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1 •**Andritz Oy,** Tammasaarenkatu 1, 00180 Helsinki, SUOMI - FINLAND, (FI)

(72) Keksijä - Uppfinnare

1 •**McKeough, Paterson,** TÄHTELÄ, SUOMI - FINLAND, (FI)
2 •**LEFFLER, Joel,** STOCKHOLM, SVERIGE, (SE)

(74) Asiamies - Ombud

ANDRITZ Oy, Patent Department P.O. Box 500, 48601 Kotka

(54) Keksinnön nimitys - Uppfinningens benämning

Menetelmä mustalipeästä erotetun ligniinin pesemiseksi
Förfarande för tvättning av lignin som separerats från svartlut
METHOD FOR WASHING LIGNIN SEPARATED FROM BLACK LIQUOR

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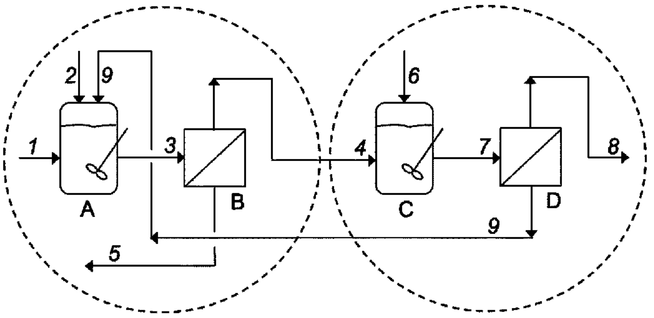
The subject of the invention is a new method for washing solid particles, comprised primarily of lignin, that have been obtained by first acidifying spent alkaline pulping liquor and then separating the particles thus precipitated from the liquor. According to the new method, the first washing step is conducted so that the pH is in the range 6 - 12, the wash ratio is at least 1, and the extent of re-dissolution of lignin is in the range 5 % - 50 %. Suitable aqueous washing media and suitable washing conditions are employed to achieve this end. In the case of a freshly introduced washing medium, the washing medium typically employed in the first washing step of the new method is comprised essentially of water, only.



Keksinnön kohde on uusi menetelmä pääasiassa ligniinistä koostuvien kiintoainepartikkeleiden pesemiseksi, jotka partikkelit on saatu hapottamalla ensin alkalinen jätekeittolipeä ja sen jälkeen erottamalla siten saostuneet kiintoainepartikkelit lipeästä. Uuden menetelmän mukaan ensimmäinen pesuvaihe suoritetaan niin, että pH on välillä 6–12, pesusuhde on vähintään 1 ja ligniinin uudelleenliukenemisaste on välillä 5–50 %. Sopivia vesipitoisia pesuväliaineita ja sopivia pesuolosuhteita käytetään tämän saavuttamiseksi. Vastasyötetyn pesuväliaineen kyseessä ollessa uuden menetelmän ensimmäisessä pesuvaiheessa tyypillisesti käytettävä pesuväliaine koostuu olennaisesti vain vedestä.

METHOD FOR WASHING LIGNIN SEPARATED FROM BLACK LIQUOR

Technical field

Lignin may be removed from spent alkaline pulping liquor (black liquor) by first acidifying the liquor and then separating the resulting lignin precipitate. The present method deals with the washing of the lignin so removed. The objective of the overall washing process is to produce a solid product comprised primarily of lignin and having a reasonably low level of impurity. The lignin product thus obtained may be utilized as a fuel, as a material or as a chemical in a range of potential applications.

Prior art

The most commonly applied method for recovering reasonably pure lignin from spent alkaline pulping liquor, commonly termed black liquor, involves the following steps:

1. Acidification of the liquor to a pH typically in the range 9 – 10 (value at 25 °C) using an acid such as carbon dioxide (CO_2) or sulphuric acid (H_2SO_4)
2. Separation of the solid particles thus precipitated by e.g. filtering the slurry
3. Washing the separated material, usually a filter cake, at low pH, typically around pH 2 (value at 25 °C), using an acidified aqueous solution
4. As required, further washing of the separated material, usually at low pH.

