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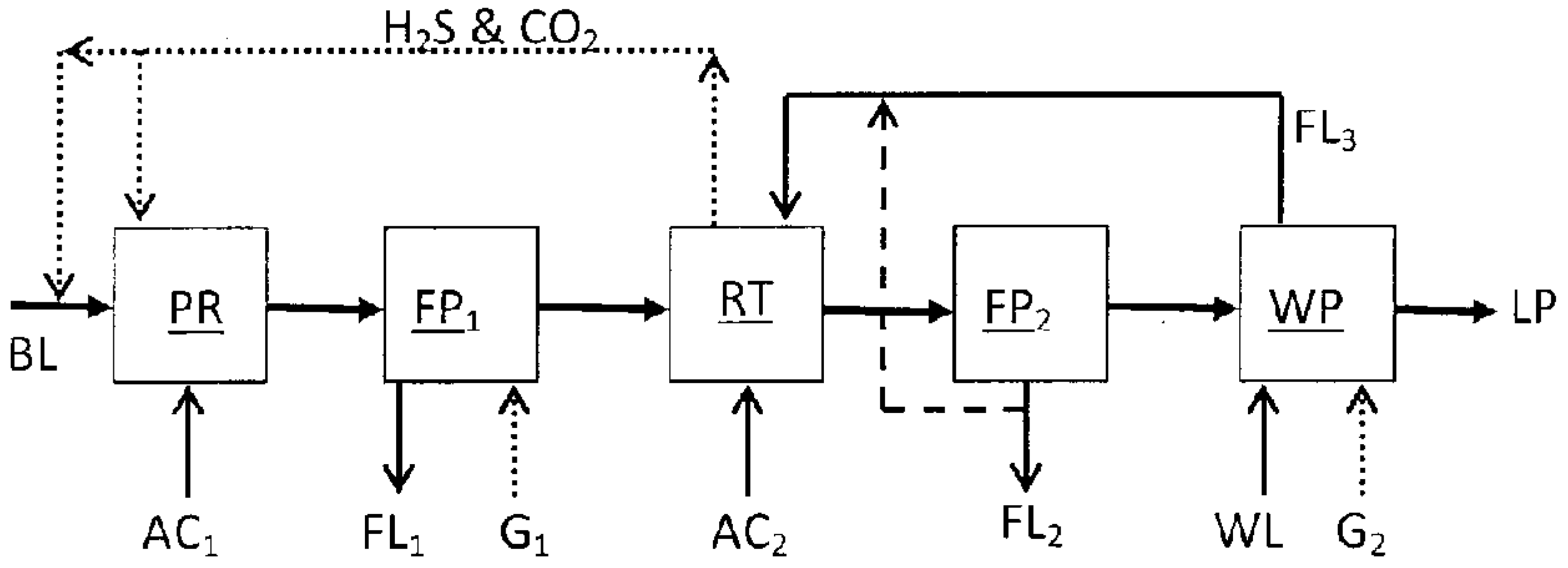
(72) **Inventeurs/Inventors**:
LITTORIN, ANDERS, SE;
WALLMO, HENRIK, SE;
NORBERG, HANNA, SE;
WIMBY, MARTIN, SE

(73) **Propriétaire/Owner**:
VALMET AB, SE

(74) **Agent**: FETHERSTONHAUGH & CO.

(54) **Titre** : PROCEDE DE PRECIPITATION DE LIGNINE A PARTIR DE LESSIVE NOIRE EN UTILISANT DES DECHETS DE GAZ

(54) **Title**: METHOD FOR PRECIPITATING LIGNIN FROM BLACK LIQUOR BY UTILIZING WASTE GASES



(57) **Abrégé/Abstract**:
The present invention relates to a method for separation of lignin from original black liquor comprising the following steps in sequence: a) Precipitation of lignin by a first acidification stage of the original black liquor, b) followed by dewatering while forming a first filter cake with high content of lignin, c) suspending the first lignin filter cake obtained in step b in a second acidification stage, d) whereupon a second lignin suspension is obtained, e) dewatering of the second lignin suspension forming a second filter cake with high content of lignin, f) washing the second filter cake water and finally, g) dewatering of the washed second lignin cake obtaining a lignin product. According to the invention is the waste gases emitted from the second acidification stage re-circulated and mixed with the original black liquor at the latest in the first acidification stage. By this recirculation is a waste gas disposal problem solved while also reducing the amount of fresh acidifier charged in the first acidification stage.

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(71) **Applicant** *(for all designated States except US)*: **METSO POWER AB** [SE/SE]; Box 8734, S-402 75 Göteborg (SE).

(72) **Inventors; and**

(75) **Inventors/Applicants** *(for US only)*: **LITTORIN, Anders** [SE/SE]; Kärra Kapellvägen 11, S-425 30 Hisings Kärra (SE). **WALLMO, Henrik** [SE/SE]; Kyrkvägen 38, S-441 60 Alingsas (SE). **NORBERG, Hanna** [SE/SE]; Doktor Sydows Gata 1 C, S-413 24 Göteborg (SE). **WIMBY, Martin** [SE/SE]; Flygtornsvägen 20, S-423 36 Torslanda (SE).

