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(54) **METHOD FOR PREHYDROLYSIS OF LIGNOCELLULOSIC MATERIAL**

(57) According to an example aspect of the present invention, there is provided a method for hydrolysing lignocellulosic chip material in a reaction vessel by a hydrolysis reaction carried out in the presence of steam at a reaction temperature. The method comprises the steps of heating the lignocellulosic material to the reaction temperature by feeding a medium pressure steam flow into the reaction vessel, introducing during a preselected period of time a liquid SO₂ flow into the medium pressure steam flow and vaporizing the liquid SO₂ flow to form gaseous SO₂ in the medium pressure steam flow, continuing the feed of the medium pressure flow into the reaction vessel after said preselected period of time, and maintaining the reaction temperature for hydrolysing the lignocellulosic material.

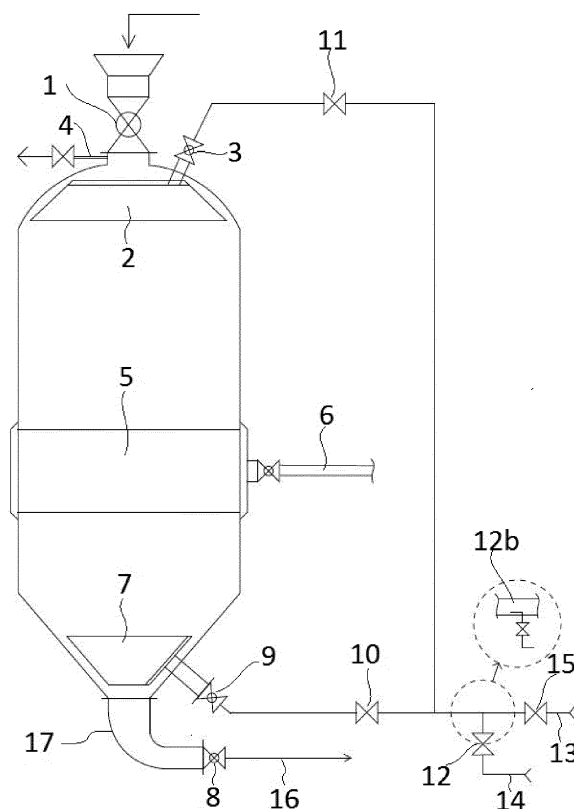


Fig. 1

Description

FIELD

- 5 **[0001]** The present invention relates to prehydrolysis of lignocellulosic material. In particular, the present invention concerns a method of hydrolysing lignocellulosic chip material with SO₂-steam mixture.

BACKGROUND

- 10 **[0002]** Mankind is looking for possible avenues to replace fossil raw material based products by renewable lignocellulose based products. More particularly this means that the lignocellulosics' main components, namely cellulose, hemicelluloses and lignin must be separated from each other and the fractions to be turned into corresponding intermediate bioproducts, cellulose fiber, carbohydrate solution (hydrolysate) and lignin solution or solids.

- 15 **[0003]** As described by Sixta, H., "Handbook of Pulp" prehydrolysis is generally used to selectively remove the hemicelluloses from the lignocellulosic biomass by hydrolysis in water at 160-180 °C, in dilute acid (0.3-0.5% H₂SO₄) at 120-140 °C or in concentrated acid (20-30% HCl) at 40 °C. Dilute mineral acids are efficient in hemicellulose hydrolysis, but they bring along corrosion and material problems and have an adverse effect on lignin - deactivation by acid catalyzed condensation reactions - which makes it more difficult to remove lignin and bleach the cellulosic fibers. So far, all the few industrial continuous prehydrolysis-kraft pulp processes use water prehydrolysis prior to the kraft cooking stage.

- 20 **[0004]** Prehydrolysis products from wood are considered to be a source for chemicals, fodder, food additives and pharmaceutical products. Both the high yield of the prehydrolysis released carbohydrate compounds and the efficient production of the high value products are prerequisites for a feasible industrial process.

