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Backlund

[11] **3,881,986**

[45] **May 6, 1975**

[54] **METHOD OF PRODUCING HOMOGENEOUSLY DELIGNIFIED PULP FROM FIBROUS MATERIAL, CONTAINING LIGNOCELLULOSE, IN A DIGESTER PARTIALLY FILLED WITH LIQUOR**

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[21] Appl. No.: **150,017**

Related U.S. Application Data

[63] Continuation-in-part of Ser. Nos. 56,121, July 2, 1970, abandoned, and Ser. No. 857,620, Aug. 29, 1969, abandoned, and Ser. No. 529,887, Feb. 24, 1966, abandoned.

[30] **Foreign Application Priority Data**

Feb. 25, 1965 Sweden 2468/65

[52] **U.S. Cl.** **162/19;** 162/41; 162/68

[51] **Int. Cl.** **D21c 1/02**

[58] **Field of Search** 162/39, 42, 43, 44, 46, 162/63, 68, 248, 19, 17, 41

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Assistant Examiner—Alfred D'Andrea, Jr.
Attorney, Agent, or Firm—Stevens, Davis, Miller & Mosher

[57] **ABSTRACT**

AN improved method is disclosed for impregnating lignocellulosic fibrous material in the cooking of pulp from wood chips in a digester partially filled with cooking liquor which is circulated through the digester for the purpose of obtaining a homogeneous delignification of the entire charge of chips within the digester, the improvement comprising conveying cooking liquor to the top of the digester, distributing the liquor substantially uniformly over substantially the entire cross-sectional area of the digester by means of at least one centrifugal pressure nozzle and subjecting the charge to pressure-impregnation by means of steam, the pressure being applied during the period in which the mixture is brought to cooking temperature but after the chips present at the top of the digester have been brought into contact with the cooking liquor, the circulation of the cooking liquor being stopped during the pressure-impregnation phase.