(74) **Agent**: **FURHEM, Hans**; c/o Metso Fiber Karlstad AB, Box 1033, S-651 15 Karlstad (SE).

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(54) **Title**: METHOD FOR PRECIPITATING LIGNIN FROM BLACK LIQUOR BY UTILIZING WASTE GASES

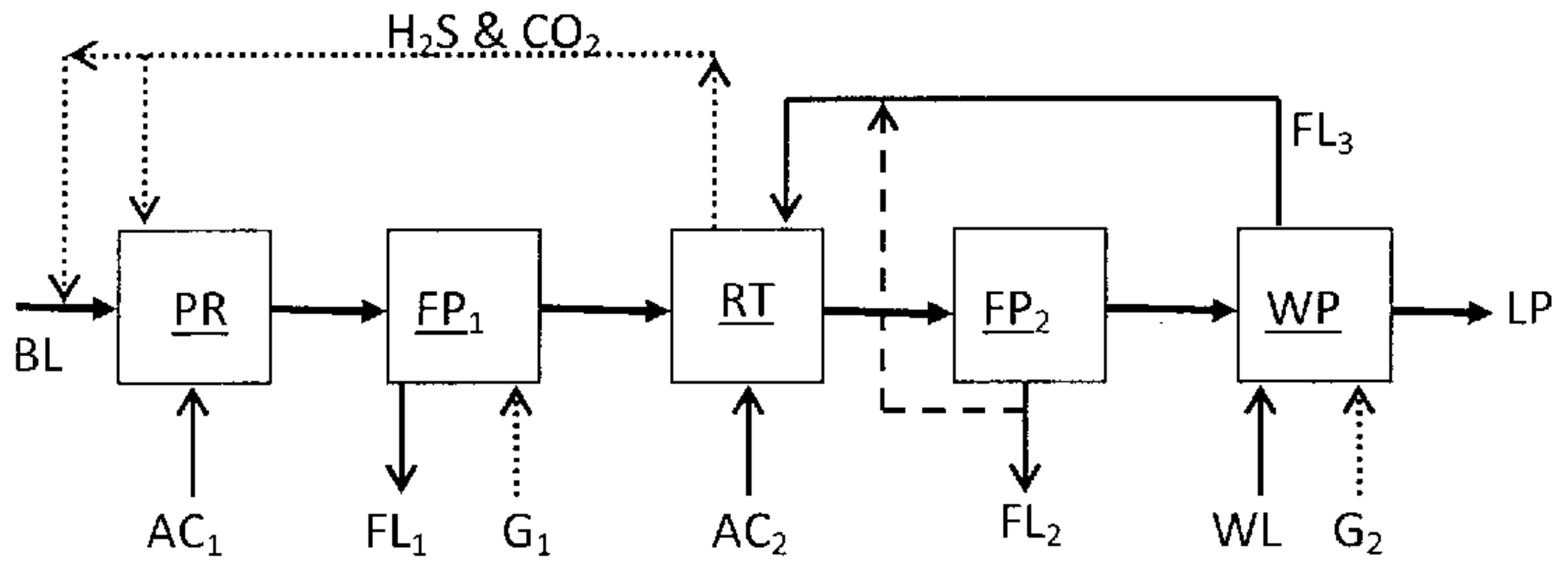


Fig. 2

(57) **Abstract**: The present invention relates to a method for separation of lignin from original black liquor comprising the following steps in sequence: a) Precipitation of lignin by a first acidification stage of the original black liquor, b) followed by dewatering while forming a first filter cake with high content of lignin, c) suspending the first lignin filter cake obtained in step b in a second acidification stage, d) whereupon a second lignin suspension is obtained, e) dewatering of the second lignin suspension forming a second filter cake with high content of lignin, f) washing the second filter cake water and finally, g) dewatering of the washed second lignin cake obtaining a lignin product. According to the invention is the waste gases emitted from the second acidification stage re-circulated and mixed with the original black liquor at the latest in the first acidification stage. By this recirculation is a waste gas disposal problem solved while also reducing the amount of fresh acidifier charged in the first acidification stage.

WO 2010/143997 A1

Method for precipitating lignin from black liquor by utilizing waste gases

Technical Field

This invention relates to a method for lignin separation from spent cooking liquor, called original black liquor, using a 2-stage acidic separation process.

Background

The advantages with lignin separation from black liquor is already described in WO 2006/031175 and WO2006/038863. These patents disclose the novel process ***LignoBoost*™** that is now sold by Metso, and wherein WO 2006/031175 disclose the basic two stage acidic process and WO2006/038863 disclose an improvement of the process where sulphate or sulphate ions are added to the process.

An important aspect of the process is that the required charge of chemicals for the acidification may be high. . If this is the case the cost of fresh chemicals is a large part of the operational cost and the commercial viability of the process is lower. These problems could be reduced, if the process is optimized for minimum requirement for charges of fresh chemicals, making the lignin product commercially sound.

Another problem with the process disclosed in WO 2006/031175 is that there may be a disposal problem with the strongly odorous H_2S gases that are emitted from the reslurrying tank and bled out from the process, and it is suggested that these hydrogen sulfides could be added to the pulping process in order to increase sulfidity and possibly increase the yield in the pulping process. However, such rerouting of the strongly odorous H_2S gases to another part of the pulp mill introduces risks for emissions of these gases during transport and storage. It is far better to use these gases at the location or process producing these gases.