- 25 **[0005]** However, at present there is no commercial utilization of the water prehydrolyzate during the course of a prehydrolysis-kraft pulping operation. The running of a water prehydrolysis step, without extensively utilizing the degraded wood byproducts, is economically not feasible because recovery of the large amounts of prehydrolysate requires a lot of additional equipment and evaporation capacity. In addition, the water based wood prehydrolysate contains many chemical compounds which cause severe solid depositions and scaling on process equipment metal surfaces, especially on heat exchange surfaces, thus requiring special control and maintenance lowering the overall process efficiency.

- 30 **[0006]** One way to improve the above described situation of water prehydrolysis or dilute mineral acid prehydrolysis is to use sulphur dioxide as a low concentration water solution at 140-150 °C. According to Radiotis et al. and Sixta, H., "Handbook of Pulp" it has several advantages: the acidity by bisulfite ions in water is higher than that in water prehydrolysis. Thus the hemicellulose degradation and dissolution is higher and the resulting monomeric sugar proportion is higher. However, the acidity is still lower than that of the reactive protons of dilute mineral acids which damage lignin and lower the pulp yield. SO₂-water based prehydrolysis has a unique advantage: it sulphonates the low-molecular phenolic compounds to some extent, thus making them hydrophilic and lowering the scaling plugging tendency in prehydrolysis as well as in the hydrolysate refining operation.

- 35 **[0007]** Documents US 3,132,051; US 5,139,617 and FI 121237B use SO₂-water, more precisely water solution of SO₂ gas containing HSO₃⁻ bisulfite ions and dissolved SO₂. However, the solutions in these documents add 8 - 12 tons of water per ton of pulp to be processed and evaporated.

- 40 **[0008]** In addition to the evaporation and energy usage burden of large water volume, the water dilutes the SO₂ charge. However, chemical reactions, i.e. hydrolysis, are controlled by reagent concentration which requires large quantity of charged SO₂. For example in the publication by Radiotis, T., et al. a SO₂ dosage of 0.5 - 1.0 % on dry wood was used to achieve satisfactory hydrolytic reaction. This is 5 - 10 kg SO₂ per ton of dry wood and about 15 - 30 kg SO₂ per ton produced pre-hydrolysed pulp. The excess sulphur which cannot be further utilized in the pre-hydrolyse-pulp plant goes into the plant's chemicals recovery cycle and accumulates disturbing the normal chemical balance. Eventually the plant must invest in sulphur removing technology increasing cost and complexity of the plant.

- 45 **[0009]** Steam-phase prehydrolysis has been one way to decrease the water usage. The heat needed to heat-up wood chips to prehydrolysis temperature, 170-175 °C, is introduced as direct "medium pressure" 10-12 atm mill process steam and hydrolysis takes place in steam phase, without liquid water in the voids within the wood chip charge. For 1 ton dry wood 0.3 t steam is needed. Chip moisture being 0.65 t H₂O, the liquid-to-wood ratio is 0.3+0.65 = 0.95 t H₂O / 1 t dry wood. As wood chips can carry up to 2 t H₂O per 1 t dry wood, this is an amount that the chips carry inside and there is practically no free water in the void volume of the chip column.. In contrast, in water prehydrolysis the liquid-to-wood ratio is 4:1 - 5:1 t H₂O/t dry wood.

- 50 **[0010]** So far, in prehydrolysis kraft batch cooking, the steam-phase prehydrolysis has provided for the technical and economic feasibility of the production. The prehydrolyzate and degradation products have occurred only inside the chips in the digester vessel, where the following alkaline cooking has neutralized and dissolved them away together with the yield loss in the kraft cooking part. Thus, all dissolved solids and liquids are gathered without extra burden in the black liquor, which is evaporated and run through the kraft chemical/energy recovery cycle with only a minor capacity increase

demand. The drawback of the steam-phase prehydrolysis is that it is technically only possible as a batch operation.

[0011] The above described segregates prehydrolysis technology into two areas, the continuous prehydrolysis-kraft process using the water based prehydrolysis and the discontinuous batch prehydrolysis-kraft process using steam-phase prehydrolysis. The steam-phase has been a winner so far, but it has one major drawback: it is not possible to use any hydrolyzing agents such as acid SO₂-water, etc., because there is no aqueous phase to enable transfer, carrying and mixing of chemicals.

