



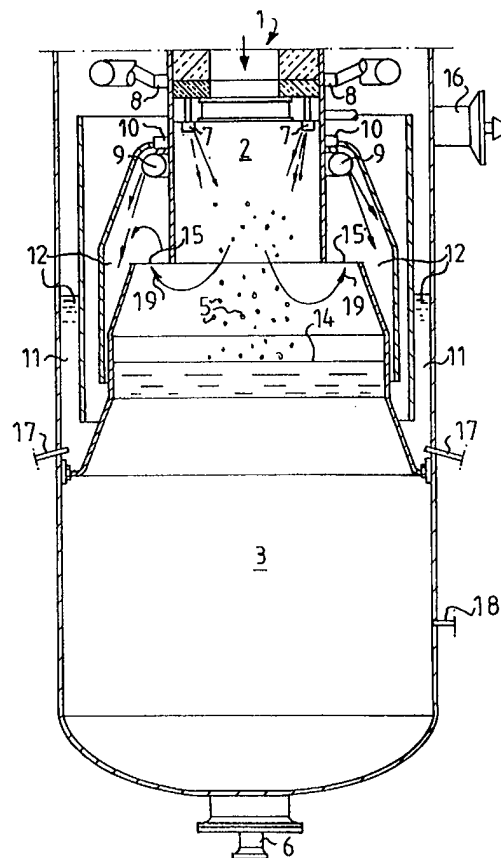
## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: SEPARATING ALKALI METALS FROM GASIFIED BLACK LIQUOR

## (57) Abstract

A process for separating alkali compounds entrained in a superheated gas which has been formed from partial combustion of spent liquor from a cellulose pulping process, where at least cooling medium is injected into a gas/salt smelt mixture in such an amount that the resulting mixture remains superheated and that salt particles are solidified or partially solidified, whereby the gas is lead away for separate cooling washing and the salt particles are dissolved in a receiving liquor.



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## SEPARATING ALKALI METALS FROM GASIFIED BLACK LIQUOR

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Technical field

The present invention relates to a process for recovering of chemicals and energy from spent liquors which are obtained during pulp production by means of chemical delignification of fibre raw material. The invention particularly relates to a process for separating alkali metal salts from gasified black liquor.

State of the art and problems

15 When pulp is produced in accordance with the sulphate method, a spent liquor, generally termed black liquor, is obtained which contains organic material and the residual chemicals which have been used when cooking the fibre raw material. In general, this black liquor is evaporated and conveyed to a separate process for recovering of the energy content of the organic material and recovering the cooking chemicals as so-called green liquor. For a long time, the so-called Tomlinson process has been the commercially dominant method used for this recovery of energy and chemicals. However, a disadvantage of this process, which is now very old, is that it requires very large combustion ovens which are complicated both from the technical point of view and as regards their operation.

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Swedish patent SE 448,173 describes a more recent process which, besides requiring process equipment which is appreciably simplified, achieves an improved recovery of both energy and chemicals. This process is based on a pyrolysis reaction in which the black liquor is gasified in a reactor, resulting in the formation of an energy-rich gas, principally comprising carbon monoxide, carbon dioxide, methane, hydrogen and hydrogen sulphide, and of inorganic chemicals in the form of small

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drops of smelt, principally comprising sodium carbonate, sodium hydroxide and sodium sulphide. The resulting mixture of gas and smelt drops is rapidly cooled, in a first stage, by means of direct contact with a cooling liquid consisting of water and green liquor, which is formed when the smelt chemicals dissolve in the cooling liquid. The gas is subsequently washed, in a second stage, in a gas-washing apparatus of the scrubber type. The gas is then used as a fuel for generating steam and/or electric power. The physical calorific value of the gas can also be utilized when the gas is cooled down from the gasification temperature to the saturation temperature for aqueous steam at the selected pressure. At a saturation temperature of 252°C, corresponding to 40 bar, for example, steam having a pressure of 3-8 bar can be generated when the green liquor is cooled and when the gas is cooled and its water content is condensed downstream of the gas-washing tower.

