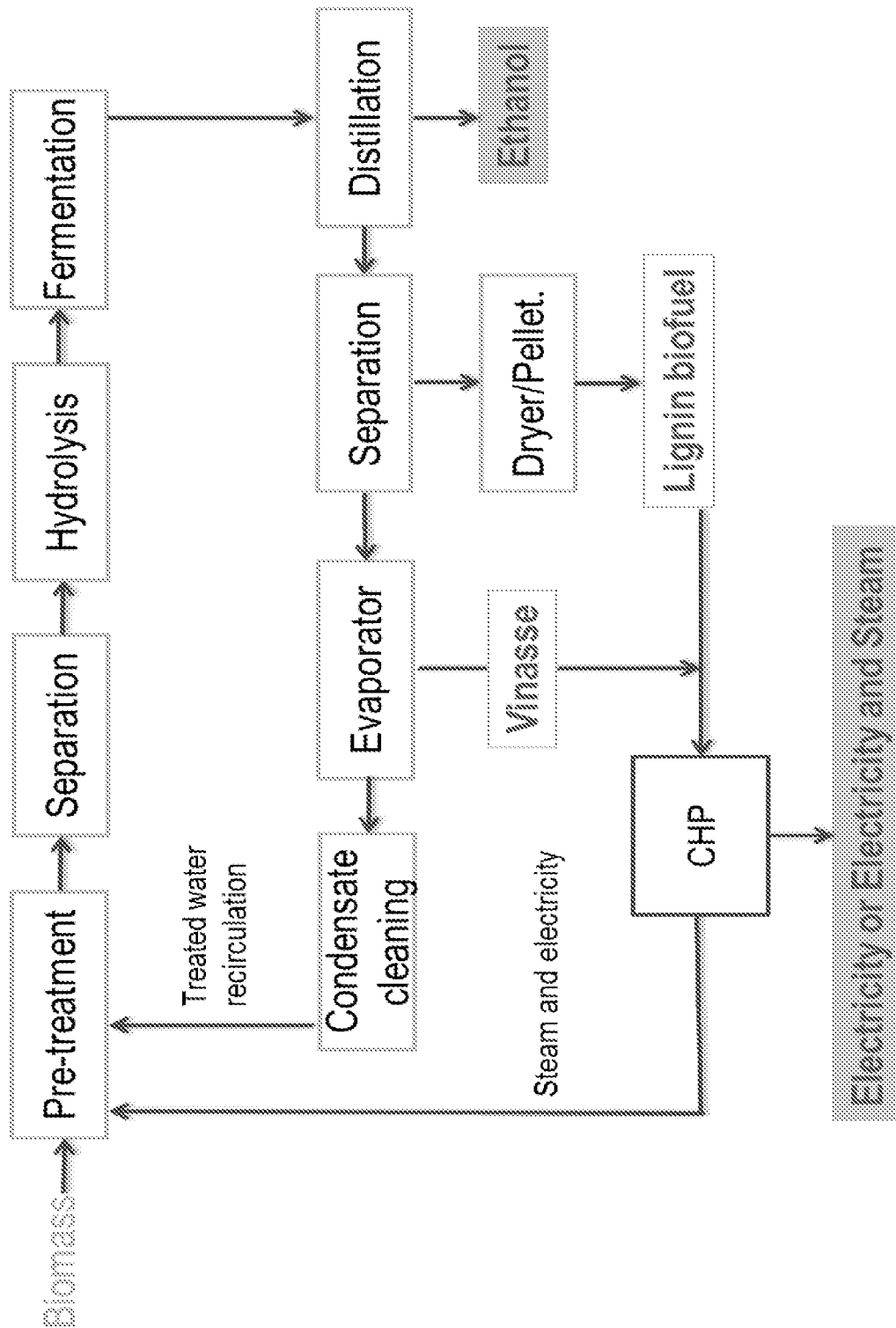


Figure 49b

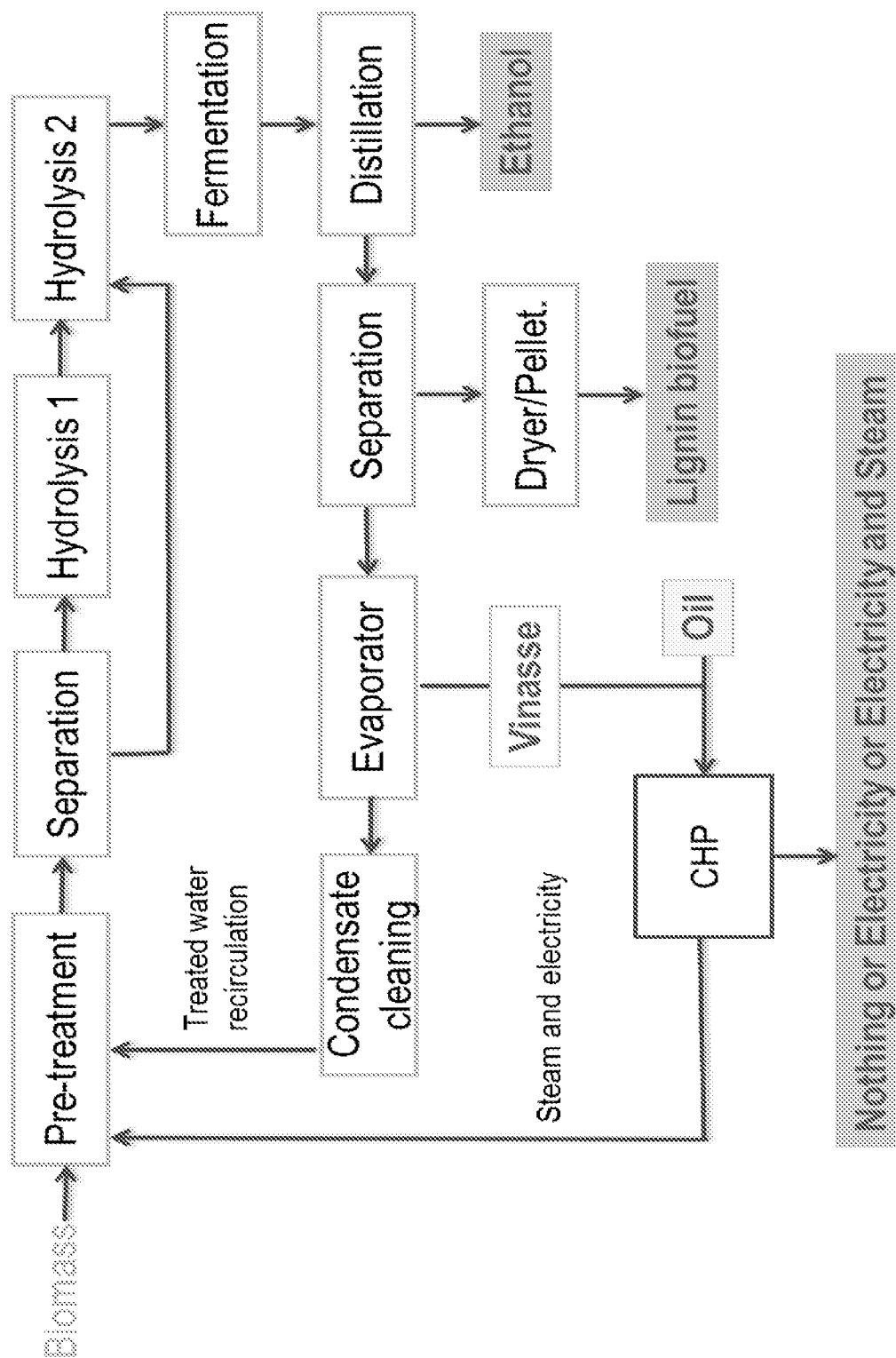


Figure 50a

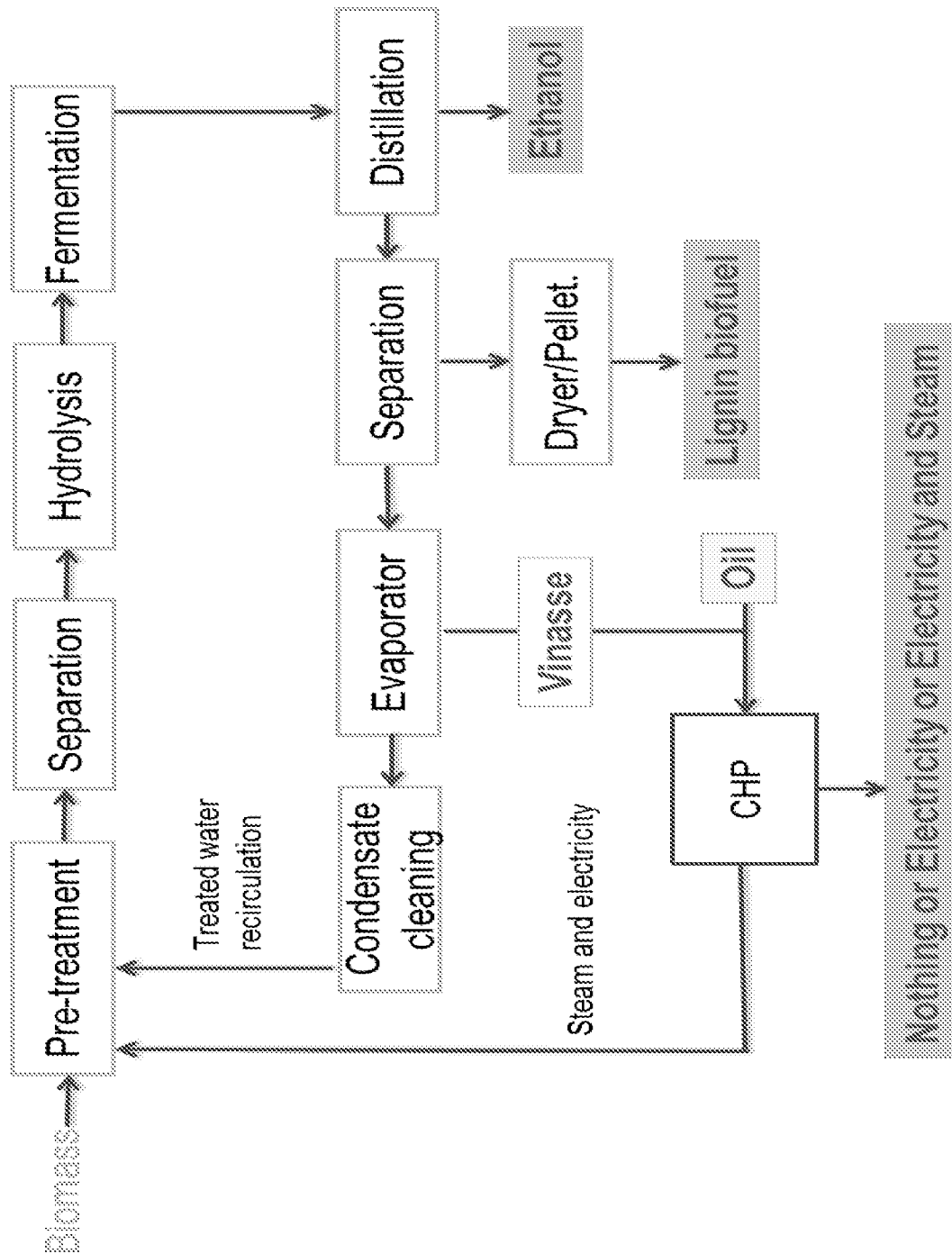


Figure 50b

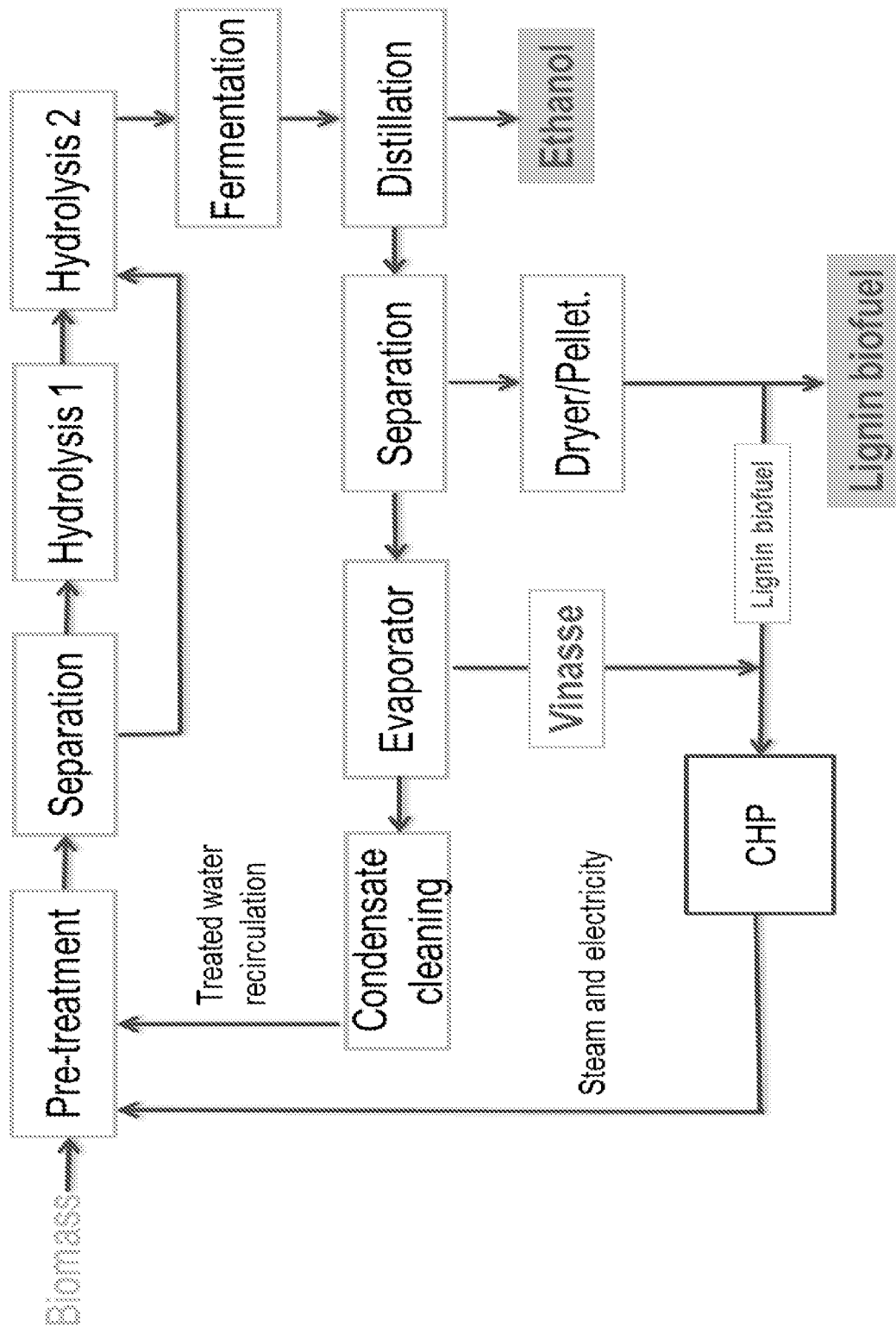


Figure 51a

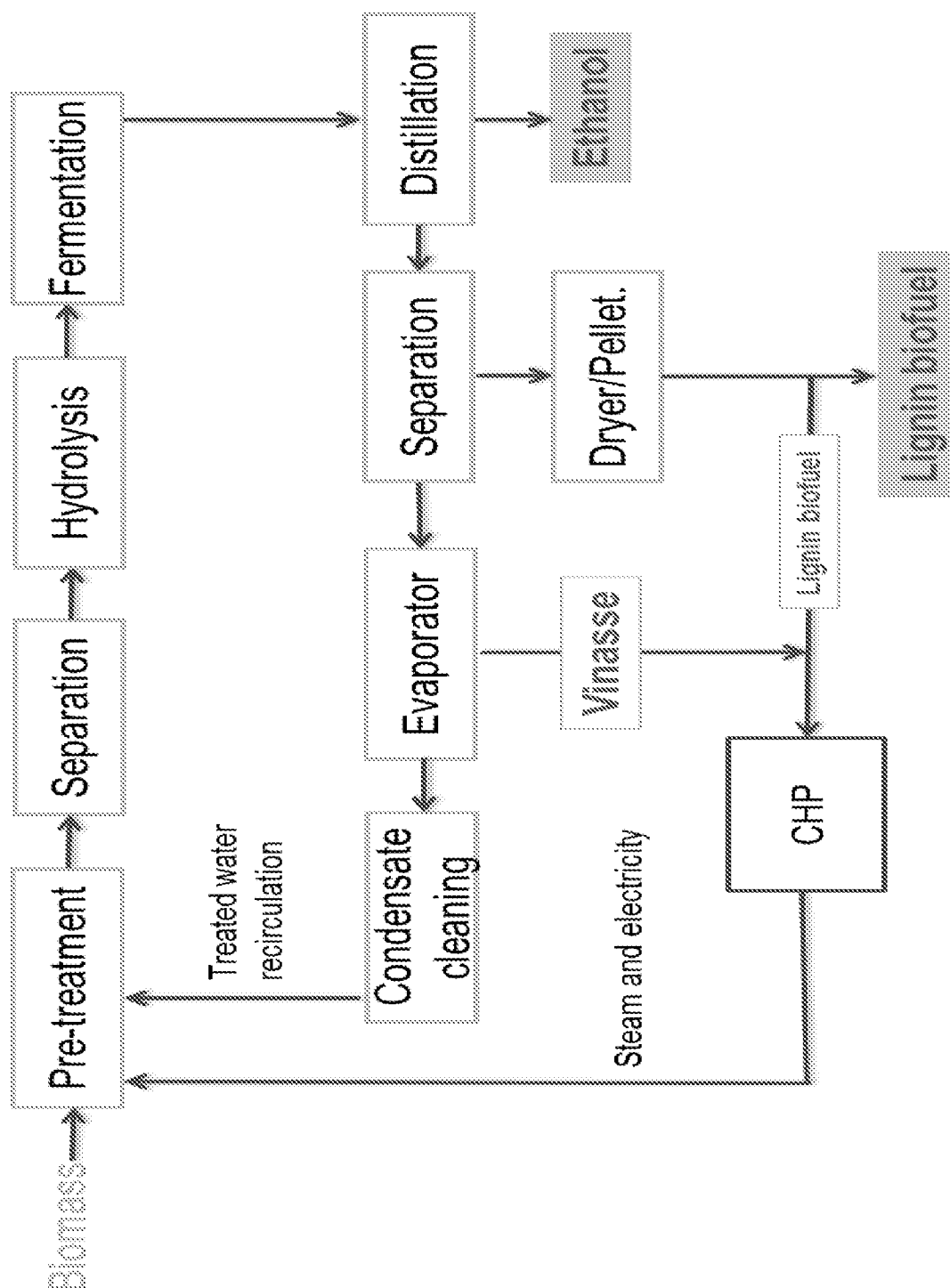


Figure 51b

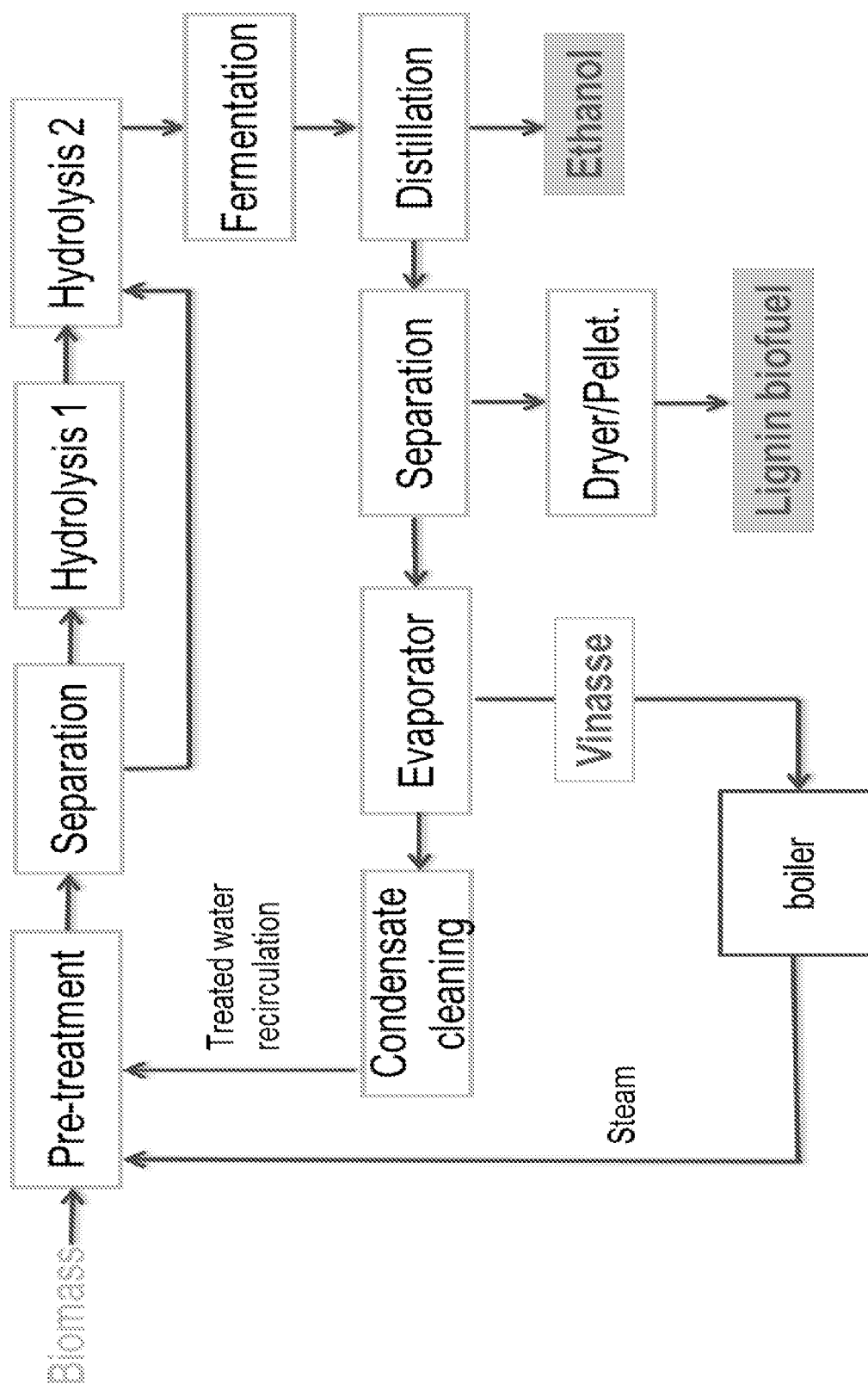


Figure 52a