SUMMARY OF THE INVENTION

[0012] The invention is defined by the features of the independent claims. Some specific embodiments are defined in the dependent claims.

[0013] It is the aim of the invention to remove at least some of the problems relating to the known technical solutions.

[0014] According to a first aspect of the present invention, there is provided a method for hydrolysing lignocellulosic material in a reaction vessel by a hydrolysis reaction carried out in the presence of steam at a reaction temperature.

The method comprises the steps of:

- heating the lignocellulosic material to the reaction temperature by feeding a steam flow into the reaction vessel,
- introducing during a preselected period of time a liquid SO₂ flow into the steam flow and vaporizing the liquid SO₂ flow to form gaseous SO₂ in the medium pressure steam flow,
- continuing the steam feed into the reaction vessel after said preselected period of time, and
- maintaining the reaction temperature in the reaction vessel to achieve hydrolysis of the lignocellulosic material.

[0015] Considerable advantages are obtained by this invention. The present invention provides a method that reduces the use of highly corrosive SO₂-water and introduces a feasible hydrolysing method with the use of SO₂-steam mixture in controlled conditions.

[0016] It is to be noted the new unexpected founding: the SO₂-steam hydrolysis does not reduce the pulp's yield, viscosity, kappa number or pentosane content. Especially is surprising that the pulp yield does not decrease when using the SO₂-steam hydrolysis.

[0017] It is to be noted here, that the use of SO₂ according to the present invention is the most efficient in view of the SO₂ concentration in the small amount of condensate and chip water (about 1 ton water per 1 ton of dry biomass); this is quite contrary to the situation in the water liquid-phase prehydrolysis, where the SO₂ dilutes in the big amount of water filling the entire reactor (4-5 tons water per 1 ton dry biomass). In process technology the reactions are most effective when the reagent concentration is high. Achieving high concentration in small amount of diluting reaction media also enables the lowest possible reagent dosage and chemical cost as well as saving in the heating energy needed.

[0018] The present invention combines the hydrolysis reactor input streams, the heating direct steam serves simultaneously as the carrying media for the SO₂ to enter and spread into the reactor content; a very simple operation saves process time, extra equipment and operational steps.

[0019] Another benefit of using SO₂ in prehydrolysis is that the hydrolysate liquid contains much more monomeric carbohydrates than is the case in water-based auto-hydrolysis. This is technically an important advantage as the water-hydrolysis dissolved poly- and oligomeric carbohydrates must be hydrolysed to monomers later anyway.

[0020] The SO₂ charge needed for the prehydrolysis is allocated so that the last part of the direct steam input flows without SO₂ in order to flush the input pipe/inlet connection and prehydrolysis reaction temperature is reached. In this new way, the SO₂ addition becomes a part of the heating media, i.e. of the input flow of the steam, and is being transferred on the chip particles with condensing steam and thereby absorbed into the chip particles.

[0021] Next, embodiments will be examined in more detail with reference to the attached drawings.

BRIEF DESCRIPTION OF THE DRAWING

[0022] FIGURE 1 illustrates a batch reactor in accordance with at least some embodiments of the present invention.

EMBODIMENTS

DEFINITIONS

[0023] In the present context, the term "hydrolysate liquid" is where the hydrolysis reaction products accumulate.

[0024] In the present context, the term "odw" means oven dry wood weight. It is the weight after all the moisture has been evaporated.

[0025] In the present context, the term "EA" means effective alkali. It is the sum of the sodium hydroxide and half of the sodium sulphide in the white liquor.

[0026] In the present context, the term "dry" means non-aqueous.

[0027] In the present context, the pressure unit "bar" is absolute pressure. When referring to pressure difference "bar" is the differential pressure.

[0028] In the present context, the term "hydrolysis" is used for the chemical reaction of removal of hemicelluloses from lignocellulosic material.

[0029] In the present context, the term "prehydrolysis" is the hydrolysis reaction before a cooking process.

[0030] As briefly discussed above, presented is a method for hydrolysing lignocellulosic material by using SO₂-steam in controlled conditions. Figure 1 presents a batch digester in accordance with at least some embodiments of the present invention. In the starting situation the digester is empty. The process starts by wood chips filling, through the feeding hopper and digester capping valve 1. Often chip packing is intensified by steam jets giving more speed to falling chips. During the chip fill, gases are evacuated out from the middle screens 5, through the cooking circulation pipeline 6. In an embodiment, air is removed from the digester.

