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(54) Benævnelse: **FREMGANGSMÅDE TIL BEHANDLING AF LIGNOCELLULOSEHOLDIG BIOMASSE**

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Description

Field of the invention

The invention relates to a process for the treatment of lignocellulosic biomass for producing "second-generation" (2G) sweet juice. This sweet juice may be used to produce other products via a biochemical pathway (as an example: alcohols such as ethanol, butanol or other molecules, for example solvents such as acetone, etc). This process generally comprises three steps, namely the preparation of liquor, impregnation of the biomass and pretreatment of the impregnated biomass, for example by cooking, optionally coupled with steam explosion.

Prior art

Lignocellulosic biomass represents one of the most abundant renewable resources on Earth. The substrates considered are very varied, they concern both ligneous substrates such as various woods (hardwoods and softwoods), co-products derived from agriculture (wheat straw, corn cobs, etc) or other agrifood, paper, etc industries.

The process for the biochemical conversion of the lignocellulosic material into 2G sweet juice in particular comprises a pretreatment step and a step for enzymatic hydrolysis with a cocktail of enzymes. These processes also usually comprise an impregnation step before the pretreatment. The sweet juice resulting from the hydrolysis is then treated, for example by fermentation, and the process also comprises separation steps and/or a step for purification of the final product.

Lignocellulosic biomass is composed of three principal polymers: cellulose (35% to 50%), which is a polysaccharide essentially constituted by hexoses; hemicellulose (20% to 30%), which is a polysaccharide essentially constituted by pentoses; and lignin (15% to 25%), which is a polymer with a complex structure and a high molecular weight, composed of aromatic alcohols connected by ether bonds. These various molecules are responsible for the intrinsic properties of the plant wall and organize themselves into a complex entangled structure.

Of the three base polymers that make up lignocellulosic biomass, cellulose and hemicellulose are those which can be used for the production of 2G sweet juice.

Usually, hemicellulose is predominantly broken down into sugar during pretreatment and cellulose is converted into glucose by enzymatic hydrolysis. However, access to crude cellulose remains difficult for enzymes to access, hence the need for a pretreatment. This pretreatment makes it possible to modify the physicochemical properties of the lignocellulosic material in order to improve the accessibility of the cellulose to enzymes and its reactivity to enzymatic hydrolysis.

Numerous technologies which are advantageous to the invention for carrying out this pretreatment exist; they shall be grouped hereinbelow under the generic term "cooking": acidic cooking, alkaline cooking, steam explosion, organosolv pulping processes. The latter process concerns a pretreatment in the presence of one or more organic solvents and generally water. The solvent may be an alcohol (ethanol), an acid such as acetic acid or formic acid, or indeed acetone. Organosolv pulping processes lead to at least partial dissolution of lignin and partial dissolution of hemicelluloses. There are thus two outlet streams: the pretreated substrate with residual cellulose, hemicellulose and lignin, and the solvent phase which contains the dissolved lignin and a portion of the hemicelluloses. There is generally a step for regeneration of the solvent, which makes it possible to extract a lignin stream. Certain organosolv pulping treatments (in particular with ethanol) are coupled with the addition of a strong acid (of the H_2SO_4 type). Bringing the biomass into contact with the solvent via an impregnation reactor before the cooking phase or bringing the biomass into contact with the acid catalyst before carrying out organosolv pulping cooking may also be envisaged.

Various configurations have been reported, for example, in the document "Production of bioethanol from lignocellulosic materials via the biochemical pathway: A review", M. Balat, Energy Conversion and Management 52 (2011) 858-875, or in fact in the document "Bioethanol production from agricultural wastes: an overview", N. Sarkar, S. Kumar Ghosh, S. Bannerjee, K. Aikat, Renewable Energy 37 (2012) 19-27.

One of the most effective pretreatments is steam explosion under zoneic conditions, which means that almost complete hydrolysis of hemicellulose and a significant improvement in the accessibility and reactivity of cellulose with respect to enzymes can be obtained. This pretreatment may be preceded by other treatment(s).

All these pretreatments are applicable to biomasses which are initially in the solid form: the aim of the pretreatment is to destructure them.

One of the most effective pretreatments is steam explosion, which enables almost complete hydrolysis of hemicellulose and a significant improvement in the accessibility and reactivity of cellulose with respect to enzymes. This pretreatment may be preceded by other treatment(s).

Patents US 8 057 639 and US 8 512 512 propose a process comprising a first step for hydrolysis of hemicellulose to C5 sugars under mild conditions which therefore protect them from degradation. This step is carried out in a first reactor at a pressure of 1.5 bar or more by injection of steam, at a temperature of 110°C or more, and optionally in the presence of a weak acid. After this step, washing is carried out in order to extract and recover the C5 sugar liquor before sending the remaining biomass, which is enriched in cellulose and lignin, to a second step (second reactor) where steam explosion takes place. This second reactor operates at a higher pressure than that of the first reactor with an injection of high-pressure steam which causes a sudden expansion of the biomass (steam explosion).

Patent application WO 2013/141776, in the papermaking field, describes an impregnation process in a vertical reactor (impregnator) containing basic impregnation liquor, defining therein a first zone in which the impregnation is carried out. The lignocellulosic material is received at the bottom of the impregnator, and it is transferred to the top of the impregnator by means of two screw conveyors. During its transfer into the second zone of the impregnator located above the level of the liquid, the biomass drains and the liquid falls back into the first zone. In this configuration, the liquid level is controlled by the introduction of basic liquor.

When a treatment requires a pressure step (impregnation, pretreatment of the cooking type or the like), it is necessary to make use of means for introducing solid biomass which are compatible with these pressure steps. This is, for example, the case of compression screws, one embodiment of which is described in patent application CA 2 77322.

The aim of the invention is thus to improve the treatment of lignocellulosic biomass. In particular, the aim of the invention is to improve the introduction of the biomass into a reactor carrying out one or more of the biomass treatment steps.

More particularly, the aim of the invention is to improve the steps for biomass impregnation and steam explosion as described in the aforementioned prior art documents. An aim of the invention is also to make the treatment process, and in particular these two steps, more efficient in terms of energy and/or treatment fluid consumption.

Throughout the present text, the abbreviation "SC" denotes the solids content, which is measured according to the standard ASTM

E1756-08(2015) "Standard Test Method for Determination of Total Solids in Biomass".

Summary of the invention

The subject matter of the invention is, firstly, a process for treating a lignocellulosic biomass or a lignocellulosic substrate comprising a solids content SC of not more than 90% by weight, said process comprising the use of at least one reactor for treating said biomass, in which the or at least one of said reactors is fed with biomass via a feed means generating a pressure increase between the biomass inlet and the biomass outlet of said feed means, and in which said feed means is washed by circulation of a washing fluid between a washing inlet and a washing outlet.

In accordance with the invention, at least a portion of the washing fluid exiting the fluid outlet of the or of at least one of the feed means is reintroduced into the washing inlet of the same feed means or of another of said feed means.

In accordance with the invention, the term "lignocellulosic biomass comprising a solids content of X%" means either a biomass which naturally comprises a solids content of X% (a "native" biomass) or a biomass which has this content after one or more operations prior to the process in accordance with the invention.

It in fact proved to be very advantageous to thus "recycle" all or a portion of the washing water of the feed means which may, for example, be a feed screw: at the very least, when the biomass does not contain any catalyst, a saving in washing fluid (water) consumption is obtained. It is also possible to obtain a saving in energy when this washing water has to be heated before introduction into the washing circuit, since a "spent" fluid which already has a temperature above ambient temperature is then reinjected into the washing circuit. In addition, when it is a matter of washing a feed means for biomass supplemented with a catalyst (for example after an operation of impregnation with a catalytic liquor using an acidic, basic or oxidizing catalyst), it has been shown that the washing water leaving the feed means could contain a certain content of catalyst (when the biomass undergoes a liquid extraction by passing through the feed means), but that this presence of "spent" washing water also containing a catalyst at the inlet of the washing circuit did not cause any problems.

It should be noted that the implementation of the invention is simple: the facility simply needs to envisage one or more additional pipes for ensuring direct fluid connection between the washing outlet and the washing inlet of the same feed means or between the washing outlet of one feed means with the inlet of another means.

In accordance with the invention, it is also possible to retain the possibility of purging the optional portion of exiting washing water which is not recycled.