Nevertheless, this process too, despite being appreciably simpler and smoother than the Tomlinson process, still leaves room for improvement. For example, unwanted carbonate and hydrogen carbonate is formed in the green liquor when carbon dioxide in the pyrolysis gas comes into contact with the green liquor when the gas and smelt droplets are quenched and dissociated in the aqueous solution of the first stage. Hence, pH of the green liquor is decreased. It also leads to formation of H<sub>2</sub>S which leaves together with the rest of the gases.

Consequently, there is a need for a process where the amount of time is minimized during which it is possible for carbon dioxide to come into contact with the green liquor and gaseous and/or liquid hydroxide salts.

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#### Summary of the invention

Now it has turned out that the above mentioned problems can be solved by conducting the superheated gases from the gasifier (1) through a downcoming tube (2) which ends above the liquid surface of a quench vessel (3) containing a receiving liquor. One or more cooling media (13), preferably comprising condensate and satu-

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rated steam, is injected into the stream of superheated gas within or near said tube in such an amount that the resulting mixture remains superheated and as a result solid salt particles are formed in the hot gas. The gaseous components of the flow from the gasifier including the injected cooling medium/media, will leave the quench vessel without any substantial contact with the receiving liquor, so that the contact time with the receiving liquor is prevented or minimized. The solid salt particles are, however, forced by gravity to continue down into the receiving liquor of the quench vessel for dissociation.

#### 10 Brief description of the figures

The invention will now be described below in more detail on the basis of a preferred embodiment and with reference to the attached figures, where:

15 Figure 1 shows a preferred embodiment of the concept according to the invention.

Figure 2 is a simplified sketch illustrating the need of causticizing the salts of green liquor produced by gasifying spent liquor as a function of the degree of superheating in the zone where the salt particles are separated from the gaseous compounds in the gasifier product.

#### Detailed description of the invention

As disclosed herein, the term "receiving liquor" relates to an aqueous liquid comprising alkali metal salts, such as green liquor or white liquor.

As disclosed herein, the term "cooling medium" relates to steam and/or an aqueous liquid substantially free from alkali metal ions, such as a condensate from a counter-current condenser or a sulphur-containing condensate from evaporation of spent liquor. Several different types of cooling media may be used at the same time. Steam is preferred.

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As disclosed herein, the term "washing liquid" relates to an aqueous liquid which preferably comprises a condensate from indirect cooling.

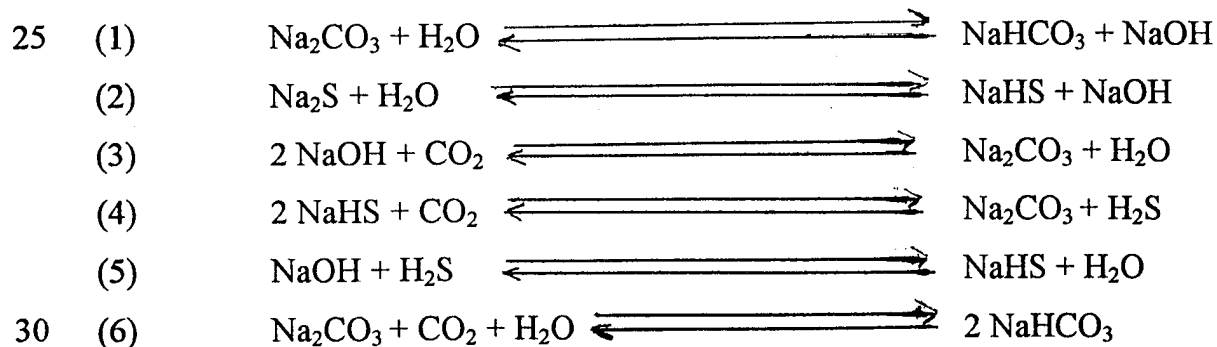
- 5 As disclosed herein, the term "quench vessel" relates to a vessel containing the receiving liquor. The quench vessel is arranged in such a way that salt particles solidifying or close to solidifying as a result of injection of cooling medium in the stream from the gasifier, can be separated and dissolved in the receiving liquor.
- 10 As disclosed herein, the term "gasifier product" relates to the hot mixture of gases and salt smelt that enters the down-coming tube from the gasifier.

