APPLICATION FOR A STANDARD PATENT

Tag Pulp Industries, S.A.
Kirchstrasse 10, Postfach 130, FL9490 Vaduz, LIECHTENSTEIN

hereby applies for the grant of a standard patent for an invention entitled:

DIGESTION METHOD AND INSTALLATION WITH PREHEATING OF LIGNOCELLULOSE MATERIALS IN SOLID PHASE

which is described in the accompanying complete specification.

Details of basic application(s):
90 05701 FRANCE FR 7 May 1990

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DATED this TWENTY NINTH day of APRIL 1991

PHILLIPS ORMONDE & FITZPATRICK
Attorneys for:
Tag Pulp Industries, S.A.

By:

Our Ref: 213002
POF Code: 1164/80648
DECLARATION FOR A PATENT APPLICATION

In support of the (a) Convention application made by
Société Anonyme TAG PULP INDUSTRIES
(hereinafter called "applicant(s)" for a patent (c) for an invention entitled (d)
DIGESTION METHOD AND INSTALLATION WITH PREHEATING OF LIGNOCELLULOSE MATERIALS IN SOLID PHASE

1. [e] JEAN GALICHON, Administrator of
Société Anonyme TAG PULP INDUSTRIES
Kirchstrasse 10 Postfach 1130 FL9490 VADUZ Liechtenstein
do solemnly and sincerely declare as follows:

1. I am/we are authorized to make this declaration on behalf of the applicant(s).
2. [f] Nivelleau de La Brunière Patrick Galichon Jean Philippe
45 bd de Montmorency 16 avenue Hoche
75116 PARIS 75008 PARIS
France France

I swear the actual inventor(s) of the invention and the facts upon which the applicant(s) is/are entitled to make the application are as follows:

Applicant is the assignee of the invention from the actual inventors

(Note: Paragraphs 3 and 4 apply only to Convention applications)

3. The basic application(s) for patent or similar protection on which the application is based is/are identified by country, filing date, and basic applicant(s) as follows:

France n° 90 05701 07/05/90
Société Anonyme TAG PULP INDUSTRIES

4. The basic application(s) referred to in paragraph 3 hereof was/were the first application(s) made in a Convention country in respect of the invention the subject of the application.

To: The Commissioner of Patents
Jean Galichon Administrateur
Délegué

[Signature]

PHILIPS ORMONDE & FITZPATRICK
Patent and Trade Mark Attorneys
367 Collins Street
Melbourne, Australia
1. A method for the pulping of raw lignocellulose material which comprises the steps of:

   impregnating said lignocellulose material with a solution of hydroxides or salts of alkalki or alkaline-earth metals;

   adjusting the weight of the retained impregnating liquid, so that the ratio of the weight of said retained impregnation liquid to the dry weight of said lignocellulose material does not exceed 2;

   subjecting the thus impregnated material in a pre-cooking step to live steam under a pressure equal to or greater than that present in the subsequent cooking step; and

   cooking the material consisting of the resulting solid mass in an exclusively externally heated closed vessel without exerting any mechanical disintegrating action on the mass.
4. An installation apparatus when used for the carrying out of the method according to any of Claims 1 to 3, comprising an externally heated cylinder provided with a central screw with blades or vanes, wherein the installation apparatus includes a device for the feeding of the cylinder with impregnated material which comprises an air lock connected to a live steam conduit.
DIGESTION METHOD AND INSTALLATION WITH PREHEATING OF LIGNOCELLULOSIC MATERIALS IN SOLID PHASE

Our Ref: 213002
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The following statement is a full description of this invention, including the best method of performing it known to applicant(s):
The present invention relates to a method and installation for the digestion of a pasty or semi-solid mass of lignocellulose materials impregnated with solutions of hydroxides or salts of alkaline or alkali earth metals.

Methods of preparing of cellulose pulp by treatment of lignocellulose materials in which the digestion is effected in solid phase are already known, for instance from French Patent A-2 542 021.

The method described in said patent consists in effecting the digestion within a cylinder which is heated exclusively from the outside, without direct addition of steam, of a pasty mass of cellulose materials impregnated with alkaline or alkali-earth bases the content of which in the impregnation solution used is adjusted to a relatively low value, and the content of hydroxides or salts of which, absorbed during the impregnation, is relatively high. The prior impregnation is carried out, in particular, under conditions such that the ratio of the impregnation liquid retained (I.L) and of the dry substance (D.S.) is equal to or less than 2.