The two main washing principles, well known and widely used, are (1) the re-slurry principle, according to which the separated material is taken from the separation device, mixed with the washing medium and subsequently re-separated, and (2) the displacement principle, according to which the separated material is left in/on the separation device, usually a filter, and the solution entrained in the separated material is pushed out of the material by the washing medium. Some of the known methods for recovering lignin from black liquor specify one or other of these washing principles.

The general known method outlined above is described, for example, in the reference book “Lignins – Occurrence, Formation and Reactions” edited by K.V. Sarkanen and

C.H. Ludwig, John Wiley & Sons, New York, 1971, in Chapter 16, Section V.A., page 671. A re-slurry type of wash at pH 2 – 3 is recommended for Step 3 in this particular case. Another known method employing a re-slurry wash for Step 3 is described in European patent EP1794363B1. For Step 3, this method employs a pH below 6, the preferable range for the pH being 1 – 3.5. A displacement wash for Step 3 is incorporated, for example, in the method proposed in the publication, Loutfi, H. & Blackwell, B., "Lignin recovery from black liquor: preliminary process design", Tappi Journal, January 1991, pp. 203-210.

Filtration is the usual method employed for separating the lignin precipitated in the acidification step and, in the case of a re-slurry wash, for separating the washed solids from the wash slurry. One type of filtration device that is suitable for these purposes is the so-called filter press. Another is the belt filter.

Use of acid in the first washing step – Step 3 above – liberates sodium still bound to the lignin after the primary acidification stage and restricts re-dissolution of lignin in the aqueous washing medium. For example, re-dissolution may be essentially prevented by carrying out the wash at a pH of 2 or less. However, a relatively large amount of acid is required in order to reach this low pH level in the first washing step. An alternative strategy for preventing re-dissolution is to increase the ionic strength of the aqueous washing medium, for example, by adding soluble salts such as sodium sulphate (Na_2SO_4) prior to, or in conjunction with, the washing step. This strategy has been incorporated, for example, in the methods described in US Patent 2623040 and US patent application US2008/0214796A1. In the former document it is proposed that a salt solution be employed in the first washing step expressly for the purpose of preventing lignin re-dissolution. In the method described in the latter document, salt and/or acid is added, as needed, to a washing medium employed in conjunction with the first filtration step (by either diluting the slurry before filtration or carrying out a displacement wash on the filter). The need, as stated in the description of the method, is to prevent re-dissolution of lignin during the washing step. If more acid is employed, less salt is required. A disadvantage of adding salt rather than acid is that a much larger amount of added impurity (salt) is left entrained in the post-wash solid material. When a relatively pure lignin is sought for, this means a greater demand for further washing in comparison to the corresponding washing chain that employs acid, only, in the first step. In addition, there are costs associated with the procurement of the salt and the further handling of the spent salt-containing solution.

In US Patent 3048576, a water wash is proposed for the first washing step of the lignin separated from acidified black liquor. The type of washing step in question is disclosed in the Example 2 given in the document. A very limited amount of water is added to the filter cake. The ratio of the weight of water to the weight of cake dry solids is only 0.69 in this example. The ash content of the cake dry matter is reduced from 33 % to only 21 %. It is stated that further washing with water is possible but would lead to some re-dissolution of lignin. Thus, in this method, a limited amount of water is utilized in the first washing step and, as a result, the ionic strength of the solution is maintained at a level high enough to prevent significant re-dissolution of lignin.

The wash ratio employed in a washing step is usually defined, and is defined herein, as the ratio of the volume of the washing medium to the volume of the liquid initially in the moist separated solids, e.g. a filter cake. In the case of lignin separated by filtration from acidified black liquor, the dry-solids content of the filter cake is typically of the order of 50 %, in which case the ratio of the weight of aqueous wash medium to the weight of cake dry solids is roughly the same as the wash ratio as defined above. Thus, in Example 2 of US 3048576, the applied wash ratio is clearly less than 1. This is in stark contrast to conventional washing operations that employ significantly higher wash ratios. For example, in the case of displacement washing, the minimum theoretical wash ratio for displacement is 1 and the wash ratio applied in practice is typically at least 1.5. Thus, the type of water wash proposed in patent document US 3048576 deviates significantly from conventional washing at wash ratios greater than 1.