The lignocellulosic biomass treated in accordance with the invention has a solids content SC of between 5% and 80% by weight. Preferably, it contains not more than 75%, in particular not more than 70% or 65% by weight, and in particular less than 50% by weight of solids.

The biomass treated in accordance with the invention thus preferably has a certain moisture content before introduction into the feed means. This water may be naturally present, as is the case, for example, for a biomass in the form of wood chips. It may also be in naturally low or very low quantities, as may be the case for wheat or corn straw. If the moisture content is too low, the biomass may be humidified beforehand to reach a solids content of not more than 90% or 80% which is desirable for the implementation of the invention.

The feed means in accordance with the invention places the biomass under compression, the general aim of this being to introduce the biomass into a zone (a reactor) at higher pressure.

This compression, from this pressure increase in the feed means, may bring about extraction or expulsion of at least a portion of the liquid contained in the biomass, which optionally becomes mixed with the washing fluid at the fluid outlet from the feed means. Optional extraction of at least a portion of the liquid contained in the biomass during its passage through the or one of the feed means may thus be carried out; this is a consequence of the choice of the biomass feed means.

Thus, if the solids content of the biomass is high, or rather in the upper values of the range mentioned above (for example of the order of 70% or 75% to 90%), there will be little or no extraction of fluid from this biomass out of the extraction means via the washing fluid outlet, and the invention in particular makes it possible to recycle the washing fluid injected into the feed means.

In addition, if the solids content is lower, or rather in the lowest values of the range mentioned above (for example below 70%, 65% or even below 50%), then there will be a certain amount of extraction of fluid from the biomass, and the invention then makes it possible to further reduce the supply of washing fluid, or even to eliminate it, washing then being able to take place entirely or in part with the fluid extracted from the biomass.

Preferably, the solids content SC of the lignocellulosic substrate leaving the feed means is then greater than or equal to 20% by weight, and preferably greater than or equal to 40% by weight.

Preferably, the pressure increase generated in the or at least one of the feed means is at least 0.05 MPa, and is in particular between 0.05 and 4 MPa, for example between 0.1 and 2.5 MPa.

This or these feed means are, for example, in the form of a feed screw, which is in particular at least partly conical, comprising a cowling equipped with a grate allowing circulation of the washing fluid. The conical shape of the general end portion of this type of screw will thus compress the biomass, creating a pressure difference. This type of feed means has a major advantage: the screw makes it possible to form a hermetic plug of biomass which provides a seal and in particular prevents any surges of liquid from the reactor into which it emerges.

An example of this type of screw is described in the document CA 2 77322 cited above.

The invention is therefore of application to any treatment of biomass in a reactor using a pressurized biomass feed means, and in particular any treatment at a pressure above atmospheric pressure or above the pressure which the biomass is at before introduction into said reactor.

In accordance with one embodiment, the process of the invention comprises a step for impregnating the biomass with an impregnation liquor containing a chemical catalyst. This step may be followed (directly or by envisaging one or more intermediate steps) by a cooking step, this cooking step being carried out by the/by one of the reactors equipped with the feed means.

In accordance with one embodiment, the process of the invention comprises a step for impregnating the biomass with an impregnation liquor containing a chemical catalyst by passing the biomass through a bed of liquor or by spraying the biomass with the liquor on a conveyor of the belt type or by passing the biomass through a stirred reactor.

In accordance with another embodiment, the process of the invention comprises a step for impregnating the biomass with an impregnation liquor containing a chemical catalyst, by introduction into a reactor via said or at least one of said feed means.

In accordance with one embodiment, the process of the invention comprises a step for treating the biomass by cooking, in particular in the or one of the reactors equipped with a feed means.

One example of the implementation of the process of the invention is as follows: it involves treating a lignocellulosic biomass, said process comprising the following steps:

a. Preparing an impregnation liquor containing a chemical catalyst intended for the impregnation of the biomass, chosen from an acid catalyst, a basic catalyst and an oxidizing catalyst, and preferably an acid catalyst, in a preparation zone

b. Introducing the milled biomass via an inlet of an impregnation reactor by means of a first feed means, said first feed means being washed by circulation of a first washing fluid between an inlet and an outlet of said means

c. Introducing the liquor into the impregnation reactor via a first liquor inlet of the reactor

d. Transferring the impregnated and drained biomass from an impregnation reactor outlet to an inlet of a cooking pretreatment reactor, the introduction into said pretreatment reactor taking place via a second feed means, said second feed means being washed by circulation of a second washing fluid between an inlet and an outlet of said feed means

e. Pretreating said biomass in said cooking reactor.

In addition, the process also comprises the following step or steps:

f. Reintroducing at least a portion of the first and/or of the second washing fluid leaving the fluid outlet of said first or of said second feed means into the washing inlet of the first feed means and/or of the second feed means.

Preferably, the impregnation and the steam cooking pretreatment take place continuously. However, the invention also applies to a process in which these two steps are discontinuous, in batch mode. Preferably, cooking is followed by steam explosion, but this explosion step remains optional.

Preferably, the impregnation in this embodiment may be carried out in a reactor the biomass inlet of which is located in a first impregnation zone of said impregnation reactor which comprises two superposed zones, said first impregnation zone and a second "draining" zone which is above the impregnation zone and which is equipped with an outlet for impregnated and drained biomass. More preferably in this reactor configuration, the introduction of the liquor into said reactor takes place via a first liquor inlet of the reactor which is located in said first impregnation zone of the reactor.

In this example, therefore, the invention proposes recycling or re-using all or a portion of the washing fluids from the two feed screws. This is particularly advantageous because it has been shown that one and/or the other of the washing fluids, or both of them, can be recycled without the quality of the impregnation or the quality of the cooking being affected in any way.

In addition, this recycling may take place in various manners, in a highly flexible fashion, and in fact for each of the washing fluids:

Thus, the washing fluid of the feed means for the impregnation reactor is generally based on water, and thus "spent" water is predominantly recovered at the outlet of the feed means. Recycling this spent water by reintroducing it into the inlet of one or the other of the feed means of the screws means that the water consumption for the process is significantly reduced. When, in addition, this water is heated, an energy saving is also observed since the washing water then requires less heating overall. Circulation of washing fluid in a closed loop is thus highly advantageous.

As regards the washing fluid of the feed means of the cooking pretreatment reactor, the situation is slightly different because the washing fluid, which is also generally water, will have a tendency to become loaded with catalyst on washing the feed means which transports the biomass already impregnated with the catalytic liquor: expulsion of a portion of the liquid from the impregnated biomass containing the catalyst takes place in this screw, and this liquid becomes mixed, at the screw outlet, with the washing water therefrom. This is then usually referred to as "press liquor" at the outlet of this feed means. This press liquor may also be recycled, either to return to the washing inlet of the feed means of the pretreatment reactor, or to go to the washing inlet of the first feed means. Even when, in addition, the impregnation liquor is very acidic, and when the press liquor therefore is as well, but to a lesser extent, this does not pose any problem as regards the equipment, since the biomass feed means of the impregnation reactor is generally designed to withstand contact with the catalytic liquor without deterioration (to combat any surges of liquor into the biomass feed means).

In both cases, it remains possible for some of the spent water and/or press liquor to be purged as well. These two cases are, in addition, alternative or cumulative.

In accordance with a preferred embodiment, the process of the invention also comprises the reintroduction of a portion of the first and/or of the second washing fluid leaving the fluid outlet of the first or the second feed means into the impregnation liquor preparation zone for the purposes of step a). for preparing said liquor.

In accordance with the requirements of the process, it is possible to adjust and to vary, as required, the proportion of the exiting washing fluid which will be reintroduced into the feed means with respect to the proportion which is used for preparing the catalytic liquor or indeed with respect to the proportion which is purged.

In the case in which a portion of the press liquor which is reintroduced into the liquor preparation zone, the advantage is twofold, since the consumption of water for the preparation of the liquor is reduced, along with the consumption of catalyst, since the press liquor contains it. The liquor preparation step generally takes place with a variety of regulation means as regards certain physicochemical parameters, such as the temperature, the catalyst content in the liquor (for example by regulating the pH when acid or basic catalysis is involved), this supply of additional catalyst may therefore be taken into account by these regulation means in order to maintain the desired characteristics for the liquor.