[0031] When the digester is full with chips the capping valve 1 is closed. Air removal can be intensified by steaming the chips by introducing low pressure steam through bottom screens 7 and valves 9, 10, 15. At the same time gases are evacuated from the top through degassing line 4, thus so far digester has been kept practically at atmospheric pressure.

[0032] The first heating stage towards reaction temperature and pressure is carried out by low pressure steam through top screens 2, bottom screens 7 and valves 3, 11, 15 and 9, 10, 15. The first low pressure steam heating stage elevates the temperature close to the corresponding saturated steam temperature at the prevailing pressure.

[0033] According to one embodiment the first heating stage pressure is 3 - 5 bar and the first heating stage temperature is 125 - 150 °C. The time for this heating stage is 15 - 45 minutes

[0034] Then heating is continued by medium pressure steam in the same way as in the first heating stage. According to one embodiment the medium pressure is at least 1 bar higher than the corresponding pressure of saturated steam at reaction temperature of the hydrolysis. According to one embodiment the medium pressure is 6 - 10 bar. The time for this heating stage is 5 - 25 minutes. The total time for heating to the desired hydrolysis temperature is 20 - 60 minutes, preferably about 45 minutes.

[0035] According to one embodiment of the present invention SO₂ is introduced into the flowing heating steam. First, the pure steam is allowed to fill the reaction vessel and condense onto the chips, after this liquid SO₂ is injected to the steam, then ending the SO₂ injection so that a clear pure steam feed phase is the last part of the heating steam flow. Thus, SO₂ injection is placed into the steam flow securing that all the pipe lines and valves etc. are above condensing temperature, but on the other hand there is enough pure steam flow to purge all pipelines and valves etc. clean from SO₂ before the end of steam flow.

[0036] According to one embodiment the predetermined SO₂ dosage in the SO₂ injection is at least 0.15 % odw, preferably at least 0.20 % odw, for example 0.25 % odw.

[0037] According to one embodiment, the hydrolysis of the lignocellulosic material is partial.

[0038] According to one embodiment, the temperature is maintained until a predetermined degree of hydrolysis of the lignocellulosic material has been reached.

[0039] In one embodiment, the pentosan content of the pulp (pulp after cooking) is - as a result of the prehydrolysis - less than 4.5 % by weight, in particular less than 4.2 % by weight, for example about 4 to about 3.5 % by weight of the pulp.

[0040] In one embodiment, hydrolysis of the lignocellulosic material will lead to a pentosan content of 4 wt-% or under of the mass of the fiber.

[0041] The injection of SO₂ charge takes place by pumping a predetermined mass-flow of liquid SO₂ from pipeline or SO₂ (1) storage 14, through valve 12 into the steam pipe 13 through injection pipe 12b over a predetermined period of time. As the mass flow of liquid SO₂ is very small compared to the tons of steam flow, the liquid SO₂ is immediately and completely vaporized in the steam flow. This means that the flow of steam and gaseous flow is dry, avoiding corrosion and difficult material selections. The steam flow into the chip bed digester carries the SO₂ in all the space of the digester as the heating-by-condensing-steam is the main mechanism, local steam pressure decreases and the gradient is withdrawing and spreading steam and SO₂ everywhere.

[0042] Once the target reaction temperature has been reached, that temperature will be maintained for a predetermined time. According to one embodiment the target reaction temperature is 150-175 °C, preferably 160-165 °C and the desired reaction time is 15-60 minutes, preferably about 30 minutes.

[0043] Sulphur dioxide, SO₂, in the steam, absorbed on the chip surface is quickly transferred to the chip interior together with the heat. A uniform SO₂ enhanced prehydrolysis is achieved in all parts of the digester - without using any external SO₂-water, water phase hydrolysis, re-circulations, indirect heating etc.

[0044] After prehydrolysis, the chips are subjected to kraft cooking. As a result of the prehydrolysis carried out as described in the fore-going using steam containing gaseous SO₂, there is practically no free condensate or water-phase in the digester, and the burden of removing this liquid phase is completely avoided.