8 Claims, 8 Drawing Figures

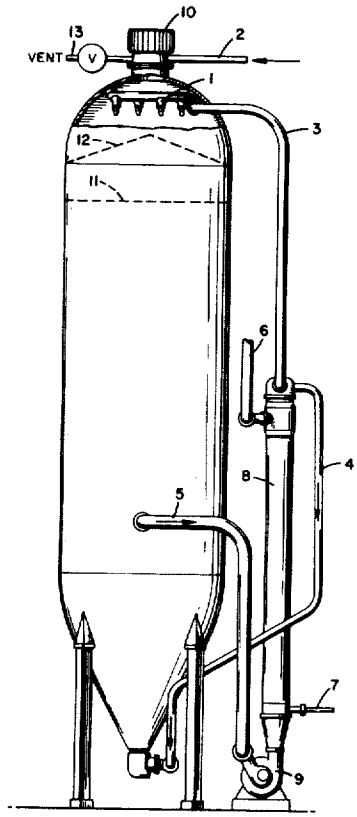


FIG. 3a

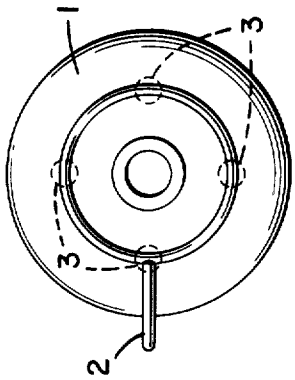


FIG. 3b

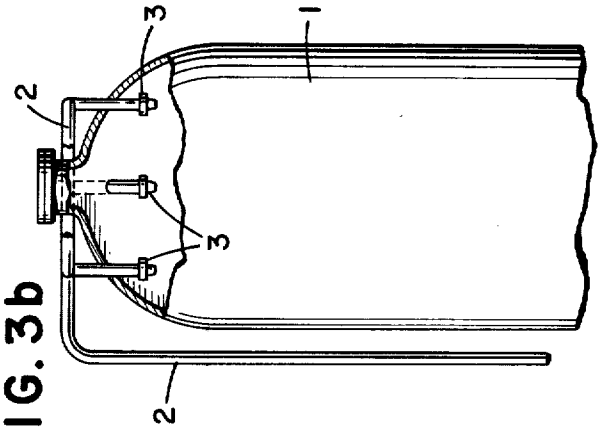


FIG. 2a

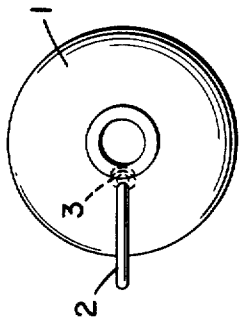


FIG. 2b

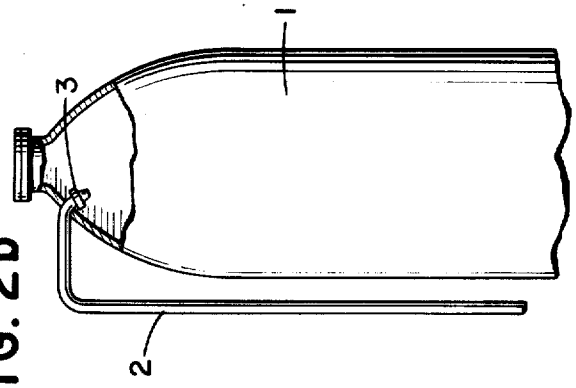


FIG. 1a

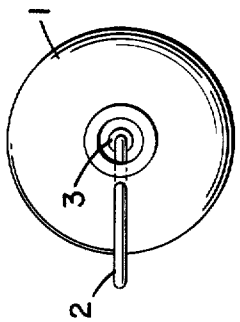
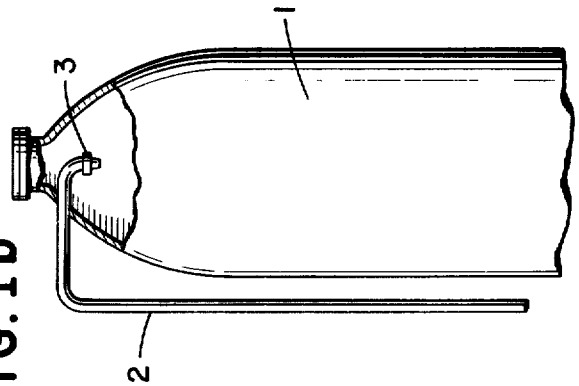


