

UNITED STATES PATENT OFFICE.

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OF BERLIN, NEW HAMPSHIRE, A CORPORATION OF MAINE.

PROCESS OF MAKING WOOD PULP.

No Drawing.

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To all whom it may concern:

Be it known that I, GEORGE A. RICHTER, a citizen of the United States, residing at Berlin, in the county of Coos and State of New Hampshire, have invented new and useful Improvements in Processes of Making Wood Pulp, of which the following is a specification.

This invention has relation to the liberation of cellulose pulp from raw material and comprises the recovery of valuable constituents contained in the waste liquor resulting from the digestion of the raw cellulosic material, and the use thereof in the preparation of a cooking acid liquor for the acid digestion of raw cellulosic material. The invention comprises also the treatment of unbleached pulp with recovered constituents of the waste liquor to produce a product having a high proportion of resistant or alpha cellulose.

In accordance with the present process an acid liquor comprising a solution of sodium sulphite or sodium sulphate or both is employed for digesting the raw material such as spruce chips. After the cooking operation, which takes place under proper conditions of time, temperature and pressure, has been completed, the digester contents are blown or discharged into a blowpit, and the acid liquor is separated from the pulp.

The acid liquor is neutralized with a sufficient quantity of alkali, caustic soda or sodium carbonate, so that it will not injure the instrumentalities with which it comes in contact. It is then evaporated and concentrated (preferably, though not necessarily, by multiple-effect evaporators) to about the consistency of treacle, and is preferably subjected to heat and external pressure sufficient to generate an explosive force therein before it is delivered to a smelting furnace, wherein the combustible content is consumed, and the sodium compounds smelted in a reducing atmosphere. The smelted compounds, comprising sodium sulphide, sodium carbonate and some unreduced sodium sulphate, are received in a water bath, and provide an alkaline liquor.

The alkaline liquor, resulting from the previous operation consisting essentially of a solution of sodium carbonate and sodium sulphide, may be converted to an acid cooking liquor by the use of sulphur dioxide. But since the direct treatment thereof with

the sulphur dioxide gas would result in the precipitation of sulphur, because of the contained sodium sulphide, the liquor is subjected to treatment to convert the sodium sulphide component to sodium carbonate. This is preferably accomplished by passing CO_2 through the liquor, or by causing the liquor and the gas to pass in countercurrent flow through a tower containing inert interstitial material such as spiral brick or its equivalent.

The liquor, resulting from the treatment of the dissolving tank liquor by carbon dioxide, is now converted to an acid sulphite liquor by causing SO_2 to react therewith. This is preferably done by causing the liquor and SO_2 to pass in countercurrent flow through one or more towers provided with inert surface or interstitial material such as spiral brick, until a cooking acid is produced having the desired percentages of free and combined SO_2 .

From the time the smelted sodium compounds are received in the dissolving tank liquor (i. e. the water into which the molten sodium compounds are delivered from the smelting furnace and are dissolved) until the liquor is acidified by the employment of SO_2 , the liquor is alkaline and thus is employed for the digestion of unbleached pulp to produce a product having a very large percentage of resistant or alpha cellulose, the selective reaction of the alkali with the ligneous and resinous components and the less stable celluloses of the pulp as to form various organic sodium compounds such as sodium oxalate, acetate, or saccharates or other soluble organic compounds.

Consequently either before the sodium sulphide of the alkaline dissolving tank liquor (containing the sodium compounds recovered from the waste acid liquor resulting from the digestion of the raw chips) is converted to sodium carbonate, or after the liquor is treated with CO_2 , more or less is withdrawn, and is employed with the unbleached pulp,—such as that resulting from the acid digestion,—to produce a product containing the desired high proportion of resistant or alpha cellulose. The waste alkaline liquor, resulting from the alkaline digestion of the unbleached pulp, is now returned, either for treatment by CO_2 in case it had not undergone the carbonation, or else directly to the towers for treatment

with SO_2 in the event that the liquor had been subjected to carbonation for the conversion of sodium sulphide.

In case the acid produced by passing SO_2 into the combined alkaline liquors is turbid or contains some organic matter resulting from the precipitation of unstable cellulose which was originally dissolved from the alkaline-treated pulp, such organic matter may be removed by filtering, or by settling, thus clarifying the acid liquor.

Any additional appropriate raw materials to make up losses in the cycle may be added at any point to the cycle. Thus sodium sulphate may be added to the neutralized acid liquor after it has been concentrated and before it is delivered to the smelting furnace, or sodium carbonate or caustic soda may be added to the alkaline liquor before or after the digestion of the unbleached pulp.