As disclosed herein, the term "superheated gas" relates to a gaseous mixture containing steam and does not contain any condensed water, i.e. steam of a certain  
 15 pressure which is heated above the condensation/boiling temperature of that pressure.

All pressure values in the following description and the appended claims refer to absolute values.

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The following equilibrium reactions in the quench are of special importance for the composition of the aqueous solution of alkali metal salts produced according to the present invention.



By contacting a process gas containing carbon dioxide with an alkaline solution using prior art quench vessels, the gas tends to absorb into the solution and the resulting pH value of green liquor is decreased.

- 5 In accordance with reactions (1) and (6), alkali is partly converted to alkali hydrogen carbonate, which is a highly undesirable compound in green liquor as it puts an extra load on the causticizing system. Twice as much lime is needed for conversion of alkali hydrogen carbonate to hydroxide compared to conversion from alkali carbonate.

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The concept behind the method which has been devised is to bring about the possibility of producing green liquor and/or white liquor without unwanted hydrogen carbonate being formed in this liquor, and unwanted hydrogen sulfide being formed in the gas.

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- The principle is that the gasifier product leaving the reactor is cooled by means of direct contact with one or more cooling media, causing mostly solidification of the salts. Any extensive contact between the gas and the receiving liquor is avoided to the greatest extent possible. According to the present invention carbon dioxide in
- 20 the gasifier product is prevented from reacting with sodium carbonate (reaction (6) above), and carbon dioxide is prevented from reacting with sodium hydroxide (reaction (3) above) and forming sodium carbonate. Furthermore, carbon dioxide is prevented from reacting with sodium hydrogen sulfide (reaction (4) above) and forming sodium hydrogen carbonate and also hydrogen sulfide desorption from the
- 25 reaction between sodium hydrogen carbonate and sodium hydrogen sulfide is avoided. It is advantageous if the sodium hydroxide which has been formed is not converted to sodium carbonate, since sodium hydroxide is the desired final product following causticizing of the green liquor. During the causticizing, sodium carbonate is converted to sodium hydroxide by reacting with slaked lime.

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The need of causticizing of green liquor produced according to the present invention is illustrated in fig. 2. The figure discloses a simplified sketch showing the need for causticizing the produced green liquor as a function of the temperature in the quench region where the alkali metal salts in the gasifier product are separated from the gaseous compounds in said gasifier product.

Preferably the above mentioned superheated gaseous compounds are quenched in a second stage, which is configured so that the maximum degree of contact is achieved between the gas and the washing liquid. Since the alkali metal salts were separated from the gas in the first stage the above mentioned equilibrium reactions cannot occur in this stage, and thus the intense contact between gas and liquid does not impair the result. The gas is quenched by being allowed to bubble through a liquid bath which principally consists of washing liquid. In this way, the originally superheated hot gas can be efficiently cooled and moist saturated.

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Fig. 1 discloses a preferred embodiment of the present invention. Spent liquor is gasified in a ceramically lined gasification reactor (1). The reactor (1) is provided with an inlet for black liquor (not shown) and an inlet for oxygen or oxygen-containing gas (not shown), and a burner (not shown). The temperature of the gasifier is kept within the range of 500°C-1600°C, preferably 800°C-1200°C. The pressure in the gasifier is held in the range of 1-150 bar. There are two separate kinds of gasification processes, namely low-pressure (or atmospherical) gasification or high-pressure (or pressurized) gasification. Low-pressure gasification (e.g. sulphide reactor) is carried out at 1.5-5 bar, preferably 1.5-3 bar. High pressure gasification (e.g. green liquor reactor), which is preferred, is carried out at 1.5-150 bar, preferably 10-80 bar, and most preferably 25-40 bar. The bottom of the reactor opens as a down-coming tube (2), which in turn ends above the resting surface of the receiving liquor (14) in the quench vessel (3). The down-coming tube (2) ends (19) more than 0.5 m, preferably more than 0.7 m and most preferably more than 1.0 m above the surface of the receiving liquid. One or more cooling media are transported in at least one conduit (8) to at least one nozzle (7) and is then injected into the hot gasifier pro-