FIG. 1b



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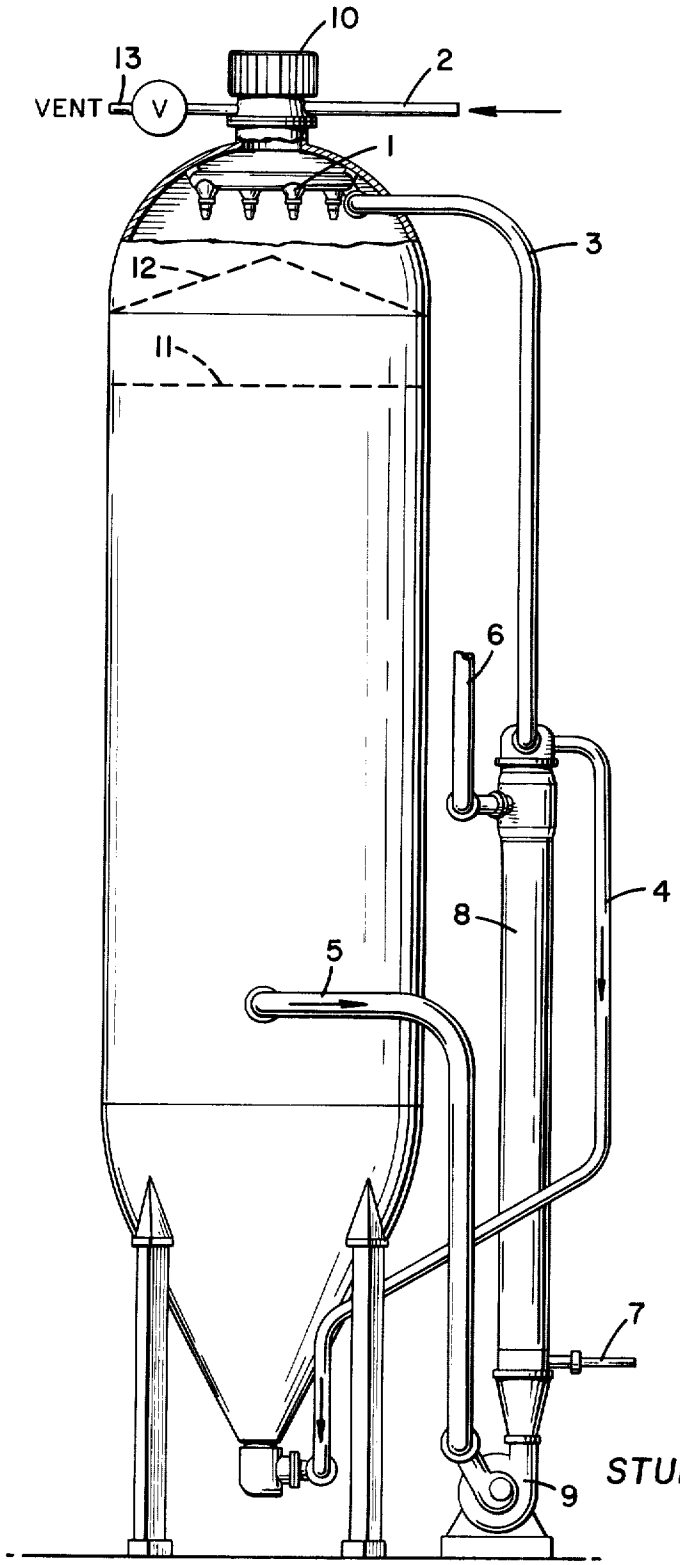


FIG. 5

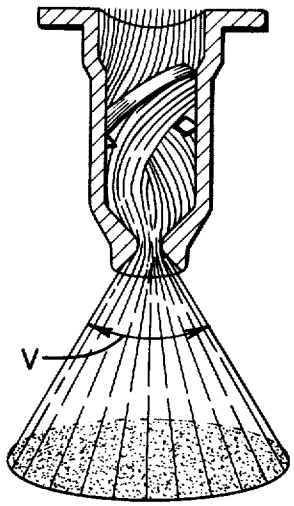


FIG. 4

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METHOD OF PRODUCING HOMOGENEOUSLY DELIGNIFIED PULP FROM FIBROUS MATERIAL, CONTAINING LIGNOCELLULOSE, IN A DIGESTER PARTIALLY FILLED WITH LIQUOR

This application is a continuation-in-part of Ser. No. 56,121, filed July 2, 1970 Ser. No. 857,620, filed Aug. 29, 1969, and Ser. No. 529,887, filed Feb. 24, 1966, all of which are now abandoned.

The manufacture of pulp comprising lignocellulosic fibrous material is frequently carried out in digesters that are not entirely filled with liquor. This method is used either during the entire cooking cycle or only during some phase or portion of the cycle. The advantages of such a cooking procedure as compared with cooking in a digester entirely filled with liquor include, among others, a reduction in the amount of heat needed to bring the contents of the digester up to the cooking temperature and to concentrate the chemicals used for the cooking, thus making it possible to use a smaller dose of chemicals per unit of production.

A comparison between the two methods of cooking mentioned above is not, however, wholly in favor of the first-mentioned method when a detailed analysis is made of how said first-mentioned method is generally carried out. Unsatisfactory distribution of the cooking liquor within the digester and the lack of pressure-impregnation of the fibrous material (chips), according to this method, result in an unevenly delignified pulp. This pulp, in turn, yields a large amount of screenings, thus increasing the risk of too high a content of shives in the screened pulp. However, the major part of the screenings comprises good fiber that is poorly delignified, and only a small part of the screenings consists of actual knots. When cooking to produce high-yield pulps, such a non-homogeneously cooked pulp is difficult to refine and hence is impure.

