(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
05 December 2024 (05.12.2024)

(10) International Publication Number
WO 2024/246762 A1

(51) International Patent Classification:
D21H 11/18 (2006.01) *D21C 9/00* (2006.01) *C08L 1/02* (2006.01) *C12N 9/24* (2006.01) *D21C 5/00* (2006.01)

(21) International Application Number: PCT/IB2024/055201

(22) International Filing Date: 29 May 2024 (29.05.2024)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2330251-6 02 June 2023 (02.06.2023) SE

(71) Applicant: STORA ENSO OYJ [FI/FI]; P.O. Box 309, 00101 Helsinki (FI).

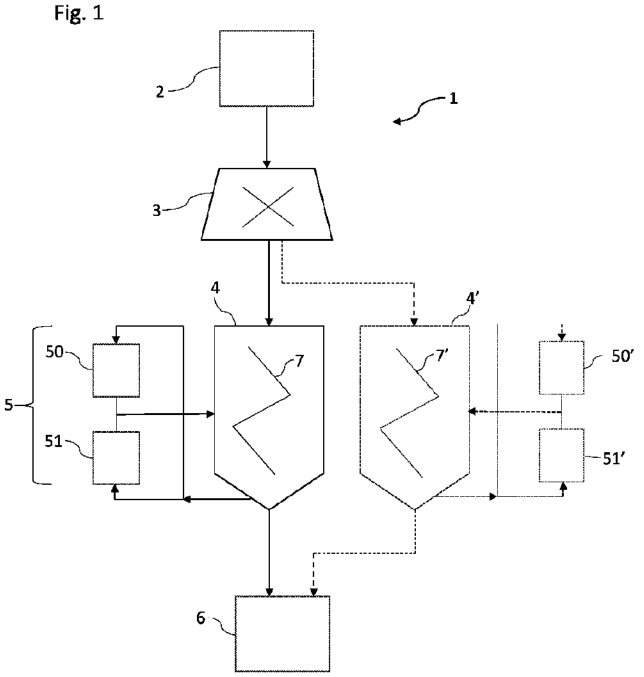
(72) Inventors: KARPPINEN, Anni; Nassakkakuja 6 E, 02230 Espoo (FI). SUHONEN, Mikko; Auraajantie 3A, 54100 Joutseno (FI). BACKFOLK, Kaj; Imatrantie 13 A6, 53100 Lappeenranta (FI).

(74) Agent: FORSBERG, Karin; Stora Enso AB, Group Intellectual Property, Box 9090, 65009 Karlstad (SE).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CV, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IQ, IR, IS, IT, JM, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, MG, MK, MN, MU, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH,

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

Fig. 1



(57) **Abstract:** The present document discloses a method and a system for producing MFC. The method according to the invention comprises the following steps: a) providing a pulp suspension comprising cellulose fibers to a buffer tank (2) wherein the temperature of said pulp suspension is controlled and adjusted for optimized enzymatic function, preferably ≥20°C b) transferring the pulp suspension to a refining unit (3) and refining said pulp to obtain a pre-refined pulp suspension, wherein a hydrolysing agent in the form of an enzyme is added to the pulp suspension before and/or after pre-refining; c) feeding said pre-refined and temperature-regulated pulp suspension to a reactor tank (4, 4'); d) hydrolyzing the refined pulp suspension in said reactor tank (4, 4') at a temperature between 40-70°C under controlled mixing using an agitating member (7); e) deactivating the enzyme in said reactor tank by means of heating the hydrolysed, pre-refined pulp suspension to a temperature >80°C f) cooling said hydrolysed, pre-refined pulp suspension; g) discharge

WO 2024/246762 A1

[Continued on next page]

TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, CV, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SC, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, ME, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report (Art. 21(3))*

— *in black and white; the international application as filed contained color or greyscale and is available for download from PATENTSCOPE*

the hydrolysed, pre-refined pulp suspension and subjecting it to further mechanical treatment to obtain MFC.

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 also includes 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.