Washing the first and/or second feed means may take place continuously or discontinuously, i.e. with permanent washing or washing at given time intervals, for example. The washing circulation flow rate may also vary, in particular as a function of the flow rate of the weight of biomass introduced into one or other of the reactors.

Reintroducing a portion of the first and/or of the second washing fluid leaving the fluid outlet of said first or of said second feed means into the washing inlet of the first feed means and/or of the second feed means may also take place continuously or discontinuously.

In accordance with one embodiment, a portion of the first washing fluid at the washing outlet of the first feed means is reintroduced into the washing inlet of the first or of the second feed means, and another portion of said washing fluid is reintroduced into the liquor preparation zone for the purposes of preparing it.

In similar manner, according to a second embodiment that is compatible with the preceding one, a portion of the second washing fluid at the washing outlet of the second feed means is reintroduced into the washing inlet of the first or of the second feed means, and another portion of said washing fluid is reintroduced into the liquor preparation zone for the purposes of preparing it.

In accordance with one embodiment, when washing of the feed means is carried out in the presence of biomass supplemented with a catalyst, in particular an acid, basic or oxidizing catalyst, the washing fluid leaving the fluid outlet of said feed means contains a certain content of said catalyst when the biomass undergoes liquid extraction by passing through said feed means, and at least a portion of said washing fluid containing this catalyst is reintroduced into the washing inlet of the same feed means or of another of said feed means, in particular conveying biomass which is free of catalyst. The term "catalyst" should be understood here in the broad sense to mean any reagent which is capable of interacting with the biomass, in particular for the purposes of modifying one of its physicochemical or rheological properties. With the invention, it has therefore been ascertained that it is possible to recycle washing water, even when supplemented with such a catalyst, to reintroduce it either into the same feed means, or into another, which in turn transports only biomass: the fact that the reintroduced washing water is not pure water but water supplemented with catalyst has been shown to have no harmful impact on the process as a whole, which was unexpected.

The invention also concerns the facility carrying out the process described previously, in particular a facility which comprises at least one reactor for treating a lignocellulosic biomass having a solids content of not more than 90% by weight with feeding with biomass of said or of at least one of said reactors by a feed means creating an increase in pressure between the biomass inlet and the biomass outlet of said feed means. Washing said feed means is carried out by circulating a washing fluid between a washing inlet and a washing outlet, and a means for reintroducing at least a portion of the washing fluid leaving the fluid outlet of the or of at least one of the feed means into the washing inlet of the same feed means or of another of said feed means.

The invention also concerns an embodiment of a lignocellulosic biomass treatment facility, comprising:

- A zone for preparing an impregnation liquor containing a chemical catalyst for the impregnation of the biomass, chosen from an acid, basic or oxidizing catalyst, and preferably an acid catalyst, and equipped with a liquor outlet

- An impregnation reactor, in particular comprising a first impregnation zone equipped with a biomass inlet and a second zone superposed on the first, termed the draining zone, said reactor being equipped with a biomass inlet and outlet

- A first means for feeding milled biomass to the impregnation reactor via the biomass inlet of the impregnation reactor, in particular located in the first impregnation zone

- A means for feeding impregnation liquor to the reactor connecting a liquor outlet of the liquor preparation zone to a first liquor inlet in the impregnation reactor, in particular in its first impregnation zone

- A reactor for pretreatment of the impregnated biomass by steam explosion, a biomass inlet of which is connected to the biomass outlet of the impregnation reactor

- A second means for feeding impregnated biomass to the pretreatment reactor via the biomass inlet of said pretreatment reactor

- A circulation of washing fluid between an inlet and an outlet of the first milled biomass feed means of the impregnation reactor

- A circulation of washing fluid between an inlet and an outlet of the second impregnated biomass feed means of the pretreatment reactor

the facility also comprising

- A means for reintroducing at least a portion of the first and/or of the second washing fluid leaving the fluid outlet of said first or of said second feed means into the washing inlet of the first feed means and/or of the second feed means.

Advantageously, the facility also comprises a means for reintroducing a portion of the first and/or of the second washing fluid leaving the fluid outlet of the first or the second feed means into the impregnation liquor preparation zone.

Advantageously, it may envisage a means for reintroducing a portion of the first washing fluid at the washing outlet of the first feed means into the washing inlet of the first or of the second feed means, and a means for reintroducing another portion of said fluid into the liquor preparation zone.

The facility in accordance with the invention may also envisage a means for reintroducing a portion of the second washing fluid at the washing outlet of the second feed means into the washing inlet of the first or of the second feed means, and a means for reintroducing another portion of said fluid into the liquor preparation zone.

These reintroduction means are advantageously pipes or a set of pipes enabling the desired hydraulic connection, the production of which is known per se, to be made.

The invention also concerns the use of the process or of the facility described above for the treatment of any lignocellulosic biomass such as wood, straw, agricultural residues, and all dedicated energy crops, which may be annual or perennial plants such as miscanthus, with a view to the production of sugars, biofuels or biosourced molecules.

Detailed description

The invention will be described in detail below with the aid of non-limiting examples of the process in accordance with the invention, illustrated by the following figures:

Figure 1: a diagrammatic overview, of block diagram type, of the general principle of the biomass treatment process in accordance with the invention,

Figure 2: a diagrammatic overview, of block diagram type, of a first embodiment of the biomass treatment process in accordance with the invention,

Figure 3: a diagrammatic overview, of block diagram type, of a second embodiment of the biomass treatment process in accordance with the invention,

Figure 4: a diagrammatic overview, of block diagram type, of a third embodiment of the biomass treatment process in accordance with the invention.

Figure 1 is therefore the representation of the principle of the invention, Figure 2 concentrates on two biomass treatment steps: catalytic impregnation and then cooking. Figures 3 and 4 describe the whole process for treating biomass up to its enzymatic hydrolysis, after impregnation and cooking.

The invention is more particularly focussed on the first two steps, namely impregnation of the milled biomass and treatment by cooking of the biomass once it has been impregnated.

Description of the figures using the following references

The same references correspond to the same components/fluids/products on all of the figures:

1: Entry of water into the liquor preparation tank

2: Entry of acid into the liquor preparation tank

3: Liquor preparation tool (tank)

4: Acidic liquor to the impregnation tool (reactor)

5: Milled biomass

6: Screw or plug-screw feeder of the impregnation tool

7: Water for washing the plug-screw feeder of the impregnation tool

8: Washing liquid outlet of the plug-screw feeder of the impregnation tool

9, 9': Impregnation tool (reactor)

9a: Impregnation zone of the impregnation tank 9

9b: Draining zone of the impregnation tank 9

10: Impregnated and drained biomass

11: Screw or plug-screw feeder of the pretreatment tool

12: Water for washing the plug-screw feeder of the pretreatment tool

13: Press liquor of the plug-screw feeder of the pretreatment tool

13.a: Press liquor to the purge

13.b: Recycle for the press liquor for washing the plug-screw feeder of the pretreatment tool

13.c: Recycle for the press liquor into the acidic liquor preparation tank 3

13.d: Recycle for the press liquor for washing the plug-screw feeder of the impregnation tool

14: Pretreatment cooking tool (explosion reactor)

15: Injection of steam for the pretreatment

16: Pretreated biomass and steam

17: Tool (cyclone) for separating steam and pretreated biomass

18: Steam to condensation

19: Pretreated biomass

20: Enzymatic hydrolysis

21: Enzymatic hydrolysis must containing sugars

22: Alcoholic (ethanolic) fermentation

23: Fermentation wine containing ethanol (alcohol)

24: Distillation

25: Concentrated alcohol

26: Crude vinasse

Figure 1 very diagrammatically represents the principle of the invention in its simplest embodiment (the details and various steps of this process will be subsequently described with the aid of Figures 2 to 4): the biomass 5, 10 (impregnated or not impregnated with a catalytic liquor) is introduced into a reactor 9, 14 for a treatment, by a pressurized feed means 6, 11 which is a screw (also known as a plug screw feeder) the end portion of which is conical, which has a cowling with a draining grate, a washing water inlet 7, 12 and a washing water outlet 8, 13. A hermetic biomass plug is generated in the downstream portion of the screw, which generates compression on the biomass reflected by a pressure difference between the biomass inlet and the biomass outlet of the screw of at least 0.05 MPa, for example about 0.5 MPa. The compression being applied to the biomass may lead to the expulsion of a portion of the liquid contained in the biomass, in particular when the SC of the biomass is less than 80% before it enters the pressurized feed means 6, 11. The liquid which is thus extracted becomes mixed with the washing water and is withdrawn with the spent washing water 8, 13.

