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(54) Title: A METHOD AND A SYSTEM FOR PRODUCING MICROFIBRILLATED CELLULOSE

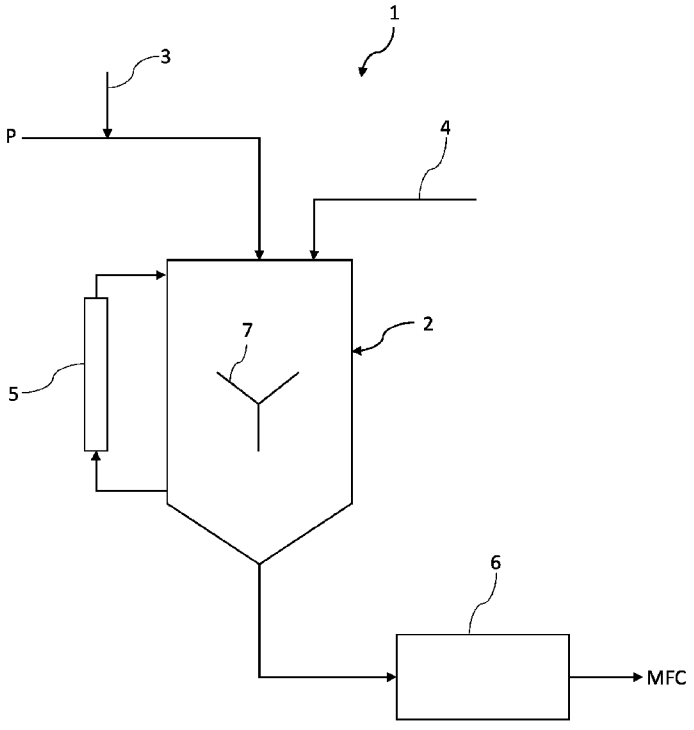


Fig. 1

(57) Abstract: The present document discloses a method for producing microfibrillated cellulose (MFC) comprising the steps of: a) provide a refined pulp suspension mixed with an enzyme to a reactor tank; b) treat the refined pulp suspension with said enzyme in the reactor tank at a temperature between 40-80°C under controlled mixing by means of an agitating member thus forming a pre-treated pulp suspension; c) adding at least one oxidant to said pre-treated pulp suspension simultaneous to mixing; d) perform oxidation of said pre-treated pulp suspension under elevated temperatures >80°C in said reactor tank for deactivating said enzyme; and e) discharge the pre-treated, pre-refined pulp suspension and subjecting it to further mechanical treatment to obtain MFC.

[Continued on next page]

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A METHOD AND A SYSTEM FOR PRODUCING MICROFIBRILLATED CELLULOSE

Technical field

The present invention relates to a method for producing microfibrillated cellulose.

Background

Microfibrillated cellulose ("MFC") is a material that consists of cellulose microfibrils that can be separated from cellulose fiber walls. The liberated fibrils have a diameter less than 1000 nm, whereas the actual fibril diameter or particle size distribution and/or aspect ratio (length/width) depends on the source and the manufacturing methods. The smallest fibril is called elementary fibril and may have a diameter of approximately 2-4 nm, while it is common that the aggregated form of the elementary fibrils is the main product that is obtained when making MFC, e.g., by using an extended refining process or high pressure-drop disintegration process, such as high pressure homogenization or fluidization.

Various methods exist to make MFC, such as single or multiple pass refining, pre-hydrolysis or enzymatic treatment followed by refining or high shear disintegration or liberation of fibrils. MFC can be produced from wood cellulose fibers, both from hardwood and softwood fibers. It can also be made from microbial sources, agricultural fibers such as wheat straw pulp, bamboo, bagasse, or other non-wood fiber sources. It is preferably made from pulp, including pulp from virgin fiber, e.g., mechanical, chemical and/or thermomechanical pulps. It can also be made from broke or recycled paper. The term MFC includes also parenchymal MFC. MFC can also be obtained from vegetable fibers, e.g., sugar beet or potato based MFC.

There are further synonyms for MFC such as cellulose microfibrils, fibrillated cellulose, nanofibrillated cellulose (NFC), fibril aggregates, nanoscale cellulose fibrils, cellulose nanofibers, cellulose nanofibrils, cellulose

microfibers, cellulose fibrils, cellulose nanofilaments, microfibrillar cellulose, microfibril aggregrates and cellulose microfibril aggregates.

