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

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
02 January 2025 (02.01.2025)

WIPO | PCT

(10) International Publication Number
WO 2025/003577 A1

(51) International Patent Classification:
C08L 97/00 (2006.01)

(21) International Application Number:
PCT/FI2024/050364

(22) International Filing Date:
28 June 2024 (28.06.2024)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
20235772 30 June 2023 (30.06.2023) FI

(71) Applicant: **CHEMPOLIS OY** [FI/FI]; Lääketehtaantie 1, 90660 Oulu (FI).

(72) Inventors: **ROUSU, Päivi**; c/o Chempolis Oy, Lääketehtaantie 1, 90660 Oulu (FI). **ROUHIAINEN, Maija**; c/o Chempolis Oy, Lääketehtaantie 1, 90660 Oulu (FI). **ANTTILA, Juha Rainer**; c/o Chempolis Oy, Lääketehtaantie 1, 90660 Oulu (FI). **KUPIAINEN, Laura**; c/o Chempolis Oy, Lääketehtaantie 1, 90660 Oulu (FI).

(74) Agent: **KOLSTER OY AB**; (Salmisaarenaukio 1), P.O. Box 204, 00181 Helsinki (FI).

(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, 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).

Declarations under Rule 4.17:
— *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*

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

WO 2025/003577 A1

(54) **Title:** METHOD FOR PRODUCING A LIGNIN PRODUCT

(57) **Abstract:** It is disclosed a method for producing a precipitated lignin product comprising the steps of: providing a soluble lignin-containing organic solution; providing an aqueous precipitant solution comprising about 5% to about 40% of acid(s); adding the aqueous precipitant solution to the soluble lignin-containing organic solution to provide a mixture having a temperature in the range of about 40°C to about 90°C; cooling the mixture containing a precipitated lignin product for about 2 sec to about 2 hours; separating and recovering the precipitated lignin product from the mixture.

METHOD FOR PRODUCING A LIGNIN PRODUCT

FIELD OF THE INVENTION

The present invention relates to a method for producing a lignin product. More particularly, the invention relates to an improved method for producing a precipitated lignin product with high purity in an efficient and controlled way.

BACKGROUND OF THE INVENTION

Precipitation of lignin from a lignocellulosic material with water is known. WO 2011/154293 A1 discloses a process for separation of lignins and sugars from an extraction liquor involving concentrating the extraction liquor, mixing the concentrated liquor with water in equal parts, stirring the resultant mixture to obtain a stable suspending of the lignins in the solution, filtering the solution comprising the suspended lignins.

WO 2021/198555 A1 discloses a method for the separation of lignin from an organic solvent, comprising multiple two-step cycles involving a first step of adding an aqueous solution to a lignin-containing organic solvent to precipitate lignin, and a second step of subjecting the resultant lignin-reduced solution to the first step of addition of an aqueous solution. Precipitated lignin is separated and recovered in each step. The multi-step addition of an aqueous solution to a lignin-containing solution provides lignin precipitates with a different molecular weight distribution.

WO 2019/162277 A1 discloses a method of production of lignin and hemicellulose from a plant lignocellulosic material comprising contacting a plant lignocellulosic material with an extraction solution comprising acetic acid, formic acid and water to obtain a solid fraction and a liquid fraction. The liquid fraction is separated, concentrated and mixed with equal part by weight of water to provide a suspension comprising solid particle in suspension.

Use of water in precipitation of lignin from an organic spent cooking liquor from organosolv pulping of a lignocellulosic material generates high amount of waste water or a diluted hemicellulose solution. High losses of organic acids and/or high processing costs to purify hemicellulose solution and further treatment of waste water cannot be avoided to recover water and organic acids.

There has also been challenges in recovering of a precipitated lignin product by filtration, e.g., due to the wide range of particle sizes and shapes.

BRIEF DESCRIPTION OF THE INVENTION

We have now found an improved method for efficient and controlled production of a precipitated lignin product having targeted properties.

The method is based on an organosolv pulping of a lignocellulosic material in which cellulose, lignin and hemicellulose present in the lignocellulosic material are separated in two main streams, i.e., cellulosic pulp and a solution of soluble lignin and hemicellulosic sugars, by using an aqueous organic solvent as a cooking liquor. The cooking liquor is acidic and includes formic acid and acetic acid, water and furfural. During cooking, hemicellulosic sugars and a portion of lignin are dissolved in the cooking liquor.

