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DESCRIPTION

[0001] The present application pertains to a process for the decomposition of lignocellulose-containing biomass-material.

[0002] Due to limited resources of mineral oil and demands to reduce CO₂ emissions the chemical industry seeks more sustainable production routes for the manufacture of commodity chemicals such as liquid fuels and base chemicals. Part of that strategy focusses on the conversion of lignocellulosic biomass into versatile chemicals or fuels such as ethanol. Lignocellulosic biomass contains cellulose (~ 25-40% w/w d.s.), hemicellulose (~ 15-25% w/w d.s.) and lignin (~ 15-30% w/w d.s.) as major components and minor amounts of other carbohydrates, waxes, proteins and inorganic compounds. Among forms of plant biomass, lignocellulosic biomass derived from any forestry and agricultural waste streams, such as wood residues and cereal straw are particularly well suited for conversion to commodity chemicals and fuels because of their availability, low cost and environmentally sound production. Additionally, life cycle analyses of production processes utilising lignocellulosic feedstocks indicate reduced greenhouse gas emissions compared to processes based on other feedstocks.

[0003] Various process options that describe the conversion of lignocellulosic biomass to ethanol and other base chemicals have been described (Pejo et al., 2008). To realize these processes on an industrial scale it is particularly desirable to transfer the maximal amount of energy, carbon and mass content contained in the renewable feedstock to the end products. At present none of the described conversion processes have realised this to the full extent.

[0004] Exemplary unit operations for the biotechnological conversion of lignocellulosic material (e.g. straw) to value-adding products (e.g. ethanol) are: mechanical de-sizing and/or physicochemical pretreatment, enzymatic hydrolysis, fermentation and product recovery. For example, Vandenbossche et al: "A new lignocellulosic biomass deconstruction process combining thermo-mechano chemical action and bio-catalytic enzymatic hydrolysis in a twin-screw extruder", *Industrial Crops and Products*, vol. 55, April 2014 (2014-04), pages 258-266, discloses a process for the decomposition of lignocellulose-containing biomass-material, comprising the steps of providing lignocellulose-containing biomass-material and subjecting the lignocellulose-containing biomass-material to comminution. Reference is also made to WO 2009/158709 A2. Regarding industrial scale cellulosic ethanol production, one key barrier is still the expenditure for cost and thus a low-energy process and efficient biomass conversion are of major importance. Therefore, the object of the present invention is the provision of an improved highly efficient process for the decomposition of lignocellulose-containing biomass-material. The inventors of the present invention have now surprisingly found that this object of the present invention may be achieved by a process in accordance with independent claim 1 for the decomposition of lignocellulose-containing biomass-material, comprising the steps

1. (a) providing lignocellulose-containing biomass-material;

2. (b) subjecting the lignocellulose-containing biomass-material to comminution;
3. (c) subjecting the comminuted lignocellulose-containing biomass-material to a sifting to separate a particle-fraction consisting of particles with an average particle diameter of less than 2500 μm ;
4. (d) subjecting the remaining comminuted lignocellulose-containing biomass-material to a pretreatment which is selected from mechanical pretreatment, biological pretreatment, chemical pretreatment, physical pretreatment and combinations thereof;

wherein before, during or after pretreatment from 1 to 15 weight-% of particles are added to the lignocellulose-containing biomass-material wherein at least 70 wt.-% of the particles have an average particle diameter of less than 1.6 mm.

[0005] Within the present invention the term "biomass-material" is a "lignocellulose-containing material". The term "lignocellulose-containing material" is to be understood to comprise all kind of material known to a person skilled in the art as comprising lignocellulose. Terms "lignocellulose-containing material", "lignocellulose-containing biomass", "lignocellulosic material" and "lignocellulosic biomass" are to be understood as synonyms within the present invention. Particularly preferred lignocellulose-containing material according to the present invention include wood, cereal straw and/or husks, bagasse, oat hulls, switch grass, cellulose, raw paper pulp (obtained from pulp and paper production) and mixtures thereof. Alternative sources or additional components may comprise one or more of the following components: purified cellulose, pulp, milk whey, molasses or sugars such as glucose and lactose. In a preferred embodiment the lignocellulose-containing material contains at least 25 wt.-%, preferably at least 40 wt.-%, more preferably at least 70 wt.-%, even more preferably at least 80 wt.-% and most preferred at least 90 wt.-% lignocellulose. It is to be understood that the lignocellulose-containing material may also comprise other compounds such as proteinaceous material, starch, sugars, such as fermentable sugars and/or non-fermentable sugars.

