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NEUTRAL TO WEAKLY ALKALINE SULFITE PROCESS FOR THE EXTRACTION OF CELLULOSE FROM CELLULOSIC MATERIAL

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The present invention generally relates to the release of cellulose fibers from solid cellulosic materials, and more particularly, to an improvement in the neutral to weakly alkaline sulfite process used for extracting cellulose from solid cellulosic material.

In the pulping of cellulosic materials in order to obtain cellulose fibers for different uses, various processes have been devised which may be generally classified into three groups. One such group may be termed alkaline digestion the most common example of which is the sulfate process. This process essentially comprises the use of a strongly alkaline solution consisting essentially of NaOH or other alkalis such as sulfides or polysulfides having a pH value greater than 12. As a result of this process the cellulose fibers have good strength properties but poor whiteness qualities. Accordingly, expensive and time consuming bleaching processes must be employed in order to obtain a proper whiteness of the cellulose fibers for use in the manufacture of high grade papers.

In order to obtain cellulose fibers having a greater whiteness, use is made of a second group of processes which comprise sulfite processes wherein the main digestive agent is SO_2 in free and/or combined form. While the cellulose fibers produced by these processes are much whiter than the cellulose fibers produced by the alkalis, the fibers are generally not as strong as the fibers produced by the alkaline processes.

Within recent years a third group of processes has gained technical importance, which processes may be generally described as the neutral sulfite digestion process. In this process the solution has an alkalinity which is so selected that at the end of the cooking process the liquor will have been rendered neutral by the acids liberated during the cooking process. Generally, this process has been previously used only for the production of so-called semichemical pulps. These are pulps containing appreciable amounts of lignin. The main advantages of this group of processes are the relatively high yields and the short reaction times of 10 to 20 minutes as compared to the 8 to 11 hours for the sulfite process, or 2½ to 8 hours of the alkaline process.

The main disadvantage of this process is the relatively poor quality of the pulp product which must invariably be mechanically fiberized in order to separate the fibers.

In the alkaline digestion process it has been proposed to reduce the cooking time by a preliminary impregnation of the cellulosic materials with the cooking liquor, sometimes supported by a steam treatment preceding the preliminary impregnation of the cellulosic materials so as to effect digestion at temperatures up to 220 to 225° C. which are above those generally used for this purpose. In comparison, in the usual sulfite process the temperature ranges from 135 to 175° C. and in the alkaline and neutural sulfite processes the temperature is increased to only about 185° C.

It is therefore a principal object of the present invention to provide an improved neutral to weakly alkaline sulfite process for the extraction of cellulose fibers from cellulosic materials.

It is another object of the present invention to provide a neutral sulfite process wherein the resulting cellulose fibers have improved strength properties or greater whiteness than was previously obtainable.

It is a further object to attain a fast digestion in order to be able to operate with small apparatus thereby obtaining a high production rate.

Generally speaking, the process of the present invention comprises the reduction of logs of wood as supplied to the mill to a consistency of fairly small chips, cooking the chips with a hot aqueous solution, commonly referred to as liquor, cooking the chips and liquor under pressure so that the pulping agent component of the liquor attacks the lignins and other organic material in the wood releasing the cellulose for washing, filtering and refining and making into paper or other uses.

It has been unexpectedly found that by a thorough preliminary impregnation of the cellulosic material with the cooking liquor and subsequently cooking the impregnated cellulosic material at greater temperatures it is possible to obtain by the neutral to weakly alkaline sulfite process cellulose fibers having improved strength properties or greater whiteness than was previously the case. Reasons for these unexpected results are not as yet fully known, but the results have been verified through numerous experiments an example of which will be described subsequently.

As a further result of the process of this invention the required time for the digestion is appreciably lower than for the conventional methods and ranges between 5 and 30 minutes with a usual period of between 8 to 10 minutes. Thus, the time required for the process of the present invention is less than that required for the production of semichemical pulp or for carrying out the alkaline digestion process at higher temperatures and with a thorough preliminary impregnation. Further, the resulting cellulose fibers are of a considerably better quality and no mechanical fiberizing is required.