Current research and development indicate that MFC may be a suitable material for packaging and coating of packaging substrates, due to its strength and barrier properties. Hence, MFC has the potential of replacing or supplementing currently used barrier films, including polymer and metal films, and also internal strength chemicals, surface coating and impregnation chemicals as well as barrier coating chemicals.

However, present methods for producing MFC in an energy and material efficient way have limited capacity, and upscaling as well as industrial use has proven to be a challenge. Also, there are high investment costs associated with manufacturing industrial scale volumes of MFC which leads to expensive production cost.

Objects

It is an object of the present invention to provide a method and system of producing MFC in which the disadvantages and problems of prior art are at least minimized.

It is also an object of the present invention to provide an improved method and system in which a new, alternative way of producing MFC is provided, especially in view of production capacity as well as energy and material efficiency.

Summary

The invention is defined by the appended independent claims, with embodiments being set forth in the dependent claims, in the following description and in the attached drawing.

According to a first aspect of the invention, there is provided a method for producing microfibrillated cellulose (MFC) comprising the steps of:

a) provide a refined pulp suspension mixed with an enzyme to a reactor tank;
b) treat the refined pulp suspension with said enzyme in the reactor tank at a temperature between 40-80°C under controlled mixing by means of an agitating member thus forming a pre-treated pulp suspension;
c) adding at least one oxidant to said pre-treated pulp suspension simultaneous to mixing;
d) perform oxidation of said pre-treated pulp suspension under elevated temperatures >80°C in said reactor tank for deactivating said enzyme; and
e) discharge the pre-treated, pre-refined pulp suspension and subjecting it to further mechanical treatment to obtain MFC.

Thanks to the invention, there is provided an improved method for pre-treating pulp which enables for improved control of enzyme pre-treatment of the pulp, applicable for pre-treated pulp. Thus, the invention enables for an efficient regulation/control of enzyme treatment with fast temperature increase for promoting enzyme hydrolysis, as well as a quick, even and efficient deactivation by means of oxidation under elevated temperatures for denaturation of the enzyme. This enables for industrially applicable high-yield production of MFC with predictable and high quality with small variation in fiber properties, such as fiber length, between different batches. It has been noted that the method and system according to the invention enables for elimination of unpredictable variation in fiber length between batches, and that a chosen fiber length is repeatable, i.e., the method leads to predictable fiber quality which is advantageous.

Other advantages achieved thanks to the inventive method and system are improved flow behavior of the pulp suspension, efficient mixing and refined control over temperature gradients during hydrolysis as well as deactivation of enzyme by means of oxidation, while keep the overall process energy-efficient (i.e., not consuming excessive energy for the deactivation of the enzyme only by heat). As a result, the production costs are minimized, and the end-product gets a competitive market price.

The MFC material obtained from the method according to the invention may be used for strength enhancement, e.g. in wet end, and other applications such as for creating barriers, paper coatings, surface sizing, adhesives, molded pulp, paper and/or paperboard making as well as in pulp production.

According to another aspect of the invention, the consistency of the pulp suspension in step a) is between 2-15wt%, more preferably 3.5-8wt %.

According to another aspect of the invention, the pulp suspension used in step a) may comprise a mixture of different types of fibers, such as kraft pulp, sulphite, fines, reinforcement fibers, dissolving pulp, TMP, CTMP or PGW. MFC can be produced from wood cellulose fibers, both from hardwood or softwood fibers. It can also be made from microbial sources, agricultural fibers such as wheat straw pulp, bamboo, bagasse, or other non-wood fiber sources. It is preferably made from pulp including pulp from virgin fiber, e.g. mechanical, chemical and/or thermomechanical pulps. It can also be made from broke or recycled paper. The pulp may be bleached or unbleached. The microfibrillar cellulose may contain some hemicelluloses; the amount is dependent on the plant source. The microfibrillated cellulose can be produced from never dried pulp. It has been found that never dried MFC has much higher accessibility for enzymes compared to MFC produced from dried pulp. It is also preferred that the microfibrillated cellulose has a very low lignin content since lignin could negatively affect the enzymatic activity.

According to another aspect of the invention, said refined pulp suspension in step a) is bleached kraft pulp with Kappa <50, more preferably <35, even more preferably <25, and hemicellulose content >5% such as >10%. Said refined pulp may also be partially recycled material including post-consumer waste or pre-consumer waste.