US Patent 4946946 discloses a process, in which lignin is precipitated by acidifying spent liquor to a pH in the range 1.8 to 4. The separated lignin is washed with water. In this case, the lack of acid compounds and/or salt compounds in the washing medium does not lead to significant re-dissolution of lignin, because, at a pH less than 4, all, or nearly all, of the sodium originally bound to the lignin has been liberated and the lignin has become hydrophobic. However, acidifying the black-liquor feed stream to a pH of 4 or less consumes a very large amount of acid, much more than is consumed when washing lignin that has been precipitated at the commonly applied pH level of 9 – 10.

Ultrafiltration, also known as membrane filtration, has been utilized as an alternative method for recovering lignin from spent alkaline pulping liquors. However, it should be noted that ultrafiltration serves as a pre-fractionation method. Sodium remains bound to the lignin so that, when a relatively pure lignin product is required, the lignin-rich solution needs to be acidified in a later step in any case.

Accordingly, there is a need for a method whereby a reasonably pure solid lignin material may be recovered from black liquor without a large consumption of acid in the washing process.

Description of the invention

The present invention represents a new approach to washing solid particles, comprised primarily of lignin, that have been obtained by first acidifying spent alkaline pulping liquor to a pH not less than 7 thereby precipitating solid particles, and then separating solid particles thus precipitated from the liquor, after which a part or all of the solid particles is/are washed with aqueous media in at least two steps to yield a solid product. According to the new method, the first washing step is conducted so that the pH is in the range 6 – 12, preferably in the range 7 – 10, the wash ratio is at least 1, preferably in the range 1 – 100, and most preferably in the range 1.5 – 10, and the extent of re-dissolution of lignin is in the range 5 % - 50 %, preferably in the range 10 % - 30 %. Suitable aqueous washing media and suitable washing conditions are employed to achieve this end. Typically, the first washing step is conducted without significant addition of either acid compounds or salt compounds to the washing medium.

In addition to black-liquor streams available, as such, at the pulp mill, certain fractions of the black liquor, in particular fractions that have been obtained without significant liberation of the sodium bound to the lignin components, may be suitable feed materials for the lignin-recovery process outlined above.

The main problem which the invention sets out to solve is that of the high acid consumption which is usually encountered when washing lignin separated from black liquor. One known solution is based on the addition of a significant amount of salt in the first washing step. The present invention provides a solution which does not incorporate significant addition of salt, thus avoiding the detrimental consequences of such addition.

For the purposes of this document, the pH of a wash is defined as the pH, measured at 25 °C, of the total mixed spent washing solution withdrawn from the washing step. Usually filtration is the method of solids separation, in which case the spent washing solution is the filtrate from the filtration step. A definition of the wash pH is necessary because, in the case of displacement washing with an acidic solution, the pH of the spent washing solution (filtrate) typically varies with time during the wash.

When precipitated particles are separated from acidified spent liquor, the separated material contains, in addition to the actual solid particles, a certain amount of entrained liquor, which, in turn, contains dissolved solids. The actual solid particles are comprised primarily of lignin. In the present method, a part of the total mass of the actual solid particles re-dissolves in the first washing step. In this document, 20 % re-dissolution of lignin, for example, means that 20 % of the total mass of the actual solid particles re-dissolves in the washing step in question.

The new method defies conventional wisdom by purposefully allowing significant re-dissolution of lignin in the first washing step. As discussed further below, remarkable benefits accrue as a result of this novel approach. One general type of washing scheme is based on the use of a fresh washing medium in the first washing step. When this washing scheme is adopted in conjunction with the new method, the washing medium typically used in the first washing step is comprised essentially of water, only. When a reasonably pure lignin is sought for, a multistep washing procedure is usually adopted. In this case a counter-current washing scheme may be applied, which means that the spent washing solution from the second washing step is led to the first washing step. In the known methods, a significant amount of acid and/or salt is added to this washing solution either prior to, or during, the first washing step. When a multistep counter-current washing scheme is adopted in conjunction with the new method, the spent washing solution from the second washing step is typically used, as such, as the washing medium in the first washing step.