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) providing a pulp suspension comprising cellulose fibers to a buffer tank wherein the temperature of said pulp suspension is controlled and adjusted for optimized enzymatic function, preferably ≥20°C;

b) transferring the pulp suspension to a pre-refining unit and refining said pulp to obtain a pre-refined pulp suspension, wherein a hydrolysing agent in the form of an enzyme is added to the pulp suspension before and/or after pre-refining;

c) feeding said pre-refined and temperature-regulated pulp suspension to a reactor tank;

d) hydrolyzing the refined pulp suspension in said reactor tank at a temperature between 40-75°C under controlled mixing using an agitating member;

e) deactivating the enzyme in said reactor tank by means of heating the hydrolysed, pre-refined pulp suspension to a temperature >85°C;

f) cooling said hydrolysed, pre-refined pulp suspension; and

g) discharging the hydrolysed, 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 a semi-continuous production and improved temperature management and mixing, and also applicable for medium and/or high consistency pulp or pre-treated pulp. Furthermore, the invention enables for an efficient temperature profile in the reactor tanks with fast temperature increase for promoting enzyme hydrolysis, as well as an even temperature control in the hydrolyzed pulp for efficient, even and fast enzyme deactivation. 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, while keep the overall process energy-efficient (i.e., not consuming excessive energy). As a result, the production costs are minimized, and the end-product gets a competitive market price.

According to another aspect of the invention, the pulp suspension is refined in said pre-refining unit at a consistency between 0.5 – 12wt%, preferably between 4 – 7wt%.

According to another aspect of the present invention, the pulp suspension is refined in said pre-refining unit to 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”.

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 fibers, 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 microfibrillar cellulose may contain some hemicelluloses;

the amount is dependent on the plant source. The microfibrillated cellulose can also 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 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, the agitating member is arranged to generate an axial flow circulation pattern in said 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. One example of an agitating member suitable for use in a method according to the invention is a spiral mixer. 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.

The invention also relates to a system for producing MFC comprising:
-a buffer tank which is configured to receive refined pulp and control and adjust pulp temperature for optimized enzymatic function, preferably ≥20°C;
-a pre-refining unit coupled to the buffer tank;
-a reactor tank coupled to said pre-refining unit, said reactor tank being configured to receive heated pulp together with added enzyme, and to perform hydrolysis of the refined pulp at temperatures between 40-75°C

under controlled mixing using an agitating member (i.e., spiral mixer or the like), thus forming a pre-treated pulp suspension;

-at least one heat exchanger unit coupled in a closed loop to the reactor tank, arranged to control the temperature of the reactor tank content and deactivate enzyme present in the pulp;

-discharge means for discharging deactivated, pre-treated pulp suspension for further refining to obtain MFC.

According to another aspect of the invention, the system comprises at least one homogenizer arranged downstream of said reactor tank and arranged to subjecting pulp to mechanical treatment to obtain MFC.

According to another aspect of the invention, the system comprises at least one post-refiner unit arranged downstream of said reactor tank and upstream of said at least one homogenizer.

According to another aspect of the invention, the system comprises at least one screening unit arranged downstream of said reactor tank and upstream of said at least one homogenizer.

According to another aspect of the invention, the system comprises more than one reactor tank arranged in parallel to each other, and configured to alternatingly discharge pre-refined, hydrolysed pulp suspension to said at least one homogenizer(s).

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 optimized pulp treatment including hydrolysis.

Brief description of the drawings

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

Fig. 2 schematically illustrates a preferred circulation pattern inside a reactor tank.