According to yet another aspect of the invention, said refined pulp suspension in step a) has a Schopper Riegler (SR) value between 15-40, preferably between 15-35.

According to yet another aspect of the invention, said enzyme is a hydrolysing agent, preferably cellulases, hemicellulases, lignases, swollenin or a mixture thereof. The cellulases can be exo or endoglucanases. The enzyme mix can also contain β-Glucosidases. It is understood that "enzyme treatment" can also be referred to as "enzymatic treatment" or "enzymatic cellulose hydrolysis". The amount of enzyme can be 20-500 ECU/g dry pulp but is not limited to such amount.

According to yet another aspect of the invention, the method comprises addition of starch to the hydrolysed pulp suspension before further oxidation, i.e. an amount of starch is added between steps b) and c). According to this aspect, both starch and enzyme-treated pulp are subjected to subsequent oxidation treatment at elevated temperature. This is beneficial because the resulting oxidized starch improves runnability during later homogenization step. Starch also improves fluidization and stability of the MFC.

According to yet another aspect of the invention, said oxidant is selected from the group comprising hydrogen peroxide (H_2O_2), peracetic acid (PAA), oxygen (O_2), ozone (O_3), sodium hydroxide (NaOH), sodium hypochlorite, potassium permanganate and combinations thereof. The hydrogen peroxide charge may vary between 2 – 40 kg/ADt, preferably between 2 – 16 kg/ADt. The higher charges require acidic or chelating stage for the used pulp in order to reduce the metal concentration of the pulp, mainly Copper, Manganese and Iron, which cause decomposition of the hydrogen peroxide. The ozone charge may vary between 3 – 20 kg/ADt, preferably between 3 – 12 kg/ADt. The usage of ozone requires pressurized reactor and acidic or chelating stage for the used pulp in order to reduce the metal concentration of the pulp, mainly Copper, Manganese and Iron, which cause ozone to degrade the degree of polymerization of the pulp. The charge of NaOH depends on the required pH of the main oxidizing chemical.

According to yet another aspect of the invention, the temperature of the content in the reactor tank in step c) is between 45-100°C, such as between 70-90°C. This means that the temperature of the pulp in the reactor tank is at least 45°C when the oxidant is added. It may also be between 45-70°C when the oxidant is added, and then the reactor tank is heated by means of a heat exchanger to a peak temperature between 90-100°C. Elevated temperatures will enhance oxidation and speed up denaturation of enzyme.

According to yet another aspect of the invention, the further mechanical treatment in step e) is homogenization and/or refining. The further mechanical treatment may comprise any suitable devices for processing the pulp to the MFC suspension. For example, the further mechanical treatment arrangement may comprise at least one mechanical fiber treatment apparatus. Each mechanical fiber treatment apparatus may be selected from the group of refiner, homogenizer/fluidizer, defibrator, deflaker, beater, friction grinder, high shear fibrillator (such as cavitron rotor/stator system, steam explosion system or high consistency refining or milling system), disperger, ball mill and other known mechanical fiber treatment apparatuses suitable to be used in processing of pulp to MFC, or combinations thereof. The pulp may be passed one or more times through each utilized mechanical fiber treatment apparatus. Optionally, the fibrillation treatment arrangement may further comprise one or more pretreatment apparatuses, wherein each pretreatment apparatus is arranged for pretreating the pulp mechanically, enzymatically or by chemical modification. For example, the fibrillation treatment arrangement may comprise at least one mechanical fiber pretreatment apparatus. Each mechanical fiber pretreatment apparatus may be selected from the group of refiner, defibrator, deflaker, beater, shredder, ball mill, rotor-stator mixer, ultrasonic treatment device, steam explosion device and other known mechanical fiber pretreatment apparatuses suitable for pretreating the cellulose fibers mechanically.

According to yet another aspect of the invention, said agitating member is a spiral mixer or the like. In one example of the invention, the agitating member

is arranged to generate an axial flow circulation pattern in the reactor tank, wherein the flow direction at the center of the tank is opposite compared to the flow direction at adjacent to the tank wall. The flow speed determined at, e.g., 1 cm from the reactor tank wall is at least 0.03 m/s and preferably at least 0.04 and most preferably at least 0.05 m/s, such as 0.05-0.3 m/s. The flow is preferably determined at least from the midpoint of the container (height/2) but can also be positioned at additional locations in order to determine flow stability and flow variations.