In accordance with the invention, at least a portion of the washing water leaving the screw 8, 13 is reinjected into the washing water inlet 7, 12 of the screw. It should be noted that it remains possible for a portion of the exiting washing water to be simply purged, and for a portion of the entering washing water to originate from a water supply as in the prior art, in order to supplement the amount of water recycled into the screw inlet, periodically or systematically.

In this representation the washing water recycle is applied to the same screw but, as will be detailed with the other figures, the invention also relates to complete or partial recycling of the washing water at the outlet of one screw to the inlet of another feed screw, when the process envisages several feed screws for several reactors.

The solids content of the biomass entering the feed screw is 50% in this case. It may occur that a portion of the liquid of the biomass is extracted during its transportation in the screw, this extracted water becoming mixed with the spent washing water at the washing outlet of the screw. The lower the solids content of the biomass, the more extraction of fluid there will be from the biomass in the feed screw, this fluid supplementing or even possibly completely replacing the washing fluid at the screw inlet.

Figure 2 represents a step for impregnation of the biomass with an acidic liquor, followed by a cooking step forming part of a biomass treatment:

The process proceeds in the following manner: The water and the acid are introduced into the acidic liquor preparation tank 3 via pipes 1 and 2 respectively. The acidic liquor from pipe 4 is then injected into an impregnation device 9′ to be mixed with the milled biomass. The impregnation device in this case is a conveyor, of the belt conveyor type driven in translation by the rotation of rollers supporting the belt, the biomass being placed on the belt to a given thickness. While it is being conveyed, the biomass is subjected to spraying with acidic liquor taken from the tank 3, via nozzles placed above the belt. The excess liquor may be recovered under the belt in a collection zone, to be optionally totally or partly recycled.

The impregnated biomass 10, which has optionally been drained beforehand, is introduced into the treatment unit 14 by means of another feed screw of "plug screw feeder" type 11 which compresses the biomass to form a biomass plug. This hermetic biomass plug provides a seal and prevents the escape of steam, when the cooking reaction in this biomass reactor 14 takes place by steam explosion. During this compression in the screw 11, a solid/liquid separation takes place, and the spent acidic liquor is evacuated through the cowling grate of the screw. This screw is washed with water from the pipe 12, which then becomes mixed with spent acidic liquor, and the mixture of washing water and of spent acidic liquor (known as press liquor) is evacuated in the pipe 13. The press liquor may also contain solid obtained from washing the screw 11.

Optionally, a solid/liquid separation step is carried out on this stream 13, for example with a device using a grate with a curved shape.

The stream 13 can then be directed towards:

- a purge via pipe 13a

- a recycle via pipe 13b in order to replace all or a portion of the water 12 for washing the screw 11

- a recycle into the acidic liquor preparation tank 3 via pipe 13c;

Various possibilities then become available:

- in accordance with one embodiment, all of the stream 13 is sent into pipe 13b to replace all or a portion of the washing water 12,

- in accordance with another embodiment, only a portion of the stream 13 is sent to the pipe 13b to replace all or a portion of the washing water 12, and the rest is sent via the pipe 13c to contribute towards the manufacture of liquor (for example in a proportion by volume between the two which varies between 20/80 and 80/20).

The biomass is treated in the treatment tool 14 by cooking and steam explosion in the manner described in the following figures.

Figure 3 presents a variation compared with Figure 2 in the manner of carrying out the impregnation of the biomass, and it also represents the steps subsequent to the cooking/steam explosion of the biomass.

Only the differences with the process according to Figure 2 will be described here, the two processes otherwise being carried out in an identical manner.

The water and the acid are introduced into the acidic liquor preparation tank 3 via the pipes 1 and 2 respectively. The acidic liquor from pipe 4 is then injected into an impregnation device 9 to be mixed with the milled biomass. In this case it is a reactor which is positioned substantially vertically here.

The pre-milled biomass is introduced into the process via pipe 5 into a feed screw 6, similar to the screw 11 already described in the preceding figure, also with a water washing circuit. This screw makes it possible to form a hermetic biomass plug which ensures the seal and prevents the escape of acidic liquor. This screw is washed with water which enters via the pipe 7 and leaves via the pipe 8. At the screw outlet, expansion of the plug takes place in the bottom zone of the impregnation reactor 9. The biomass becomes impregnated with acidic liquor in the impregnation zone 9a before being drained in the draining zone 9b. It is conveyed into the reactor 9 via one or two conveying screws.

The stream 13 can then be directed, as previously, towards:

- a purge via pipe 13a

- a recycle via the pipe 13b to replace all or a portion of the water 12 for washing the screw 11

- a recycle into the acidic liquor preparation tank 3 via the pipe 13c;

- but another possibility also becomes available because in this case, the stream 13 can also be recycled via the pipe 13d to replace all or a portion of the water 7 for washing the screw 6.

Continuing with the more detailed description of the reactor 14 common to Figures 2 to 4 and to the steps following the treatment in this reactor 14:

The steam required for heating the reactor 14 is introduced via the pipe 15. At the reactor outlet, the biomass/steam mixture is expanded and conveyed via the line 16 to the separation tool 17. The separation tool 17 may be of the cyclone type: it can separate the steam 18 from the pretreated biomass 19.

The pretreated biomass is then transformed in the transformation tool 20 into a must 21 containing sugars by using a cocktail of enzymes. The sugars are converted into alcohol (for example ethanol, acetone, butanol) by fermentation in the fermentation step 22. The fermentation wine 23 is sent to a separation and purification step 24. Step 24, which is carried out by distillation, for example, enables a stream 25 containing the concentrated alcohol to be separated from the crude vinasse (spent water, residual lignin) 26.

The details of the operating conditions for the steam explosion, must conversion and fermentation steps, which are considered to be known to the person skilled in the art, will not be provided here.

Figure 4 shows a variation compared with Figure 3

The additional references with respect to Figure 3 are:

8: Spent washing liquid outlet of the impregnation tool screw 6

8.a: Spent washing liquid to the purge

8.b: Recycled washing liquid for washing the impregnation tool screw

8.c: Washing liquid recycled to the acidic liquor preparation tank 3

8.d: Recycled washing liquid for washing the screw 11 of the pretreatment tool 14

Only the differences with respect to the process according to Figure 3 will be described here, the two processes otherwise being carried out in identical manner:

In this case, the water 8 for washing the feed screw 6 is also recycled in accordance with various alternative or cumulative possibilities. The washing liquid 8 for the transfer screw 6 may be either:

- purged from the system via the pipe 8a

- recycled via the pipe 8b to replace the washing water 7 for the feed screw 6 of the impregnation reactor 9

- recycled into the acidic liquor tank 3 via the pipe 8c

- recycled via the pipe 8d to replace the washing water 12 for the feed screw 11 of the explosion tool/reactor 14.

In accordance with another embodiment of the invention, all of the stream 8 is sent to the pipe 8b or to the pipe 8d to replace all or a portion of the washing water 12 or 7. In accordance with another embodiment, a portion is sent to the pipe 8b or 8b, and the rest is sent via the pipe 8c to contribute towards the preparation of the impregnation liquor. The ratio between the two portions, by volume, may be between 80/20 and 20/80.

In any case, both as regards recycling either the spent water from the screw 6 or the press liquor from the screw 11, a portion may also be taken to be purged (pipes 13a, 8a).

Naturally, it is also within the context of the invention to combine recycling the spent water from washing the screw 6 with that of the press liquor from the screw 11, or to recycle only one or the other.

Some operating details or variations are given below for the first two biomass treatment steps described above:

<u>The impregnation step</u> is carried out at a temperature ranging from 10°C to 95°C, and the residence time for the biomass in said impregnation step is comprised between 20 seconds and 12 hours. Preferably, the residence time for the biomass is comprised between 30 seconds and 60 minutes.

The impregnation step may be carried out in batch or in continuous mode. Preferably, this step is carried out in continuous mode. The impregnation step is preferably carried out at atmospheric pressure. Preferably, the impregnation step is carried out in a single step.

The impregnation step involves placing the biomass and the acidic liquor in contact. It may, for example, be carried out by dipping or by spraying.