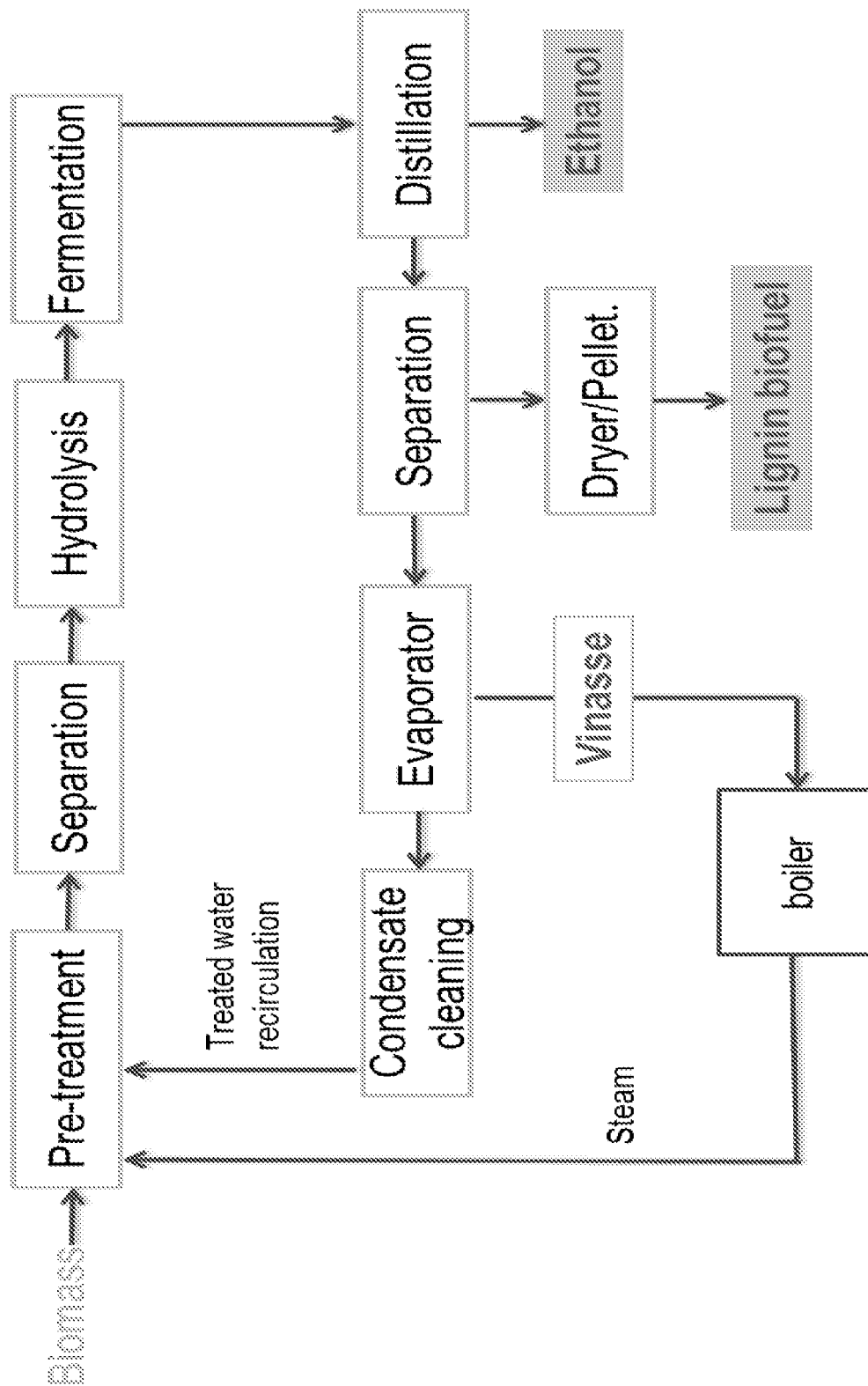


Figure 52b

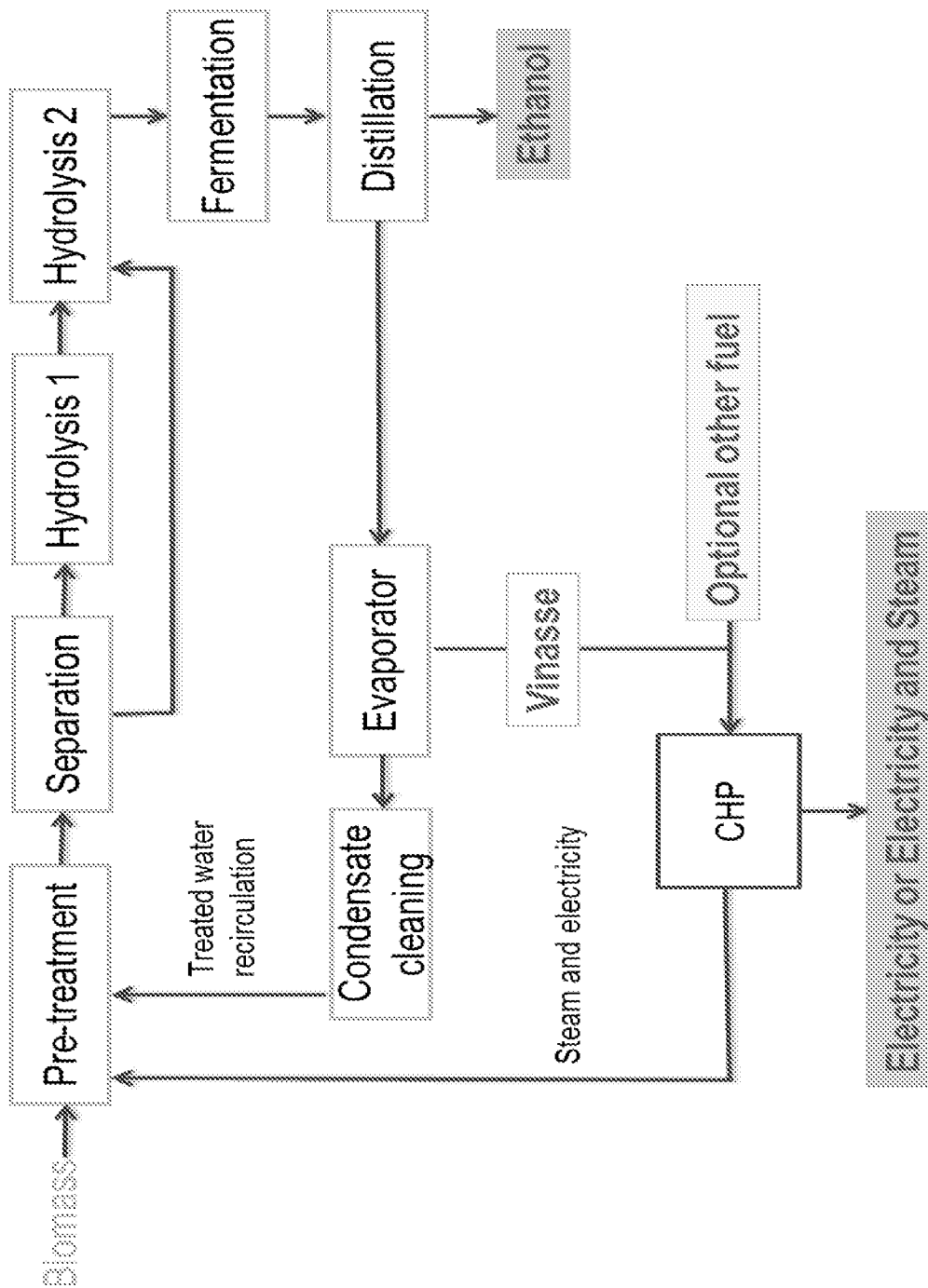


Figure 53a

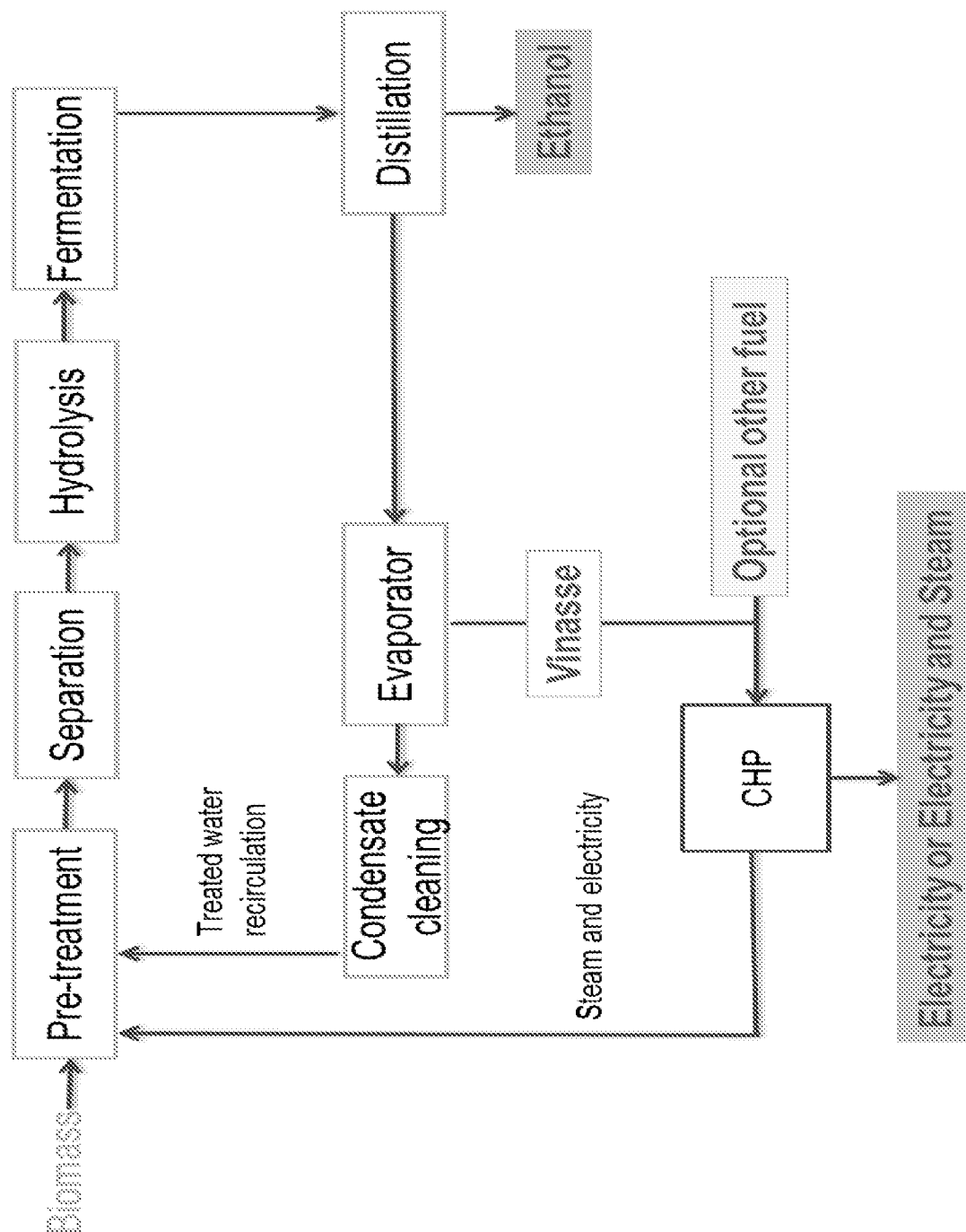


Figure 53b

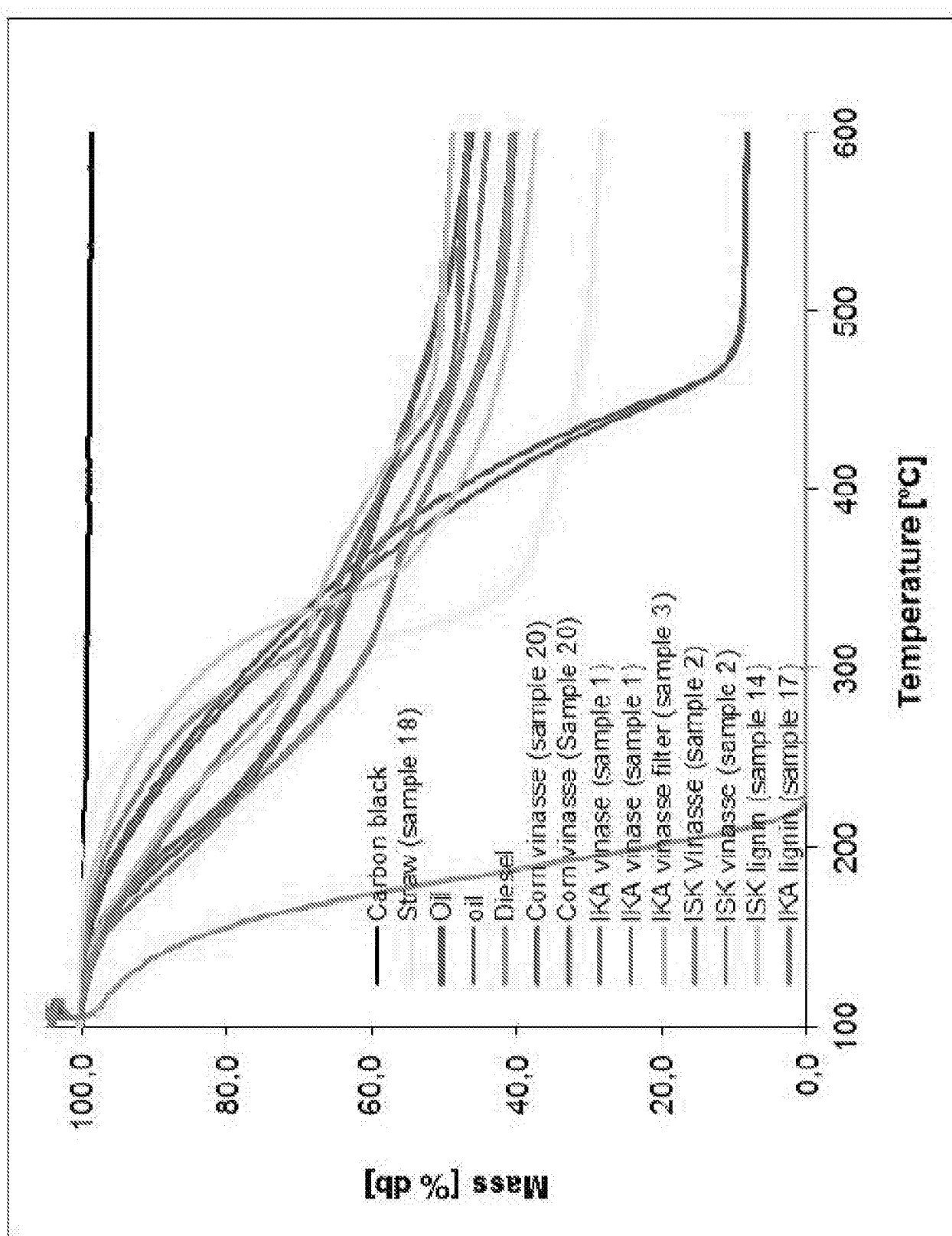


Figure 54

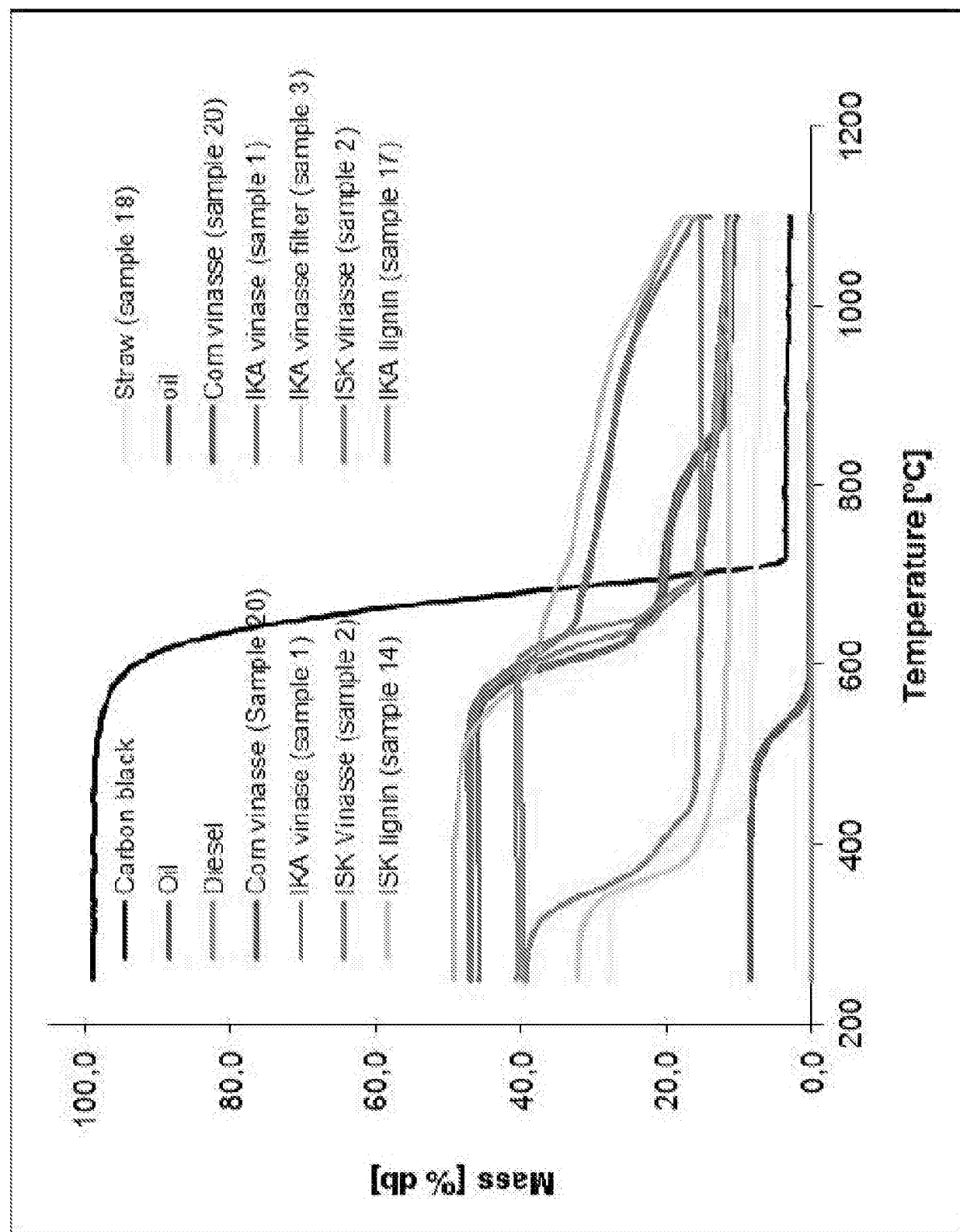


Figure 55

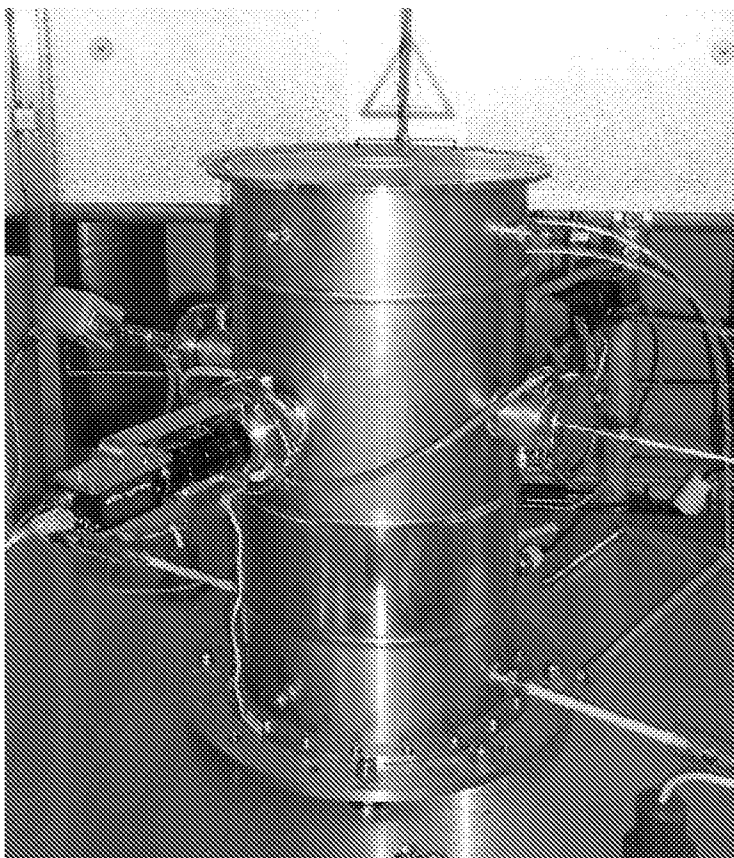


Figure 56