The main advantage of the new method over known methods that employ acidic compounds, only, as additives is a significantly lower consumption of acid in the overall washing chain. In the case of multistep washing aiming at achieving a reasonably pure lignin product, embodiments of the invention employ an acidic wash at low pH in at least one of the later washing steps. Even in these embodiments, the overall washing chain has a total consumption of acid that is significantly lower than that of a conventional washing chain that employs acidic compounds, only, as additives. Note that, because of the need to liberate all, or nearly all, of the sodium originally bound to the lignin, acidic washing at low pH, typically in the range 1 – 4, in at least one of the washing stages is a precondition for obtaining a reasonably pure lignin product. In processes based on the present method, such a product is obtained by employing acidic washing at low pH in at least one of the washing steps subsequent to the first washing step.

In the case of a multistep counter-current washing scheme, acidic washing at low pH is commonly employed as the second washing step in processes based on the invention. It follows that the spent washing solution that is led to the first washing step has, in that case, a relatively low pH. However, it is essential to note that if a pH level of, say, 2 is applied in the second washing step, the amount of free acid in the spent washing solution from the second washing step is small compared to the amount of acid that would have to be added in the first washing step in order to perform the first washing step at the same low pH level. For example, if the acid in question is sulphuric acid (H_2SO_4), the concentration of free H_2SO_4 in a pH 2 solution is only about 0.5 g/L. If, in the first washing step, the wash ratio is, say, 4, then a washing medium at pH 2 brings roughly 2 g of H_2SO_4 per kg of lignin to the first washing step. This is two orders of magnitude smaller than the typical H_2SO_4 consumption level – around 200 g of H_2SO_4 per kg of lignin – that is encountered when lignin, separated from black liquor after acidification by CO_2, is washed at pH 2 with an H_2SO_4-water solution. In the present method the pH in the first washing step is at least 6.

The spent washing solution from the second washing step in a multistep washing scheme also contains a variety of other soluble compounds that have been washed out of the cake treated in the second washing step. However, the concentrations of these compounds in the spent washing solution are low and, in a counter-current washing scheme, their effect on the solubility of lignin in the first washing step is, at most, minor. Obviously, with a multistep counter-current washing scheme, a low pH in the first washing step could be achieved by employing a very large excess of acid in the second washing step. In terms of lignin yield and acid consumption, the end result would be a washing process with a performance similar to those of known methods employing acid addition in the first washing step. This would defeat the purpose of the present invention, distinguishing features of which are significant re-dissolution of lignin in the first washing step and considerably reduced acid consumption in the overall washing chain. Thus a multistep counter-current washing process, which does not employ addition of chemicals, or employs only limited addition of chemicals, in the first washing step but employs a very large excess of acid in the second washing step is excluded from the realm of the present invention. This is realized by setting a minimum pH of 6 for the first washing step of the new method. Similarly, a multistep counter-current washing process that employs significant addition of salt in the second wash step is excluded from the realm of the present invention. This is realized by stipulating that the extent of re-dissolution of lignin in the first washing step is in the range 5 % - 50 %.

Obviously, the higher the effective price of the acid used in the lignin washing process, the greater is the advantage of reduced acid consumption. In the case of the dominant chemical pulping process, the kraft process, H_2SO_4 has usually been proposed as the acid for washing lignin separated from black liquor. Not only is H_2SO_4, generally speaking, the least costly mineral acid, but also, because sulphur is a process element in the kraft process, negligible amounts of foreign elements are introduced into the chemical recovery cycle as a result of the utilization of H_2SO_4. On the other hand, the introduction of sulphur disturbs the sodium-sulphur balance of the mill. The end result is an increase in the requirement for sodium make-up in the form of sodium hydroxide (NaOH) or sodium carbonate (Na_2CO_3). Thus, the effective price of the H_2SO_4 used for lignin washing becomes much higher than its purchase price. So, when the new method is applied at a kraft pulp mill and H_2SO_4 is the acid to be used in any acidic washing steps, the costs associated with the use of H_2SO_4 for washing lignin are remarkably reduced compared to those of known methods employing an acidic wash at low pH in the first washing step.