Summary of the invention

The invention is based upon the finding that the H_2S gases that are emitted from the reslurrying tank also contains a large amount of residual carbon dioxide, CO_2. By re-circulating this H_2S and CO_2 rich gas back to the first acidification stage a corresponding reduction of addition of the fresh carbon dioxide is obtained.

carbonates content in the lignin cake. These compounds reacts with the acidifier and forms carbon dioxide (CO_2) and hydrogen sulfide (H_2S), according to:

$$CO_3^{2-} + 2H^+ <-> CO_2 + H_2O$$

$$HCO_3^- + H^+ <-> CO_2 + H_2O$$

$$S^{2-} + 2H^+ <-> H_2S$$

$$HS^- + H^+ <-> H_2S$$

The formation of carbon dioxide in this process enables a further source for carbon dioxide needed for the first acidification stage, and the hydrogen sulfide is also a net contributor to the acidification as the pK_a value of hydrogen sulfide is 6,89.

This invention relates to a two-stage acidic separation process and the scientific definition of acidity is as follows. Reactions of acids are often generalized in the form HA $H^+ + A^-$, where HA represents the acid and A^- is the conjugate base. Acid-base conjugate pairs differ by one proton, and can be interconverted by the addition or removal of a proton (protonation and deprotonation, respectively). Note that the acid can be the charged species and the conjugate base can be neutral in which case the generalized reaction scheme could be written as HA^+ $H^+ + A$. In solution there exists equilibrium between the acid and its conjugate base. The equilibrium constant K is an expression of the equilibrium concentrations of the molecules or ions in solution. Brackets indicate concentration, such that $[H_2O]$ means *the concentration of* H_2O. The acid dissociation constant K_a is generally used in the context of acid-base reactions. The numerical value of K_a is equal to the concentration of the products over the concentration of the reactants, where the reactant is the acid (HA) and the products are the conjugate base and H^+. The stronger of two acids will have a higher K_a than the weaker acid; the ratio of hydrogen ions to acid will be higher for the stronger acid as the stronger acid has a greater tendency to lose its proton. Because the range of possible values for K_a spans many orders of magnitude, a more manageable constant, pK_a is more frequently used, where $pK_a = -\log_{10}(K_a)$. Stronger acids have a smaller pK_a than weaker acids. Experimentally determined pK_a at 25°C in aqueous solution are often quoted in textbooks and reference material. This value of the pK_a is used in following description of the weak and strong acids used. As a comparison the pK_a value for water is 15.7.

embraces any acidifier comparable to carbon dioxide in aspects of acidic strength, i.e. a positive pK_a number in the order of 1-15, preferably 5-12. Often is carbon dioxide the preferred choice for the weak chemical as it is a low impact on environmental loading, and has a well proven capability of improving wash-out performance in wash stages and improves drain ability of the lignin cake. Carbon dioxide is also relatively inexpensive and would not introduce new chemicals into the liquor cycle of the pulp mill. This weak acid is used in the first separation stage of the invention, as the volumes of black liquor are considerable.

It is intended throughout the present description that the expression strong acid embraces any acidifier comparable to sulfuric acid in aspects of acidic strength, i.e. a pK_a number in the order of -3, i.e. in the negative range and preferably in the range -1 to -5. An alternative strong acidifier could be found in acidifying is performed by adding SO2(g) , organic acids, hydrochloric acid (HCl), nitric acid (HNO_3), hydroiodic acid (HI), hydrobromic acid (HBr) or perchloric acid (HCLO4), even if the alternatives with chlorides most often is less preferred due to environmental load. The fresh sulfuric acid added to the process could be obtained from so called "spent acid" from a chlorine dioxide generator or fresh sulfuric acid from a chemical supplier, or mixtures thereof.

The present invention solves above problems, both the disposal problem of the odorous sodium sulfide as well as a reduced charge of acidifier in the first acidification stage.

It is intended throughout the present description that the expression "dewatering" embraces any means of dewatering. Preferably the dewatering is performed by using centrifugation, a filter press apparatus, a band filter, a rotary filter, such as a drum filter, or a sedimentation tank, or similar equipment, most preferred a filter press apparatus is used.

It is intended throughout the present description that the expression "original black liquor" embraces spent cooking liquor from a digester, having most of the lignin from the original cellulose material dissolved in the "original black liquor". The "original black liquor" may also have a large content of organic and inorganic material, but other specific constituents, while keeping the bulk volume of dissolved lignin unaltered.

According to the basic aspects of the inventive method it is related to a method for separation of lignin from an original black liquor comprising the following stages in sequence: (a) precipitation of the lignin by a first acidification stage of the original black liquor by adding a first acid or mixture of acids; (b) followed by dewatering while forming a first filter cake with a high content of the lignin; (c) suspending the first lignin filter cake obtained in stage (b) in a second acidification stage using a second acid or mixture of acids; (d) whereupon a second lignin suspension is obtained; (e) dewatering of the second lignin suspension forming a second filter cake with a high content of the lignin; (f) washing the second filter cake and finally; (g) dewatering of the washed second lignin filter cake obtaining a lignin product, wherein waste gases are emitted from the second acidification stage, and wherein the waste gases are re-circulated and mixed with the original black liquor at the latest in the first acidification stage.