Under such conditions, the material which is subjected to the digestion is almost solid and the digestion presents difficulties, in particular due to the non-uniform distribution of the heat given up by the wall of the furnace. It has already been proposed, in particular in accordance with French Patent Application 80 15085, to effect better
control of the temperature during the course of the digestion and to reduce to an utmost the variations in temperature within the treated mass by controlling the conditions of filling and the rate of advance of the mass in an externally heated horizontal cylinder provided with a central screw with blades or vanes. The consumption of heating power being related to a large extent to the water content of the impregnated lignocellulose materials, it is advantageous to decrease the I.L/D.S. ratio as much as possible, for instance by operating with a ratio of 0.7 to 1.3.

It is noted, however, that below 1.3 problems of heat exchange with the wall of the digester arise very sharply, which has the effect of increasing the digestion time.

Now it has surprisingly been found that one can overcome this difficulty and even operate with lignocellulose materials impregnated with an I.L/D.S. ratio of less than 1 up to values of 0.7 to 0.8, by subjecting the impregnated materials to the action of live steam.

The method of the present invention is characterized by subjecting the impregnated materials, before introduction into the digester, to the treatment with live steam under a pressure equal to or greater than that present in the digester so that a part of the steam by giving off its sensible heat to the impregnated material and condensing assures the preheating and penetration of the impregnation liquor up to the center of the material.
Accordingly the present invention provides a method for the pulping of raw lignocellulose material which comprises the steps of:

impregnating said lignocellulose material with a solution of hydroxides or salts of alkali or alkaline-earth metals;

adjusting the weight of the retained impregnating liquid, so that the ratio of the weight of said retained impregnation liquid to the dry weight of said lignocellulose material does not exceed 2;

subjecting the thus impregnated material in a pre-cooking step to live steam under a pressure equal to or greater than that present in the subsequent cooking step; and

cooking the material consisting of the resulting solid mass in an exclusively externally heated closed vessel without exerting any mechanical disintegrating action on the mass.

The present invention further provides an installation apparatus when used for the carrying out of the method described above according to any of Claims 1 to 7, comprising an externally heated cylinder provided with a central screw with blades or vanes, wherein the installation apparatus includes a device for the feeding of the cylinder with impregnated material which comprises an air lock connected to a live steam conduit.

The method of the present invention is characterized by subjecting the impregnated materials, before introduction into the digester, to the treatment with live steam under a pressure equal to or greater than that present in the digester so that a part of the steam by giving off its sensible heat to the impregnated material and condensing assures the preheating and penetration of the impregnation liquor up to the center of the material.
Such a preheating also results in a slight increase in the I.L./D.S. ratio, since a part of the steam condenses.

Impregnated materials whose I.L./D.S. ratio may vary between 0.7 and 1.3 are preferably subjected to the action of the live steam.

The steam pressure may vary between 5 and 10 kg/cm².

Thus, as the impregnated material, as a result of the action of the live steam, experiences an increase in temperature of about 100°C, there is an increase in the I.L/D.S. ratio in the following proportions.

For an initial I.L./D.S. value of 0.7, this value increases after the application of the live steam to 0.92.

For an initial I.L./D.S. value of 0.8, said ratio is 1.04 after the steam treatment.

This manner of operation makes it possible considerably to reduce the time of digestion, that is to say, by one-half or more of the time necessary without prior action of the steam.

Another object of the invention is an installation for the carrying out of the above method comprising an externally heated cylinder provided with a central screw with blades or vanes, which is characterized by the fact that the device for feeding the cylinder with impregnated material comprises a chamber lock connected to a live steam conduit.

Other features of the invention will become evident from the description of the examples of application of the
invention given with reference to a digestion installation shown in the accompanying drawing.

The digester of the invention comprises a cylinder with double jacket 1 traversed by hot oil, within the axis of which cylinder there is a horizontal screw 2 the inclined blades 3 of which are separated by non-inclined blades 4. The rotation of the screw is effected by a motor 5.

The entrance and discharge of materials are located at the opposite ends of the cylinder, each having a chamber lock, 6 and 7, provided with push pistons. The entrance chamber lock 6, at the openings of which the valves 8 and 9 are located, is connected via a conduit provided with a valve 10 to a source of live steam. The same conduit can also be connected to the open air via a valve 11 for the evacuation of the steam which has been used.