Detailed description

The drawings schematically illustrate 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 buffer tank 2 configured to receive pulp, and control/adjust the temperature of said pulp for optimized enzymatic function, preferably ≥20°C depending on the enzyme. For instance, the pulp inside the buffer tank can be adjusted to a temperature between 20-85°C. Sometimes the pulp inside the buffer tank 2 may be warmed to a temperature ≥50°C, preferably between 50-85°C, or between 50-75°C. A pre-refining unit 3 is coupled to the buffer tank 2, and pulp is fed to said unit 3 at controlled flow. The pulp suspension is refined in said pre-refining unit 3 at a consistency between 0.5 - 12wt%. A hydrolysing agent in the form of an enzyme is added to the pulp suspension before and/or after the pre-refining step. Preferably, the enzyme is added under high shear mixing in order to become evenly mixed with the pulp. At least one reactor tank 4 is coupled to said pre-refining unit 3, said reactor tank 4 being configured to receive the refined, heated pulp together with the added enzyme. The pulp is subjected to hydrolysis in said reactor tank 4 at temperatures between 40-75°C under controlled mixing using an agitating member 7, i.e., a spiral mixer or the like, thus forming a pre-treated pulp suspension. The enzyme may be added at one or several dosing points in order to make mixing more efficient. Also, the dosing of enzyme may be controlled based on the desired MFC quality and the measured properties of the pulp that is received in the buffer tank 2 (pulp consistency, pH, temperature) or after pre-refining in the pre-refining unit 3 (fiber length, Schopper Riegler number). A temperature regulating unit, such as at least one heat exchanger 50, 51 is/are coupled in a closed loop to the reactor tank 4, arranged to control and adjust the temperature of the reactor tank content. The temperature and mixing in the reactor tank are optimized to achieve an efficient and even enzymatic hydrolysis treatment of the pulp.

After the desired treatment time, i.e., when the desired fiber length and fine content is achieved in the slurry, the enzyme present in the reactor tank 4 is to be deactivated to stop hydrolysis. Fiber length may be measured e.g. by means of an FS5 fiber analyser. Deactivation of enzyme is achieved by means of increasing the temperature of the pulp to at least >85°C while ensuring an even mixing of the pulp to secure deactivation of enzyme in the entire pulp volume. Deactivation temperature may even be >90°C such as between 90-99°C. According to the invention, the spatial temperature control in the reactor tank 4 is optimized such that temperature variations in the reactor tank content once a heating or cooling operation has been performed are less than 25°C preferably less than 15°C, such as between 0.5-15°C, measured in different parts of the reactor or over time during at least 30 minutes and preferably at least 60 min. In other words, the temperature profile in the reactor tank is kept homogeneous and thus full deactivation of enzyme is achieved within a short time. 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”).

The system also comprises discharge means for discharging the deactivated, pre-treated pulp suspension for further refining to obtain MFC.

In one embodiment, the system 10 comprises at least two reactor tanks 4, 4’ arranged in parallel, where each of them is coupled both to said upstream pre-refining unit 3 as well as to downstream continued treatment. In Fig. 2, a second reactor tank 4’ is illustrated in dashed lines, the second reactor tank 4’ being arranged in parallel to the first reactor tank 4. According to the invention, the plurality of reactor tanks 4, 4’ are arranged to provide an alternating treatment of pulp such that a semi-continuous production of MFC can be obtained. This means when a first reactor tank 4 has been filled with pulp, enzyme has been added and hydrolyzation under controlled mixing and temperature-regulation has been performed, the second reaction tank 4’ is ready for discharge of the pre-treated, hydrolysed pulp. Once discharge of the second tank 4’ is done, new pulp is fed and enzyme dosing is done, the first tank 4 is ready to discharge its batch, and so on. Evidently, more than two reactor tanks may be arranged in parallel to increase capacity of the system.

At least one homogenizer 6 is coupled to said reactor tank(s) 4, 4’ 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 one embodiment, the system 10 according to the invention comprises at least one post-refiner unit (not shown) arranged between said reactor tank(s) 4, 4’ and said at least one homogenizer 6.

The pre-treated, hydrolysed pulp discharged from the reactor tank(s) 4, 4’ is to be subjected to mechanical treatment in order to obtain MFC. 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.

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

a) providing a pulp suspension comprising cellulose fibers the buffer tank 2 wherein said pulp suspension is warmed up/heated and kept at a temperature ≥20°C;

b) transferring the pulp suspension to the pre-refining unit 3 and refining said pulp to obtain a pre-refined pulp suspension, wherein a hydrolysing agent in the form of an enzyme is added to the pulp suspension before and/or after pre-refining;

c) feeding said pre-refined and temperature-regulated pulp suspension to one of the reactor tank(s) 4, 4’;

d) hydrolyzing the refined pulp suspension by means of enzyme treatment in said reactor tank 4, 4’ at a temperature between 40-75°C under controlled mixing using an agitating member 7;
e) deactivating the enzyme in said reactor tank by means of heating the hydrolysed, pre-refined pulp suspension to a temperature >85°C;
f) cooling said hydrolysed, pre-refined pulp suspension;
g) discharge the hydrolysed, pre-refined pulp suspension and subjecting it to further treatment to obtain MFC. The targeted fibrer length for the hydrolysed pulp is from 15 to 90 % from the original fiber length of the raw material.