The cyclic operation herein described is economical. The sodium compounds in the acid cooking liquor by which the raw wood chips are digested are recovered in an alkaline liquor in which the pulp resulting from the acid digestion is again digested under proper conditions of time, temperature and pressure to produce a substantially alpha cellulose pulp which is available for nitration purposes. The sodium compounds contained in the waste liquor from the alkaline digestion and those in the dissolving tank liquor not employed in the alkaline digestion are reemployed in an acid cooking liquor for the digestion of a fresh batch of raw wood chips.

By using the alkaline dissolving tank liquor for the alkaline treatment of the unbleached cellulose pulp, there is a saving of heat in the alkaline digestion, for this liquor either before or after carbonation is hot, and consequently less steam is required.

As indicating generally the concentration and strength of the sodium compound solutions employed both in the acid digestion of the raw material, and the alkaline digestion of the resulting unbleached pulp, the following may be cited:—

The acid used for the initial digestion of the raw cellulosic material may test from 1% to 2% combined SO_2 and from 3% to 6% free SO_2 . The alkaline liquor for treating the pulp to produce a high alpha cellulose stock may contain from 3% to 8% alkali, figured as NaOH and based on the pulp treated. Any sodium sulphide or sodium carbonate present will be in amounts equivalent to the percentages noted. For example, for each 100 pounds of pulp treated there may be present in the alkaline digestive liquor from 3 to 8 pounds of NaOH or its equivalent as sulphide or carbonate.

It will be understood, of course, that, be-

fore the cellulose pulp resulting from the acid liquor digestion is subjected to the alkaline liquor digestion, it is washed and otherwise subjected to any suitable treatment including in some cases its bleaching.

I have stated that the smelting of the sodium content of the spent acid cooking liquor is in a reducing atmosphere. This is preferable though not essential, as an oxidizing atmosphere could be maintained in the smelting furnace. But, by reason of reducing conditions, the alkaline dissolving tank liquor contains notable proportions of sodium sulphide which has a more powerful reaction than sodium carbonate on the less resistant components of the cellulose pulp. If oxidizing conditions are maintained in the retort, the losses in sodium compounds in the cycle are made up by the addition of caustic soda to the alkaline dissolving tank liquor before it is delivered to the digester for the alkaline digestion of the cellulose pulp.

What I claim is:—

1. A process comprising the following steps:—digesting raw cellulosic material in an acid sodium-compound cooking liquor; separating, neutralizing and concentrating the spent acid liquor, and smelting and recovering in an alkaline solution the sodium components thereof; carbonating the alkaline liquor for the conversion of certain sodium compounds thereof to sodium carbonate; and acidifying such alkaline liquor with sulphur dioxide to produce an acid cooking liquor.

2. A process comprising the following steps:—digesting raw cellulosic material in an acid sodium-compound cooking liquor; separating, neutralizing and concentrating the spent acid liquor, and smelting and recovering in an alkaline solution the sodium components thereof; digesting the pulp resulting from the first herein-mentioned step in such alkaline liquor; and acidifying the spent alkaline liquor with SO_2 to produce an acid cooking liquor for the repetition of the first-mentioned step.

3. A process comprising the following steps:—digesting raw cellulosic material in an acid sodium-compound cooking liquor; separating, neutralizing and concentrating the spent acid liquor, and smelting and recovering in an alkaline solution the sodium components thereof; carbonating the alkaline liquor for the conversion of certain sodium compounds thereof to sodium carbonate; digesting the pulp resulting from the first herein-mentioned step in such alkaline liquor; and acidifying the spent alkaline liquor with SO_2 to produce an acid cooking liquor for the repetition of the first-mentioned step.

4. A process comprising the following steps:—digesting raw cellulosic material in

an acid sodium-compound cooking liquor; separating, neutralizing and concentrating the spent acid liquor, and smelting and recovering in an alkaline solution the sodium components thereof; carbonating the alkaline liquor for the conversion of certain sodium compounds thereof to sodium carbonate; acidifying such alkaline liquor with sulphur dioxide to produce an acid cooking liquor, and clarifying the said liquor.

5 A process comprising the following steps:—digesting raw cellulosic material in an acid sodium-compound cooking liquor;

separating, neutralizing and concentrating the spent acid liquor, and smelting and recovering in an alkaline solution the sodium components thereof; digesting the pulp resulting from the first herein-mentioned step in such alkaline liquor; acidifying the spent alkaline liquor with SO_2 to produce an acid cooking liquor for the repetition of the first-mentioned step, and clarifying such acid liquor.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.