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3,013,933 METHOD FOR PREPARATION OF WOOD CELLULOSE

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This invention relates to the manufacture of high-alpha cellulose pulp from wood and has for its object the provision of an improved method for manufacturing high-alpha cellulose pulp from wood in a presteaming and alkaline digestion procedure. More particularly, the invention is advantageously concerned with improvements in the method of my U.S. Patent 2,583,994, issued January 29, 1952.

I have now made the significant discovery that, notwithstanding the importance of air removal from the digester as described in my patent, gases generated during pretreatment accumulate in those parts of the digester where condensation is most rapid and that their removal effects an improvement in the control of the operation.

I have found also that, regardless of the completeness of air removal during the presteaming temperature rise period, the temperature at points farthest removed from the steam inlet tends to fall below that of the main chip mass even though digester pressure is maintained. This condition results in less complete pretreatment of those chips lying within this lower temperature zone, and pulp from this part of the digester is correspondingly lower in alpha content, higher in pentosans content, and less suitable for the product of viscose tire cord, cellulose acetate, and other products where chemical purity is important. The admixture of this quantity of pulp, however small, detracts from the quality of the entire production.

Such materials as carbon dioxide, formic acid, and acetic acid are formed in the presteaming and my invention provides an effective means for their removal with a consequent diminution in the amount of alkaline chemicals expressed as Na_2O required to obtain equivalent pulping in the alkaline stage.

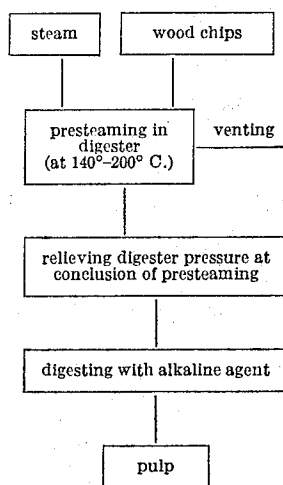
The improvements of the present invention are accomplished, in general, by controlled relief of gases throughout the steam pretreatment and relieving the digester to low pressure at the conclusion of the steam pretreatment, prior to pumping in the alkaline liquor. The quantity of steam which must be relieved during the pretreatment depends on the design of the equipment but in any case should be adequate to effect removal of the volatile products of the steam pretreatment from the digester at a rate to prevent accumulations of these materials. Maintenance of normal and uniform temperatures can be taken as adequate evidence of sufficient relief. Normally where steam is injected at one end of the digester, the gases are vented from the other, and the desired temperature uniformly can be realized by relieving not over about 2,000 pounds of steam per ton of pulp during the pretreatment temperature rise and time at maximum temperature periods.

It is well known that turpentine and furfural can be recovered from wood by a suitable steam treatment. In accordance with my invention however, I may effect a recovery of these important chemicals while relieving the digester for the maintenance of uniform temperature and reducing the pressure at the conclusion of presteaming. Steam thus relieved together with the steam vented during the relief at the end of the pretreatment can be condensed to recover turpentine and furfural, as well as other condensed volatile materials.

An alternative procedure which effectively maintains uniform temperatures during the steam pretreatment step

comprises circulating the gaseous contents of the digester during the presteaming operation through the digester, as by a blower equipped with external duct work, to prevent local accumulation of the gaseous products of the pretreatment and consequent low temperature zones. In order to accomplish the other objects of the invention by this procedure, the digester must be blown down to low pressure at the conclusion of the presteaming operation and the relieved vapors are advantageously condensed.