In an embodiment of the invention the waste gases emitted from the second acidification stage are treated in a conversion process wherein at least a part of the waste gas components is converted to a stronger acid having a lower pK_a value before being re-circulated and mixed with the original black liquor. The conversion process preferably takes place in a combustion chamber wherein the combustion is assisted with air or oxygen supply. In this embodiment could also the waste gases emitted from the second acidification stage be collected by a pump that establish a sub atmospheric pressure during the second acidification stage in order to increase degassing and collection of all gases emitted and avoid leakage of odorous gases to the environment.

In a preferred embodiment of the inventive method is the first acid a weak acid having a positive pK_a, preferably carbon dioxide. As the first acid is used in the total effective and yet environmental friendly acidifier that also do not add any dangerous chemicals into the liquor cycle is preferable. This would, however, not exclude the possibility of using the same acidifier as used as the second acid under the basic aspects of the invention.

In yet a preferred embodiment of the inventive method is the second acid a strong acid having a negative pK_a, preferably sulfuric acid. As the second acid is used in the relurrying stage of the first lignin cake, the amount of acidifier needed is dramatically reduced as the total volume of the lignin cake is less than 1/10 of the original black liquor volume. This would not exclude the possibility of using the same acidifier as used as the first acid under the basic aspects of the invention.

In a further preferred embodiment of the inventive method is the entire separation process, from the first acidification stage and until obtaining the final lignin product, kept at acidic conditions below pH 6. Preferably the entire separation process from the first acidification stage is kept at acidic conditions even below pH 4. The pH level throughout the process is most preferred a pH from 1 to 3.5. This would prevent any separated lignin from being dissolved again, and the precipitated lignin would be subjected to repeat leaching of metals and other unwanted components, meeting the objectives of obtaining a clean lignin product at high yield.

In yet a further preferred embodiment of the inventive method is at least the dewatering of stage b) but possibly also stage f) including blowing trough of the first, or second, filter cake by gas or a mixture of gases, preferably flue gases, air or vapor, most preferred air or overheated vapor, in order to dispose of the remaining black liquor. This gas could also be re-circulated to the original black liquor if needed, as these gases may contain components that have a net contributing effect upon the acidification.

The inventive method may also include the additional steps of combining the pH level adjustment with an adjustment of the ion strength, preferably by using alkali metal ions or alkaline earth metal ions, most preferred calcium ions.

The inventive method may also include the additional steps of that at least a part of the filtrate from the dewatering in step g), or filtrate from washing in step f) is returned to the second acidification stage c) to further reduce the consumption of acid and water.

Short description of the figures

Fig. 1 shows the prior art 2-stage lignin separation process according to WO 2006/031175.

Fig. 2 shows the inventive modification of 2-stage lignin separation process.

Detailed description of the invention

In figure 1 is the known prior art process according to WO 2006/031175 shown. The separation of lignin from original black liquor BL comprising the following stages in sequence:

a) Precipitation of lignin by a first acidification stage of the original black liquor by adding a first acid or mixture of acids AC_1, in any suitable precipitation reactor PR,

b) followed by dewatering while forming a first filter cake with high content of lignin, said dewatering made in any suitable filter press FP_1, which may drain a first filtrate FL_1 from the lignin suspension and have addition of gas blow trough G_1 of the lignin cake in order to displace any residual acidic liquor,

c) suspending the first lignin filter cake obtained in stage b in a second acidification stage using a second acid or mixture of acids AC_2, said suspension made in any suitable reslurry tank RT while discarding the odorous gases H_2S emitted,

d) whereupon a second lignin suspension is obtained in the reslurry tank RT,

e) dewatering of the second lignin suspension forming a second filter-/lignin cake with high content of lignin, said dewatering made in any suitable filter press FP_2, which may drain a second filtrate FL_2 from the lignin suspension, and at least a portion of this second filtrate FL_2 may be re-circulated back to stage c, wash apparatus WP, adding a wash liquid WL to this washing stage, and finally

g) dewatering of the washed second lignin cake obtaining a lignin product LP, said dewatering preferably made in the last stages of the wash apparatus WP, which may drain a third filtrate FL_3 from the second filter/lignin cake, and at least a portion of this third filtrate FL_3 may be re-circulated back to stage c, and may also have addition of gas blow trough G_2 of the lignin cake in order to displace any residual acidic liquor.

In figure 2 is the inventive improvement of the prior art process according to WO 2006/031175 shown. The separation of lignin from original black liquor BL comprises the same basic steps as shown in figure 1, but here the waste gases emitted from the second acidification stage, i.e. from reslurrying tank RT, is re-circulated and mixed with the original black liquor at the latest in the first acidification stage.

The filtrate from the first dewatering stage in FP_1 is preferably re-circulated directly to a recovery system, preferably after re-alkalization.

The gas used in FP_1 or WP for blowing through the filter cake is preferably air or flue gases, preferably flue gases from a recovery boiler, a bark boiler or a lime kiln.