[0045] As mentioned above, in steam-phase batch reactor process the use of acid or SO₂-water has not been possible, as there is no liquid reaction medium to inject and mix the acid or SO₂-water into. However, now it has been discovered that also steam-phase hydrolysis can be improved and intensified by using SO₂ gas. Further, the use of SO₂ gas can be made very simple and the preparation, storage and handling of highly corrosive SO₂-water is completely avoided. Liquid dry SO₂ is injected from a storage tank directly into the steam pipe/inlet connection into the hydrolysis reactor (batch digester) and the inflowing heating steam carries and distributes the SO₂ all over the reactor space as a part of the steam flow into every part of the chip filled reactor pulled by the pressure gradient generated by the condensing steam on the wood chips. The spread SO₂ gas absorbs on the moist chip particle surfaces and subsequently penetrates and diffuses into the wood chips.

[0046] The kraft cooking is carried out as known in the art. It can be a conventional kraft batch cooking, where first white liquor is added and then the reaction vessel is heated, next the white liquor is circulated through screen 5 to cooking circulation pipeline 6 and pumped up to valve 3 and down to valve 9 and fed back into the reaction vessel through screens 2 and 7. An option to the conventional kraft batch cooking is a so called "displacement kraft batch cooking" process where several filling and displacements take place. The prehydrolysed and cooked - chemically defibrated pulp - is discharged through bottom curve 17 and valve 8 to fibre line feed 16.

[0047] It goes without explaining that the prehydrolysis part may have different process equipment and instrumentation embodiments, such as separate steam connections and lines, different screen designs, flow of steam and SO₂(g) injection only from bottom or from top, or both and multiple points.

[0048] The present invention has preferred embodiments in batch reactor processes, but it is not limited to only batch process. It is evident that steam-phase hydrolysis could be constructed to be a continuous process when the chip feeding and discharging machine organs are designed and constructed to operate continuously. In that case SO₂ would be injected into the entering steam, in particular medium pressure steam.

EXAMPLE 1

[0049] Experimental results obtained in the following examples show the effect of gas phase SO₂ - water steam hydrolysis compared to a reference process without SO₂ in water steam hydrolysis. The process was the same in all examples, the prehydrolysis conditions were varied and the kraft cooking procedure and conditions were constant. The varying prehydrolysis conditions are shown in table 1. The process steps were as follows:

- chip steaming and air evacuation at 100 °C for 10 min
- direct steam injection into digester up to desired hydrolysis temperature
- staying at temperature the desired reaction time
- charging first volume of kraft cooking white liquor (8.3% EA on odw) for neutralization of reactor content
- charging a volume of hot white liquor (7.4% EA on odw) and hot black liquor (20 gEA/l) for kraft cooking stage
- adjusting cooking temperature 160 °C by indirectly heated reactor re-circulation
- staying at cooking temperature the desired cooking time 45 min
- stopping cooking reactions by displacing hot black liquor from the reactor using cold washing liquid

[0050] The results of a reference prehydrolysis-kraft cook with no SO₂ gas and two tests with (1) 0.2 % SO₂ gas charge and (2) 0.25 % SO₂ gas charge are presented in table 1 below.

Table 1

Prehydrolysis	Reference	(1)	(2)
SO ₂ gas, charge, %	0	0.2	0.25
Hydrolysis temperature, °C	172	162	162

(continued)

Prehydrolysis	Reference	(1)	(2)
Heating time, min	45	45	45
Time at reaction temperature, min	55	55	55

[0051] The results of these experiments are shown in table 2 below.

Table 2

Pulp yield, % on wood	39.7	38.8	39.5
Pulp kappa number	8.0	8.1	8.1
Pulp viscosity, ml/g	1020	1020	1030
Pulp pentosan content, %	4.6	4.0	3.8

[0052] The pulp technical results of these experiments are characterized by pulp pentosan content. This is the main quality parameter of this type pulp and it is controlled by the prehydrolysis reaction degree (hydrolytic removal of xylan hemicelluloses from the wood). The pulp buying textile industry's standard for prime pulp is pentosan content 3.5-4.0 %. It is evident that this pentosan range is well met at 0.2-0.25 % SO₂(g) charge.