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duct. The cooling medium is injected at a pressure above the pressure in the down-coming tube (2). It is necessary that the temperature of the resulting mixture formed by the injected cooling medium and the gasifier product remains superheated in order to prevent dissociation of the salts and unnecessary formation of carbonates and hydrocarbonates, and that this temperature preferably is lower than the smelt temperature of the alkali metals in order enable the smelt to solidify or partially solidified or form sufficiently sized drops of smelt in the separation zone before reaching the surface of the receiving liquor (14). Preferably said mixture temperature is within the range of 250-800°C, preferably 250-600°C and most preferably 250-400°C. When the pressure in the down-coming tube (2) is atmospheric the temperature of the mixture of the cooling medium and gasifier products in the down-coming tube (2) is in the range of 150-800°C, preferably 150-600°C and most preferably 150-400°C. The injected cooling medium may at least partially be in liquid state, provided that the resulting mixture is superheated. The cooling effect is stronger if a liquid cooling medium is injected because evaporation of the liquid requires energy. The skilled person can easily determine the amount of cooling medium that can be mixed with the above mentioned hot gasifier product so that this superheating requirement is fulfilled. In some cases it may be advantageous just to inject a minimal amount of cooling medium, especially when the gasification is carried out at a relatively low temperature or when a higher degree of superheating is desired. Preferably the cooling medium/media is/are injected perpendicularly in relation to the gasifier product stream, or at an angle towards the quench vessel. Furthermore, it sometimes might be advantageous to inject the steam tangentially in order to create a rotational movement. In an alternative embodiment, cooling medium is introduced into a space (annulus, tubes) around the reactor in order to cool it before being supplied. The solidified or partially solidified salt particles (5) are moved downwards by the resulting gas stream and into the liquid by means of gravity and are finally dissolved in the receiving liquor (14). The concentration of the receiving liquor in quench vessel (3) is controlled by adding water or a suitable aqueous solution through pipe (18) and transporting receiving liquor away through conduit (6). The gaseous constituents of the mixture of cooling medium and gasifier products are

passed through at least one opening (15) after the down-coming tube (2). The outlet (15) is arranged at least 0.5 m, preferably 0.7 m and most preferably 1 m downwards from nozzle (7) in order to obtain a suitable level of superheating of the gaseous compounds/smelt drops within the tube (2).

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In a preferred embodiment of the present invention the superheated gases are subsequently quenched, and optionally cooled and moist-saturated in a second stage after passing through opening (15). The gases are forced into at least one preferably annular cavity (12) at least partially filled with washing liquid (11). Washing liquid is introduced into the side cavity (12) by means of at least one conduit (10) and at least one nozzle (9). It is preferable to inject the washing liquid in a manner so as to not dilute the receiving liquor. When the gases are passed through the side cavity (12) an intense contact with the washing liquor (11) is obtained. The gas is let out via conduit (16) together with at least a part of the washing liquid. It is also possible to let out excess washing liquid via pipe (17).

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The present invention can, of course, be applied to recovering alkali metal salts from different steps in different pulping processes, such as recovering alkali metal salts from spent bleach liquor, or spent liquor from production of CTMP, or spent liquor from a pulping process based on potassium hydroxide.

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The invention will now be further illustrated by the following example.

#### Example

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Gasified black liquor was quenched by a process according to the present invention. Gaseous compounds and small smelt drops were let in a down-coming tube ending 1 m above the resting surface of green liquor. The temperature of the gas/smelt mixture was 950 °C and the pressure was 32 bar. As cooling media were injected condensate downstream followed by moist saturated steam of 40 bar, 250°C, in the down-coming tube, whereby the temperature of the resulting superheated mixture

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became 410°C. The superheated steam and the gaseous compounds were let out through the down-coming tube to a second quenching step and the solidified smelt or partially solidified salt particles were separated from the gas stream by means of gravity and subsequently dissolved in green liquor. The results are disclosed in the table below.