The object of the present invention is to eliminate the above-mentioned drawbacks and relates to a method of obtaining a uniform cooking of the entire change of chips and, respectively, thoroughly cooked individual chips by providing for a good distribution of the cooking liquor throughout the digester and/or a pressure-impregnation carried out in a special manner.

There are various previously-known devices for distributing cooking liquor throughout the digester in connection with the cooking of pulp. For example, Swedish Pat. No. 71,512 discloses the provision of dispersion cones or dispersion plates in front of the discharge pipe; while Swedish Pat. No. 74,480 shows that large perforated plates have been used as distributors. However, experience has shown that these known devices do not meet the demands which are made with regard to good distribution of cooking liquor within a pulp digester to guarantee a satisfactory degree of delignification. Primarily, this is due to the fact that the distribution efficiency of the devices is too low or to the fact that the devices are easily clogged by chips, small particles of wood, or by the pulp itself.

According to this invention, it is possible to improve the distribution of the cooking liquor throughout a digester, or an equivalent closed chamber of treatment, by using one or more centrifugal pressure nozzles for introducing into the digester that part of the cooking liquor which is to circulate within the digester and/or is injected at the top of the digester. Such a nozzle is of such a design and construction and is so connected to

the interior of the digester, that the cooking liquor, when discharged from the nozzle, will be effectively distributed over the entire cross-sectional area of the digester, or over at least a major portion thereof. Good distribution of the cooking liquor accomplished in this manner leads to a uniform temperature and to a distribution of chemicals throughout the entire digester.

Of the accompanying drawings,

FIGS. 1*a* and 1*b*, 2*a* and 2*b*, 3*a* and 3*b* illustrate diagrammatically some examples of how the centrifugal pressure nozzles can be arranged in the digester in accordance with this invention. The figures designated *a* show top views of the various arrangements; while the figures designated *b* show side views of the corresponding arrangements with a portion of the top of the digester cut away.

FIG. 4 shows a longitudinal section taken through a centrifugal pressure nozzle of known design and construction that is suitable for use in connection with this invention and which per se is similar, for example, to that shown in U.S. Pat. No. 2,305,210.

FIG. 5 shows still another digester arrangement described more fully hereinafter.

In FIGS. 1*a*–3*b*, numeral 1 designates digesters arranged for discontinuous or continuous cooking of pulp of liqnocellulosic fibrous material, particularly wood chips; numeral 2 designating circulation or injection lines for the cooking liquor; and 3 designating centrifugal pressure nozzles. In FIG. 4, *v* is the angle of spread for cooking liquor at the centrifugal nozzle, marked with double arrows. This angle should preferably exceed 50° so that the liquor will be spread over a sufficiently large area.

For spraying the cooking liquor by means of a centrifugal spray nozzle, a certain amount of energy is required to develop the spraying pressure required for each particular application of the nozzle. In order to save pumping energy, part of the cooking liquor sent to the top of the digester can be led through centrifugal pressure nozzles while the rest of the cooking liquor can be introduced into the digester by other means. Cooking liquor circulation, carried out as described above, results in uniform cooking in respect of the various portions of the charge of chips.

In itself, this results in a considerable improvement; but if the desired pulp is to be defined as being a homogeneously cooked pulp, it is necessary that uniform delignification be obtained in respect of each individual chip in the charge. To obtain such a homogeneously cooked pulp, some form of pressure-impregnation is, however, required in addition, particularly if the chips include oversize pieces and/or if the chips possess a relatively high dry content.

A common method of pressure-impregnating the charge of chips in connection with discontinuous cooking involves pumping the chip-filled digester full of cooking liquor, whereupon the pump itself is caused to develop the pressure in the digester. As previously pointed out, however, several difficulties are involved in the process of cooking with large quantities of liquor.