In the context or the present invention, the term "organosolv pulping" means that delignification of the lignocellulosic material is performed with organic acids in an aqueous solution constituting a cooking liquor. The organic acids include formic acid and acetic acid. In the invention, the organosolv pulping of a lignocellulosic material is thus performed in acidic conditions wherein the total content of organic acids in the cooking liquor is about 65% wt.% to about 90% wt.%.

In the present invention, the cooking liquor used in the organosolv pulping may contain a low amount alcohol(s), such as methanol and ethanol, of at most 8 wt.%. Due to the low content of alcohol in the cooking liquor, organosolv pulping in the present invention does not correspond to organosolv delignification with alcohols.

In context of the invention, a soluble lignin-containing organic solution obtained from cooking of a lignocellulosic material with an aqueous organic solvent is described here as "a spent cooking liquor". The spent cooking liquor thus contains, e.g.., soluble lignin, hemicellulosic sugars, inorganics, proteins and extractives, and also components of original cooking liquor. As used herein, the terms "soluble lignin-containing organic solution" and "spent cooking liquor" are interchangeable.

In the method of the invention, soluble lignin is precipitated from the spent cooking liquor using an aqueous solution which comprises organic acids. It was surprisingly found, firstly, that a lignin precipitate of high purity was obtained using specific precipitation conditions involving employing an aqueous organic solution comprising acids as a precipitant solution, and secondly, using a dynamic mixer in mixing the spent cooking liquor and an aqueous organic solution in a continuous manner affected the precipitated lignin product yield and properties, and thirdly, quick cooling of the lignin precipitate provided beneficial filterability of the

lignin precipitate. In particular, use of an aqueous solution comprising organic acids for lignin precipitation, and quick cooling of the lignin precipitate have a favorable effect on quality of the lignin precipitate. Acidic precipitant solution obtainable from the organosolv pulping process provides a lignin precipitate of high purity and diminishes need of fresh water.

In the context of the invention, the term “lignin precipitate of high purity” means a precipitated lignin product which contains a decreased amount of sugars, inorganics and proteins. The term "beneficial filterability" means a precipitated lignin product which filtrates with an increased rate.

Generally, it is challenging to achieve a lignin product with high purity without compromising process effectiveness and savings in energy and in use of fresh water. The method of the invention employs specific precipitation conditions whereby lignin filterability of a lignin precipitate is improved. Significant energy savings are achieved with method of the invention by utilizing acidic process streams, and a spent cooking liquor of high dry matter content in the precipitation, while a high quality lignin product is achieved.

The present invention provides an efficient and economic method for producing and recovering a pure precipitated lignin product having minor amounts of impurities, such as protein, by filtration using an acidic precipitant solution and quick cooling after the precipitation. The lignin precipitate of high purity ensures efficient recovery of the lignin product by filtration whereby clogging of the filter is avoided. The method of the invention efficiently employs acidic process streams avoiding need for fresh water thus providing savings in energy costs.

DETAILED DESCRIPTION OF THE INVENTION

An object of the present invention is to provide a method for producing a lignin product, comprising the steps of:

- providing a soluble lignin-containing organic solution,
- providing an aqueous precipitant solution comprising about 5% to about 40% of acid(s),
- adding the aqueous precipitant solution to the soluble lignin-containing organic solution to provide a mixture having a temperature in the range of about 40°C to about 90°C,
- cooling the mixture containing a precipitated lignin product about 2 sec to about 2 hours,
- separating and recovering the precipitated lignin product from the

mixture.

The percentages of the various constituents used in the method of the present invention are given on weight basis, i.e. wt.%.

Soluble lignin-containing organic solution is originated from an organosolv pulping method of a lignocellulosic material.

In the method of the invention, the soluble lignin-containing organic solution may contain a low amount of alcohol(s), such as methanol and ethanol. In an embodiment, the amount of ethanol is at most 5 wt.%. In an embodiment, the amount of methanol is at most 6 wt.%. The total content of alcohol(s) in the soluble lignin-containing organic solution is at most 8 wt.%.