In figure 3 is an alternative embodiment of the inventive improvement of the prior art process according to WO 2006/031175 shown. Here is the re-circulated gas from the reslurring stage in RT passed to an oxidization reactor where the gas is oxidized with or without a catalyst in a combustion process with a flame in an oxidation reactor OR, while being added with oxygen, O_2. In such oxidization a conversion of the H_2S to Sulfur dioxide or Sulfur trioxide takes place, which in a water solution have a stronger acidity than H_2S. The oxidation reactor could alternatively be a catalytic process or a combination of a combustion and a catalytic process similar to a sulfuric acid production process. The acidic strength of H_2SO_3 and H_2SO_4 corresponds to pK_a values in the order of 1.8 and -3, respectively. If a large part of the H_2S is converted to SO_2 and/or SO_3, then the need for charging fresh carbon dioxide decreases even further in order to establish the low pH value for the lignin precipitation. The savings in charge of fresh carbon dioxide could then be reduced by some 10-20% further, depending upon the conversion efficiency in the oxidization and total amount of H_2S

preferably be collected by a vacuum pump P, pressurizing the gases and positively assuring that all emitted gases from the reslurrying tank is vented away. As oxygen/air may be included in the stream of gases, due to slight sub atmospheric pressure in reslurrying tank, it would be advantageous to add the treated gases before charging the carbon dioxide to the original black liquor, i.e. in the in feed pipe to precipitation reactor PR as shown in figure, or any other suitable vessel.

An additional procedure for stabilizing the lignin during the 2-stage process is, in combination with a pH-decrease, to adjust the ionic strength in the slurry stage, preferably with multivalent alkali metal ions or alkaline earth metal ions (e.g. calcium). At a given pH, a higher ionic strength in the suspension stage reduces the lignin yield losses. Here also the ionic strength and pH of the wash water preferably essentially correspond to the conditions in the slurry stage to avoid gradients during the washing process. A higher ionic strength in the slurry and in the wash water gives a stable lignin and high lignin yield even at pH- values in the upper acidic range.

The method according to the first aspect of the present invention may further be performed, as set out above, whereby first the lignin is precipitated with carbon dioxide or other suitable acids according to previously known methods. The suspension is then dewatered in some form of separation equipment (e.g. some form of filtration equipment, sedimentation tank, centrifugation etc). A filter press equipment where the filter cake can be pressed to a high dry content is preferable. Thereafter, air is preferably blown through the pressed filter cake in order to remove as much as possible of the remaining black liquor. In this way, the acid consumption and hydrogen sulfide formation in the subsequent re-slurry stage can be considerably reduced.

The filter cake is reslurried in tank RT preferably equipped with a suitable stirring device and equipped with an exhaust to take care of the hydrogen sulfide formed as well as residual and formed carbon dioxide.

Examples

Example 1 (Comparative)

In the prior art process shown in figure 1 was an original black liquor used with a dry lignin product had a dry matter concentration of 70% at a production rate of 348 t/day.

Following charges was made in the process:

Charge of Fresh CO_2 (ton/day)	Filtrate (FL_1) (ton/day)	Charge of H_2SO_4 (m^3/day)	Filtrate (FL_2) (ton/day)	Wash Liquor (WL) (ton/day)
120	4441	12	541	541

As shown in the figure was the charge proportions of fresh carbon dioxide more than 1/3 (>33%) of the produced final lignin product, i.e. charge of 120 ton per 348 ton of final lignin product.

As the costs for carbon dioxide is almost 100 Euro per ton, the costs for chemicals then a great part of the production costs for the final lignin product.

Example 2, the invention

If the process instead is modified according to the invention, using re-circulation of the gases emitted in the reslurrying stage, as shown in figure 2, but using the same original black liquor with a dry matter concentration of 30% and at a feed rate of 4790 t/day, and where the final lignin product had a dry matter concentration of 70% at a production rate of 348 t/day following charges was made in the process:

Charge of Fresh CO_2 (ton/day)	Filtrate (FL_1) (ton/day)	Charge of H_2SO_4 (m^3/day)	Filtrate (FL_2) (ton/day)	Wash Liquor (WL) (ton/day)
78	4441	12	541	541

As shown in the figure was the charge proportions by weight of fresh carbon dioxide reduced to about 1/5 (22%) of the produced final lignin product, i.e. a charge of 78 ton per 348 ton of final lignin product. The actual charge of carbon dioxide was reduced by 35% in comparison with the prior art process without recirculation of emitted gases from the reslurrying stage.

Various embodiments of the present invention have been described above but a person skilled in the art realizes further minor alterations, which would fall into the should not be limited by any of the above-described exemplary embodiments, but should be defined only in accordance with the following claims and their equivalents. For example, any of the above- noted methods can be combined with other known methods for separating lignin from black liquor using equivalent weaker or stronger acids. Other aspects, advantages and modifications within the scope of the invention will be apparent to those skilled in the art to which the invention pertains.