Hydrolysation treatment by means of enzymes is performed in said reactor tank(s) 4, 4’. The enzyme is a wood degrading enzyme, preferably cellulase enzyme. Enzymatic hydrolysis of cellulose is preferably, but not limited to cellulases. These typically catalyze the hydrolysis of the beta-1,4-glucosidic linkages in cellulose chains of the pulp fibers. The cellulases can be exoglucanase or endoglucanase or a mixture of these. The enzyme or enzyme mixture or enzyme “cocktail” might preferably contain other wood degrading or hydrolysis chemicals such as hemicellulases such as xylanase and/or mannase. The amount of enzyme can be 20-500 ECU/g dry pulp but is not limited to such amount.

The pulp is preferably hardwood or softwood. Preferably, the pulp is bleached kraft pulp with Kappa <25 and hemicellulose content >5%.

An agitating member such as a mixer 7, 7’ is provided in said tank(s) 4, 4’ to obtain an even mixing of the tank content and a homogenous temperature control of the suspension. An example of a suitable agitating member is a spiral mixer which reaches the bottom and the inner walls of the tank 4, 4’. Preferably, the agitating member 7, 7’ is arranged to generate an axial flow circulation pattern **F** in the reactor tank 4, wherein the flow direction at the center of the tank is opposite compared to the flow direction at adjacent to the tank wall. An example of preferred flow pattern is illustrated schematically in

Fig. 2 (no mixer shown herein). The flow speed determined at, e.g., 1 cm from the reactor tank wall 40 (see Fig. 2) is at least 0.03 m/s and preferably at least 0.04 m/s, 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 4 (height/2) but can also be positioned at additional locations in order to determine flow stability and flow variations.

By generating a homogenous mixing of the pulp suspension inside the reactor tank 4, 4', the enzyme distribution is kept even throughout the material and the pulp will be evenly treated/hydrolysed throughout its volume. Also, the temperature, enzyme concentration and pH of the pulp can be controlled and adjusted if needed and the pulp which is discharged from the tank will have constant properties. Moreover, the deactivation of the enzyme is more efficient when the temperature can be increased rapidly and evenly in the whole volume of the tank 4, 4'. Cooler areas in the tank 4, 4' would risk leaving some of the enzyme active, which could lead to further hydrolysis of the fibers/fibrils and decrease in the MFC quality. The deactivation level of the enzyme is achieved when no detectable residual enzyme remains in the pulp. The minimum capacity of the MFC plant may start from 1 ton dry MFC per day.

<u>Examples</u>

Example 1

Pre-refined pulp with SR value of 32 and enzyme were mixed in a 28 m^3 tank with propeller mixer in the bottom and 2 circulation loops of the material from the bottom to the top of the tank. The dry content of the material was 5.3wt%.

After enzymatic treatment, the temperature of the material was increased to over 90 ºC with a heat exchanger in one of the circulation flows. After the material had reached this temperature according to a temperature sensor placed in the heat exchanger loop, the temperature in different areas of the tank was observed with an IR camera on the surface of the tank. There were large colder areas in the bottom of the tank and near the wall further away from the circulation loops. Part of the material had flow rate of zero (i.e. not moving).

This uneven temperature profile results in uneven deactivation of enzymes but also means that kinetics of cellulose hydrolysis will vary. Uneven hydrolysis will further increase the amount of reject and intact fiber and possible risk of higher content of dissolved low molecular weight material in the water phase.

Example 2

Pre-refined pulp and enzyme were mixed in the same tank as in the Example 1, but the dry content of the pre-refined pulp was lowered to 4.3wt%. At this concentration, the viscosity of the pulp was so low that the propeller mixer moved the pre-refined pulp in the whole tank volume. After the enzymatic treatment, the temperature was increased to over 90 ºC. When the temperature sensor in the heat exchanger loop showed that the temperature had reached 90 ºC, the temperature was observed with an IR camera from the outside of the tank. Compared to Example 1, the temperature was even throughout the inspected area.