[0006] According to step b) of the process according to the present invention, the provided lignocellulose-containing biomass-material is subjected to a comminution. The term "comminution" is thereby understood to comprise any kind of comminution known to a person skilled in the art as suitable for the inventive purpose. Within a preferred embodiment, the comminution comprises any kind of mechanical processing, maceration, shredding, grinding, chopping, crushing, cutting, irradiation, milling such as dry milling, wet milling and vibratory ball milling, and any combinations thereof. Within a particularly preferred embodiment the comminution comprises or consists of milling which is further preferably carried out by a hammer-mill. The comminution is preferably carried out in a continuous fashion.

[0007] According to step c) of the inventive process, the comminuted lignocellulose-containing biomass-material is subjected to a sifting to separate a particle-fraction consisting of particles with an average particle diameter of less than 2500 μm , preferably with an average diameter of less than 2200 μm , further preferred of less than 1800 μm and particularly preferred of less than 1600 μm , even further preferred of less than 1200 μm and most preferred of less than 800 μm . In an alternative embodiment, a particle-fraction consisting of particles with an

average particle diameter of from 1 to 2500 µm, preferably 1 to 1800 µm, particularly preferred 1 to 1600 µm and most preferred of from 1 to 1200 µm is separated within step c) of the inventive process.

[0008] Within this separation step c), it is particularly preferred to separate at least 65 % (wt. separated particle fraction /wt. initial biomass-material) of the particles with the respective average particle diameter as defined before, preferred at least 75 % (wt./wt.), further preferred at least 85 % (wt./wt.) particularly preferred at least 95 % (wt./wt.), further particularly preferred at least 99 % (wt./wt.) and most preferred 100 % (wt./wt.). Separating particles with an average particle diameter of less than 1200 µm, preferably with an average diameter of less than 1100 µm, further preferred of less than 1000 µm and particularly preferred of less than 800 µm, even further preferred of less than 300 µm and most preferred of less than 100 µm is particularly advantageous as general dust exposure and particularly the risk of dust explosion are significantly minimized.

[0009] Within the present invention the term "sifting" is to be understood as comprising any kind of separation of the respective particle fraction - as defined before - known to a person skilled in the art as suitable for the inventive purpose. Within a particularly preferred embodiment, the term "sifting" is to be understood as "sieving" and/or "filtrating". The sifting is preferably carried out within a process wherein during comminution the respective particle fraction as defined before comprising or mainly consisting of particles light enough to stay in the air are carried to a filter or sieve by pneumatic air or aspiration to separate the respective fraction according to step c) of the inventive process. Within a preferred embodiment, the respective particle fraction is separated during comminution - thus steps b) and c) are carried out concurrently, preferably in a continuous fashion - or right after comminution which is also preferably carried out in a continuous fashion.

[0010] The separation according to step c) of the inventive process is preferably carried out in a continuous fashion during comminution of the lignocellulose-containing biomass-material.

[0011] According to step d) of the inventive process, the remaining lignocellulose-containing biomass-material is subjected to a pretreatment. The term "remaining lignocellulose-containing biomass-material" is to be understood as comprising any lignocellulose-containing biomass-material not separated within the particle-fraction with an average particle diameter of less than 2500 µm within step c) of the inventive process.

[0012] Within the present invention the term "pretreatment" is to be understood as a process leading to at least partial removal and separation of hemicellulose from cellulose and disruption and removal of the lignin sheath, in order to decrease the crystallinity of cellulose and thus to increase the accessible surface area of cellulose and/or to increase the pore size of cellulose. The pretreatment preferentially mobilises the pentose fraction of the lignocellulose-containing material, while at the same time it enhances the digestibility of the solid cellulose-containing fraction.

[0013] Methods suitable for the pretreatment of the lignocellulose-containing material according to step (a) of the present invention include any kind of mechanical, biological, chemical and/or physical pretreatment methods known to a person skilled in the art. Within a preferred embodiment, the pretreatment method is selected from the methods of mechanical comminution, treatment with acids and/or alkalines, wet oxidation, pH-controlled hydrothermolysis and/or steam explosion.