Comparative example

Gasified black liquor was quenched according to the teachings of SE-C-448,173. The gasification process were performed in the same manner as in the example. The results are disclosed in the table below.

Table

Compound	Example	Comparative example
NaHCO <sub>3</sub>	-	40 g/l
Na <sub>2</sub> CO <sub>3</sub>	105 g/l	108 g/l
NaHS	15 g/l	12 g/l
<u>NaOH</u>	<u>40 g/l</u>	<u>0</u>
Total	160 g/l	160g/l

Read g/l counted as NaOH.

The results show that green liquor produced according to the present invention is more alkaline than green liquor produced according to the method of the state-of-the-art. Causticizing of the green liquor of the state-of-the-art requires 120 % more slaked lime than the green liquor produced according to the present invention.

Claims

1. A process for the preparation of an aqueous solution of alkali metal salts and for separating alkali compounds entrained in a superheated gas containing carbon monoxide, carbon dioxide, methane and hydrogen, which mixture of gas and entrained alkali compounds (gasifier product) has been formed from partial combustion of spent liquor from a cellulose pulping process, the process comprising the steps of:
- 5
- a) passing the gasifier product down through a down-coming tube (2), which tube (2) ends above the resting surface of a receiving liquor (14) in a quench vessel (3);

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  - b) injecting one or more cooling media into the gas in such an amount that the resulting mixture of cooling medium and gasifier products is superheated and has a temperature that is sufficiently low to enable solidification or partially sodification of alkali metal salt particles (5) from liquid and gaseous alkali metal salts present in the gasifier product;

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  - c) forcing the resulting superheated mixture of steam and gaseous components in the gasifier product to pass through at least one opening (15) at the down-coming tube (2) thereby avoiding contact with a receiving liquor (14) and collecting this gas steam mixture; and

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  - d) collecting the salt particles (5) in the receiving liquor (14) in the quench vessel (3); whereby an aqueous solution of alkali metal salts is formed;

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  - e) adjusting the concentration of alkali metal salt solution by adding a controlled amount of water or a suitable aqueous solution; and

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  - f) collecting the aqueous solution of alkali metal salts.

2. A process according to claim 1 further comprising the steps of:

- 5 g) conducting the superheated mixture of steam and the gaseous components that has been passed through the opening(s) (15) at the down-coming tube (2) to at least one side cavity (12) at least partially filled with a washing liquid (11);
- h) introducing a washing liquid into the side cavity (12);
- 10 i) passing the gaseous components through the washing liquid (11) in order to obtain intense contact with the washing liquid; and
- k) collecting the gas mixture so obtained.

3. A process according to claim 1 or claim 2 **characterized** in that the pressure in the down-coming tube (2) is in the range of 1.0-50 bar.

4. A process according to claim 3 **characterized** in that the pressure in the down-coming tube (2) is in the range of 10-80 bar.

20 5. A process according to claim 4 **characterized** in that the pressure in the down-coming tube (2) is in the range of 25-40 bar.

6. A process according to any of the preceding claims, **characterized** in that the temperature of the gasifier is kept within the range of 500-1600°C.

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7. A process according to claim 6, **characterized** in that the temperature of the gasifier is kept within the range of 800-1200°C

8. A process according to any of the preceding claims, **characterized** in that the injected cooling medium is saturated with moisture.