According to this invention, pressure-impregnation is carried out in a digester, partially filled with liquor, by introducing gas and/or steam into the digester or by heating steam, already present within the digester, to a temperature sufficiently high to provide a pressure which is above the pressure level created by the aver-

age temperature prevailing in the digester at any given moment.

When pressure-impregnation is accomplished by means of a gas, air is preferred; and when impregnating with vapor, steam is usually used. Before applying the pressure in connection with discontinuous cooking, the digester is first filled with chips, and the desired amount of cooking liquor added, and the circulation of the cooking liquor then started.

Pressure-impregnation preferably should be performed at an early stage of the cooking process while the cooking liquor still contains a high percentage of active chemicals. In order that the pressure-impregnation shall yield a fully satisfactory result, the cooking liquor is brought into contact with the entire charge of chips before the impregnation is started. When steam—which condenses very easily—is used, it is of advantage to interrupt the circulation of the cooking-liquor while the actual pressure-impregnation is going on so that the required pressure can be developed with reasonable amounts of steam. In certain cases, it may be advantageous to repeat the pressure-impregnation several times within one and the same cooking cycle.

In carrying out continuous cooking in a digester partially filled with cooking liquor, the pressure-impregnation, in accordance with this invention, can be carried out by temporarily raising the pressure above the operating pressure of the digester. This temporary increase in pressure is repeated at intervals of 0^{00}–4^{00} hours, suitably 0^{10}–2^{00} hours, and preferably, 0^{15}–0^{30} hour.

When centrifugal pressure nozzles are used in connection with the cooking of pulp, the required heat may be supplied to the contents of the digester by injecting direct steam into the vapor phase; i.e., above the liquor level in the digester. In that case, a pressure pulsation can be obtained within the digester by temporarily varying the steam supply or the amount of circulating and/or injected liquor. This pressure pulsation enables the cooking liquor to penetrate the chips better.

According to this invention, another way of utilizing the vapor phase for generating pressure consists in increasing the pressure in the vapor phase by heating. This can be accomplished in the following manner: The majority of modern pulp digesters is heated indirectly, in which case part of the circulated cooking liquor is conveyed to the top of the digester via a heat exchanger. By temporarily interrupting the circulation of cooking liquor while maintaining the supply of steam to the heat exchanger, generation of steam can be obtained from the heat exchanger to the top of the digester; and hence, also a desired increase of pressure.

In connection with the pressure-impregnation in accordance with the present invention, the pressure is increased by 0.1–10, suitably 1–6, and preferably 2–4 kg/cm^2; and the duration of the impregnation is fixed to 0^{00}–6^{00} hours, suitably 0^{10}–1^{00} hour, and preferably 0^{05}–0^{30} hour.

The table lists some results of the application of this invention in connection with discontinuous cooking in a 20-liter vertical laboratory digester with external forced circulation of the cooking liquor via a heat exchanger, using steam as the heating medium. The cooks were carried out in accordance with the sulphate method with 160 kg of active Na_2O/ton of moisture-free wood and with a wood: liquor ratio of 1 : 3.5. Cooking cycle: 80°–169°C for 3 hours; varying periods at 169°C. The initial material used was pine chips in the condition intended for factory cooking. The cooks were blown from the digesters; and after washing, they were screened in flat screens with slots of 0.20 mm width.