[0053] Most interesting are the other pulp parameters: pulp yield, pulp delignification degree (kappa number), pulp viscosity are at quite the same level regardless of using SO₂(g). This is a new advantage: the state-of-the-art water-based SO₂ prehydrolysis-kraft cooks have a clear yield loss. However, the prehydrolysis conditions were significantly milder with SO₂(g): hydrolysis temperature 10 degrees lower.

[0054] The examples demonstrate that good quality effects can be achieved by using clearly less SO₂(g)(0.2-0.25% on odw) when it is introduced with the steam into steam phase process. It also proves that the SO₂ gas dosage achieves high enough concentration of SO₂ in wood chips - reflecting almost exactly the difference in the total amount of water and dilution in the prehydrolysis. In the flow of steam and SO₂(g) phase about 1 t liquid/lt wood versus about 4 t liquid/lt wood.

[0055] It is to be understood that the embodiments of the invention disclosed are not limited to the particular structures, process steps, or materials disclosed herein, but are extended to equivalents thereof as would be recognized by those ordinarily skilled in the relevant arts. It should also be understood that terminology employed herein is used for the purpose of describing particular embodiments only and is not intended to be limiting.

[0056] Reference throughout this specification to "one embodiment" or "an embodiment" means that a particular feature, structure, or characteristic described in connection with the embodiment is included in at least one embodiment of the present invention. Thus, appearances of the phrases "in one embodiment" or "in an embodiment" in various places throughout this specification are not necessarily all referring to the same embodiment.

[0057] As used herein, a plurality of items, structural elements, compositional elements, and/or materials may be presented in a common list for convenience. However, these lists should be construed as though each member of the list is individually identified as a separate and unique member. Thus, no individual member of such list should be construed as a de facto equivalent of any other member of the same list solely based on their presentation in a common group without indications to the contrary. In addition, various embodiments and example of the present invention may be referred to herein along with alternatives for the various components thereof. It is understood that such embodiments, examples, and alternatives are not to be construed as de facto equivalents of one another, but are to be considered as separate and autonomous representations of the present invention.

[0058] Furthermore, the described features, structures, or characteristics may be combined in any suitable manner in one or more embodiments. In the following description, numerous specific details are provided, such as examples of lengths, widths, shapes, etc., to provide a thorough understanding of embodiments of the invention. One skilled in the relevant art will recognize, however, that the invention can be practiced without one or more of the specific details, or with other methods, components, materials, etc. In other instances, well-known structures, materials, or operations are not shown or described in detail to avoid obscuring aspects of the invention.

[0059] While the forgoing examples are illustrative of the principles of the present invention in one or more particular applications, it will be apparent to those of ordinary skill in the art that numerous modifications in form, usage and details of implementation can be made without the exercise of inventive faculty, and without departing from the principles and concepts of the invention. Accordingly, it is not intended that the invention be limited, except as by the claims set forth below.

[0060] The verbs "to comprise" and "to include" are used in this document as open limitations that neither exclude nor require the existence of also un-recited features. The features recited in depending claims are mutually freely combinable unless otherwise explicitly stated. Furthermore, it is to be understood that the use of "a" or "an", i.e. a singular form, throughout this document does not exclude a plurality.

REFERENCE SIGNS LIST

[0061]

- 1 Digester capping valve
- 2 Top screens
- 3 Valve
- 4 Degassing line
- 5 Middle screens
- 6 Cooking circulation pipeline
- 7 Bottom screen
- 8 Valve
- 9 Valve
- 10 Valve
- 11 Valve
- 12 Valve 12b injection pipe
- 13 Steam pipeline
- 14 SO₂ Storage pipeline
- 15 Valve
- 16 Fiber feed line
- 17 Bottom curve

CITATION LIST

[0062] Patent Literature

US 3,132,051
US 5,139,617
FI121237B

Non Patent Literature

[0063]

Sixta, H., "Handbook of Pulp", Vol.1, p. 325-365
Radiotis et al., NWBC 2011, Stockholm, p.92-99