In the new method, the extent of re-dissolution of lignin in the first washing step is very dependent on the washing conditions, such as the temperature, the residence time and the wash ratio. The observed degree of dependence of lignin re-dissolution on the washing temperature and washing time was unexpected and explains, in part, why the new method has been overlooked previously. For example, the extent of re-dissolution of lignin in the first washing step increases steeply with increase in wash temperature. In the new method the temperature employed in the first washing step is typically below 90 °C, preferably below 60 °C, and most preferably below 30 °C. Furthermore, it was observed that, particularly at lower temperatures, the extent of re-dissolution of lignin is sensitive to the washing time, with longer times tending to increase the extent of re-dissolution. When the new method is conducted under favourable conditions, the extent of re-dissolution of lignin in the first washing step is typically in the range 10 % – 30 %.

A remarkable feature of the new method is that, compared to the known methods that employ acidic compounds, only, as additives, the loss of income associated with the lower lignin yield is, in many cases of practical interest, more than offset by the savings in the costs attributable to the washing acid.

For each of the washing steps in processes incorporating the new method, either of the two main washing principles, viz. the re-slurry principle or the displacement principle, or a combination of the two principles, may be applied. Another remarkable feature of the new method is that, in spite of the significant re-dissolution of lignin in the first washing step, the filtration resistance of the filter cake formed or washed in the first washing step can be kept at an acceptably low level. In other words, an acceptably short filtration time can be attained.

In general, lignin recovery is applied to only part of the total black-liquor stream of the pulp mill, in which case the main option for the further handling of the spent washing solution of the first washing step (after separation of the solids) is leading the solution back to the main black-liquor stream, usually at some location in the black-liquor evaporation plant of the mill. An interesting alternative is to recycle the spent washing solution from the first washing step to the first step in the overall lignin-recovery process, viz. the black-liquor acidification step, or to the black-liquor stream that is fed to this acidification step. This alternative would increase somewhat the final lignin yield but, on the other hand, would, for the same final lignin purity, also increase the extent of washing needed in steps subsequent to the first washing step.

The new method has further potential advantages over known methods when applied in conjunction with sulphur-free alkaline pulping processes. For the known methods employing acidic washing in the first step, use of H_2SO_4 as the washing acid is largely ruled out because the sulphur-containing spent washing solution from the first washing step cannot be sent to the chemical recovery cycle of the sulphur-free pulping mill and, typically, neither can the spent solution be discharged via the effluent treatment plant of the mill because of the large amount of dissolved organics in the solution. The new method could be exploited by using freshly introduced water as the washing medium in the first washing step and sending the spent washing solution so produced to the recovery cycle of the mill. H_2SO_4 would be employed as an additive in a later washing step. The content of organics in the spent washing solution from this later step could be low enough to permit discharge of this spent solution via the effluent treatment plant.

The present new method is described in more detail with reference to the drawings, FIG. 1 and FIG. 2, each depicting one embodiment of the invention. The numbers and letters in FIG. 1 and FIG. 2 refer to the following streams and processing stages:

1. Solids separated from acidified black liquor
2. Water
3. Slurry formed in the wash step
4. Filter cake
5. Filtrate

6. Acidic aqueous solution
7. Slurry formed in the wash step
8. Filter cake
9. Filtrate

A. Re-slurry wash vessel
B. Filter
C. Re-slurry wash vessel
D. Filter

The embodiment depicted in FIG. 1 employs a two-step washing scheme with newly introduced washing medium in each of the two stages. The dotted circle on the left delineates the first washing step and the one on the right the second washing step. In this embodiment, both washing steps employ the re-slurry wash principle. The material (*1*) fed to the washing process is made up of solid particles that have been obtained by first acidifying spent alkaline pulping liquor and then separating the particles thus precipitated from the liquor. Filtration is the usual method of separation, in which case the feed material (*1*) is in the form of a moist filter cake. This material is re-slurried with water (*2*) in the first wash vessel (A). The slurry (*3*) formed in the wash vessel (A) is led to a filtration device (B). The filtrate (*5*) is led, for example, to the black-liquor evaporation plant of the pulp mill or, for example, to the black-liquor acidification stage. The filter cake (*4*) produced in the first washing step is re-slurried with an acidic aqueous solution (*6*) in the second wash vessel (C). Enough acid is added to reach, after mixing, a low pH, typically around 2. The slurry (*7*) formed in the wash vessel (C) is led to a filtration device (D). The filtrate (*9*) is, for example, led to the black-liquor evaporation plant or is, for example, discharged from the mill via the effluent treatment plant. The filter cake (*8*) produced in the second washing step is the product of the process. The dry matter in this cake is comprised of lignin and some residual impurities.

The embodiment depicted in FIG. 2 employs a two-step counter-current washing scheme. The filtrate (*9*) exiting the second washing step is recycled to the first wash vessel (A) to be utilized as washing medium in the first washing step. Water (*2*) may be added as supplementary washing medium. The other features of this embodiment are the same as those of the embodiment depicted in FIG. 1, described previously. Note that the amount of free acid in the filtrate (*9*) from the second washing step is very small compared to the amount of acid that would need to be added in the first washing step in order to reach a pH as low as that used in the second washing step, i.e. a pH of around 2.

Variants of these embodiments include those employing the same two-step washing schemes but with the washes carried out as displacement washes rather than as re-slurry washes. In each case, the particles that have precipitated from the feed black liquor in the primary acidification stage are separated by filtration and the filter cake thus formed is left on the filter for the two subsequent displacement washes.

The embodiments of the present invention are not limited to those mentioned or described herein.

Example

Lignin was precipitated by lowering the pH of kraft black liquor to a value around 10 (measured at 25 °C) by addition of CO_2 to the liquor. The slurry was filtered and a displacement wash using water at 20 °C was carried out on the filter. The wash ratio was 4. The pH of the total (mixed) filtrate from this washing step was roughly 10. A second wash was performed as a re-slurry acidic wash using an H_2SO_4-water solution. The wash ratio was again 4 and, after mixing, a pH of 2 was reached. The slurry was filtered to yield a filter cake as the product of the precipitation and washing chain.

A reference experiment, simulating a known method, was also conducted. The only difference compared to the above experiment was an altered first washing step. The above displacement wash using water was substituted by an acidic re-slurry wash at pH 2 using an H_2SO_4-water solution. The wash ratio was kept at 4.

When compared to the reference processing chain, the processing chain based on the new method exhibited:

- a yield of lignin about 20 % lower

- a consumption of H_2SO_4 about 50 % lower.

In many cases of practical interest, the above differences render the process based on the new method more economic than the corresponding known process employing acidic washing at low pH in the first washing step.

Claims

1. A method for washing solid particles, comprised primarily of lignin, that have been recovered from spent alkaline pulping liquor, or from a fraction thereof, by first acidifying the liquor, or fraction thereof, to a pH not less than 7 thereby precipitating solid particles, and then separating precipitated solid particles from the accompanying liquid, after which a part or all of the solid particles is/are washed with aqueous media in at least two steps to yield a solid product; wherein the washing medium and the conditions employed in the first washing step are such that, in that step, the pH is in the range 6 - 12, the wash ratio is at least 1, and the extent of re-dissolution of lignin is in the range 5 % - 50 %, and acid is added in the second washing step.
2. A method as in Claim 1, wherein the washing medium employed in the first washing step is a newly introduced process stream and is comprised essentially of water, only.
3. A method as in Claim 1, wherein the solid particles are washed using a multistep counter-current washing scheme and part or all of the washing medium employed in the first washing step is comprised of part or all of the spent washing solution from the second washing step.
4. A method as in Claim 1, wherein the washing medium employed in the first washing step is a mixture of water and part or all of the spent washing solution from a second washing step.
5. A method as in any one of Claims 1 - 4, wherein the washing principle employed in the first washing step is the re-slurry wash principle.
6. A method as in any one of Claims 1 - 4, wherein the washing principle employed in the first washing step is the displacement wash principle.
7. A method as in any one of Claims 1 - 6, wherein the temperature employed in the first washing step is typically below 90 °C, preferably below 60 °C, and most preferably below 30 °C.
8. A method as in any one of Claims 1 - 7, wherein the spent washing solution from the first washing step is recycled either to (1) the spent alkaline pulping

liquor stream that is fed to the acidification stage or to (2) the acidification stage itself.