Example 1

500 kg of bagasse of 65% dryness are impregnated with 17 kg of powdered anhydrous Na₂CO₃ and 40 kg of powdered caustic soda.

Onto the vigorously agitated mass there are sprayed 300 ml of water at 90 or 95°C and the agitation is maintained for about 10 minutes. A fibrous homogeneous mass which is easy to manipulate and is of a straw-yellow color is obtained in which the I.L./D.S. is 0.8.

The impregnated bagasse, which weighs 587 kg, is separated into two parts.
The first part is treated in the digester without preheating by means of steam with a digestion time of 20 minutes at a temperature of 165-168°C. The second part is treated with steam preheating for 30 seconds at a pressure of 7.5 kg/cm² before introduction into the digester. The digestion time is 10 minutes at a temperature of 165 to 168°C. The results were as follows.

<table>
<thead>
<tr>
<th>Time of stay</th>
<th>Temp. Steam treatment</th>
<th>Kappa Index</th>
<th>Undigested percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st lot</td>
<td>20 min. 165-168°C no</td>
<td>27</td>
<td>1.5%</td>
</tr>
<tr>
<td>2nd lot</td>
<td>10 min. 165-168°C 37 sec, 7.5 kg</td>
<td>15</td>
<td>0.3%</td>
</tr>
</tbody>
</table>

Despite a reduced period of digestion, there is noted better digestion (lower kappa) and a more uniform pulp (lower percentage of undigested matter).

Example 2

Kenaf cut to an average length of 3 cm is used. The entire plant is employed, with the exception of the leaves. The cortical fiber is not removed.

400 kg of kenaf of 84% dryness are mixed vigorously with 20 kg of powdered anhydrous sodium carbonate and 47 kg of powdered caustic soda.

While maintaining the agitation, 121 liters of water of 90 to 95°C are sprayed onto the mass and the agitation is continued for 10 minutes.
The I.L./D.S. ratio is 0.75.

As previously, the impregnated kenaf is separated into two lots. The first is introduced into the digester without prior treatment with steam with a time of digestion of 30 minutes at 165 to 168°C.

The second lot is treated for 30 seconds with steam under a pressure of 7.5 kg/cm² and then introduced into the digester. The time of digestion is 15 minutes at 165-168°C.

The results obtained are summarized in the following table.

<table>
<thead>
<tr>
<th>Time of stay</th>
<th>Temp.</th>
<th>Steam treatment</th>
<th>Kappa Index</th>
<th>Undigested percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st lot</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>30 min.</td>
<td>165-168°C</td>
<td>no</td>
<td>31</td>
<td>1.6%</td>
</tr>
<tr>
<td>2nd lot</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>15 min.</td>
<td>165-168°C</td>
<td>30 sec, 7.5 kg</td>
<td>18</td>
<td>0.2%</td>
</tr>
</tbody>
</table>

In accordance with this example, it is also noted that, despite a shorter period of digestion, the steam treatment permits better digestion and the obtaining of a more homogeneous pulp.

The invention is not limited to the embodiments shown; it is capable of variations within the scope of the person skilled in the art.
The claims defining the invention are as follows:

1. A method for the pulping of raw lignocellulose material which comprises the steps of:
   - impregnating said lignocellulose material with a solution of hydroxides or salts of alkali or alkaline-earth metals;
   - adjusting the weight of the retained impregnating liquid, so that the ratio of the weight of said retained impregnation liquid to the dry weight of said lignocellulose material does not exceed 2;
   - subjecting the thus impregnated material in a pre-cooking step to live steam under a pressure equal to or greater than that present in the subsequent cooking step; and
   - cooking the material consisting of the resulting solid mass in an exclusively externally heated closed vessel without exerting any mechanical disintegrating action on the mass.

2. A method according to claim 1, wherein the weight of the retained impregnation liquid to the dry weight of said lignocellulose material is between 0.7 and 1.3.

3. A method according to claim 1, wherein the live steam is employed at a pressure of 5-10 kg/cm².

4. An installation apparatus when used for the carrying out of the method according to any of Claims 1 to 3, comprising an externally heated cylinder provided with a central screw with blades or vanes, wherein the installation apparatus includes a device for the feeding of the cylinder with impregnated material which comprises an air lock connected to a live steam conduit.

DATED: 10 July 1992
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Dated 10 July 1992

[Signature]