In an embodiment, the soluble lignin-containing organic solution comprises formic acid, acetic acid, and water. In another embodiment, the soluble lignin-containing organic solution comprises formic acid, acetic acid, water and furfural.

In an embodiment, the soluble lignin-containing organic solution comprises about 65 wt.% to about 90 wt.% of organic acids in total based on the liquid part (i.e., volatile components including formic acid, acetic acid, furfural, alcohol(s), water) of the soluble lignin-containing organic solution. In an embodiment, the organic acids are formic acid and acetic acid.

In an embodiment, the dry matter content of the soluble lignin-containing organic solution is in the range of about 50% to about 90%. In another embodiment, the dry matter is from about 60% to about 85%. In a further embodiment, the dry matter is from about 70% to about 80%.

The aqueous precipitant solution comprises about 5% to about 40% of acid(s). In another embodiment, the aqueous precipitant solution comprises about 20% to about 30% of acid(s). The acid(s) is(are) organic acid(s). In an embodiment, the acid is selected from formic acid, acetic acid and a mixture thereof.

In an embodiment, the aqueous precipitant solution comprises an acidic process stream from organosolv pulping process of the lignocellulosic material. In an embodiment, the aqueous precipitant solution consists of an acidic process side stream from organosolv pulping process of the lignocellulosic material. Using at least partly acidic process side streams originated from organosolv pulping process in the lignin precipitation, savings in fresh water and in energy costs are achieved.

The aqueous precipitant solution is added to the soluble lignin-containing organic solution in a weight ratio of about 0.7:1 to about 2.5:1. In an embodi-

ment, the aqueous precipitant solution is added in a weight ratio of about 1:1 to about 2:1.

Addition of the aqueous precipitant solution to the soluble lignin-containing organic solution provides a mixture. In an embodiment, the content of organic acids in total based on the liquid part (i.e., volatile components including formic acid, acetic acid, furfural, alcohol(s), water) of the mixture is at most 60 wt.%. In an embodiment, the total organic acid content is in the range of about 5 wt.% to about 60 wt.% based on the liquid part.

The temperature of the mixture of the soluble lignin-containing organic solution and the aqueous precipitant solution before cooling is in the range of 40°C to about 90°C. In an embodiment, the temperature is about 50°C to about 90°C. In an embodiment, the temperature is about 60°C to about 85°C. In another embodiment, the temperature of the mixture is in the range of about 65°C to about 80°C. It is not necessary that the temperature of the soluble lignin-containing organic solution and the aqueous precipitant solution are initially the same. The temperature of the precipitant solution may be lower than that of the soluble lignin-containing organic solution. For example, the precipitant solution and the soluble lignin-containing organic solution may have a temperature of about 60°C and about 95°C, respectively.

After the aqueous precipitant solution has been added, cooling of the mixture is started. The mixture containing precipitated lignin is cooled to about 20°C to about 50°C. In an embodiment, cooling of the mixture is performed or about 2 sec to about 2 hours. In another embodiment, cooling of the mixture is performed within 60 minutes. Duration of cooling is dependent on the efficiency of a heat exchanger employed in the method. Shorter cooling time is achieved with a more efficient heat exchanger. Fast cooling ensures that uniform particle size of a lignin is achieved providing, in turn, enhanced filterability of the precipitated lignin product.

The precipitated lignin product is separated and recovered. In an embodiment, the separation is carried out by filtration.

If desired, the precipitated lignin product may be washed, e.g., with water to remove any residual impurities in the product.

The precipitated lignin product produced by the method of the invention comprises at least 50% of acid insoluble lignin determined with NREL TP-510-42618 method (August 2012). Lignin determined by this method is also known "Klason lignin".

The content of the impurities including protein, acids, pentosan hemicellulose, inorganics, e.g., ash of biomass, of the precipitated lignin product produced by the method of the invention is in the range of about 2 wt.% to about 30 wt.%.

In a further aspect, the invention provides a precipitated lignin product comprising at least 50% of acid insoluble lignin determined with NREL TP-510-42618 method (August 2012). Lignin determined by this method is also known "Klason lignin".

In an embodiment, the precipitated lignin product of the invention contains about 2 wt.% to about 30 wt.% of impurities including protein, acids, pentosan hemicellulose, inorganics, e.g., ash of biomass.

