United States Patent Office 3,177,110 Patented Apr. 6, 1965

3,177,110

PROCESS FOR PRETREATING WOOD FOR THE PRODUCTION OF CELLULOSE

Alfred Ogait, Aschaffenburg, Main, Germany, assignor to Aschaffenburger Zellstoffwerke Aktiengesellschaft, Aschaffenburg, Germany

No Drawing. Filed July 19, 1962, Ser. No. 211,111
Claims priority, application Germany, July 24, 1961, A 37,957, A 37,958, A 37,959
1 Claim. (Cl. 162—45)

The present invention relates to the treatment of wood, more particularly, to the method of pretreating wood with milk of lime prior to the sulfite cooking of the wood in the production of cellulose.

It is known that by pretreating wood prior to subjecting the wood to the sulfite cooking process, a higher yield of cellulose products can be obtained from which improved parchments can be produced. The wood or wood fibers or other cellulose-containing raw materials are pretreated with an aqueous alkali or alkaline earth substance. This pretreating process is carried out by adding to the wood fibers aqueous alkali or alkaline earth solutions in a quantity having several times the weight of the weight of the wood. The wood fibers are subjected to the effect of this solution for a period of several hours so as to impregnate the wood as thoroughly as possible with the alkali or alkaline earth lye.

The impregnating process can be improved by circulating the lye and subjecting the wood fibers to a pressure or vacuum treatment. The excess lye is then removed after the impregnation process has been completed. With such a process an index of saturation of 2.0 can be obtained. The index of saturation can be defined as the ratio of the amount by weight of water contained in a determined amount by weight of theoretically dry wood after the impregnation. While this known process results in a very thorough impregnation, the impregnation process itself is time-consuming.

It is therefore the principal object of the present invention to provide a novel and improved process for pretreating wood prior to the sulfite cooking process for the production of cellulose.

It is another object of the present invention to provide a process for pretreating wood prior to the sulfite cooking process wherein the pretreating process is carried out concurrently with the transportation of the wood.

It is another object of the present invention to provide a process for pretreating wood in batches prior to the sulfite cooking process for the production of cellulose.

It is an additional object of the present invention to provide a process for concurrently pretreating the wood prior to the sulfite cooking process and for storing the wood prior to the use thereof.

It has been unexpectedly discovered that the comparatively small amounts of water received by the wood fibers after an impregnating treatment of only a few minutes are sufficient to considerably improve the sulfite cooking process in the presence of lime whereby the yield is increased and the parchment properties of the wood fibers are improved. The process of the present invention essentially comprises adding milk of lime to the wood fibers and agitating the wood fibers with the quantity of the milk of lime being such that after the treatment, one weight unit of the theoretically dry wood contains—including the natural water contents of the wood—not more than one weight unit of water. The impregnated wood is then left for a period of several hours under the influence of the lime.

The milk of lime should contain an amount of CaO ranging from 0.5 to 5% of the theoretically dry wood with the contents preferably being 1 to 2%. The milk

of lime can be replaced by solutions containing equivalent amounts of alkaline substances or ammonia.

The pretreating process can be carried out by flowing wood chips in an aqueous suspension of calcium hydroxide as the wood chips are being transported to either a storage container or the container for the sulfite cooking process.

The impregnation of the wood chips can also be carried out in stages or by a batch process.

Also, the pretreating process can be carried out simultaneously with the storage of the wood in the form of chips.

In order that the unexpected results of the present invention may be fully appreciated, reference is made to a specific test wherein chips of fir wood as conventionally used for producing cellulose and having a water content of 10% were dumped into a rotary cylindrical drum. 0.9 part of milk of lime was then added to the wood fibers. After various periods of impregnation the following indices of saturation were observed.

Period of time in minutes	Index of saturation
0.5	0.60
1	0.65
1.5	0.70
2.5	0.75
6	0.80
15	0.86
60	1.00

As will be apparent from the above table an index of saturation of 0.65 is obtained after only one minute and an index of saturation of 0.8 is obtained after 6 minutes. This relatively small degree of impregnation is adequate to produce an increased yield of cellulose and a cellulose having greatly improved parchment properties after the subsequent sulfite cooking process.