[0014] "Steam explosion" according to the present invention preferably comprises a pressurised hydrothermal treatment at a temperature of from 60 to 350 °C, preferably from 80 to 300 °C, particularly preferred from 100 to 250 °C and most preferred from 110 to 220 °C of the lignocellulose-containing material in the absence or presence of acid (such as H₂SO₄, HCl, H₃PO₄) or base/alkaline (i.e. NH₄OH, NaOH, KOH, lime) catalysts, which are added at concentrations from 0.01 to 15 % (wt./wt.), preferably from 0.05 to 12.5 % (wt./wt.), more preferred from 0.1 to 10 % (wt./wt.) and most preferred from 0.25 to 7.5 %. In a preferred embodiment of the present invention the pressure is preferably selected from 1 to 100 bar, preferably from 2 to 50 bar, also preferred from 3 to 25 bar and most preferred from 5 to 15 bar. Reaction times during steam explosion have to be selected from 10s to 2h, preferably from 1 minute to 1.5 hours, and most preferred from 5 minutes to 1 hour to provide for efficient transformation of the biomass components in preparation for enzymatic hydrolysis. Within a particularly preferred embodiment a "mechanical comminution" pretreatment of the lignocellulose-containing material is carried out before or during the steam explosion pretreatment, wherein the mechanical comminution is selected from the group consisting of mechanical processing, grinding, shredding, chopping, crushing, cutting, irradiation, milling and combinations thereof.

[0015] "Acid pretreatment" according to the present invention preferably constitutes a continuous dilute and/or mild acid treatment, such as, treatment with sulfuric acid, or another organic acids, such as acetic acid, citric acid, tartaric acid, succinic acid, hydrogen chloride or mixtures thereof.

[0016] Other acids may also be used. A "mild acid treatment" according to the present invention is to be understood as carried out at a pH of from 1 to 5, preferably pH from 2 to 3 (relative to the lignocellulose-containing material). In a preferred embodiment the acid is added in concentrations from 0.01 to 15 wt.-% (wt./wt.), preferably from 0.05 to 12.5 wt.-% (wt./wt.), more preferred from 0.1 to 10 wt.-% (wt./wt.) and most preferred from 0.25 to 7.5 wt.-%. The acid is preferably sulfuric acid. The acid may be contacted with the lignocellulose-containing material at a temperature in the range of from 120 to 280 °C, preferably from 135 to 225°C and most preferred from 150 to 200 °C for a period from 1 to 60 minutes, preferably 2 to 30 minutes and most preferred from 5 to 15 minutes. Addition of strong acids, such as sulphuric acid, may be applied within particularly preferred embodiments to remove hemicellulose.

[0017] "Chemical pretreatment" according to the present invention also pertains to treatment of the lignocellulose-containing material with H₂O₂, ozone, Lewis acids, FeCl₃, (Al)₂SO₄ in

aqueous alcohols, glycerol, dioxane, phenol, ethylene glycol, NaOH, Na₂CO₃ and/or ammonia. Preferred concentrations, temperature and duration are chosen analogous to the conditions referenced above regarding acid pretreatment.

[0018] "Wet oxidation pretreatment" according to the present invention involves the use of oxidizing agents, such as sulphite based oxidizing agents.

[0019] The term "mechanical comminution" regarding the "pretreatment" refers to any mechanical treatment which promotes the separation and/or release of cellulose, hemicellulose and/or lignin from lignocellulose-containing material. Mechanical comminution is preferably selected from the group consisting of mechanical processing, grinding, chopping, shredding, crushing, cutting, irradiation, milling such as dry milling, wet milling and vibratory ball milling, and combinations thereof.

[0020] "Biological pretreatment" according to the present invention refers to any biological pretreatment which promotes the separation and/or release of cellulose, hemicellulose, and/or lignin from the lignocellulose-containing material. Biological pretreatment techniques can involve applying lignin-solubilizing microorganisms such as actinomycetes (e.g. Streptomyces strains), white rod fungi.

[0021] Pretreatment methods suitable for the process of the present invention are to be carried out within suitable devices known to a person skilled in the art. A device suitable for carrying out chemical pretreatment may be any kind of vessel such as a batch reactor. A device suitable for carrying out steam explosion may be any kind of vessel such as a batch reactor but may also be carried out within a screw reactor, preferably a continuous screw reactor.

[0022] In a preferred embodiment the solids content of the pretreated lignocellulose-containing material is up to 75 % (wt./wt.), preferably from 25 to 65% (wt./wt.) and particularly preferred from 40 to 55 % (wt./wt.).