The following examples are presented for further illustration of the invention without limiting the invention thereto.

The content of acid soluble lignin was measured according to technical report of NREL TP-510-42618 (August 2012).

The content of pentosans was determined according to standard "TAPPI T223 cm-10 Pentosans in wood and pulp", wherein furfural is analysed with HPLC.

Example 1

Wheat straw lignocellulosic material was delignified with a cooking liquor containing water, formic acid and acetic acid to provide a soluble lignin-containing spent cooking liquor. The soluble lignin-containing spent cooking liquor was concentrated to a dry matter content of 57%. The content of formic acid and acetic acid in total based on the liquid part of the soluble lignin-containing spent cooking liquor was 77%.

Two acidic aqueous solutions containing 20% and 35% of formic acid and acetic acid in total were used as precipitant solutions to precipitate lignin in the concentrated spent cooking liquor.

Water was used as a reference precipitant solution (0% acid). The precipitant solutions were added to the concentrated spent cooking liquor in a ratio of 1:1 (w/w) to provide a mixture.

Addition of water (0% acid) gave a mixture containing 23% of formic acid and acetic acid in total based on the liquid part of the mixture. Addition of 20% acidic solution gave a mixture containing 37% of formic acid and acetic acid in total based on the liquid part of the mixture. Addition of 35% acidic solution gave a mixture containing 48% of formic acid and acetic acid in total based on the liquid part of the mixture.

The resultant mixture had a temperature of 40°C. After precipitation, the mixture was cooled for about 15 min to reach the temperature of about 35°C. Precipitated lignin was filtered using a filter paper having a retention capacity of 0.6 µm.

Table 1 shows the effect of the precipitant solutions on the purity of the precipitated lignin products. The percentages are based on weight and on dry matter of the precipitated lignin.

Table 1

Precipitant	Protein-corrected acid insoluble lignin (%)*	Bound acids (%)	N as protein (%)	Pentosans (%)
0% acid	81.2	7.7	7.2	3.1
20% acid	82.1	7.0	5.4	2.9
35% acid	83.3	6.8	3.1	2.4

* acid insoluble lignin minus amount of protein

The results show that less protein, acids and pentosan hemicellulose impurities are bound to precipitated lignin, and thus the purity of the lignin product is increased when the acid content of the precipitant solution increased.

Reference example 2

The example illustrates an effect of different cooling times on filterability of lignin precipitates.

Two soluble lignin-containing spent cooking liquors were tested having a different dry matter content. Pure water was used as a precipitant solution. The precipitant solution was added to the spent cooking liquors with vigorous stirring to provide mixtures. The temperature of the mixtures was 80°C. The spent cooking liquors containing precipitated lignin were cooled to a room temperature of about 22°C before filtering.

Precipitated lignin was filtered using a filter paper having a retention capacity of 0.6 µm.

Total filtration time describes the minimum time required to stop dropping of a filtrate.

The results of Table 2 relate to a lignin precipitation from a soluble lignin-containing spent cooking liquor having a dry matter content of 35% before lignin precipitation. The precipitant solution (water) was added to the spent cooking

liquor in a ratio of 1:1.

The results of Table 3 relate to a lignin precipitation from a soluble lignin-containing spent cooking liquor having a dry matter content of 79% before lignin precipitation. The precipitant solution was added to the spent cooking liquor in a ratio of 1:1 and 2:1.

Table 2. DM 35% of the lignin-containing spent cooking liquor concentrate

	Slow cooling	Medium-speed cooling	Fast cooling
Cooling time, h *	4.5	2.5	0.2
The amount of precipitate, g	141	148	148
Filtration time ("surface dry"), min	8.4	5.7	3.5
Total filtration time, min	14.9	7.3	8.2

* cooling time to lower the temperature from 80°C to 22°C

Table 3. DM 79% of the lignin-containing spent cooking liquor concentrate

	Slow cooling	Medium-speed cooling	Fast cooling	Slow cooling
Spent cooking liquor:water	1:1	1:1	1:1	1:2
Cooling time, h *	3.9	2.3	0.19	5.1
The amount of precipitate, g	68	70	69	100
Filtration time ("surface dry"), s	14	15	9	20
Total filtration time, s	50	51	31	30

* cooling time to lower the temperature from 80°C to 22°C

The results of Tables 2 and 3 show that filtration of the precipitated lignin product became faster when the cooling time was shortened after the precipitation of the lignin product. This indicates that the precipitated lignin product desirably has a beneficial particle size distribution allowing fast filtration of the precipitated product.