According to yet another aspect of the invention, the fiber length L_c (l) of the pulp after the step d) is 0.2-2 mm, preferably 0.3-1.7 mm measured with Valmet Fiber Image Analyzer (Valmet FS5) UHD using Valmet analyzer client 2.25 software.

According to yet another aspect of the invention, the amount of Fe in the pulp after treatment is less than 30 mg/kg and more preferably less than 17 mg/kg based on dry pulp, measured according to standard ISO 12830:2019.

According to yet another aspect of the invention, the amount of Copper in the pulp after treatment is less than 3.0 mg/kg preferably below 1.2 mg/kg based on the dry pulp measured according to standard ISO 12830:2019.

According to yet another aspect of the invention, the amount of Manganese in the pulp after treatment is less than 1.5 mg/kg preferably below 1.0 mg/kg based on the dry pulp measured according to standard ISO 12830:2019.

According to yet another aspect of the invention, the method is arranged to be operated as a semi-continuous process. Thanks to the set-up according to the invention, two or more reactor tanks can be used parallel and designed so that the enzymatic treatment and deactivation occurs in one of them and one of them is feeding material to the further mechanical treatment to obtain MFC.

According to another aspect of the invention, said system comprises a control arrangement including a self-learning artificial intelligence unit based on Convolutional Neural Network arranged to assist in temperature control of the pulp material. It is also within the ambit of the invention to include deep learning and/or machine vision or other applicable systems that can be used for IR imaging of the reactors and provide improved feedback loop for improved temperature regulation and thus optimize pulp treatment including e.g. hydrolysis and oxidation.

Brief description of the drawings

Fig. 1 schematically illustrates a system according to one example of the invention.

Detailed description

The drawing schematically illustrates a system in which the present invention can be practiced.

Fig. 1 schematically shows a system 1 according to the invention for pre-treating cellulose pulp before production of MFC. As seen herein, said system 1 comprises a reactor tank 2 configured to receive refined pulp suspension P. A hydrolysing agent in the form of an enzyme 3 is added to the pulp suspension before said pulp P is added to the reactor tank 2 meaning that enzyme and pulp will enter the reactor tank 2 as a mixture. The enzyme may also be added to the reactor tank 2 separately. Said pulp suspension is treated in the reactor tank 2 at a temperature between 40-80°C under controlled mixing by means of an agitating member 7, thus forming a pre-treated pulp suspension. The amount of enzyme can be 20-500 ECU/g dry pulp but is not limited to such amount. When the hydrolyzation of pulp has reached a desired level, treatment is interrupted by means of denaturating the enzyme. According to the present invention, this is achieved by means of adding at least one oxidant 4 to said pre-treated pulp suspension simultaneous to mixing and perform oxidation of said pre-treated pulp

suspension under elevated temperatures >80°C in said second reactor tank 2. A temperature controlling unit such as at least one heat exchanger 5 is coupled in a closed loop to the reactor tank 2 and is arranged to control and adjust the temperature of the reactor tank content. The temperature and mixing in the reactor tank 2 are thus optimized to achieve an efficient and even enzymatic hydrolysis treatment of the pulp, as well as to elevate the temperature during denaturating by means of adding oxidant 4. A sufficient deactivation level of the enzyme is achieved when no detectable residual enzyme remains. Deactivation of enzyme in the pulp slurry can be measured by means on conventional methods known to the person skilled in the art, such as by use of e.g. Megazyme Cellulase Assay Kit (Product code: "K-CellG5-4V"). Once deactivation is completed, the pre-treated pulp inside the reactor tank 2 may be cooled by means of the heat exchanger 5.

The pre-refined and pre-treated pulp is subsequently discharged and subjected to further mechanical treatment 6 to obtain MFC. The further mechanical treatment 6 may be achieved in a homogenizer or a refining unit. In this regard, at least one homogenizer 6 is coupled to said reactor tank 2 and arranged to subject the pulp to mechanical treatment to obtain MFC by circulating the material for a required time with targeted fibrillation gap, pressure and flow. It is conceivable to install multiple homogenizers, such as 2 – 5 homogenizers, in series with intermediate heat exchangers for cooling the material. The number of homogenizers in series depends on the required average size and distribution (i.e., fibrillation degree) required for the MFC in the application for which it is intended.