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9. A process according to any of the preceding claims, characterized in that the down-coming tube (2) ends (19) more than 0.5 meter, preferably more than 0.7 meter and most preferred more than 1.0 meter above the surface of the receiving liquid.
- 5 10. A process according to any of the preceding claims, characterized in that the cooling medium is injected at least 0.5 m, preferably 0.7 m, and most preferably 1.0 m above the outlet(s) (15).
- 10 11. A process according to any of the preceding claims, characterized in that at least a part of the cooling medium in step b) is steam.
12. A process according to claim 11, characterized in that all the cooling medium in step b) is steam.
- 15 13 A process according to claim 1, characterized in that the mixture of the cooling medium and gasifier products in the down-coming tube (2) has a temperature within the range of 250-800°C, preferably 250-600°C and most preferably 250-400°C.
- 20 14. A process according to claim 1, characterized in that the pressure in the down-coming tube (2) is atmospheric and in that the mixture of the cooling medium and gasifier products in the down-coming tube (2) has a temperature within the range of 150-800°C, preferably 150-600°C and most preferably 150-400°C.
- 25 15. An apparatus for separating melted salts entrained in a hot gas, said apparatus comprising:
- a down-coming tube (2) which in its upper end is connected to a source (1) of the gas/melted salts;
  - at least one device for injecting cooling medium (7) in the down-coming tube (2); and
  - 30 at least one opening (15) located in or close to the bottom end (19) of the down-coming tube (2).

16. An apparatus according to claim 15, characterized in that the device for injecting cooling medium (7) in the down-coming tube (2) is adapted for injecting steam.

5 17. An apparatus according to claim 15, characterized in that the distance between the device(s) (7) for injecting cooling medium and the opening(s) (15) in the down-coming tube is at least 0.5 m, preferably 0.7 m and most preferably 1.0 m.

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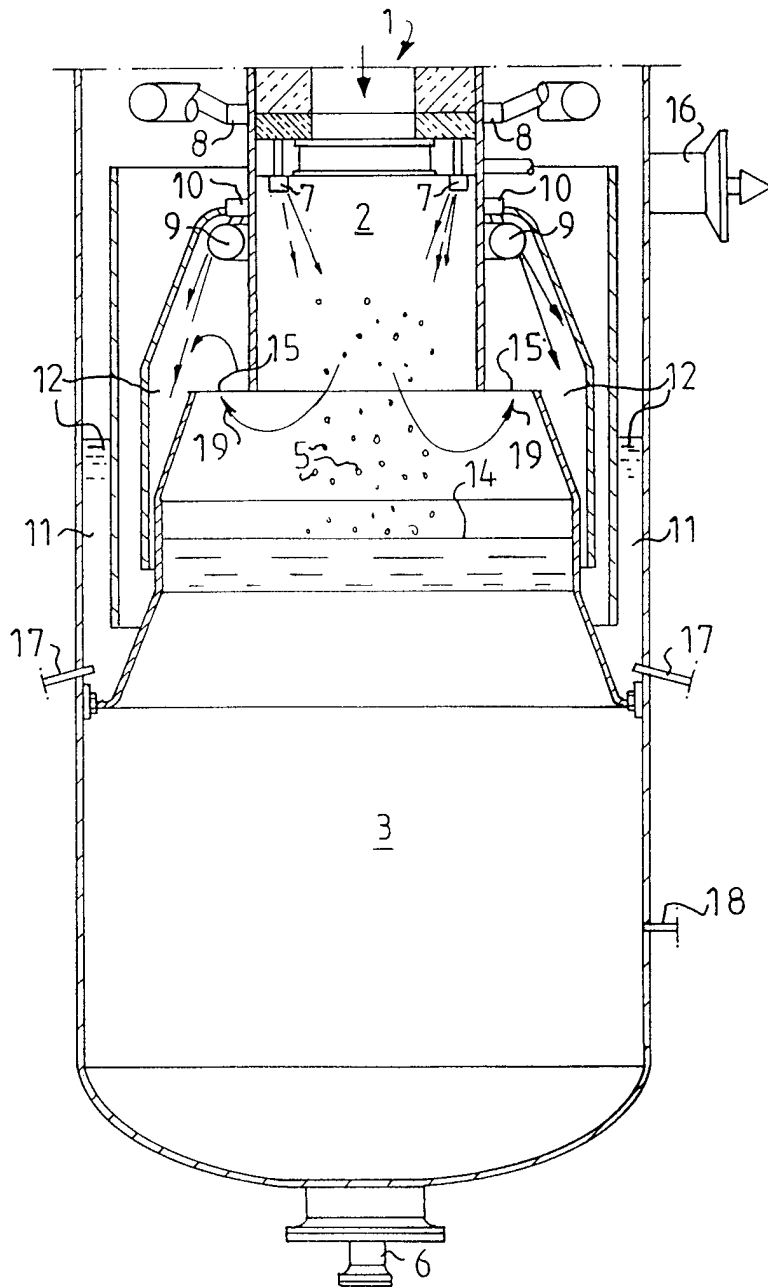