The digestion liquors may comprise solutions of alkaline sulfites or bisulfites with additions such as Na_2CO_3, $NaHCO_3$ and/or preferably NaOH, NaHS, $Na_2S_2O_3$, Na_2S, NAS_2 or other polysulfides. The pH value of the digestion liquors at the beginning of the cooking process ranges between 7.0 and 11.5, preferably between 7 to 10. The digestion or cooking temperature is between 200 and 280° C., and preferably between 225 and 280° C. This temperature is generally higher than permissible in previously known and conventional processes.

The thorough preliminary impregnation of the present process can be accomplished by known methods by the use of steam or vacuum, by mechanical or hydraulic pressure, or by agitation, or by any combination of those steps.

Prior to the impregnation of the crude cellulose material with the digestion liquir the wood chips may be treated with steam or subjected to a vacuum.

In order to further reduce the digestion time, the cellulosic raw material may be subjected to an initial comminution to form wood chips having a maximum length of 25 mm., and a maximum thickness of 5 mm. A minimum length of 3 mm. and a minimum thickness of about 0.5 mm. is advisable. This will result in obtaining cellulose fibers having a greater strength.

While the present invention is essentially directed to the use of wood, other cellulosic materials are not excluded. Such other cellulosic materials having various chip sizes and comprising, for example, waste material from plants, grasses, bamboo, can be used for producing cellulose fibers of a good quality according to the present invention.

The present invention also has the advantage of making possible the production of cellulose pulp having op-

timum qualities and good whiteness even by the use of digestion liquors containing substantial quantities of $Na_2S_2O_3$. These quantities may range up to 60% of the total chemical content of the liquor. In the known sulfite processes darker grades of cellulose pulps are produced by such small quantities of $Na_2S_2O_3$ as 2%. In the process of the present invention the presence of a relatively small quantity of Na_2S and/or polysulfides does not have any detrimental effects on the produced cellulose fibers.

Because of the short cooking time required for the present invention, this process has the further advantage of a high production rate.

In order to set forth a specific embodiment of the present invention, the following example is disclosed in detail but it is not considered to be limiting of the invention but is disclosed merely for illustrative purposes.

Example

Comminuted pine wood chips having an average length of 15 mm. and an average thickness of 2 mm. were deposited in an autoclave and steamed for about 2 minutes under a pressure of 6 atmospheres. The wood was accordingly quickly heated and the entrapped air was driven out of the wood. The digestion liquor which was heated to about 250° C. was then introduced into the autoclave. This liquor had the following composition:

	G./l.
Sodium sulfite	50
Sodium sulfide	10

5 parts of this liquor were added to one part of wood chips, whereupon the resulting temperature in the autoclave was 170° C. Within 3 minutes the contents of the autoclave were heated to 250° C. and then kept at this temperature for 3 minutes. After a rapid decompression and cooling the pulp was removed from the autoclave.

The pulp, whose yield was about 49%, was then treated in order to remove therefrom splinters and other undesirable substances. The pulp was then tested for strength in accordance with the Technical Association of the Pulp and Paper Industry (Tappi) Standards T 220 m. 60, "Critical Testing of Pulp Hand Sheets." The following values were obtained with a freeness of 35° SR:

Tensile breaking length, m.	12,200
Burst factor	105
Tear strength	97
Folds, no.	5,050

It will be understood that this invention is susceptible to modifications in order to adapt it to different usages and conditions, and accordingly, it is desired to comprehend such modifications within this invention as may fall within the scope of the appended claims.

What is claimed is:

1. In the process of the neutral to weakly alkaline sulfite type for extracting cellulose fibers from wood, the steps of comminuting the wood to wood chips having an average length of about 15 mm. and an average thickness of about 2 mm.; removing air from said wood chips; thoroughly impregnating the resultant air-freed wood chips with a sulfite solution having a pH value between 7 and 11.5, said sulfite solution having a concentration of about 50 g./l. sodium sulfite and about 10 g./l. sodium sulfide, the weight ratio of the sulfite solution to the wood chips being about 5:1, respectively; and digesting the resultant impregnated wood chips at 200–280° C. for 5–30 minutes.

2. The process of claim **1**, wherein the digestion step is conducted at 225–280° C. and for a time of about 8–10 minutes.

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