Example 3

Hydrolysed, refined pulp suspension was mixed in a 25 m^3 tank with spiral mixer in the bottom and 1 circulation loop of the material from the bottom to the top of the tank. The dry content of the material was 4.2wt%. The vertical flow speed 1 cm from the tank wall was 0.28 m/s. The material was heated to 37.2 ºC. When the temperature sensor in the heat exchanger loop showed that the temperature had reached 37.2ºC, the temperature was observed with an IR camera from the outside of the tank. Compared to Example 1, the temperature was even throughout the inspected area. The maximum measured temperature difference was 7°C. However, the measurements were effected by the mirroring effect of the tank steel surface via change in emissivity. The emissivity changes according to the distance and angle between the measurement point and FLIR-camera. The real temperature difference is smaller than 7°C.

The preferred setup according to the invention comprises a reactor and temperature sensors inside the reactor (i) as well as prior (ii), after (iii) and optionally inside the heat exchanger (iv). The temperature stability and homogeneity can be followed by monitoring the temperature at the various positions (i-iv) with one or several temperature sensors. The example below presented in Table 1 illustrates particularly the role of mixing on the temperature homogeneity in the reactor.

	Example 1 Comparative	**Example 2**	**Example 3**
Pulp consistency	5.3 wt%	4.3 wt%	4.2 wt%
Mixer	Standard	Standard	Spiral
Vertical pulp flow	Low	High	High
Temperature variations	High (>20 degrees)	Low (< 10 degrees)	Low (< 7 degrees)
Deactivation time (>90)	120 min	120 min	-
Residual enzyme activity	high	low	-

Table 1

To summarize, a simplified process for the manufacture of MFC is proposed and implemented in industrial scale thanks to the possibility of being operated in a semi-continuous manner. 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) providing a pulp suspension comprising cellulose fibers to a buffer tank (2) wherein the temperature of said pulp suspension is controlled and adjusted for optimized enzymatic function, preferably ≥20°C

b) transferring the pulp suspension to a refining unit (3) and refining said pulp to obtain a pre-refined pulp suspension, wherein a hydrolysing agent in the form of an enzyme is added to the pulp suspension before and/or after pre-refining;

c) feeding said pre-refined and temperature-regulated pulp suspension to a reactor tank (4, 4');

d) hydrolyzing the refined pulp suspension in said reactor tank (4, 4') at a temperature between 40-70°C under controlled mixing using an agitating member (7);

e) deactivating the enzyme in said reactor tank by means of heating the hydrolysed, pre-refined pulp suspension to a temperature >80°C

f) cooling said hydrolysed, pre-refined pulp suspension;

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

2. A method according to claim 1, wherein the pulp suspension is refined in said refining unit at a consistency between 0.5 - 12wt%, preferably between 4 – 7wt%.

3. A method according to any one of the previous claims, wherein the pulp suspension is refined in said refining unit to a Schopper Riegler (SR) value between 15-40, preferably between 15-35.

4. A method according to any one of the previous claims, wherein said hydrolysis is obtained by means of enzyme treatment, preferably wherein the enzyme is a wood degrading enzyme, preferably cellulase enzyme.

5. A method according to any one of the previous claims, arranged to be operated as a semi-continuous process.

6. A method according to any one of the previous claims, wherein the agitating member (7) is arranged to generate an axial flow circulation pattern (F) in said reactor tank (4), wherein the flow direction at the center of the tank is opposite compared to the flow direction at adjacent to the tank wall.

7. A method according to any one of the previous claims, wherein heating and cooling of the reactor tank content is achieved by means of at least one closed loop heat exchange system (50, 51).

8. A system for producing MFC comprising:
-a buffer tank (2), said buffer tank being configured to receive refined pulp and adjust the temperature of said pulp to a temperature ≥20°C;
-a pre-refining unit (3) coupled to the buffer tank (2);
-a reactor tank (4) coupled to said pre-refining unit (3), said reactor tank (4) being configured to receive heated pulp together with added enzyme, and to support hydrolysis of the refined pulp at temperatures between 40-75°C under controlled mixing using an agitating member (7), thus forming a pre-treated pulp suspension;
-at least one heat exchanger unit (5) coupled in a closed loop to the reactor tank (4), 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.