A simplified flow diagram of the process is as follows:



In practicing the invention, wood chips are charged into the digester and the digester is steamed directly, preferably with saturated steam. Although not an essential part of this improved method, it is desirable to displace air during the early stages of the pretreatment. Usually this is accomplished by steaming the digester from the bottom and relieving at the top or vice versa. Alternatively, one may resort to evacuation or water displacement. Regardless of what, if any, steps to remove air are taken, when the digester has been steamed to about 100° C. and steam issues from the vent, the relief line is throttled but not completely closed and the digester temperature is raised by continued direct steaming to a temperature between 140° and 200° C., and preferably between 160° and 180° C., in a total time of roughly, 20 to 90 minutes. During the time at which the digester is at superatmospheric pressure, certain amounts of steam relief are permitted. This steam is condensed if such products as turpentine and furfural are to be recovered. Relief may be practiced in short bursts at frequent intervals or at a slower continuous rate. In either case the amount of steam relieved is at least sufficient to maintain good temperature uniformly throughout the digester. Actually the amount of relief may economically be greater than this minimum quantity in order to effect recovery of greater amounts of steam distillable products, such as turpentine and furfural.

Normally the pressures corresponding to the aforementioned steaming temperature (140° to 200° C.) will be from about 55 to 235 pounds absolute per sq. in.

As stated in my said patent, it is desirable to drain any condensate from the digester as rapidly as it forms in order to provide as uniform pretreatment conditions as possible.

At the conclusion of the steam pretreatment, the digester is preferably relieved through a condenser to permit recovery of additional quantities of such volatile materials as turpentine and furfural, and furthermore to release residual quantities of such acidic materials as acetic acid, formic acid and CO_2 , in order to minimize the quan-

tity of alkaline chemicals expressed as Na₂O required in the alkaline stage to follow.

At the conclusion of the steaming treatment and after relieving the steam pressure if such step is to be practiced, an alkaline cooking liquor is pumped into the digester containing the presteamed chips and the cook proceeds in accordance with the conditions set forth in my patent and are comparable to those used in normal single stage alkaline pulping operations. The alkaline cooking liquor may be a kraft or sulfate liquor containing principally sodium sulfide (Na₂S) and sodium hydroxide (NaOH) with lesser amounts of sodium carbonate (Na₂CO₃) and sodium sulfate (Na₂SO₄), or a soda liquor in which the principal alkaline ingredient is sodium hydroxide with lesser amounts of sodium carbonate, or a modification or combination of those liquors with or without additional agents such as sodium sulfite (Na₂SO₃), etc. Generally the ratio of total Na₂O:dry wood in strongly alkaline liquors is between 0.14 and 0.30. The digester is brought up to a temperature of normally 160° to 180° C. in, roughly, one to two hours and held at this temperature for one to five hours, and the cook is blown and washed. Usually the liquid charged to the digester includes a certain amount of organic solids derived from earlier cooks, it being common practice to recycle from spent or black liquor solids for reasons of steam economy. The following examples illustrate practices of the invention:

Example 1

The following experiment illustrates the improvement in uniformity of quality of the cellulose from top to bottom of the digester to be gained by practicing this improved procedure. Two cooks were made on southern pine chips under conditions identical except in regard to relief practice during steam pretreatment. Direct steam was introduced into the digester at the bottom with the top relief valve open. Steaming rate was such that steam was observed coming from the relief line in about 15 minutes. The relief valve was closed in the one cook but left slightly open in the other. Steaming was continued at such rate as to reach 175° C. in 45 minutes. The observed gauge pressure to maintain this temperature (measured near middle of the digester) was about 120 pounds per sq. in. in the cook made with the relief valve slightly open (conditions of this invention), and 125-136 pounds per sq. in. in the one not relieved. The steam relieved from the first cook during the time to and at temperature was condensed and amounted to about 1800 pounds of steam per ton of pulp produced.

In each cook after steaming for 45 minutes at maximum temperature, the steam inlet valve was closed and the digester was relieved to atmospheric pressure. A kraft liquor was pumped in and the digestion completed under the following alkaline stage conditions:

Total liquid:wood ratio.....	4.4
AA:wood ratio.....	0.18
Sulfidity	percent.. 25
Maximum temperature.....	° C.. 170
Time to maximum temperature.....	hr.. 1.5
Time at maximum temperature.....	hr.. 2.5

At the completion of the alkaline cook, portions of unbleached pulp from the top of each digester charge were analyzed. The data illustrate the improvement in uniformity obtainable by the practice of relieving during the steam pretreatment in accordance with the present invention.