9. A method as in any one of Claims 1 - 8, wherein the washing principle employed in the second washing step is the re-slurry wash principle.

10. A method as in any one of Claims 1 - 8, wherein the washing principle employed in the second washing step is the displacement wash principle.

Patenttivaatimukset

1. Menetelmä pääasiassa ligniinistä koostuvien kiintoainepartikkeleiden pesemiseksi, jotka partikkelit on talteenotettu alkalisesta jätekeittolipeästä tai sen fraktiosta, hapottamalla ensin lipeä tai sen fraktio pH-arvoon vähintään 7 siten saostaen kiintoainepartikkeleita, ja sen jälkeen erottamalla saostuneita kiintoainepartikkeleita niitä sisältävästä nesteestä, mikä jälkeen osa tai kaikki kiintoainepartikkelit pestään vesipitoisella väliaineella ainakin kahdessa vaiheessa kiintoainetuotteen aikaansaamiseksi, jolloin pesuväliaine ja olosuhteet ensimmäisessä pesuvaiheessa ovat sellaiset, että kyseisessä vaiheessa pH on välillä 6–12, pesusuhde on vähintään 1 ja ligniinin uudelleenliukenemisaste on välillä 5–50 %, ja happoa lisätään toisessa pesuvaiheessa.

2. Patenttivaatimuksen 1 mukainen menetelmä, jossa ensimmäisessä vaiheessa käytettävä pesuväliaine on uusi syötetty prosessivirtaus ja koostuu olennaisesti vain vedestä.

3. Patenttivaatimuksen 1 mukainen menetelmä, jossa kiintoainepartikkelit pestään käyttäen monivaiheista vastavirtapesukonseptia ja ensimmäisessä pesuvaiheessa käytettävä pesuväliaine tai osa siitä koostuu osittain tai kokonaan toisen pesuvaiheen jätepesuliemestä.

4. Patenttivaatimuksen 1 mukainen menetelmä, jossa ensimmäisessä pesuvaiheessa käytettävä pesuväliaine on sekoitus vedestä ja toisen pesuvaiheen jätepesuliemestä tai osasta sitä.

5. Jonkin patenttivaatimuksen 1–4 mukainen menetelmä, jossa ensimmäisessä pesuvaiheessa käytettävä pesukonsepti on uudelleenlietteytyspesukonsepti.

6. Jonkin patenttivaatimuksen 1–4 mukainen menetelmä, jossa ensimmäisessä pesuvaiheessa käytettävä pesukonsepti on syrjäytyspesukonsepti.

7. Jonkin patenttivaatimuksen 1–6 mukainen menetelmä, jossa ensimmäisessä pesuvaiheessa käytettävä lämpötila on tyypillisesti alle 90 °C, edullisesti alle 60 °C, ja edullisimmin alle 30 °C.

8. Jonkin patenttivaatimuksen 1–7 mukainen menetelmä, jossa ensimmäisen pesuvaiheen jätepesuliemi kierrätetään joko (1) alkaliseen jätekeittolipeävirtaukseen joka syötetään hapotusvaiheeseen, tai (2) itse hapotusvaiheeseen.

9. Jonkin patenttivaatimuksen 1–8 mukainen menetelmä, jossa toisessa pesuvaiheessa käytettävä pesukonsepti on uudelleenlietteytyspesukonsepti.

10. Jonkin patenttivaatimuksen 1–8 mukainen menetelmä, jossa toisessa pesuvaiheessa käytettävä pesukonsepti on syrjäytyspesukonsepti.