[0023] Within the process according to the present invention before, during or after pretreatment particles are added to the lignocellulose-containing biomass-material wherein at least 70 wt.-% of these particles have an average diameter of less than 1.6 mm, preferably less than 1.4 mm, further preferred less than 1.0 mm and most preferred less than 0.75 mm. It is further preferred that at least 80 wt.-%, preferably at least 85 wt.-%, particularly preferred at least 90 wt.-%, further particularly preferred at least 95 wt.-% and most preferred at least 99 wt.-% of these particles have an average diameter of less than 1.6 mm, preferably less than 1.4 mm, further preferred less than 1.0 mm and most preferred less than 0.75 mm. The inventors of the present invention surprisingly found that the addition of particles of the respective average diameter as defined before improves the conveying of the lignocellulose-containing biomass-material during pretreatment which is particularly beneficial when the lignocellulose-containing biomass-material is conveyed during pretreatment by use of screw or spiral conveyors. The addition of these particles decreases the friction of the material within the

treatment device and during conveying through pipelines and thus contributes to the saving of energy but also decreases process time contributing significantly to reduce production costs.

[0024] The term "average particle diameter" refers to the D(0.50) value of a volumetric particle size distribution. The D(0.50) value describes the particle size of a volumetric particle size distribution where 50% of the particles are either smaller or larger than the respective value.

[0025] Within a preferred embodiment of the present invention, the particles are added in a continuous fashion during the pretreatment. It is, however, also possible to add these particles batch-wise, preferably at the beginning and/or the middle of the pretreatment.

[0026] According to the invention, from 1 to 15 wt.-% (weight of particles / weight of pretreated lignocellulose-containing biomass material) of particles are added to the lignocellulose-containing biomass-material, preferably from 1 to 12 wt.-%, further preferred from 2 to 11 wt.-% and most preferred from 3 to 10 wt.-%.

[0027] Within a further preferred embodiment, the particles are selected from the group consisting of graphite, charcoal, activated coal, silicate, clay mineral particles and mixtures thereof, wherein graphite, charcoal and/or activated coal are particularly preferred. To add particles from this group of materials is advantageous as the particles will substantially increase the energy value of the lignocellulose-containing biomass-material as the remaining or remnant material is usually used as combustible material for various applications. Suitable clay mineral particles are selected from the group consisting of bentonites and smectic sheet silicates, such as montmorillonite, aliettite, corrensite, kulkeite, lunijianlaite, rectorite, saliotite, tarasovite, tosudite, beidellite, brinrobertsite, nontronite, swinefordite, volkonskoite, yakhontovite, hectorite, ferrosaponite, saponite, sauconite, spadaite, stevensite, zincsilite and mixtures thereof.

[0028] Within another preferred embodiment of the present invention, the particles added are selected from the particle-fraction separated according to step c). It is thereby possible that all of the particles added are selected from the particle fraction separated according to step c) or only a part of the particles added are selected from the particle fraction separated according to step c), however, it is preferred that all particles separated are added to the biomass material. In case only a part of the particle-fraction separated according to step c) is added, it is preferred that at least 50 wt.-%, preferably at least 65 wt.-%, more preferred at least 95 wt.-% and most preferred at least 99 wt.-% are added. It is further preferred that from 50 to 99 wt.-% of the particles separated according to step c) are added, preferably from 65 to 99 wt.-% and most preferred from 85 to 99 wt.-%.

[0029] Within a further embodiment of the process of the present invention, it is also possible that part of the particles added are selected from the group consisting of graphite, charcoal, activated coal, silicate and clay mineral particles and another part is selected from the particles separated according to step c) of the inventive process. Within a particularly preferred embodiment at least 65 wt.-% (weight of particles from step c) / total weight of added particles)

of the particles added are selected from the particles separated according to step c), preferably at least 85 wt.-% and most preferred at least 95 wt.-% whereas the remaining amount of at most 15 wt.-%, preferably at most 5 wt.-% is preferably selected from charcoal, graphite, clay minerals and mixtures thereof.

[0030] Within a further particularly preferred embodiment, the particles added show a volumetric particle size distribution with a D(0.10) value of from 20 to 175 μm and a D(0.50) value from 250 to 450 μm . A particularly preferred particle size distribution shows a D(0.10) value of from 35 to 130 μm and a D(0.50) value from 300 to 450 μm and. The particle size distribution was measured with a 5917 Mastersizer 2000 Ver. 5.40 (Serial number MAL1015917), by Malvern Instruments, UK. In the following a further particularly preferred embodiment of the present invention is described. The particularly preferred embodiment is not to be understood as limiting the invention in any respect.