Not only can this pretreating process be carried out continuously in a rotating drum to which wood chips and milk of lime have been added, but the pretreating process can be carried out simultaneously with the transportation of the wood chips. Wood chips have been transported by mixing them with water and flowing the mixture of water and wood chips through pipes. This transportation is advantageous since the wood is less susceptible to abrasion than if the wood chips were transported, by example, compressed air. The present invention therefore proposes to transport the wood chips by dumping them into an aqueous suspension of calcium hydroxide and then flowing the mixture of wood chips and the solution through pipes to either the storage container or the sulfite cooker. During the flow of the wood chips through the pipelines, the wood will absorb sufficient milk of lime in order to obtain the results during its subseqeunt storing as discussed above. When the wood chips have reached either the storage container or the cooker, they may be dehydrated by straining and then left untouched for a period ranging from 2 to 15 hours. Sufficient time is then available for the lye to penetrate into the interior of the wood chips and to complete the chemical reaction with the hemicellulose of the wood in order to obtain the desired increase in yield. Upon completion of this pretreating process, the sulfite cooking step can then be carried out in a known manner.

While the foregoing pretreating process is a continuous one, this impregnation of the wood can also be carried out in batches or semi-continuously. The wood chips are dumped into a wooden storage bin and simultaneously therewith a corresponding quantity of alkaline solution is sprayed in the storage bin against the wood chips. The alkaline solution which is not absorbed by the wood chips during this initial spraying then descends to the bottom of the storage bin from where it is pumped and

recirculated through a sprayer against the wood chips. In this process a wood, such as pine wood chips, absorb such amounts of the alkaline solution that the impregnated wood contains about one part by weight of water per one part by weight of theoretically dry wood.

This process results in a uniform impregnation of the wood and where milk of lime is used as the alkaline solution, a uniform distribtuion of the undissolved lime will result upon the surface of the wood chips. This lime which firmly adheres to the surfaces of the wood thus has an opportunity of dissolving and of becoming diffused throughout the wood chips so as to replace the dissolved calcium hydroxide which has been used up.

If the wood chips are subjected for a sufficiently long period of time to the action of the alkaline solution in either a continuous or a batch process, the wood chips, particularly if caustic soda is used, may absorb somewhat more water with the alkaline solutions than one part by weight per one part by weight of dry wood. It has now been discovered that this greater impregnation is advantageous since the yield of the subsequent sulfite cooking is increased more rapidly even though the storage period of the impregnated wood chips is the same. The storage period can even be considerably shortened and a maximum yield will still be obtained. As a result, the impregnation of the wood according to the present invention, can be increased to such an extent that the wood contains *more* than one part by weight of water per one part by weight of theoretically dry wood.

Another advantage of the present invention is that the acid used for the sulfite cooking is diluted to a far lesser extent than with previously known pretreating processes.

The known impregnating processes use an excess amount of milk of lime and subject the wood chips to the influence of the milk of lime for a period of time of at least 1 hour with the liquid being agitated and the wood and liquid being under pressure. During such an impregnating process the wood chips absorb a quantity of water which is about twice as great as their theoretical dry weight. Since during the subsequent sulfite cooking process the weight ratio of wood and liquid is in the order of 1:4.5, the cooker filled with the impregnated wood chips can receive only 2.5 times the amount of weight of the weight of the wood, which is only 55% by weight of the normal amount of acid. Accordingly, the acid is diluted by about 45%. Since the SO_2 content of the acid used for the sulfite cooking process is at least 4 to 5%, it is then necessary to supply 7.5 to 9% of sulfur dioxide. This is a great disadvantage since the use of more than 7% of sulfur dioxide makes it necessary to supply liquid SO_2 which considerably increases the cost of the process.

The process carried out according to the present invention, however, results in an index of saturation of, for example, 0.8, so that for one unit weight of wood there can be supplied to the cooker 3.7 parts by weight of acid. The acid is thus diluted by only 20%. For this reason it is sufficient to supply only 4 to 5% of sulfur dioxide which is much more advantageous than the necessity of supplying 7.5 to 9% of SO_2 as required by the previously known processes. It is relatively easy to obtain acid with 5 to 6% sulfur dioxide at a normal lime content of about 1% CaO and it is thus unnecessary to supply liquid sulfur dioxide.

Comparable advantageous results are obtained if in place of the milk of lime equivalent amounts of an alkaline substance, such as sodium hydroxide or ammonia, are added to the wood chips and act upon the wood chips for at least one hour and preferably several hours.