Claims

1. Method for hydrolysing lignocellulosic material in a reaction vessel by a hydrolysis reaction carried out in the presence of steam at a reaction temperature, the method comprising the steps of:
 - heating the lignocellulosic material to the reaction temperature by feeding a steam flow into the reaction vessel,
 - introducing during a preselected period of time a liquid SO₂ flow into the steam flow and vaporizing the liquid SO₂ flow to form gaseous SO₂ in the medium pressure steam flow,
 - continuing the steam feed flow into the reaction vessel after said preselected period of time, and
 - maintaining the reaction temperature for hydrolysing the lignocellulosic material.
2. Method according to claim 1, further comprising the steps of:
 - filling a reactor with wood chips,
 - preheating and displacing air by introducing a low pressure steam flow and venting gases out of the reaction

vessel,

- closing the venting and introducing the liquid SO₂ flow into a medium pressure steam heating flow until a predetermined SO₂ dosage is reached, and
- terminating the hydrolysis reaction by degassing and introducing cold liquid into the reactor.

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3. Method according to claim 1 or 2 wherein the steam flow is fed into the reaction vessel in order to reach the predetermined hydrolysis reaction temperature.

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4. Method according to claim 2 or 3, wherein the preheating temperature is 125-150 °C.

5. Method according to any of claims 2 to 4, wherein the preheating time is 15-45 minutes.

6. Method according to any of claims 2 to 5, wherein the low pressure steam flow pressure is 3-5 bar.

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7. Method according to any of the preceding claims, wherein the hydrolysis of the lignocellulosic material will give a pentosan content of 4 wt-% or under of the mass of the fiber.

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8. Method according to any of the preceding claims, wherein the SO₂ dosage is at least 0.15 % oven dry wood weight, preferably at least 0.20 % oven wood weight, for example 0.25 % oven dry wood weight.

9. Method according to any of the preceding claims, wherein the liquid SO₂ flow is introduced into a flow of medium pressure steam having a pressure which is at least 1 bar higher than the corresponding pressure of saturated steam at reaction temperature.

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10. Method according to claim 9, wherein the steam flow pressure is 6-10 bar.

11. Method according to any of the preceding claims, wherein the time for heating is 5-25 minutes.

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12. Method according to any of the preceding claims, wherein the total time for heating to hydrolysis temperature is 20-60 minutes, preferably 45 minutes.

13. Method according to any of the preceding claims, wherein the hydrolysis temperature is 150-175 °C, preferably 160-165 °C.

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14. Method according to any of the preceding claims, wherein the time at hydrolysis reaction temperature is 15-60 min, preferably 30 min.