Particularly preferred embodiment

[0031] Process for the decomposition of lignocellulose-containing biomass-material selected from straw or bagasse, comprising the steps

1. (a) providing the lignocellulose-containing biomass-material;
2. (b) subjecting the lignocellulose-containing biomass-material to comminution selected from milling, shredding and grinding;
3. (c) subjecting the comminuted lignocellulose-containing biomass-material to a sifting to separate a particle-fraction consisting of particles with an average particle diameter of less than 2500 μm preferably less than 1800 μm ;
4. (d) subjecting the remaining comminuted lignocellulose-containing biomass-material to a pretreatment selected from steam explosion;

wherein during pretreatment from 8 to 10 wt.-% particles are added to the lignocellulose-containing biomass-material wherein at least 85 wt.-% of the particles added have an average particle diameter of less than 1.6 mm and are selected from the particles separated according to step c).

Example and Figure

[0032] The present invention is further described by the following example and figure. The example and figure are for illustrative purposes only and are not to be understood as limiting the invention.

Fig. 1 shows power consumption when carrying out the method with and without sifting and particle addition

Fig. 2 shows the volumetric particle size distribution of the particles added to the pretreated

biomass as described in example 1

Example 1

[0033] Rectangular wheat straw bales were transported with a conveyor system to a machine where the attached ribbons were removed. Next, the bales were loosened up in a bale crusher equipped with rotating scrappers operated at 3000 rpm yielding particles with particle sizes from 10 to 40 cm. This operation is needed to ensure a smooth transport of the straw and operation of the subsequent milling step. Next, the straw was pneumatically transported to a hammer mill operated at 3000rpm with 30mm sieves where the straw was cut to pieces with particle sizes from 1 to 5 cm. During the milling particles with a volumetric particle size distribution with a D(0.50) of 419 μm and particle sizes $<2500\mu\text{m}$ were removed with an aspiration system operated with a radial ventilator (model BWA-MXE, Reitz). The air containing these particles was then guided to a separator/filter system (bag filter model FTT 1-1-6, Hainke) where the particles were separated from the air. These particles were then put into a transport screw and transported to a buffer vessel.

[0034] The cut straw was transported to the thermal pretreatment system with a pin drum feeder follow by a transportation screw and plug screw. The screw system had an average power consumption of 12.1 kW. Next, the wheat straw was pretreated at 160°C for 5 min without addition of any chemicals. The pretreated material was collected in a cyclone after thermal pretreatment.

[0035] The particles with the volumetric particle size distribution with a D(0.50) of 419 μm (see Fig. 2) were then mixed with the cut straw fraction by introducing them into the pneumatic straw transport system. This was done by use of a rotary feeder.

[0036] This method lead to power savings in the range of from 3 to 5.4 % in power consumption (shown in Fig. 1) as the average power consumption of the screw transportation system of the thermal pretreatment unit dropped to 11.7 kW.

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- [WO2009158709A2 \[0004\]](#)

Non-patent literature cited in the description

- **VANDENBOSSCHE et al.** A new lignocellulosic biomass deconstruction process combining thermo-mechano chemical action and bio-catalytic enzymatic hydrolysis in a twin-screw extruder *Industrial Crops and Products*, 2014, vol. 55, 258-266 [\[0004\]](#)

Patentkrav

1. Fremgangsmåde til nedbrydning af lignocelluloseholdigt biomassemateriale, omfattende trinnene
- 5 (a) at tilvejebringe lignocelluloseholdigt biomassemateriale;
(b) at udsætte det lignocelluloseholdige biomassemateriale for findeling;
(c) at udsætte det findelte lignocelluloseholdige biomassemateriale for sining til separering af en partikelfraktion bestående af partikler med en gennemsnitlig partikeldiameter på mindre end 2500 μm ;
- 10 (d) at udsætte det resterende findelte lignocelluloseholdige biomassemateriale for en forbehandling, som udvælges blandt mekanisk forbehandling, biologisk forbehandling, kemisk forbehandling, fysisk forbehandling og kombinationer deraf;
hvor, før, under eller efter forbehandling, fra 1 til 15 vægt-% af partikler tilsættes til det lignocelluloseholdige biomassemateriale, hvor mindst 70 vægt-% af de tilsatte partikler har en gennemsnitlig partikeldiameter på mindre end 1,6 mm.
- 15
2. Fremgangsmåde ifølge krav 1, hvor de tilsatte partikler tilsættes på en kontinuerlig måde under forbehandlingen.
- 20
3. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, hvor de tilsatte partikler udvælges fra gruppen bestående af grafit-, trækul-, aktiveret kul-, silikat- og lermineralpartikler.
- 25
4. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, hvor de tilsatte partikler udvælges blandt partikelfraktioner ifølge trin c).
- 30
5. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, hvor en partikelfraktion bestående af partikler med en gennemsnitlig partikeldiameter på mindre end 1600 μm separeres i henhold til trin (c).
- 35
6. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, hvor mindst 85 vægt-% af de tilsatte partikler har en gennemsnitlig diameter på mindre end 1,6 mm.