In the present invention the wood chips are subjected to the action of the milk of lime for a considerable period of time which is at least one hour and preferably ranges from 5 to 15 hours. It has been discovered and it will become readily apparent from several of the examples contained herein that the cellulose yield after the sulfite cooking subsequent to the impregnation, when carried out according to the present invention, increases considerably in direct proportion to the increase of the period of time in which the wood chips are under the action of the milk of lime.

A further result of the present invention relates to the storing of the wood subsequently to be used in the production of cellulose. Under the present practice wood is cut up into chips and then stored as against the previous practice of cutting the wood, storing it until needed, and then chopping the wood into chips. By passing the wood chips through a bath of milk of lime in operations as previously described, the wood chips may then be stored in the open air for long periods of time without being attacked by microorganisms or fungi. This period of time of storage ranges up to several months. Even though during this storage period the calcium hydroxide is being transformed into calcium carbonate, a relatively high pH-value remains which protects the wood from putrefactive bacteria, fungi, and other agents which are destructive of wood. Even after a long storage period the cooking of the wood with sulfite still produced the higher cellulose yields described above. The preservation of the wood during storage can be increased by adding caustic acid, magnesium oxide or magnesium dioxide.

The following operating examples are given to facilitate the comprehension of the present invention and are for illustrative purposes only and are not to be considered as limiting in any way.

Example I

Pine wood is chopped into chips and then sorted in chips of various sizes. The sorted chips are then passed through a mixing drum to which milk of lime containing 30 g. of CaO/l. is added. After a thorough mixing during which the wood chips absorb sufficient quantities of milk of lime, the wood is dumped onto a conveyer or similar transportation installation to deliver the wood chips to the storage area. The wood chips are then piled in heaps ranging up to about 10 m. in height and are stored in the open air for a period of several months during which no putrefaction phenomena can be observed.

When the wood chips are subsequently needed for producing cellulose, the wood chips are mixed with water and the resulting mixture pumped through pipelines to a point immediately prior to the sulfite cookers at which point the water is removed from the mixture. The transportation of the wood chips in this manner has the additional advantage that the wood is washed so as to remove any dirt which may have accumulated thereon during the storage period. On the other hand, the increase in the production of cellulose obtained by the treatment with milk of lime, is not influenced if the calcium salts are dissolved out of the wood during the transportation of the wood chips through the water. The removal of the calcium salts can even produce certain advantages in the subsequent process when the wood chips are subjected to a sulfite cooking.

Example II

Chips of fir wood having a water content of 10% are dumped into a rotating cylindrical vessel. A milk of lime containing 12.5 g. of CaO per liter and 0.7 part by weight—with reference to the theoretically dry weight of the wood—is supplied to the wood chips. After 15 minutes the milk is completely absorbed by the wood chips which now have an index of saturation of 0.8 and contain 1.0% CaO.

The impregnated wood chips remain under the action of the lime at room temperature for a period of time ranging from 0 to 16 hours. The length of this period of time will produce different cellulose yields as indicated in the table at the end of this example. The impregnated wood is then treated with a cooking acid in a known manner containing 6.3% SO_2 and 1.1% CaO, at

a maximum temperature of 127° C. and a pressure of 5 atm. absolute.

The following results are obtained for various periods of time for the action of the lime on the wood chips:

Duration of action of lime	Degree of decomposition (Johnsen-Noll-number)	Cellulose yield in percent of wood	Additional yield with reference to untreated wood in percent of weight of pulp
Untreated	45	53.7	
1 hour	46	55.1	2.6
3 hours	46.5	56.0	4.3
6 hours	46	56.2	4.7
9 hours	47	56.4	5.0
12 hours	47	56.7	5.6
16 hours	49	56.8	5.8

Example III

Example II is repeated but with the milk of lime containing 25 g. CaO per liter and the impregnated wood containing 2.0% CaO. The sulfite cooking is carried out with an acid containing 5.5% SO_2 and 1.1% CaO for 9½ hours at a temperature of 129° C. and a pressure of 5 atm. absolute.