The impregnation step is carried out in equipment which is known to the person skilled in the art, for example in a stirred reactor, by horizontal or vertical passage of the biomass through a bed of liquor, by spraying onto a belt conveying the biomass (as seen in Figure 2) or in a conveying screw.

The impregnation reactor or impregnator is generally equipped with one or more tools which transfer(s) the lignocellulosic substrate from its entry to the outlet aperture. These tools may, for example, be screws or belts. The impregnator is, moreover, equipped with one or more pipes for conveying the acidic liquor and also, if needs be, with one or more pipes for withdrawing acidic liquor. Said acidic liquor inlet and outlet pipes are generally installed so as to function by co-current or counter-current recycling.

<u>The pretreatment step</u> (cooking):

The lignocellulosic substrate undergoes this pretreatment step, which is in particular directed towards modifying the physical and physicochemical properties of the cellulosic fraction, such as its degree of polymerization and its crystalline state.

The pretreatment includes a cooking zone. This cooking is carried out at a temperature comprised between 100°C and 250°C, and more preferably comprised between 130°C and 230°C, at a pressure of between 0.1 and 4 MPa. The residence time in the cooking zone is comprised between 10 seconds and 4 hours, and more preferably between 3 minutes and 1 hour.

Cooking may take place in batch or continuous mode. It may be carried out in any equipment known to the person skilled in the art, for example a stirred reactor, a horizontal tubular reactor equipped with a conveying screw, a non-stirred batch reactor, etc. The thermal energy required for cooking may be supplied by means of an exchange of heat with a heat-transfer fluid (indirect), by electrical heating, or by direct injection of a hot fluid, for example pressurized water or steam.

Exit of the solid at the end of cooking may take place by rapid decompression, by slow decompression, after a temperature reduction induced by direct or indirect heat exchange, etc.

In one embodiment (preferred), the cooking zone is heated with steam by direct injection and is followed by abrupt decompression of the medium, this process being known as steam explosion (or "steamex"). This is a process in which the lignocellulosic substrate is rapidly brought to a high temperature by injecting pressurized steam. The treatment is stopped by abrupt decompression.

The operating conditions for the steam explosion process are as follows:

- steam is injected directly into the reactor;
- the temperature of the reactor is generally comprised between 150°C and 250°C, preferably between 160°C and 220°C,
- the pressure is comprised between 0.5 and 2.5 MPa, more preferably between 0.8 and 2.0 MPa,
- the residence time before the expansion phase ranges from 10 seconds to 25 minutes, and preferably between 3 minutes and 15 minutes.

Steam explosion may be carried out in batch or continuous mode and the depressurization step which permits destructuring of the biomass may proceed in one or more steps.

Exemplary embodiments of this or these recycling operations using the process according to Figure 4 will now be described:

Comparative Example 1

This is an example with a recycle of only the spent washing waters as make-up water for the liquor preparation tank. The biomass to be treated is wood having the following characteristics:

Feedstock: Poplar wood, flow rate 7.47 tonne/hour, solids content (SC): 50%, mean composition (based on SC):

	% (based on SC)
Cellulose	42.6%
Hemicellulose	20.5%

Lignin and others (ash, extractable matter, etc)	36.9%

The acronym "SC" denotes the solids content, which is measured in accordance with the standard ASTM E1756-08(2015) "Standard Test Method for Determination of Total Solids in Biomass".

The wood is used in the form of chips with a characteristic dimension of 50 mm. The temperature of the chips entering the unit is ambient temperature. The chips 5 are conveyed to the impregnation reactor 9 via the conical screw 6. This screw is washed with washing water 7,8 at a flow rate of 1 tonne/hour. During the compression carried out by the screw 6, liquid is extracted from the poplar chips at a rate of 0.679 tonne/hour; the total flow rate of spent water for washing the screw 6 of the impregnation reactor 9 is therefore 1.679 tonne/hour.

The following are introduced into the impregnation reactor 9:

- the compressed wood chips, via the conical screw 6
- 2.9 tonne/hour of an acidic liquor 4 prepared with water 1 and sulphuric acid 2 in a concentration by weight of 1.57% by weight (wt), at a temperature of 90°C

The impregnated chips are removed from the impregnation reactor 9 and transferred to a steam explosion reactor 14. Entry into the pretreatment reactor takes place by transfer by means of another conical screw 11. This screw is washed with washing water at a flow rate of 0.6 tonne/hour. During the compression carried out by the screw 11, liquid is extracted from the impregnated poplar chips at a rate of 2.95 tonne/hour; the total flow rate of spent water for washing the screw 6 of the impregnation reactor 9 is thus 3.55 tonne/hour. This stream contains 0.5% by weight of H_2SO_4.

The steam explosion pretreatment is carried out at 200°C in a continuous configuration using a short residence time. The medium is abruptly expanded to a pressure of 1.3 atm.

The acidic liquor preparation tank 3 located upstream of the impregnation step delivers a flow rate of 2.9 tonne/hour of acidic liquor. This liquor is prepared with water and 96% sulphuric acid; the respective flow rates are 2.8526 tonne/hour of water and 0.0474 tonne/hour of acid at a concentration of 96% by weight. The total consumption of water in this configuration is 4.5526 tonne/hour and the total consumption of acid is 0.0474 tonne/hour of sulphuric acid at a concentration of 96% by weight.

The spent water from washing the screws 6 and 11 are purged via pipes 8a and 13a.

Comparative Example 2

This repeats Comparative Example 1, the only difference here being that a portion 13c of the stream obtained from washing the screw 11 of the explosion reactor 14 is sent to the acidic liquor preparation tank 3: this flow is equal to the liquid make-up required taking into account the make-up of acid which should be made in order to produce the target acidity. Thus, the flow rate of recycled liquid is 2.8675 tonne/hour and the flow rate of make-up acid is 0.0325 tonne/hour of sulphuric acid at 96% by weight. In this second configuration, the total consumption of water is 1.6 tonne/hour (for washing the screw 11) and the consumption of acid is 0.0325 tonne/hour of sulphuric acid at 96% by weight. A stream of 2.362 tonne/hour of spent water has to be treated.

Example 3, in accordance with the invention

In this example, a process of impregnation in accordance with the invention and of pretreatment of wood, treating the same feedstock as in the preceding Comparative Examples 1 and 2, is described. The wood is used in the form of chips with a characteristic dimension of 50 mm. The temperature of the chips entering the unit is ambient temperature.

The chips are conveyed towards the impregnation reactor 9 via the conical screw 6. This screw is washed with a washing liquid 7,8,13d at a flow rate of 1.0 tonne/hour; this liquid originates from the mixture of water 7 and liquid 13d for washing the screw 11 of the pretreatment reactor 14. During the compression carried out by the screw 6, liquid is extracted from the poplar chips at a rate of 0.679 tonne/hour; the total flow rate of spent water for washing the screw 6 of the impregnation reactor 9 is therefore 1.679 tonne/hour.

The following are introduced into the impregnation reactor 9:

- the compressed wood chips, via the conical screw 6

- 2.9 tonne/hour of an acidic liquor 4 prepared with water 1 and sulphuric acid 2 in a concentration by weight of 1.57% by weight, at a temperature of 90°C

The impregnated chips are removed from the impregnation reactor 9 and transferred to a steam explosion reactor 14. Entry into the pretreatment reactor 14 takes place by transfer by means of a second conical screw 11. This screw 11 is washed with washing water 12,13 at a flow rate of 0.6 tonne/hour. During the compression carried out by the screw 11, liquid is extracted from the impregnated poplar chips at a rate of 2.95 tonne/hour; the total flow rate of spent water for washing the screw 11 of the reactor 14 is therefore 3.55 tonne/hour. This stream contains 0.5% by weight of H_2SO_4. The steam explosion pretreatment is carried out at 200°C in a continuous configuration using a short residence time. The medium is abruptly expanded to a pressure of 1.3 atm.

The acidic liquor preparation tank 3 located upstream of the impregnation step delivers a flow rate of 2.9 tonne/hour of acidic liquor. Preferably, it is equipped with sensors for measuring the pH and the flow rate for the water, acid, spent liquor and prepared liquor.