In some cases, the oxidant may case a high pH level of the pulp. It is thus conceivable to add pH adjusting additives before mechanical treatment of the pulp.

In one embodiment, the system 1 comprises at least two reactor tanks arranged in parallel. According to the invention, such plurality of reactor tanks

is arranged to provide an alternating treatment of pulp such that a semi-continuous production of MFC can be obtained.

The invention also relates to a method for pre-treating pulp in MFC production. The method comprises the following steps:

a) provide a refined pulp suspension P mixed with an enzyme to a reactor tank 2;

b) treat the refined pulp suspension with said enzyme 3 in the reactor tank 2 at a temperature between 40-80°C under controlled mixing by means of an agitating member 7 thus forming a pre-treated pulp suspension;

c) adding at least one oxidant 4 to said pre-treated pulp suspension simultaneous to mixing;

d) perform oxidation of said pre-treated pulp suspension under elevated temperatures >80°C in said reactor tank for deactivating said enzyme; and

e) discharge the pre-treated, pre-refined pulp suspension and subjecting it to further mechanical treatment 6 to obtain MFC.

Examples

Example 1

Softwood kraft pulp was pre-refined to SR 18, and enzyme-treated with 80 ECU/dry cellulose g Ecopulp R (AB Enzymes) cellulase for 90 min. The concentration of the pulp was 5.0%. A laboratory sample was collected from the process. The initial enzyme activity was determined from the supernatant after centrifugation with Cellulase Assay Kit CellG5 (Megazyme) with the exception that the incubation time was prolonged to 16 h for improved sensitivity. After the enzyme activity had been determined, the enzyme treated pulp was heated to 90 ºC for 120 min and cooled down to room temperature. Then, 30% hydrogen peroxide solution was added to the enzyme treated pulp and the pulp was mixed with a mixing rod for 1 min. The sample was standing at room temperature 10-30 min. After that the sample

was centrifuged and the supernatant was collected and stored in the refrigerator overnight. The next day the enzyme activity of the supernatant was analyzed in the same way as above.

The enzyme activity was 0.012 in the heat and hydrogen peroxide treated sample compared to 0.035 before the treatments.

Example 2

The same enzyme treated pulp as in Example 1 was used after the enzyme activity determination. 30% hydrogen peroxide solution was added to the enzyme treated pulp and the pulp was mixed with a mixing rod for 1 min. After that, the enzyme treated pulp was heated to 90ºC for 120 min and cooled down to room temperature. Then, the pulp was centrifuged, and the enzyme activity of the supernatant was determined similarly as in Example 1.

The enzyme activity was 0.000. This shows that the hydrogen peroxide should be added before the heat treatment for the optimal deactivation of the enzyme.

Test point	Pre-treatment	T(C)	Time (min)	After-Treatment	Enzyme activity
No treatment	No	-	-	No	0.035
H2O2 addition after heat treatment	No	90	120	0.225 g/dm3 H2O2 100%	0.012
H2O2 addition before heat treatment	0.225 g/dm3 H2O2 100%	90	120	No	0.000

Table 1: Results from Example 1 and 2

To summarize, a simplified and improved process for the manufacture of MFC is proposed and implemented in industrial scale. The resulting pre-treated pulp will comprise an even quality thanks to the efficient hydrolysing in said reactor tank(s) and the careful control of process parameters such as temperature changes, mixing operation and even distribution inside the reactor tank(s). Scalability is judged to be improved compared with the current process solution due the simplification and so is the ability to keep the process at high hygienic standard.

In view of the above detailed description of the present invention, other modifications and variations will become apparent to those skilled in the art. However, it should be apparent that such other modifications and variations may be done without departing from the spirit and scope of the invention.

CLAIMS

1. A method for producing microfibrillated cellulose (MFC) comprising the steps of:

a) provide a refined pulp suspension mixed with an enzyme to a reactor tank;

b) treat the refined pulp suspension with said enzyme in the reactor tank at a temperature between 40-80°C under controlled mixing by means of an agitating member thus forming a pre-treated pulp suspension;

c) adding at least one oxidant to said pre-treated pulp suspension simultaneous to mixing;

d) perform oxidation of said pre-treated pulp suspension under elevated temperatures >80°C in said reactor tank for deactivating said enzyme; and

e) discharge the pre-treated, pre-refined pulp suspension and subjecting it to further mechanical treatment to obtain MFC.