9. A system according to claim 8, further comprising at least one fluidizer, high speed mixer, extruder, grinder, refiner or preferably one homogenizer (6)

coupled to said reactor tank (4) and arranged to subjecting pulp to mechanical treatment to obtain MFC.

10. A system according to claim 9, further comprising at least one post-refiner unit arranged between said reactor tank (4) and said at least one homogenizer (6)

11. A system according to claim 8, comprising more than one reactor tank (4, 4') arranged in parallel to each other, and configured to alternatingly discharge pre-refined, hydrolysed pulp suspension to said at least one homogenizer(s) (6).

12. A system according to claim 8, wherein said system (1) 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.

Fig. 1

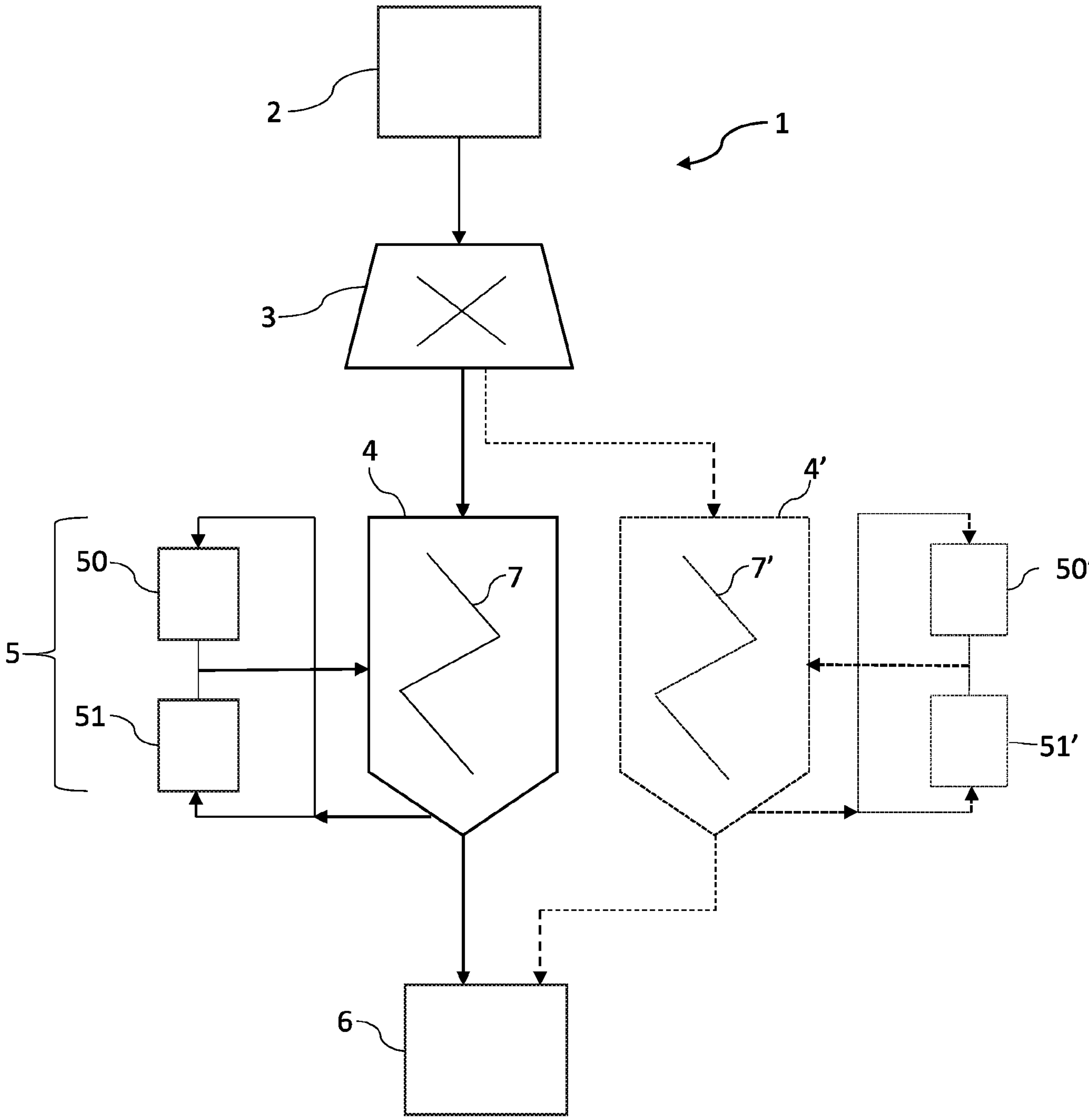
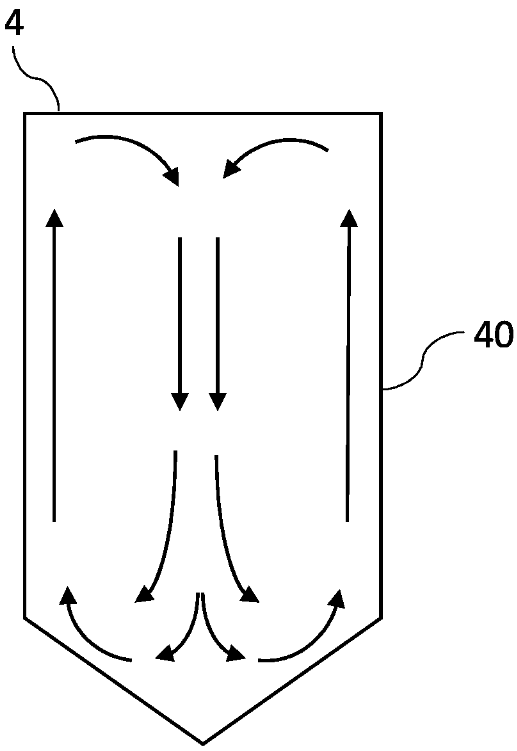