A portion of the stream obtained from washing the screw 11 of the explosion reactor 14 is sent to the acidic liquor preparation tank 3: this flow is equal to the liquid make-up required, taking into account the make-up of acid which should be made in order to produce the target acidity. Thus, as in the preceding examples, the flow rate of recycled liquid is 2.8675 tonne/hour and the flow rate of make-up acid is 0.0325 tonne/hour of sulphuric acid at 96% by weight. The part 13d of the stream of spent liquid 13 for washing the screw 11 of the pretreatment reactor 14 which is not sent as make-up liquid for the liquor tank 3 is sent to wash the screw 6 of the impregnation reactor 9, at a flow rate of 0.683 tonne/hour, as a supplement to 0.317 tonne/hour of water. The total consumption of water is thus 0.917 tonne/hour. A stream of 1.679 tonne/hour of spent water has to be treated.

Thus, compared with the configuration of comparative example 2, the process in accordance with the invention allows:

- a reduction in the consumption of water of more than 42% by weight

- a 29% reduction in the weight of spent water to be treated.

Example 4, in accordance with the invention

In this example, a process for impregnation in accordance with the invention and for the pretreatment of wood is described, treating the same feedstock as in Comparative Example 1.

The wood is used in the form of chips with a characteristic dimension of 50 mm. The temperature of the chips entering the unit is ambient temperature.

The chips are conveyed to the impregnation reactor 9 via the conical screw 6. This screw is washed with washing water 7 at a flow rate of 1.0 tonne/hour. During the compression carried out by the screw, liquid is extracted from the poplar chips at a rate of 0.679 tonne/hour; the total flow rate of spent water for washing the screw 6 of the impregnation reactor 9 is thus 1.679 tonne/hour.

The following are introduced into the impregnation reactor 9:

- the compressed wood chips, via the conical screw 2

- 2.9 tonne/hour of an acidic liquor 4 prepared with water 1 and sulphuric acid 2 in a concentration by weight of 1.57% by weight, at a temperature of 90°C

The impregnated chips are removed from the impregnation reactor 9 and transferred to a steam explosion reactor 14. Entry into the pretreatment reactor 14 takes place by transfer by means of a second conical screw 11. During the compression carried out by the screw, liquid 13 is extracted from the impregnated poplar chips at a rate of 2.95 tonne/hour. A portion 13b of this stream, 1.5 tonne/hour, is used to wash the conical screw 11, in accordance with the invention. The total flow rate of spent water for washing the screw 6 of the impregnation reactor 9 is thus 4.45 tonne/hour, 1.5 tonne/hour of which being recycled for washing ,13b. This stream contains 0.602% by weight of H_2SO_4. This stream is more concentrated in acid than the extracted stream in Example 1, since the spent acidic liquor extracted during compression has not been diluted with the screw washing water. The steam explosion pretreatment is carried out at 200°C in a continuous configuration using a short residence time. The medium is abruptly expanded to a pressure of 1.3 atm.

The acidic liquor preparation tank 3 located upstream of the impregnation step delivers a flow rate of 2.9 tonne/hour of acidic liquor.

The stream 13 obtained from washing the screw 11 of the pretreatment reactor 14 which is not used for washing the screw, 13b, is sent at 13c to the acidic liquor preparation tank 3 at a flow rate of 2.87 tonne/hour, the remainder 13a (0.08 tonne/hour) being purged. The flow rate of make-up acid is 0.0294 tonne/hour of sulphuric acid at 96% by weight.

In the process according to this example, the total consumption of water is 1.0 tonne/hour (for washing the impregnation screw 6) and the consumption of acid is 0.0294 tonne/hour of sulphuric acid at 96% by weight. A stream of 1.759 tonne/hour of spent water has to be treated.

Thus, compared with the configuration of Comparative Example 2, the process according to this example in accordance with the invention allows,:

- a reduction in the consumption of water of 37% by weight

- a 9.4% reduction in the consumption by weight of concentrated sulphuric acid

- a 25% reduction in the weight of spent water to be treated.

In conclusion, these examples demonstrate that the invention can be used to obtain very significant savings in energy/starting materials, with great flexibility of implementation. In addition, the modification induced on the facility remains modest and easy to implement, with the addition of pipes equipped, where appropriate, with means for varying the flow rate, for measuring the pH or the temperatures, the design of these additional pipes being entirely within the purview of a person skilled in the art. The invention remains beneficial even when the various operations for treating the biomass (for example the impregnation and cooking and/or steam explosion operation) are not carried out continuously: preference will be given in this case to recycling the washing water to the same feed screw (loop 8b for washing the screw 6, loop 13b for washing the screw 11).

Patentkrav

1. Fremgangsmåde til behandling af en lignocelluloseholdig biomasse, der omfatter et tørstofindhold på højst 90 vægt-%, hvilken fremgangsmåde omfatter anvendelse af mindst en reaktor til behandling af biomassen (9, 14), hvorved der til reaktoren eller mindst en af reaktorerne tilføres biomasse ved hjælp af et tilførselsmiddel (6, 11) ved at skabe en trykstigning mellem tilførselsmidlets biomasseindløb og biomasseudløb, hvorved tilførselsmidlet vaskes ved cirkulation af et vaskefluidum mellem et vaskeindløb (7, 12) og et vaskeudløb (8, 13), kendetegnet ved, at i det mindste en del af det vaskefluidum, der kommer ud af fluidumudløbet fra tilførselsmidlet eller mindst et af tilførselsmidlerne (6, 11), føres igen ind i vaskeindløbet til det samme tilførselsmiddel eller et andet af tilførselsmidlerne.

2. Fremgangsmåde ifølge det foregående krav, kendetegnet ved, at der udvindes opløsning fra biomassen under dens passage gennem tilførselsmidlet eller et af tilførselsmidlerne (6, 11).

3. Fremgangsmåde ifølge et af de foregående krav, kendetegnet ved, at den trykstigning, der frembringes i tilførselsmidlet eller mindst et af tilførselsmidlerne (6, 11), er på mindst 0,05 MPa, især mellem 0,05 og 4 MPa.

4. Fremgangsmåde ifølge et af de foregående krav, kendetegnet ved, at den omfatter et trin til imprægnering af biomassen med en imprægneringsopløsning, der indeholder en kemisk katalysator, hvilket trin udføres ved at indføre biomassen i reaktoren/en af reaktorerne (9, 1), der er forsynet med tilførselsmidlet (6,11).

5. Fremgangsmåde ifølge et af kravene 1 til 4, kendetegnet ved, at den omfatter et trin til imprægnering af biomassen med en imprægneringsopløsning, der indeholder en kemisk katalysator, ved gennemløb af biomassen i et leje med opløsning eller ved sprøjtning af biomassen med opløsningen på en båndtransportør (9') eller ved passage af biomassen gennem en omrørt reaktor.

6. Fremgangsmåde ifølge et af kravene 1 til 4, kendetegnet ved, at den omfatter et trin til imprægnering af biomassen med en imprægneringsopløsning, der indeholder en kemisk katalysator, ved indførelse i en reaktor (9) gennem tilførselsmidlet eller mindst et af tilførselsmidlerne (6).

7. Fremgangsmåde ifølge et af de foregående krav, kendetegnet ved, at den omfatter et trin til behandling af biomassen ved kogning, især i reaktoren eller en af de reaktorer (14), der er forsynet med et tilførselsmiddel (11).

8. Fremgangsmåde ifølge et af de foregående krav, hvilken fremgangsmåde omfatter følgende trin:
a. Fremstilling af en imprægneringsopløsning (4), som indeholder en kemisk katalysator (2), der er beregnet til imprægnering af biomassen (5), som er valgt blandt en sur katalysator, en basisk katalysator og en oxiderende katalysator, og som fortrinsvis er en syrekatalysator, i et fremstillingsområde (3)
b. Indføring af den knuste biomasse (5) gennem et indløb til en imprægneringsreaktor (9) ved hjælp af et første tilførselsmiddel (6), hvilket første tilførselsmiddel vaskes ved cirkulation af et første vaskefluidum (7, 8) mellem et indløb og et udløb i midlet (6)
c. Indføring af opløsningen i imprægneringsreaktoren (9) gennem et første opløsningsindløb (4) til reaktoren
d. Overførsel af den imprægnerede og afdryppede biomasse (10) fra et udløb fra imprægneringsreaktoren til et indløb til en forbehandlingsreaktor (14) til forbehandling ved kogning, idet indføringen i forbehandlingsreaktoren sker ved hjælp af et andet tilførselsmiddel (11), hvilket andet tilførselsmiddel vaskes ved cirkulation af et andet vaskefluidum (12, 13) mellem et indløb og et udløb i tilførselsmidlet
e. Forbehandling af biomassen (10) i reaktoren ved dampeksplosion (14)
f. Genindføring af i det mindste en del af det første og/eller det andet vaskefluidum, der kommer ud af fluidumudløbet fra det første eller det andet tilførselsmiddel (6, 11), ind i

vaskeindløbet til det første tilførselsmiddel (6) og/eller det andet tilførselsmiddel (11).