2. A method according to claim 1, wherein the consistency of the pulp suspension in step a) is between 3.5-8wt %.

3. A method according to claim 1, wherein said refined pulp suspension in step a) is bleached kraft pulp with Kappa <25 and hemicellulose content >5%.

4. A method according to claim 1, wherein said refined pulp is recycled material including post-consumer waste or pre-consumer waste.

5. A method according to claim 1, wherein said refined pulp suspension in step a) has a Schopper-Riegler (SR) value between 15-40, preferably between 15-35.

6. A method according to claim 1, wherein said enzyme is a hydrolysing agent, preferably cellulase.

7. A method according to claim 1, comprising addition of starch between steps b) and c).

8. A method according to claim 1, wherein said oxidant is selected from the group comprising H_2O_2, peracetic acid (PAA), oxygen (O_2), ozone (O_3), NaOH, sodium hypochlorite, potassium permanganate and combinations thereof.

9. A method according to claim 1, wherein the temperature of the content in the reactor tank in step c) is between 45-100°C, such as between 70-90°C.

10. A method according to claim 1, wherein the further mechanical treatment in step e) is homogenization and / or refining.

11. A method according to claim 1, where the agitating member is a spiral mixer.

12. A method according to claim 1, where the fiber length L_c (l) of the pulp after the step d) is 0.2-2 mm, preferably 0.3-1.7 mm measured with FS5 fiber analyzer.

13. A method according to claim 1, wherein the amount of Fe in the pulp after treatment is less than 20 mg/kg and more preferably less than 15 mg/kg based on dry pulp.

14. A method according to claim 1, wherein the amount of cupper in the pulp after treatment is less than 0.15 mg/kg based on dry pulp.

15. A system for producing microfibrillated cellulose (MFC) comprising:
-a reactor tank (2) being configured to receive refined pulp suspension (P) mixed with an enzyme (3) and being arranged to support hydrolysis of the pulp at temperatures between 40-80°C under controlled mixing by means of an agitating member (7) thus forming a pre-treated pulp suspension;
-an arrangement for adding at least one oxidant (4) to said pre-treated pulp suspension simultaneous to mixing in the tank (2);

-at least one heat exchanger unit (5) coupled in a closed loop to the reactor tank (2), arranged to control the temperature of the reactor tank content;
-discharge means for discharging the deactivated, pre-treated pulp suspension for further refining to obtain MFC.

16. A system according to claim 15, further comprising at least one fluidizer, high speed mixer, extruder, grinder, refiner or preferably one homogenizer (6) coupled to said reactor tank (2) and arranged to subjecting the hydrolysed pulp to mechanical treatment to obtain MFC.

17. A system according to claim 15, further comprising a control arrangement including a self-learning artificial intelligence unit based on Convolutional Neural Network arranged to assist in temperature control of the pulp material.