CLAIMS:

1. A method for separation of lignin from an original black liquor comprising the following stages in sequence:

(a) precipitation of the lignin by a first acidification stage of the original black liquor by adding a first acid or mixture of acids;

(b) followed by dewatering while forming a first filter cake with a high content of the lignin;

(c) suspending the first lignin filter cake obtained in stage (b) in a second acidification stage using a second acid or mixture of acids;

(d) whereupon a second lignin suspension is obtained;

(e) dewatering of the second lignin suspension forming a second filter cake with a high content of the lignin;

(f) washing the second filter cake and finally;

(g) dewatering of the washed second lignin filter cake obtaining a lignin product,

wherein waste gases are emitted from the second acidification stage, and wherein the waste gases are re-circulated and mixed with the original black liquor at the latest in the first acidification stage.

2. The method according to claim 1, wherein the waste gases emitted from the second acidification stage are treated in a conversion process and wherein at least a part of the waste gases components is converted to a stronger acid having a lower pK_a value before being re-circulated and mixed with the original black liquor.

3. The method according to claim 2, wherein the conversion process takes place in a combustion chamber, wherein the combustion is assisted with an air or an

oxygen supply.

4. The method according to claim 2, wherein the conversion process takes place in an oxidization reactor with a catalyst with or without a flame.

5. The method according to claim 1, wherein the waste gases emitted from the second acidification stage are collected by a pump that establishes a sub atmospheric pressure during the second acidification stage.

6. The method according to claim 1, wherein the first acid is a weak acid having a positive pK_a.

7. The method according to claim 6, wherein the first acid is carbon dioxide.

8. The method according to claim 6 or 7, wherein the second acid is a strong acid having a negative pK_a.

9. The method according to claim 8, wherein the second acid is sulfuric acid.

10. The method according to claim 8 or 9, wherein the entire separation process from the first acidification stage and until obtaining the final lignin product is kept at acidic conditions below pH 6.

11. The method according to claim 10, wherein the entire separation process from the first acidification stage is kept at acidic conditions below pH 4.

12. The method according to any one of claims 1 to 11, wherein at least the dewatering of stage (b) and optionally also stage (f) includes blowing through of the first, or second, filter cake by a gas or a mixture of gases in order to dispose of the remaining black liquor.

13. The method according to claim 12, wherein the gas flowing through the filter cake is a flue gas, air or vapor.

14. The method according to claim 13, wherein the gas flowing through the filter cake is air or an overheated vapor.

15. The method according to any one of claims 1 to 14, wherein a pH level adjustment is combined with an adjustment of the ion strength.

16. The method according to claim 15, wherein the ionic strength adjustment is effected by using alkali metal ions or alkaline earth metal ions.

17. The method according to claim 16, wherein the ionic strength adjustment is effected by using calcium ions.

18. The method according to any one of claims 1 to 17, wherein at least a part of a third filtrate from the dewatering step (g), or a third filtrate from washing in step (f) is returned to the second acidification stage (c) to further reduce the consumption of acid and water.