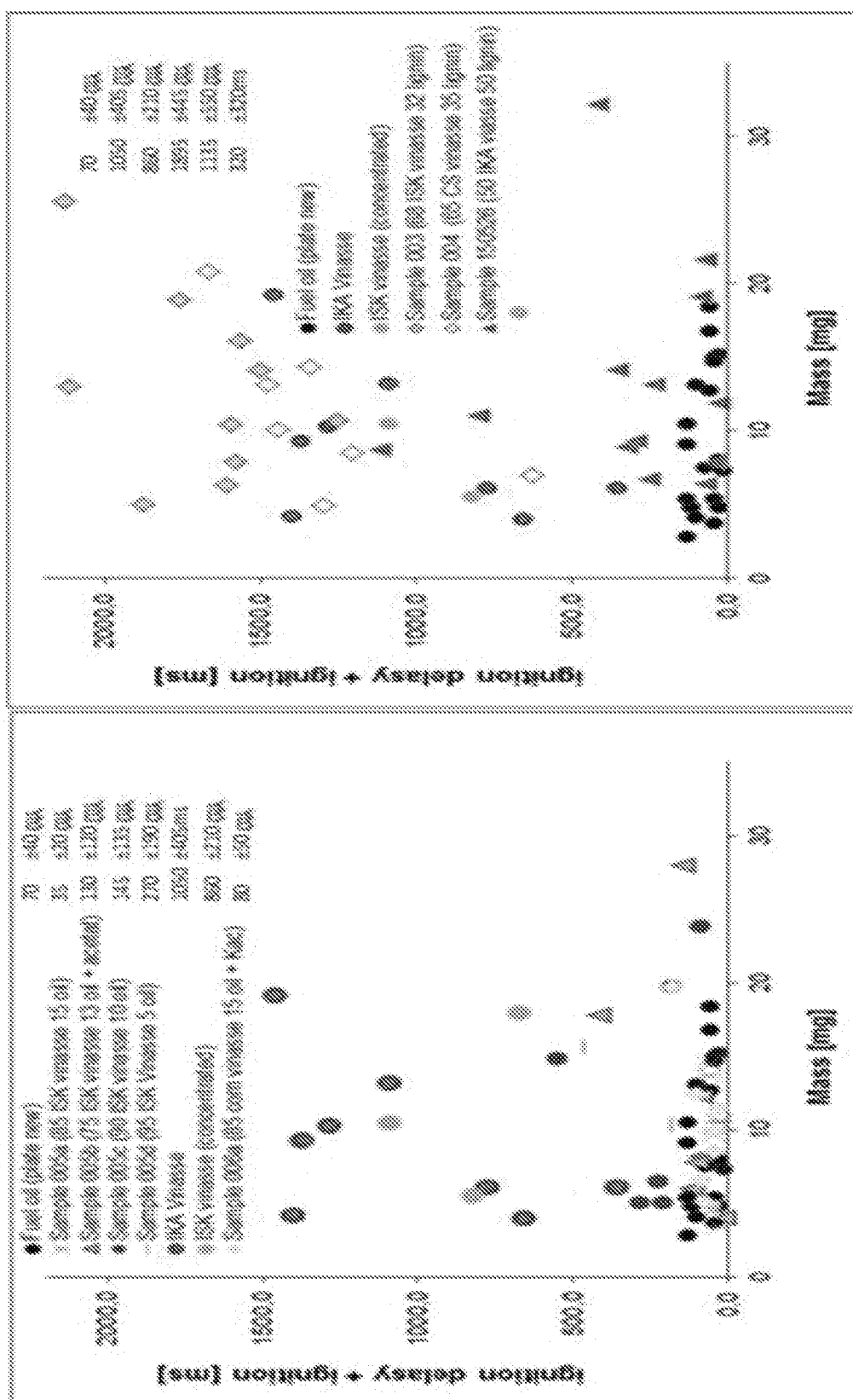


Figure 57

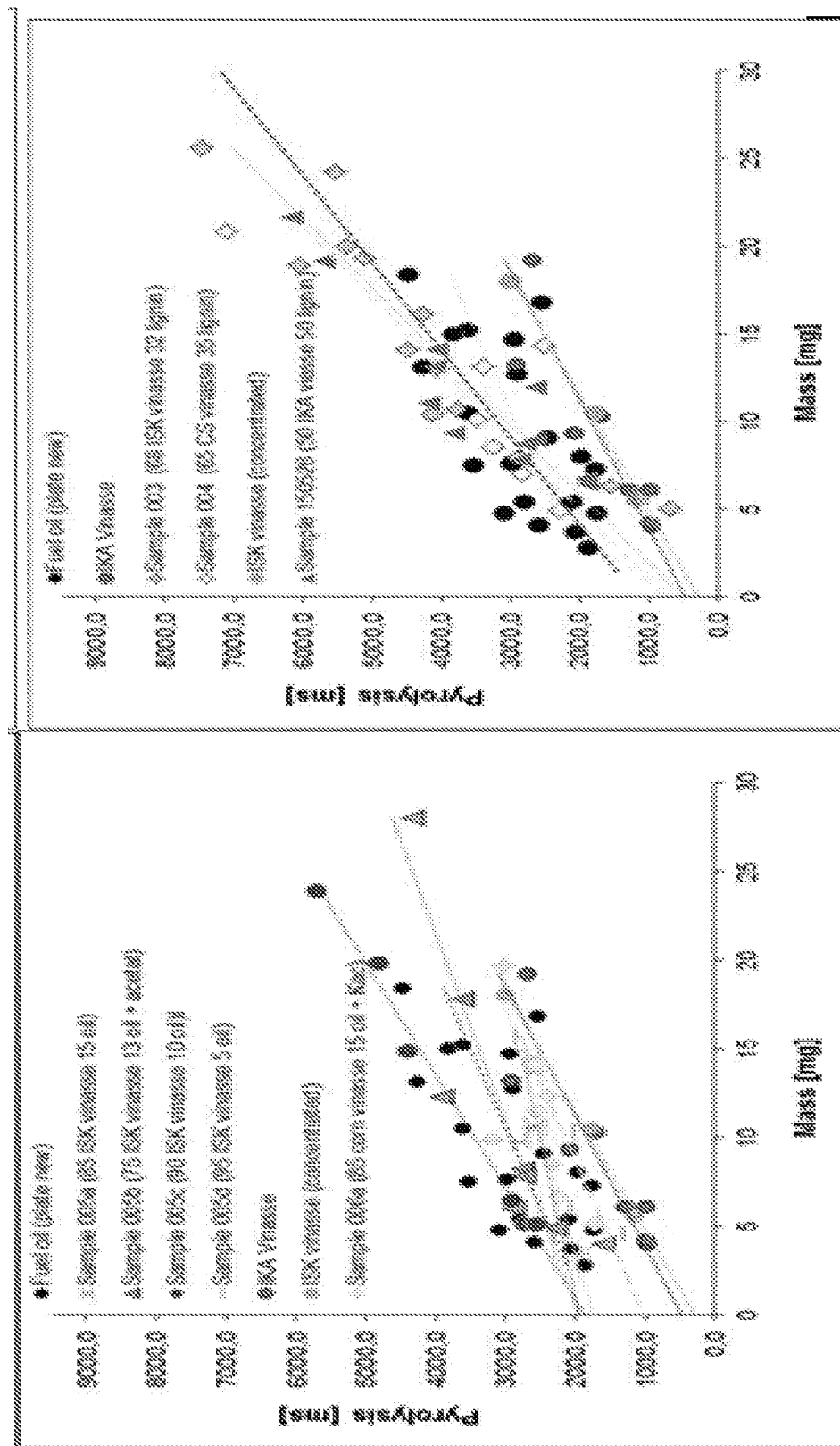


Figure 58

INTERNATIONAL SEARCH REPORT

International application No
PCT/DK2016/050452

A. CLASSIFICATION OF SUBJECT MATTER
INV. C10G1/02 C08L5/14 C08L97/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C10G C08L