7. Fremgangsmåde ifølge et hvilket som helst af de foregående krav, hvor de tilsatte partikler udviser en volumetrisk partikelstørrelsesfordeling med en D(0,10)-værdi på fra 20 til 175 μm og en D(0,50)-værdi fra 250 til 450 μm .

DRAWINGS

Fig. 1

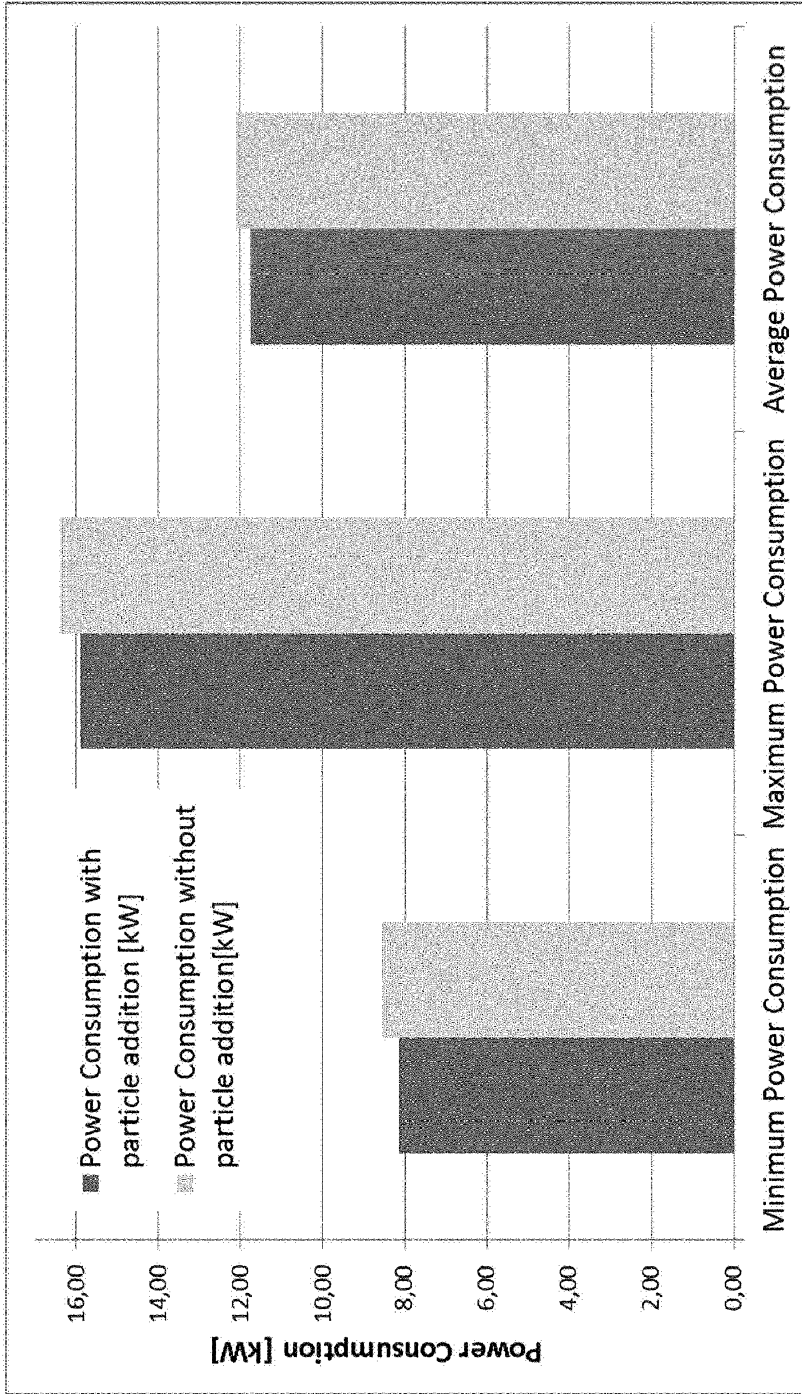


Fig. 2