9. Fremgangsmåde ifølge det foregående krav, kendetegnet ved, at den desuden omfatter:
g. Genindføring af en del af det første og/eller det andet vaskefluidum, der kommer ud af fluidumudløbet fra det første eller det andet tilførselsmiddel (6, 11), ind i området (3) til fremstilling af imprægneringsopløsningen med henblik på fremstillingen a.) af opløsningen.

10. Fremgangsmåde ifølge et af de foregående krav, kendetegnet ved, at tilførselsmidlet og/eller mindst et af tilførselsmidlerne er en tilførselsskrue (6, 11), især i det mindste delvist konisk, der omfatter en kappe forsynet med et gitter, som muliggør cirkulationen af vaskefluidummet og eventuelt udtagningen af fluidum fra biomassen.

11. Fremgangsmåde ifølge krav 7 eller 8, kendetegnet ved, at en del af det første vaskefluidum (8) ved vaskeudløbet fra det første tilførselsmiddel (6) føres ind igen i vaskeindløbet (8b, 8d) til det første eller det andet tilførselsmiddel, samt ved, at en anden del af vaskefluidummet føres ind igen (8c) i området til fremstilling (3) af opløsningen med henblik på dens fremstilling.

12. Fremgangsmåde ifølge krav 7 eller 8, kendetegnet ved, at en del af det andet vaskefluidum (13) ved vaskeudløbet fra det andet tilførselsmiddel (11) føres ind igen (13b, 13d) i vaskeindløbet til det første eller det andet tilførselsmiddel (6, 11), samt ved, at en anden del (13c) af vaskefluidummet føres ind igen i området (3) til fremstilling af opløsningen med henblik på dens fremstilling.

13. Fremgangsmåde ifølge et af de foregående krav, kendetegnet ved, at vasken af tilførselsmidlet (11) udføres i nærvær af biomasse tilsat en katalysator, især en sur, basisk eller oxiderende katalysator, ved, at det vaskefluidum, der kommer ud

af tilførselsmidlets (6, 11) fluidumudløb, indeholder en vis mængde af katalysatoren, når biomassen undergår en væskeekstraktion ved at løbe gennem tilførselsmidlet, samt ved, at vaskefluidummet indeholdende denne katalysator i det mindste delvist føres ind i det samme tilførselsmiddels eller et andet af tilførselsmidlernes vaskeindløb for, især ved transport af biomassen, der er fri for katalysator.

14. Installation til udførelse af fremgangsmåden ifølge et af de foregående krav, kendetegnet ved, at den omfatter mindst en reaktor til behandling af en lignocelluloseholdig biomasse med et tørstofindhold på højst 90 vægt-% med en tilførsel af biomasse til reaktoren eller mindst en af reaktorerne ved hjælp af et tilførselsmiddel ved at skabe en trykstigning mellem tilførselsmidlets biomasseindløb og biomasseudløb, med vask af tilførselsmidlet ved cirkulation af et vaskefluidum mellem et vaskeindløb (7, 12) og et vaskeudløb, og et middel til genindføring af i det mindste en del af det vaskefluidum (8, 13), der kommer ud af fluidumudløbet fra tilførselsmidlet eller mindst et af tilførselsmidlerne (6, 11), ind i vaskeindløbet til det samme tilførselsmiddel eller til et andet af tilførselsmidlerne.

15. Installation til kontinuerlig behandling af en lignocelluloseholdig biomasse, hvilken installation omfatter:
- Et område til fremstilling (3) af en imprægneringsopløsning (4), som indeholder en kemisk katalysator til imprægnering af biomassen (5), der er valgt blandt en sur katalysator, en basisk katalysator og en oxiderende katalysator, og fortrinsvis en syrekatalysator, og som er forsynet med et opløsningsudløb
- En imprægneringsreaktor (9), hvilken reaktor er forsynet med et biomasseindløb og et biomasseudløb
- Et første tilførselsmiddel til tilførsel af knust biomasse (6) til imprægneringsreaktoren (9) gennem imprægneringsreaktorens biomasseindløb
- Et tilførselsmiddel til tilførsel af imprægneringsopløsning (4a) til reaktoren (5), der forbinder et opløsningsudløb fra

området til fremstilling (3) af opløsningen (4) til et første opløsningsindløb i imprægneringsreaktoren

- En forbehandlingsreaktor (14) til forbehandling af den imprægnerede biomasse (10) ved kogning, af hvilken et biomasseindløb er forbundet med imprægneringsreaktorens biomasseudløb (9)

- Et andet tilførselsmiddel (11) til tilførsel af imprægneret biomasse (10) til forbehandlingsreaktoren gennem forbehandlingsreaktorens (14) biomasseindløb

- En cirkulation af vaskefluidum (7, 8) mellem et indløb og et udløb i det første tilførselsmiddel (6) til tilførsel af knust biomasse (5) til imprægneringsreaktoren (9)

- En cirkulation af vaskefluidum (12, 13) mellem et indløb og et udløb i det andet tilførselsmiddel (11) til tilførsel af imprægneret biomasse (10) til forbehandlingsreaktoren (14)

kendetegnet ved, at installationen desuden omfatter:

- Et middel til genindføring af i det mindste en del af det første og/eller det andet vaskefluidum, der kommer ud af fluidumudløbet fra det første eller det andet tilførselsmiddel (6, 11), ind i vaskeindløbet til det første tilførselsmiddel og/eller det andet tilførselsmiddel.

16. Anvendelse af fremgangsmåden eller af installationen ifølge et af de foregående krav til behandling af lignocelluloseholdige biomasser (5) af typen træ, halm, landbrugsrester og alle dedikerede energiafgrøder, især enårige eller flerårige planter, såsom miscanthus, med henblik på at fremstille sukkerarter, biobrændstoffer eller biobaserede molekyler.