The results show that the combination of the hot lignin-containing spent cooking liquors and the hot precipitant solution, and quick cooling after precipitation produce a lignin precipitate exhibiting good filterability.

Example 3

Soluble lignin-containing spent cooking liquor having a dry matter content of 79% before lignin precipitation was provided. The organic acid content of the liquid part of the soluble lignin-containing spent cooking liquor was 76 wt.%. An aqueous solution containing formic acid and acetic acid in total of 20% was used as a precipitant solution. The precipitant solution was added to the spent cooking liquor in a ratio of 2:1 (w/w) to provide a mixture. The mixture had a formic acid and acetic content in total of 25 wt.% based on the liquid part of the mixture.

The temperature of the mixture was 80°C. Cooling and filtration were carried out as described in reference example 2.

The results are shown in Table 4.

Table 4. DM 79% of the lignin-containing spent cooking liquor concentrate

	Slow cooling	Medium-speed cooling	Fast cooling
Spent cooking liquor:precipitant	1:2	1:2	1:2
Cooling time, h *	5.05	2.17	0.18
The amount of precipitate, g	101	101	107
Filtration time ("surface dry"), s	15	13	7
Total filtration time, s	52	42	25

* cooling time to lower the temperature from 80°C to 22°C

The results show that filtration of the precipitated lignin product became faster when the cooling time was shortened after the precipitation of the lignin product.

Example 4

The following Table 5 shows fresh water and energy savings obtained by using an acidic precipitant solution in accordance with the method of the invention instead of pure water.

The spent cooking liquor containing soluble lignin solution used for

precipitation had a dry matter content of 70% (w/w). Flow rate of the spent cooking liquor was 30.5 t/h.

The effect of three different precipitant solutions on fresh water and energy savings were tested. Pure water was used as a reference. Precipitant solution B was an acidic process stream containing at least 5% of acids and consisting of a condensate of hemicellulose filtrate produced in an organosolv pulping of a cellulosic material. Precipitant solution A contained 50% (w/w) of pure water and 50% (w/w) of the acidic process stream.

Each precipitant solution was added to the spent cooking liquor in the ratio of 2:1. Flow rate of the precipitant solution was 61 t/h.

Resultant lignin precipitate was washed with water. Water used for precipitation and washing was led to a distillation. Energy consumption needed in distillation is given in Table 5.

Table 5

	Reference (water)	A	B
Precipitant			
flowrate total (t/h)	61	61	61
water (t/h)	61	30.5	0
process stream (t/h)	0	30.5	61
Water to lignin washing (t/h)	18.1	18.1	17.3
Water to distillation (t/h)	85.3	52.2	23.5
Heat duty of distillation (MW)	88	54	24

The results show that use an acidic precipitant solution reduces fresh water consumption and energy consumption compared with the case when pure water is used for lignin precipitation.

It will be obvious to a person skilled in the art that, as the technology advances, the inventive concept can be implemented in various ways. The invention and its embodiments are not limited to the examples described above but may vary within the scope of the claims.

CLAIMS

1. A method for producing a precipitated lignin product comprising the steps of:

- providing a soluble lignin-containing organic solution,
- providing an aqueous precipitant solution comprising about 5% to about 40% of acid(s),
- adding the aqueous precipitant solution to the soluble lignin-containing organic solution to provide a mixture having a temperature in the range of about 40°C to about 90°C,
- cooling the mixture containing a precipitated lignin product for about 2 sec to about 2 hours,
- separating and recovering the precipitated lignin product from the mixture.

2. The method of claim 1, wherein the soluble lignin-containing organic solution is a spent cooking liquor from an organosolv pulping process of a lignocellulosic material.

3. The method of claim 2, wherein the organosolv pulping process of a lignocellulosic material is performed using an aqueous organic solvent as a cooking liquor.

4. The method of claim 3, wherein the aqueous organic solvent comprises at least one of formic acid and acetic acid.

5. The method of claim 4, wherein the aqueous organic solvent comprises furfural.

6. The method of any one of the preceding claims, wherein the soluble lignin-containing organic solution comprises formic acid, acetic acid, and water.