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

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

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2015/064762 A1 (MEDEIROS PINTO JOSÉ RICARDO [BR] ET AL) 5 March 2015 (2015-03-05) claims 1-1356-60, 79, 86 paragraphs [0056] - [0060], [0079], [0086] figures 1-6	1-91, 114-274
X	US 2015/167969 A1 (RETSINA THEODORA [US] ET AL) 18 June 2015 (2015-06-18) claims 1-16 paragraphs [0056], [0059] - [0061] figure 1 ----- -/-	1-91, 114-274



Further documents are listed in the continuation of Box C.



See patent family annex.

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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Date of the actual completion of the international search

20 February 2017

Date of mailing of the international search report

28/02/2017

Name and mailing address of the ISA/

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Fax: (+31-70) 340-3016

Authorized officer

Pardo Torre, J

INTERNATIONAL SEARCH REPORT

International application No
PCT/DK2016/050452

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2009/116070 A2 (LIGNOIL TECHNOLOGIES PRIVATE L [IN]; PRADYOT AGASKAR A [US]) 24 September 2009 (2009-09-24) claims 1-42 figure 1 -----	92-147
X	WO 2012/177138 A1 (UNIV UTRECHT HOLDING BV [NL]; ZAKZESKI JOSEPH JOHN [NL]; BRUIJNINCX PI) 27 December 2012 (2012-12-27) claims 1-15 page 9, lines 1-20 -----	92-113
X	US 2010/159520 A1 (DINER BRUCE A [US] ET AL) 24 June 2010 (2010-06-24) claims 1-23 paragraphs [0078] - [0127] -----	114-147

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/DK2016/050452

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