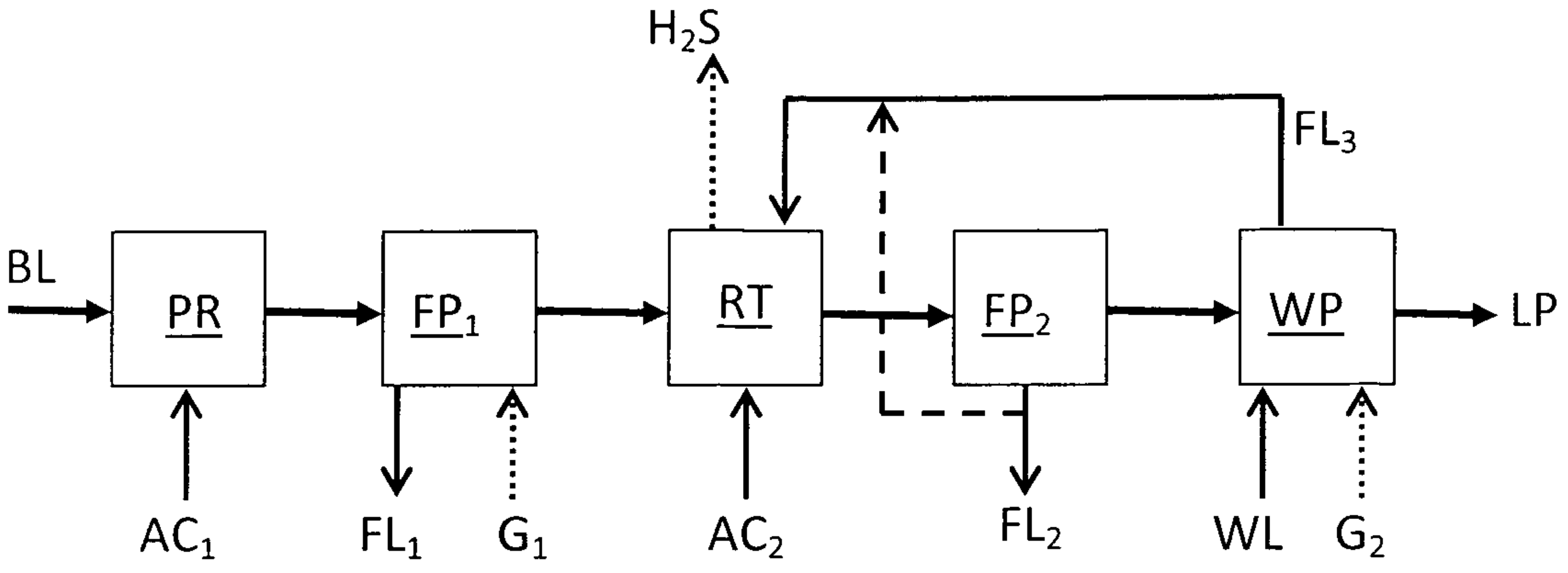


Fig. 1 Prior art

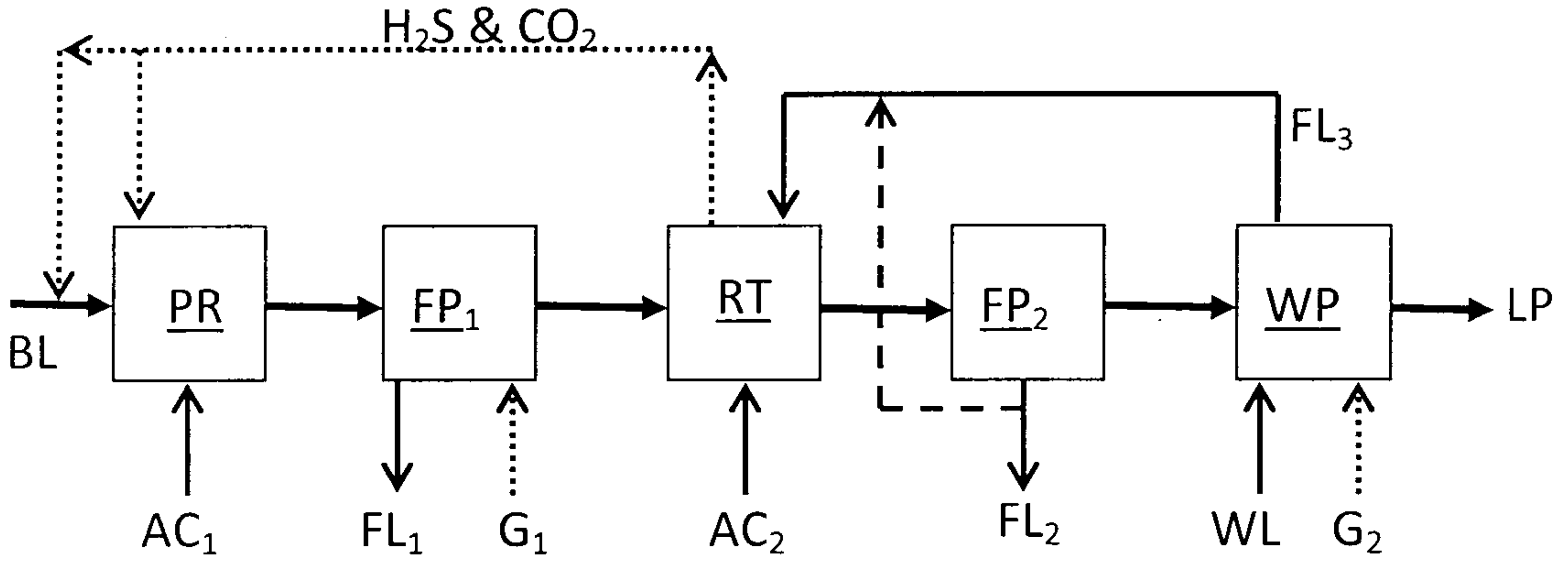


Fig. 2

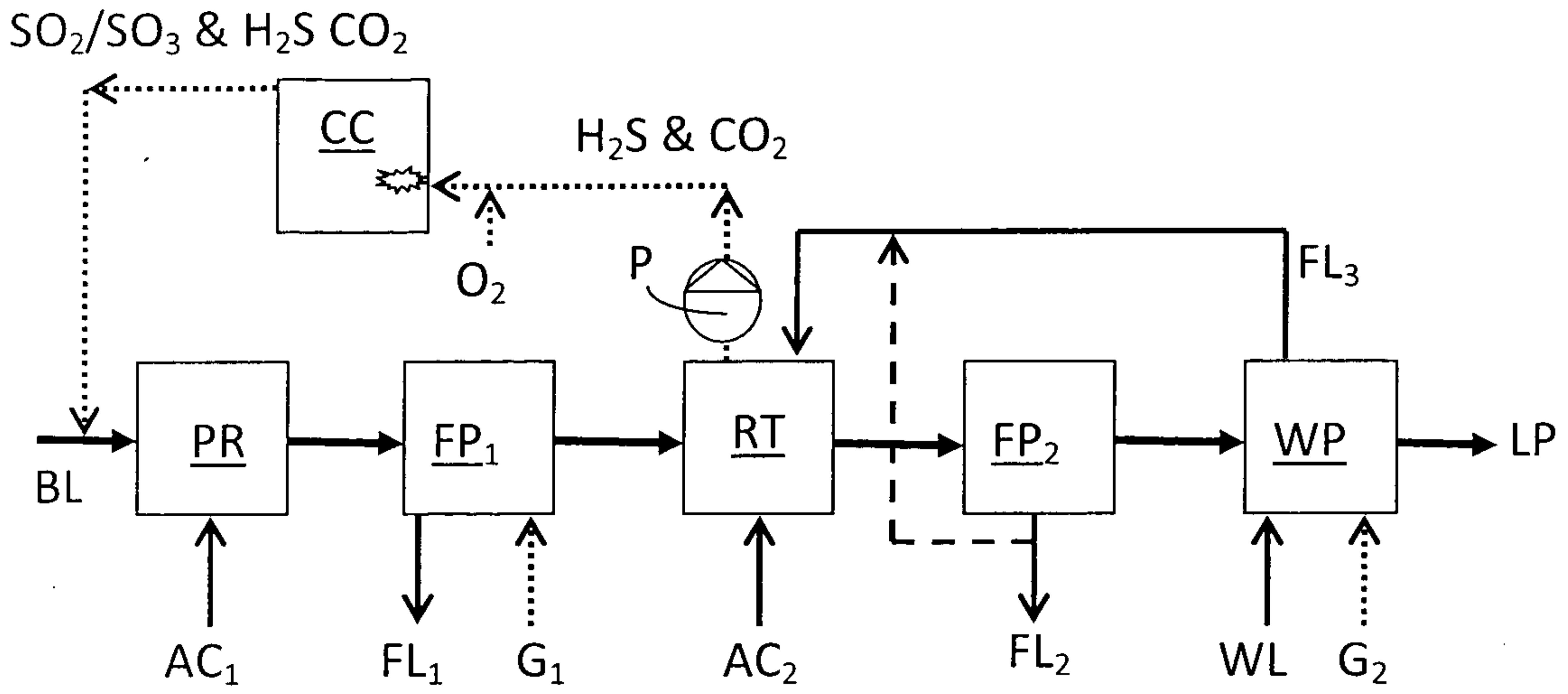
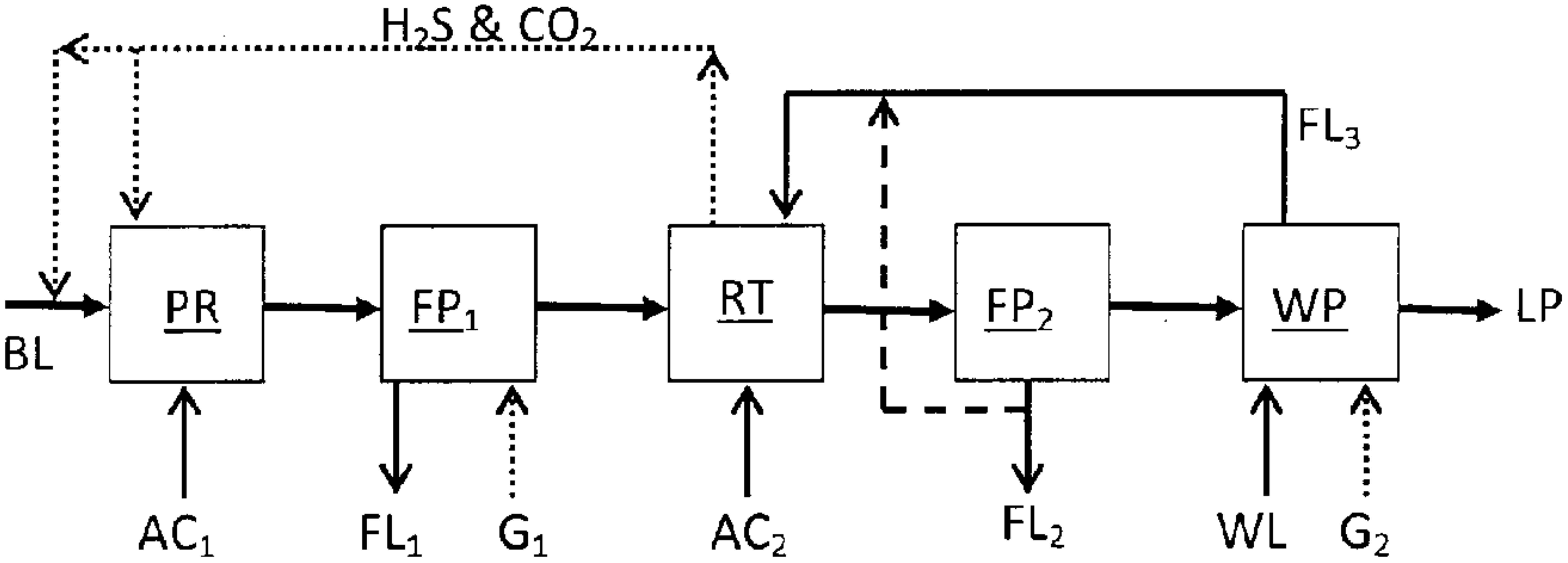

SO2/SO3 & H2S CO2
CC
H2S & CO2
O2
P
FL3
BL
PR
FP1
RT
FP2
WP
LP
AC1
FL1
G1
AC2
FL2
WL
G2

Fig. 3


H2S & CO2
FL3
BL
PR
FP1
RT
FP2
WP
LP
AC1
FL1
G1
AC2
FL2
WL
G2