7. The method of claim 6, wherein the soluble lignin-containing organic solution further comprises furfural.

8. The method of any one of the preceding claims, wherein the soluble lignin-containing organic solution comprises about 65 wt.% to about 90 wt.% of formic acid and acetic acid in total based on the liquid part of the soluble lignin-containing organic solution.

9. The method of any one of the preceding claims, wherein the soluble lignin-containing organic solution contains at most 8 wt.% alcohol(s), such as methanol and ethanol.

10. The method of any one of the preceding claims, wherein the dry matter of the soluble lignin-containing organic solution is in the range of about 50% to

about 90%, specifically about 60% to about 85%, more specifically about 70% to about 80%.

11. The method of any one of the preceding claims, wherein the aqueous precipitant solution comprises about 20% to about 30% of acid(s).

12. The method of claim 11, wherein the acid(s) in the aqueous precipitant solution are organic acid(s).

13. The method of claim 11 or 12, wherein the organic acid(s) comprise(s) at least one of formic acid and acetic acid.

14. The method of any one of the preceding claims, wherein the aqueous precipitant solution comprises an acidic process side stream from organosolv pulping process of the lignocellulosic material.

15. The method of any one of the preceding claims, wherein the aqueous precipitant solution is added to the soluble lignin-containing organic solution in a weight ratio of about 0.7:1 to about 2.5:1, specifically about 1:1 to about 2:1.

16. The method of any one of the preceding claims, wherein the mixture has a temperature of about 50°C to about 90°C, specifically 60°C to about 85°C, more specifically about 65°C to about 80°C.

17. The method of any one of the preceding claims, wherein the content of organic acids in total based on the liquid part of the mixture is at most 60 wt.%, specifically in the range of about 5 wt.% to about 60 wt.%.

18. The method of any one of the preceding claims, wherein the mixture is cooled to about 20°C to about 50°C, specifically about 25°C to about 40°C.

19. The method of any one of the preceding claims, wherein the cooling of the mixture is performed within 60 minutes, specifically about 10 minutes to about 60 minutes.

20. The method of any one of the preceding claims, wherein the separation is carried out by solid-liquid separation method, such as filtration and centrifugal means.

21. The method of any one of the preceding claims, wherein the precipitated lignin product contains about 2 wt.% to about 30 wt.% of impurities including protein, acids, pentosan hemicellulose, inorganics, e.g., ash of biomass.

22. The method of any one of the preceding claims, wherein the precipitate lignin product comprises at least 50% of acid insoluble lignin determined with NREL TP-510-42618 method (August 2012).

23. A precipitated lignin product comprising at least 50% of acid insoluble lignin determined with NREL TP-510-42618 method (August 2012).

24. The precipitated lignin product of claim 23, wherein the precipitated lignin product contains about 2 wt.% to about 30 wt.% of impurities including protein, acids, pentosan hemicellulose, inorganics, e.g., ash of biomass.

INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2024/050364

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08L97/00
ADD.

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)
C08L

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, BIOSIS, EMBASE, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CA 2 101 754 A1 (ALCELL TECH INC [CA]) 2 August 1992 (1992-08-02)	23,24
Y	paragraphs [0013], [0024]; claim 41	1-22
X	WO 2021/198555 A1 (CHEMPOLIS OY [FI]) 7 October 2021 (2021-10-07) cited in the application	23,24
Y	page 4, paragraph 4; claims 1,15	1-22

☐ 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

"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
3 September 2024	13/09/2024

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Friedrich, Christof

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/FI2024/050364

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
CA 2101754 A1	02-08-1992	AU 1140995 A	30-03-1995
		AU 1744892 A	07-09-1992
		BR 9205576 A	08-11-1994
		CA 2101754 A1	02-08-1992
		EP 0569526 A1	18-11-1993
		FI 933418 A	22-09-1993
		JP H06511043 A	08-12-1994
		WO 9213849 A1	20-08-1992
		ZA 92566 B	28-07-1993
WO 2021198555 A1	07-10-2021	EP 4127308 A1	08-02-2023
		FI 20205325 A1	01-10-2021
		WO 2021198555 A1	07-10-2021