The following results are obtained for different periods of time during which the lime acts upon the wood chips;

Duration of action of lime	Degree of decomposition (Johnsen-Noll-number)	Cellulose yield in percent of wood	Additional yield with reference to untreated wood in percent of weight of pulp
Untreated	61	54.6	
1 hour	65	57.1	4.6
3 hours	66	57.9	6.0
6 hours	66	58.4	6.9
9 hours	61	58.6	7.3
12 hours	63	58.7	7.5
16 hours	63	59.0	8.1

It will be apparent from the foregoing table that the lime must act upon the wood chips for 10 to 12 hours in order to get the maximum increase in the cellulose yield. The increase in the yield of cellulose also depends on the amount of lime absorbed by the wood chips; for example, where 1% CaO is contained in the wood chips, the yield increase is 5–6% whereas a 2% CaO content is in the wood chips, the yield increase is from 7–8%.

Example IV

In order to illustrate the effects of increases in the CaO content, Example II is repeated with the exception that the wood is treated with a milk of lime containing 37.5 g. CaO per liter. After the impregnation the wood contains 3.1% CaO. The wood is then stored for 12 hours during which time the lime acts upon the wood chips. The wood chips are then digested with an acid containing 6% SO_2 and 0.84% CaO for 9½ hours at a temperature of 126° C. under a pressure of 5 atm. absolute. The yield is 65.7% of the weight of the wood and a cellulose is obtained with a decomposition degree (Johnsen-Noll-Number) 73.

Carrying out the sulfite cooking with untreated wood, the yield is only 58.8% and the Johnsen-Noll-Number is only 70. The pretreatment with 3% CaO thus results in a yield increase of 12% of the cellulose weight.

The cellulose obtained after a pretreatment according to the present invention, and particularly after the pretreatment described in Examples II to IV, will also have greatly increased parchment properties. It has been discovered that a particular relationship exists between the increase of the yield and the improvement of the parchment property. Thus, where the cellulose yield has

increased as a result of the pretreating process of the present invention, the parchment properties will also improve with this improvement being much more pronounced as the increase in yield becomes greater.

Example V

Example II is repeated but with the milk of lime being replaced 0.7 part by weight of sodium hydroxide containing 35 g. sodium hydroxide per liter. The wood has an index of saturation of 0.8 and after the impregnation contains 0.8% of sodium hydroxide. After the sulfite cooking according to Example II, the sodium hydroxide acts upon the wood for 12 hours. The degree of decomposition (Johnsen-Noll-Number) thus obtained is 62 and the cellulose yield is 58.5% of the wood. In a treatment without impregnation the yield is only 53.5%.

The additional yield with reference to untreated wood in percent of the weight of the pulp is 9.0.

Example VI

Example II is repeated but with the milk of lime being replaced by 0.7 part by weight of an ammonia solution containing 15 g. of ammonia per liter. The index of saturation of the wood is 0.8 and after the impregnation the wood contains 1.2% ammonia (NH_3). The ammonia then acts upon the wood chips for 12 hours after which the sulfite cooking is carried out according to Example II. The cellulose yield is 58.0% of the wood and the Johnsen-Noll-Number is 50.

After a similar processing without the impregnation treatment, the cellulose yield is only 53.1% and the Johnsen-Noll-Number is 48.

The additional yield with reference to untreated wood in percent of the weight of the pulp is 9.0.

It will be understood that this invention is susceptible to modification 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 claim.

I claim:

In a process for the production of cellulose from wood comprising first pretreating the wood by impregnation with an alkaline solution and then subjecting said pretreated wood to a sulfite digestion, the steps of cutting the wood into chips and forming a mixture of the wood chips with an aqueous suspension of an alkaline solution, flowing the mixture of wood chips and alkaline solution through a pipeline to a sulfite digester so that the wood becomes impregnated with the alkaline solution, discontinuing the impregnation when up to an amount by weight of the alkaline solution is absorbed in the wood so that one part by weight of the theoretically dry wood contains at the end of the impregnation not more than one part by weight of water including the natural water content of the wood, straining excess alkaline solution from the mixture and regenerating the removed solution with a corresponding amount of alkaline solution, recirculating the regenerated solution through the pipeline together with the wood chips, and storing the impregnated wood for at least one hour prior to the sulfite digestion.

References Cited by the Examiner

UNITED STATES PATENTS

1,885,764	11/32	Richardson	162—86
2,041,745	5/36	Dreyfus	162—86
2,610,119	9/52	Magnuson	162—17
2,799,579	7/57	Messing	162—237
2,803,540	8/57	Durant	162—237

FOREIGN PATENTS

526,399	9/40	Great Britain.

DONALL H. SYLVESTER, *Primary Examiner.*

MORRIS O. WOLK, *Examiner.*