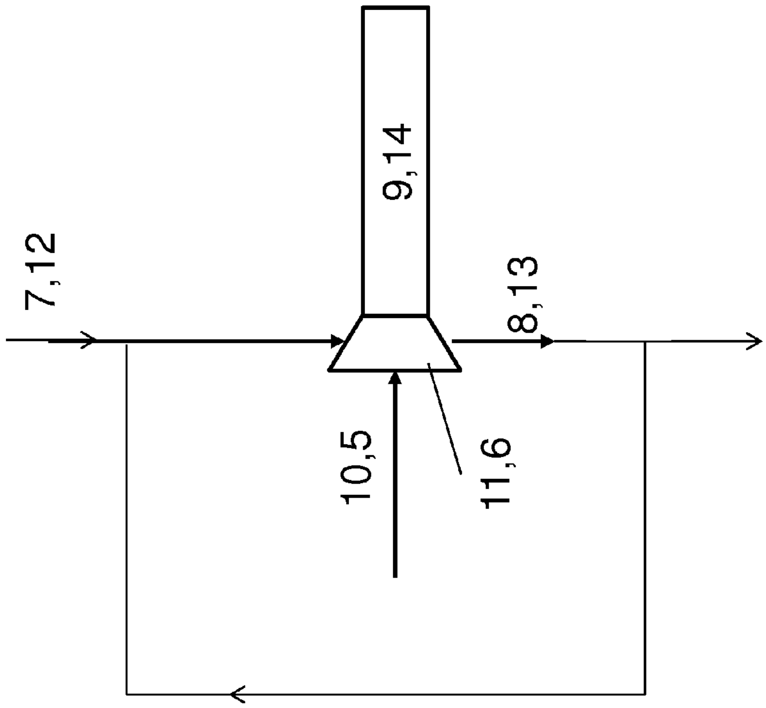

Figure 1

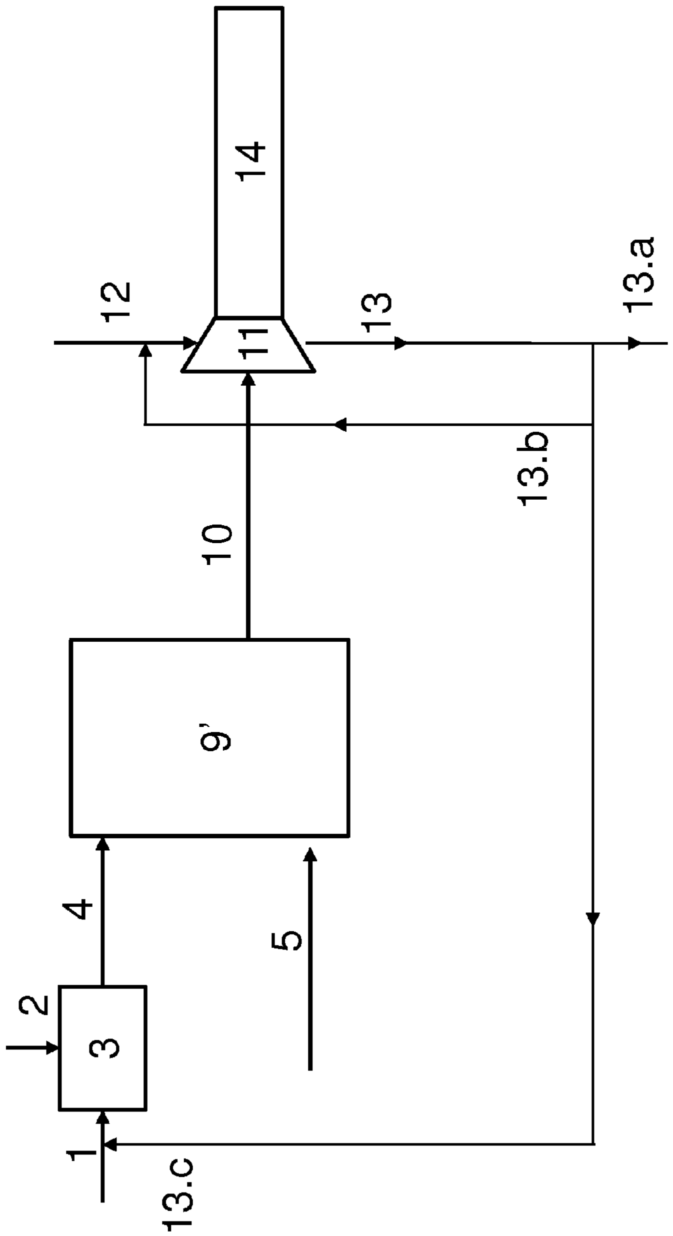

Figure 2

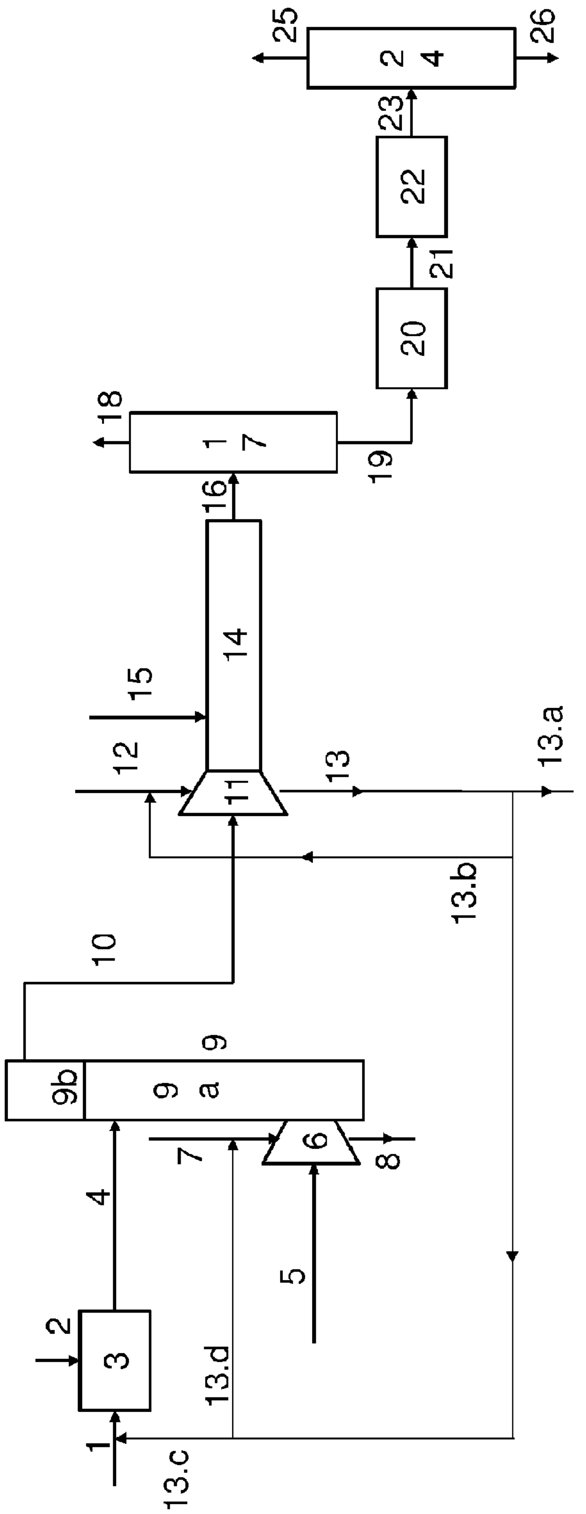
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Figure 3

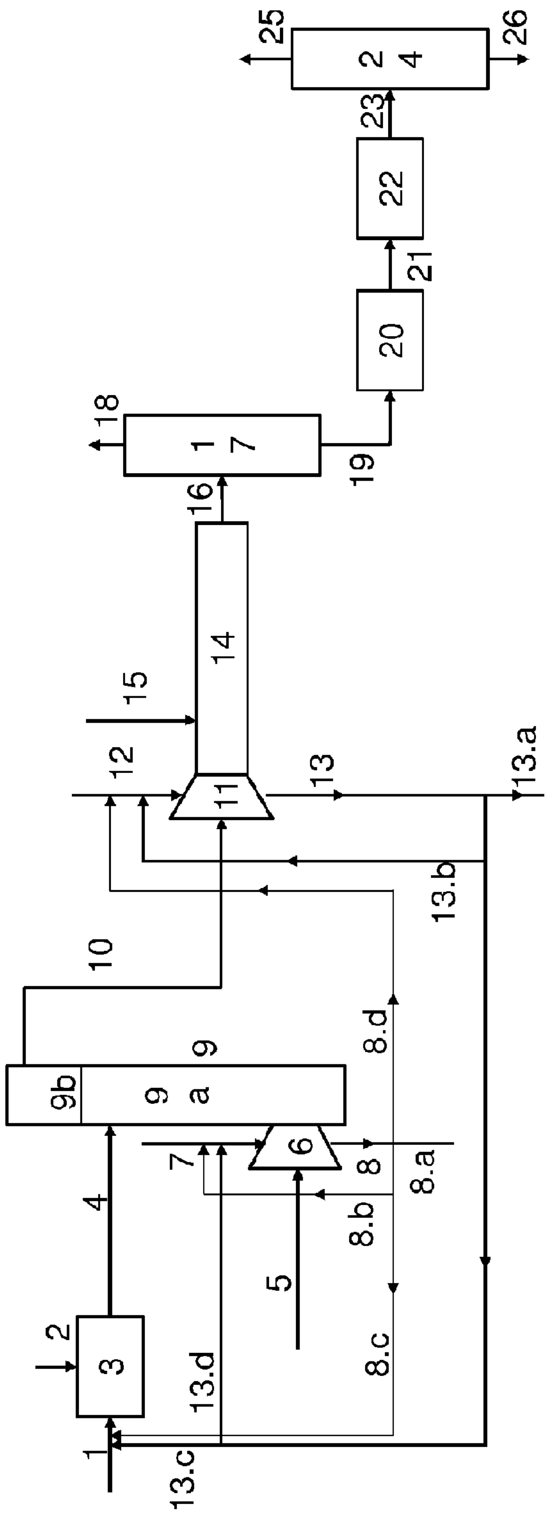
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Figure 4