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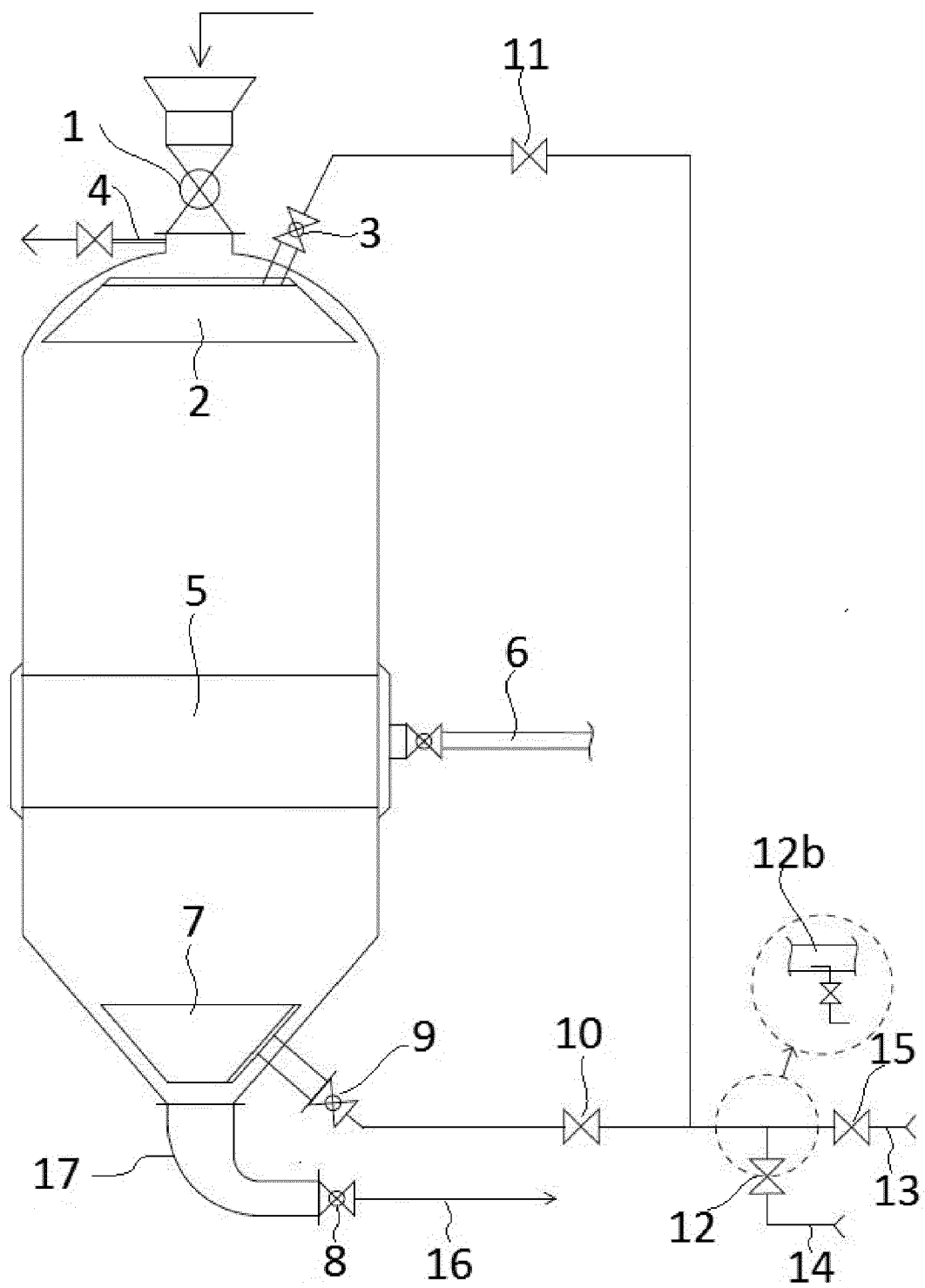


Fig. 1



EUROPEAN SEARCH REPORT

 Application Number
 EP 20 39 7506

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
Y	WO 2017/100907 A1 (IOGEN CORP [CA]) 22 June 2017 (2017-06-22) * paragraphs [0073], [00305]; example 13 * * paragraph [0157] - paragraph [0159] * -----	1-14	INV. D21C1/02
Y	SCHELL D J ET AL: "A technical and economic analysis of acid-catalyzed steam explosion and dilute sulfuric acid pretreatments using wheat straw or aspen wood chips", APPLIED BIOCHEMISTRY AND BIOTECHNOLOGY ; PART A: ENZYME ENGINEERING AND BIOTECHNOLOGY, HUMANA PRESS INC, NEW YORK, vol. 28 - 29, no. 1, 1 March 1991 (1991-03-01), pages 87-97, XP035177040, ISSN: 1559-0291, DOI: 10.1007/BF02922591 * figure 1 * * page 89 - page 90 * -----	1-14	
Y	GB 734 216 A (PROCEL SA) 27 July 1955 (1955-07-27) * page 1, right-hand column, line 60 - page 2, left-hand column, line 39 * * examples I, II * -----	1-14	TECHNICAL FIELDS SEARCHED (IPC) D21C C12M
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A	US 2012/064592 A1 (O'MULLAN PATRICK [US] ET AL) 15 March 2012 (2012-03-15) * paragraph [0227]; figure 4 * ----- -/--	1-14	
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Place of search Munich		Date of completion of the search 18 September 2020	Examiner Billet, Aina
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	

EPO FORM 1503 03.82 (P04C01)



EUROPEAN SEARCH REPORT

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			TECHNICAL FIELDS SEARCHED (IPC)
The present search report has been drawn up for all claims			
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