Fig. 1



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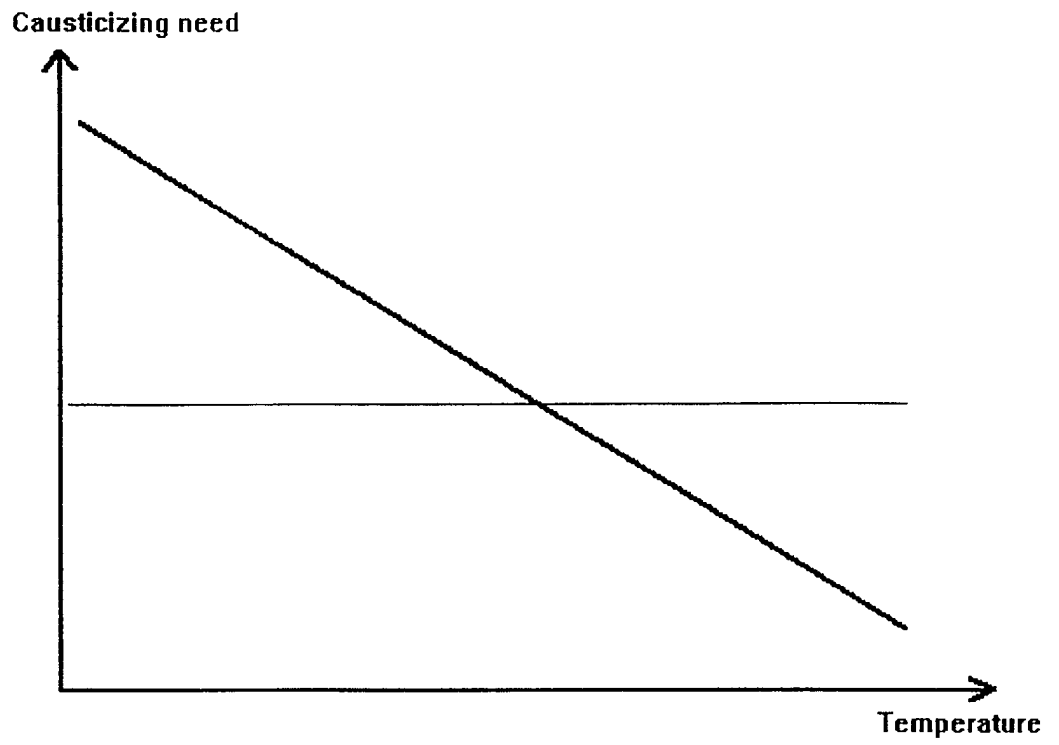


Fig. 2

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 98/00637

A. CLASSIFICATION OF SUBJECT MATTER		
<b>IPC6: D21C 11/12</b> 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)		
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<b>SE,DK,FI,NO classes as above</b>		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
<b>WPI</b>		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 9535410 A1 (KVAERNER PULPING TECHNOLOGIES AB), 28 December 1995 (28.12.95), page 3, line 19 - line 23; page 7, line 9 - line 14; page 9, line 5 - line 7, figures 1,3 --	1-17
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A	EP 459962 A1 (CHEMREC AKTIEBOLAG), 4 December 1991 (04.12.91) --	1-17
A	SE 470516 B (CHEMREC AB), 27 June 1994 (27.06.94) -- -----	1-17
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> 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 document 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		
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## INTERNATIONAL SEARCH REPORT

Information on patent family members

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International application No.

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