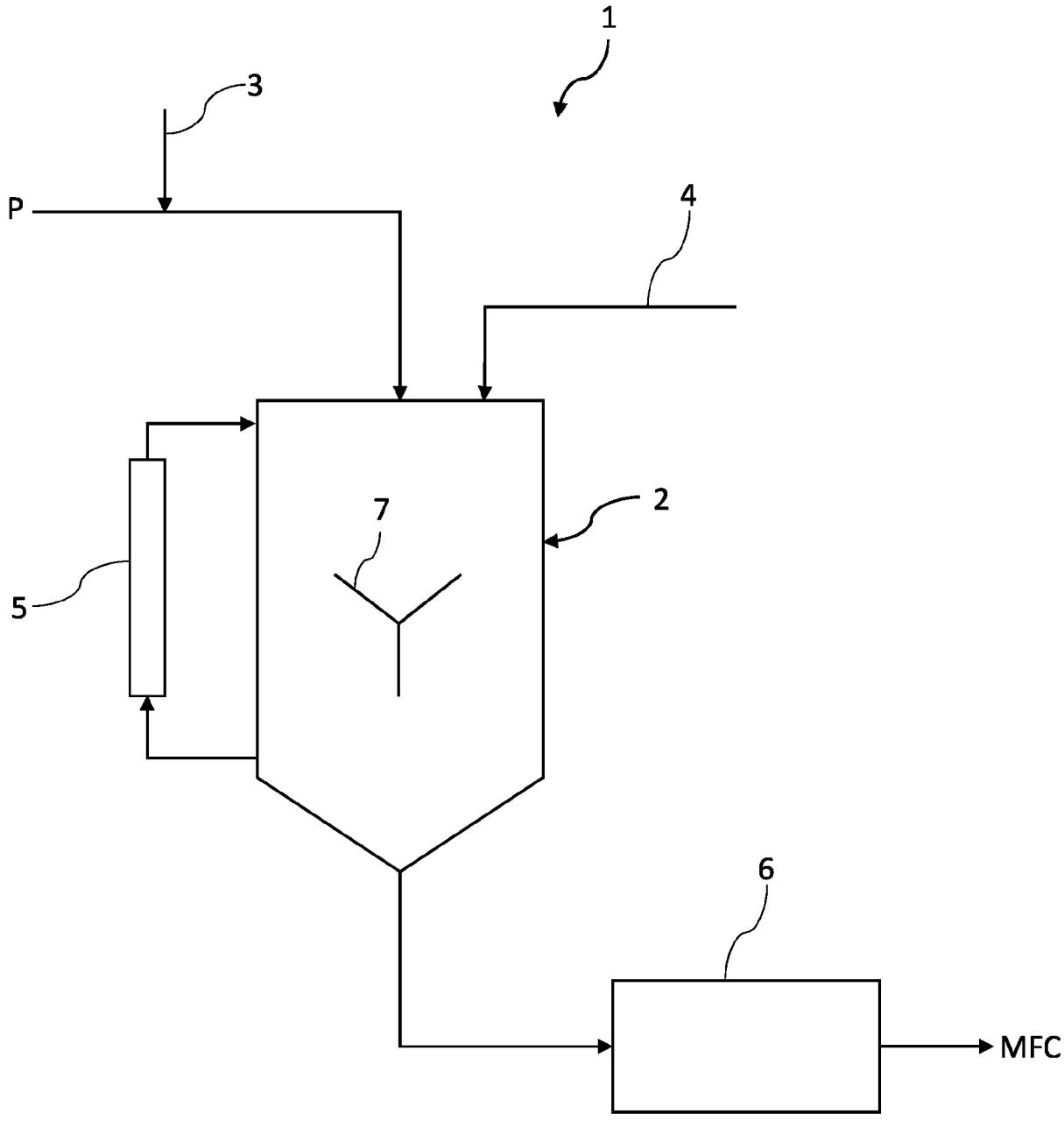


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER

IPC: see extra sheet

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C08L, C12N, D21C, D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE, DK, FI, NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, PAJ, WPI data, BIOSIS, COMPENDEX, EMBASE, INSPEC, KIME

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 2018071015 A (DAIO SEISHI KK), 10 May 2018 (2018-05-10); paragraphs [0015]-[0016], [0024], [0028], [0032]-[0039], [0049]; figures 1-2	15-17
A	--	1-14
Y	JP 2018048237 A (DAIO SEISHI KK), 29 March 2018 (2018-03-29); paragraphs [0017]-[0019], [0026]-[0032]; figure 1	15-17
A	--	1-14
A	CN 112376305 A (UNIV SOUTH CHINA TECH), 19 February 2021 (2021-02-19); whole document	1-17
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☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

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Date of the actual completion of the international search	Date of mailing of the international search report
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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2015177548 A1 (CELLUCOMP LTD ET AL), 26 November 2015 (2015-11-26); whole document --	1-17
A	HENRIKSSON, M., HENRIKSSON, G., BERGLUND, L.A., LINDSTRÖM, T. "An environmentally friendly method for enzyme-assisted preparation of microfibrillated cellulose (MFC) nanofibers" In: European Polymer Journal, 2007, Vol. 43, pp. 3434-3441, DOI: 10.1016/j.eurpolymj.2007.05.038; whole document --	1-17
A	PÄÄKKÖ, M. et al. "Enzymatic hydrolysis combined with mechanical shearing and high-pressure homogenization for nanoscale cellulose fibrils and strong gels" In: Biomacromolecules, 2007, Vol. 8, pp. 1934-1941, DOI: 10.1021/BM061215P; whole document -- --------	1-17

International application No.
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Continuation of: second sheet

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