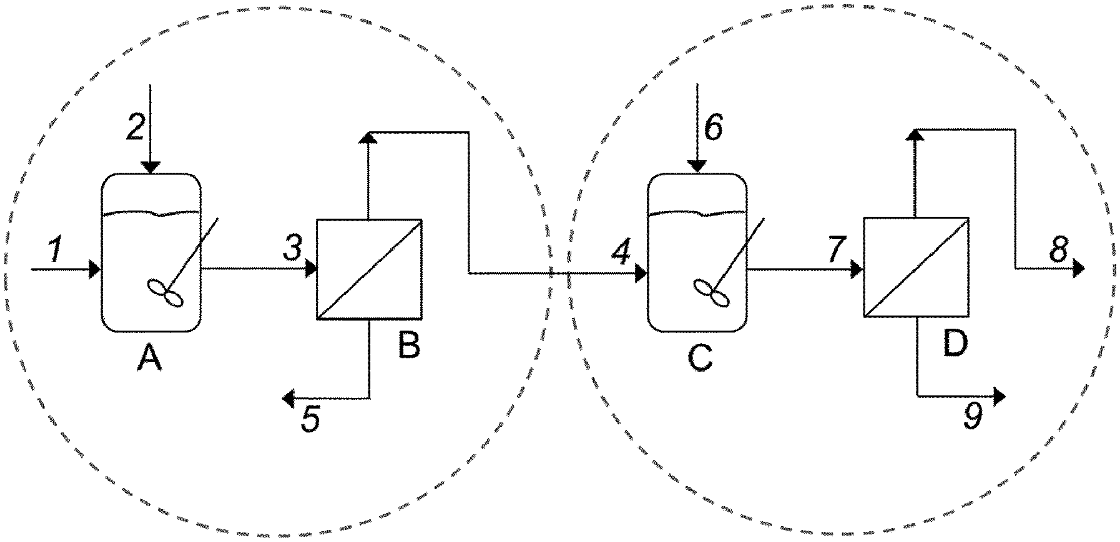


FIG. 1

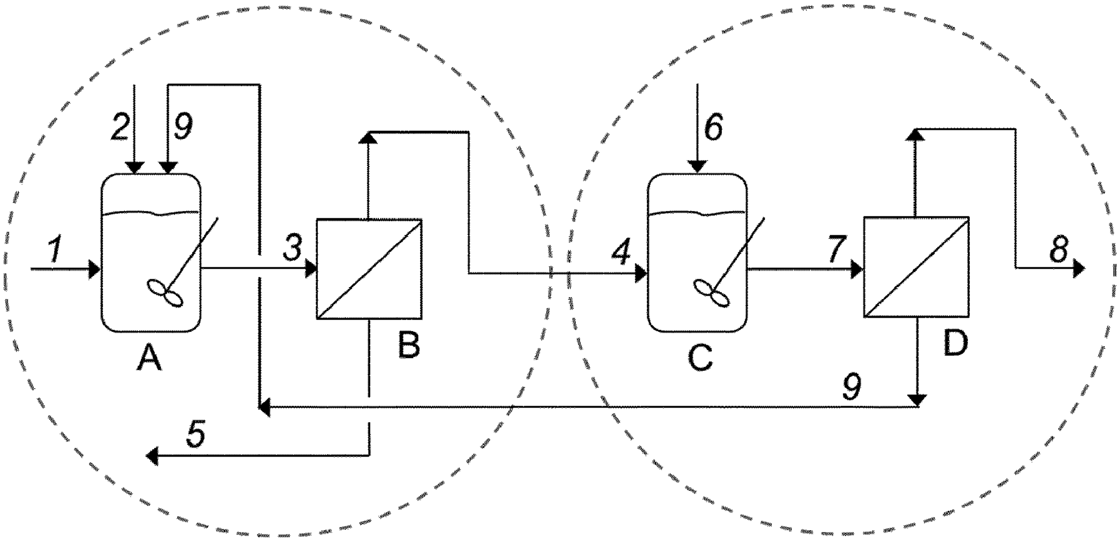


FIG. 2