	Pulp Produced by Invention	Pulp Produced by U.S. Pat. 2,583,994
Alpha cellulose, percent.....	95.8	95.3
Hot 10% KOH Solubility, percent.....	4.1	5.0
Pentosans, percent.....	1.8	2.3

Example 2

This example illustrates the production of turpentine and furfural from southern pine chips by practicing the method of the invention. Southern pine chips were charged to the pilot scale digester. The digester was steamed slowly from the bottom with the relief line open at the top. When steam was observed issuing from the relief line, 15 minutes after the start, the relief valve was throttled but not closed, and the steaming was continued to 175° C. maximum temperature. After 0.75 hour at this temperature, the digester was relieved to atmospheric pressure. The vapors relieved during the steam pretreatment and during relief following pretreatment were condensed and separated into aqueous and nonaqueous layers. The total amount of steam relieved during the temperature rise and time at maximum temperature period amounted to about 1800 pounds per ton of pulp. The yield of crude turpentine (nonaqueous layer) amounted to 2.0 gals. per B.D. ton of pulp. The furfural content of the aqueous layer determined by the barbituric acid precipitation method average 20 pounds per B.D. ton of pulp.

Example 3

This example illustrates the more effective use of alkaline stage chemicals which results when digester pressure is relieved at the end of the steam pretreatment in accordance with the preferred method of this invention. Hemlock chips were presteamed for 0.75 hour at 175° C. with vapor bleed-off. At the conclusion of the pretreatment, the digester pressure was reduced to atmospheric pressure and sufficient 40% sulfidity kraft liquor was pumped in to give an AA:wood ratio of 0.20 (as Na₂O). During the alkaline stage, the digester was steamed to 170° C. in 1.5 hours and held at this temperature for 2.5 hours.

A second cook was made under identical conditions except that at the conclusion of the steam pretreatment the digester pressure was not relieved, but instead, the alkaline liquor was pumped in under pressure.

Failure to blow down the digester pressure prior to starting the alkaline cook resulted in less thorough alkaline digestion as is evident from the following data:

	With Blowdown	Without Blowdown
Yield screened pulp, percent.....	37.2	38.5
Screenings, percent.....	0.27	0.43
TAPPI Permanganate No.....	16.3	23.7

In carrying out my improved presteaming operation, the aforementioned advantageous results are achieved when removing from 500 pounds to 2000 pounds of steam on a basis of 2000 pounds of oven dry, sometimes called B.D. or bone dry, pulp. Especially desirable results are accomplished in the way of temperature uniformity and chemical recovery when removing from 1000 to about 1800 pounds of steam from the digester per 2000 pounds of oven dry pulp.

It will be seen from the foregoing examples that the method of the invention improves the uniformity of treatment throughout the digester when using the steam pretreatment alkaline cooking process; furthermore, that the method permits recovery of valuable by-products, such as furfural and turpentine, and that it effects more efficient use of alkali in the alkaline stage.

The alternative procedure of circulating the digester gases throughout the steam pretreatment is an effective means of accomplishing the temperature uniformity desired. This alternative would require a costly system of high pressure ducts and blowers. However, if practiced with final blowdown prior to alkaline cooking, it too will permit recovery of turpentine and furfural and

will reduce the alkali requirement during alkaline cooking.

I claim:

1. In the production of pulp of high-alpha low pentosan content from wood chips in which the wood chips are subjected to presteaming followed by digestion with an alkaline cooking liquor, the improvement which comprises subjecting the chips to the direct action of the steam at a temperature of from 160° to 200° C., venting from the digester substantially continuously during the presteaming from 500 to 2000 pounds of steam per ton of bone dry chips, thereby maintaining uniform temperature conditions during the presteaming throughout the digester and removing such volatile gases as form, and at the conclusion of the presteaming relieving the pressure in the digester to substantially atmospheric pressure to remove such acids as were released during presteaming, thereby reducing the amount of chemicals required in the alkaline digestion that follows.

2. In the process of claim 1, relieving the pressure at the conclusion of presteaming by blowdown to atmospheric pressure.

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