Fig. 2



INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2024/055201

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CN 112376305 A (UNIV SOUTH CHINA TECH), 19 February 2021 (2021-02-19); page 1, line 32 - line 33; page 1, line 37 - page 2, line 9; page 2, line 20 - line 24; page 3, line 17 - line 38 --	1-12
Y	JP 2018071015 A (DAIO SEISHI KK), 10 May 2018 (2018-05-10); paragraphs [0001], [0016]-[0023] --	1-12
A	JP 2018048237 A (DAIO SEISHI KK), 29 March 2018 (2018-03-29); paragraphs [0017], [0026]-[0027]; figure 1 --	8-12

☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"D" document cited by the applicant in the international application

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
25-06-2024	25-06-2024

Name and mailing address of the ISA/SE	Authorized officer
Patent- och registreringsverket Box 5055 S-102 42 STOCKHOLM Facsimile No. + 46 8 666 02 86	Erik Johansson Telephone No. + 46 8 782 28 00

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 20170370047 A1 (NELSON KIMBERLY ET AL), 28 December 2017 (2017-12-28); whole document --	1-12
A	US 20140124150 A1 (SABOURIN MARC ET AL), 8 May 2014 (2014-05-08); whole document --	1-12
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 --	1-12
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 -- --------	1-12

Continuation of: second sheet

International Patent Classification (IPC)

D21H 11/18 (2006.01)
C08L 1/02 (2006.01)
D21C 5/00 (2006.01)
D21C 9/00 (2006.01)
C12N 9/24 (2006.01)

CN	112376305	A	19/02/2021	NONE			
JP	2018071015	A	10/05/2018	JP	6243991 B1	06/12/2017
JP	2018048237	A	29/03/2018	JP	6905318 B2	21/07/2021
US	20170370047	A1	28/12/2017	CA	3063062 A1	04/01/2018
				US	10753042 B2	25/08/2020
				WO	2018005259 A2	04/01/2018
US	20140124150	A1	08/05/2014	US	8906198 B2	09/12/2014
				US	20150090412 A1	02/04/2015
				WO	2014070452 A1	08/05/2014