Sulphate Method Cooking in a 20-liter Vertical Laboratory Digester with Varied Cooking Liquor Distribution and Impregnation

Distribution of Cooking Liquor	Pressure Impregnation	Chlorine Number	Yield % Screened	Shive	Total
Without centrifugal pressure nozzle for cooking liquor	None	5.4	45.4	2.6	48.0
		6.5	47.0	1.9	48.9
With centrifugal pressure nozzle	None	5.4	46.9	0.8	47.7
		6.6	47.9	0.8	48.7
"	Steam 2 kg/cm^2	5.8	47.5	0.4	47.9
	Time 0^{05} hour	7.3	49.0	0.4	49.4
"	Air 7 kg/cm^2	5.8	47.9	0.2	48.1
	Time 3^{00} hours	6.9	48.8	0.2	49.0

It is evident from the table that the improved distribution of the cooking liquor according to the present invention had a significant effect even with as little a distribution area as used in this example; it is also evident that in case of unsatisfactory distribution the amount of shives did vary widely from one cook to another. When cooking was carried out with centrifugal pressure nozzle connected, the shive content dropped to a more stable level. When both distribution and pressure-impregnation were used, the shives dropped further to a very low level. It is important to note that even with a chlorine number as high as about 7, the amount of shives still was very low.

Additional examples with reference to FIG. 5 of the accompanying drawings are as follows:

EXAMPLE 1

Pressure-Impregnating with Steam

Chips and cooking liquor are charged to the digester (FIG. 5) while the digester lid **10** is open. The volume of the digester is 140 m^3. Wood, white liquor, and black liquor are charged to the digester in quantities whereby the liquid contained by the wood is in the ratio of 3.5

: 1 and whereby the charge VA (NaOH) is 220 kg/ton. The expression "VA" refers to the amount of active alkaline chemicals employed for bringing the lignin into solution; i.e., the amount of NaOH and Na_2S. The circulation pump **9** is started when the liquid level rises above the suction pipe **5**. Liquid is then sucked from the digester through the suction pipe **5** and passes the pump **9**, from which it passes through the heat exchanger **8** up through the top circulation pipe **3** and out through four centrifugal pressure nozzles **1** of the type designated as spraying system Cv 6Hf 450. As a result of the liquid circulation, packing of the chips in the digester is facilitated, the filling and packing process being continued for 30 minutes. The liquid circulation pipe **4** to the bottom of the digester is closed while the digester is being filled with chips. The circulation pump has a capacity of 16 m^3/min at 15 *m* water-column; i.e., the pressure difference between the outlet and the inlet of the pump **9**.

When the digester is filled with chips and cooking liquor, the digester lid **10** is closed. For the purpose of pressure-impregnating the chips, the circulation pump **9** is stopped and steam is sprayed in through a conduit leading to the top of the digester above the liquid level **11**, so that the pressure in the digester rises to 5 kg/cm² in about 2 minutes. The pressure is held at this level for 10 minutes, after which the supply of steam is interrupted and the circulation pump **9** started. The pressure then falls practically instantaneously to roughly 0 kg/cm². The pressure-impregnation process is now completed, and heating of digester begins. The cooking liquor circulating through the heat exchanger **8** is heated indirectly by passing steam to the heat exchanger through a conduit **6**, condensate being drained off at **7**.

The cooking liquor is then heated to a temperature in the range of 80° to 170°C for 45 minutes. During this heating period, approximately 70 percent of the circulating liquid passes to the top of the digester via tube **3** and the remainder to the bottom of the digester through conduit **4**. The liquid circulating to the top of the digester is distributed by means of four centrifugal pressure nozzles **1** uniformly over the cross-sectional area of the digester, and thus, also over the chips **12** which lie above the liquid level **11**.

The digester is then allowed to stand at a maximum temperature of 170°C for 80 minutes, after which the digester pressure is lowered from 7 to 5.5 kg/cm² for 10 minutes by venting at **13**. The digester is then emptied by blowing the blow tank in the usual fashion.

Blowing time = 30 minutes.

EXAMPLE 2

Pressure-Impregnating with Air

Chips and cooking liquor are charged to the digester (FIG. **5**) in quantities whereby the ratio of liquid to wood is 3.5 : 1, and the charge of VA (NaOH) is 220 kg/ton. The digester is packed with chips in the same manner as described above in Example 1. After fully charging the digester, the digester lid **10** is closed. At the same time heating of the digester begins, compressed air is passed into the digester through tube **2** for pressure-impregnating purposes. The puressure is then raised to 4 kg/cm² over a period of 2 minutes by supplying compressed air to the digester. Circulation of the liquid is constantly taking place and heating at the same time. That is, liquid withdrawn through the pipe **5** is pumped through the heat exchanger **8**, of which liquid, 70 percent is passed to the top circulation pipe **3** and passes out through four centrifugal pressure nozzles **1**, while 30 percent of the liquid is passed to the bottom of the digester via the circulation pipe **4**. The liquid is distributed via the centrifugal pressure nozzles **1** uniformly over the chips **12** above the liquid level **11**.

The applied pressure of 4 kg/cm² is maintained while the digester is being heated at 80°–170°C for 45 minutes, and venting takes place when the pressure exceeds 7 kg/cm² through the vent tube **13**.

The digester is held at 170°C for 80 minutes, after which the pressure is lowered from 7 to 5.5 kg/cm² for 10 minutes by venting the digester. The digester is emptied by blowing the blow tank in the usual way.

Blowing time = 30 minutes.

In comparison with the results obtained when cooking by conventional methods in digesters that are not entirely filled with liquor, cooking by the method according to the present invention provides, inter alia, the following advantages:

1. Higher yield of screened pulp because of the lower amount of screenings obtained.
2. Lower shive content in the screened pulp.
3. Higher point of defibratability; i.e., the possibility of cooking the pulp to a higher chlorine number with an acceptable amount of screenings.
4. Possibility of cooking more rapidly.
5. Possibility of reducing the amount of liquor in the digester.
6. Obtaining a more easily refined product in the cooking of high-yield pulp, and a resulting final product of higher quality; or, the possibility of increasing the chlorine number and thus also the yields of pulp while substantially maintaining the quality of the product.

The applications of the present invention are, of course, not limited to cooking in vertical digesters, as specifically illustrated and described, but the invention may also be used in other types of digesters, such as horizontal digesters.

What is claimed is:

1. In a method of impregnating lignocellulose fibrous material in the cooking of pulp from wood chips in a digester, the improvement which comprises

1. introducing wood chips and a cooking liquor into a digester with the volume of cooking liquor being less than the volume of the digester whereby there is a vapor space above the liquor;
2. circulating liquor from the bottom of the digester to the top thereof and through at least one centrifugal nozzle thereby spraying liquor substantially uniformly over the chips throughout substantially the entire cross-sectional area of the digester;
3. stopping circulation of liquor; and
4. thereafter introducing steam into the space above the liquor and chips while the digester is closed thereby elevating the pressure in the digester above atmospheric pressure while simultaneously heating the chips and liquor to cooking temperature thereby impregnating liquor wet chips with liquor.

2. A method according to claim **1**, characterized in that the pressure-impregnation is carried out at the beginning of the cooking cycle.

3. A method according to claim **1**, characterized in that only part of the required cooking liquor is conveyed to the top of the digester to the charge of chips

through at least one centrifugal pressure nozzle, and separately introducing the rest of the cooking liquor to the charge of chips.

4. A method according to claim 3, characterized in that the cooking liquor supplied to the charge of chips through a centrifugal pressure nozzle is caused to leave the nozzle in a spray having an angle of spread of at least 50°.

5. A method according to claim 1, characterized in that the pressure-impregnation is carried out a plurality of times during the cooking cycle.

6. A method according to claim 1, characterized in that vapor present within the digester is heated up to such a high temperature as will generate a pressure exceeding the pressure produced by the prevailing average temperature of the contents of the digester.

7. A method according to claim 1, characterized in that the pressure-impregnation is carried out at an increase of presssure amounting to 0.1 – 10 kg/cm^2, and that the impregnation is caused to take place for a period of time of up to 6 hours.

8. A method according to claim 1, characterized in that the pressure-impregnation is carried out at an increase of pressure amounting to 1–6 kg/cm^2, and that the impregnation is caused to take place for a period of time of about 10 minutes to 1 hour.

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