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PROCESS OF CHEMICAL FIBER LIBERATION

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This invention relates to the production of so-called chemical pulps, that is, cellulose pulps which have been produced by cooking raw cellulosic material in chemical liquors until the fibers have been isolated from the cementitious components associated with the raw material.

There are various chemical liquors of acid and alkaline type which may be used to effect liberation of fiber from raw cellulosic material. The usual practice is to cook the raw material in the chemical liquor at elevated temperature and under pressure, until fiber liberation has been completed. In order that the cooking operation be completed in a practical or reasonably short period of time, it has been found necessary to use liquors containing chemicals of comparatively high reactivity and in substantial amount. For example, the acid sulphite process requires the use of a sulphurous acid solution of a sulphite such as calcium magnesium or sodium sulphite, the function of the free sulphurous acid apparently being to hydrolyze the cementitious content of the raw material so that it may be reacted upon and dissolved by the sulphite. The alkaline processes, viz., the soda or kraft process, require the use of alkaline liquors containing caustic soda, a soda liquor containing substantially only caustic soda, and a kraft liquor containing not only caustic soda but also the milder sodium sulphide as a fiber-liberating agent. In all these processes, not only does the liquor react upon and dissolve the cementitious content of the raw material, but also to some extent the cellulose fibers being isolated therefrom, the degree to which reaction upon and degradation of the cellulose fibers occur depending upon the liquor employed and the conditions under which fiber liberation is effected. Thus, a kraft cook, being milder than a soda or acid-sulphite cook, results in pulps of comparatively high strength and tear resistance; and the higher the sulphidity, up to, say, about 60% to 70%, of the kraft liquor, the better is the quality of the resulting pulp. In the usual kraft process, where the inorganic content of the spent cooking liquor is smelted under reducing conditions, a substantial amount of sodium carbonate, as well as sodium sulphide, is present in the smelt. The sodium carbonate, being inefficient as a fiber-liberating chemical, is converted to caustic soda for the preparation of the fresh cooking liquor, so that kraft cooking, as previously stated, takes place in a liquor containing caustic soda and sodium sulphide.

The object of the present invention is to provide a process of chemical fiber liberation which may be applied in the production of a high grade pulp of even better quality than kraft pulp, which results in a high yield of such pulp, and which comprises an economical cycle of steps. This object, generally stated, is realized by cooking the raw cellulosic material in successive mild chemical liquors of different types, each of which is capable of reacting upon and dissolving cementitious components from the raw material. More specifically, raw cellulosic material such as chipped wood is first cooked under atmospheric or super-atmospheric pressure in a neutral or slightly alkaline solution of sodium sulphite to effect a removal of a portion of the lignin and other cementitious components from the wood without completing fiber liberation, whereupon the wood is then cooked in an alkaline liquor preferably of lower alkalinity but of higher sulphidity than the usual alkaline liquors until fiber liberation is completed. Such a two-step fiber-liberating process may be practised economically, as the spent liquors resulting from both cooking steps may be mixed, concentrated, and their inorganic content smelted under reducing conditions to produce a smelt consisting of sodium carbonate and sodium sulphide. The sodium carbonate, being less soluble than the sodium sulphide, may be removed from a solution of the smelt by selective crystallization, whereupon it may be used for the preparation of fresh cooking liquor for the first cooking step, and the remainder may be used for the preparation of fresh cooking liquor for the second cooking step. That is to say, the crystallized sodium carbonate may be treated with sulphur dioxide and water to form a

sodium sulphite liquor, a comparatively small amount, if desired, being independently causticized and the caustic soda being used with the sodium sulphite to produce a sulphite cooking liquor of comparatively low alkalinity. Or all of the sodium carbonate may be converted to sodium sulphite, and fresh caustic soda may be added to the sodium sulphite to produce a somewhat alkaline liquor and compensate for sodium losses suffered in the cycle. The mother liquor, containing largely sodium sulphide, as previously stated, may be used for carrying out the second cooking step, residual sodium carbonate present therein preferably being converted to caustic soda. One of the important advantages of a process such as described is that a saving in lime over the usual alkaline cooking processes is made possible, as most of the sodium carbonate present in the smelt, rather than being causticized with lime, is converted into sodium sulphite for carrying out the first cooking step.

The process of the present invention may be best understood by giving a specific example of procedure such as the following. Chipped wood, e. g., spruce, may be cooked in a liquor containing from about 2% to 10% sodium sulphite for about three hours at about 300° F., and under about 67 pounds pressure. If desired, the liquor may contain a comparatively small amount of alkali such as caustic soda, sodium carbonate, or sodium sulphide. At the end of this cooking operation, the chips are somewhat darker than originally, but they cannot be reduced to a pulp even when subjected to the action of a kollergang, showing that fiber liberation is incomplete. That a substantial portion of ligneous and other cementitious material has been removed from the chips is evidenced by the distinctly brown color acquired by the spent cooking liquor. The presence of caustic soda in the cooking liquor is advantageous when it is desired to shorten the time of cooking, but the caustic soda should be insufficient to cause fiber liberation to go to completion. Under the conditions given, fiber liberation cannot go to completion, as cooking in sodium sulphite liquor does not result in pulp unless continued for a period of six to eight hours and unless a comparatively large amount of caustic soda is present in the liquor. If cooking is practised under atmospheric pressure, it is desirable that the liquor contain a high content of sodium sulphite, but if carried out under super-atmospheric pressure, the sodium sulphite content may be lower and the time of cooking may be less than with a corresponding sodium sulphite content under atmospheric pressure.

The cooked chips, after washing if desired, may then be cooked in an alkaline liquor containing caustic soda and sodium sulphide and of suitable sulphidity. For example, they may be cooked in a liquor containing one pound of active alkali per cubic foot, calculated as Na_2O, and having a sulphidity of 40%. Cooking may be carried out at a temperature of about 335° F. and under a pressure of about 110 pounds for about two to four hours, at the end of which time pulp amounting to 55% by weight, based on chips, and having approximately the following characteristics, may be obtained:

Strength (Mullen)	205
Tear resistance	294
Pentosan content	8%
Alpha cellulose content	92%

The strength and tear values of the product and its alpha cellulose content are higher than the corresponding values of a pulp obtained by the usual kraft process. Because of its excellent physical and chemical characteristics, it may advantageously be refined to high alpha cellulose content and pure whiteness by a series of steps which include treatment with chlorine water, alkaline cooking, and finally bleaching.

It is to be noted that the second cooking step is carried out in an alkaline liquor of comparatively low alkali content. If raw wood chips were cooked in such a liquor, under the conditions given, complete fiber liberation would not take place, but inasmuch as a portion of the ligneous and other cementitious components has been removed from the chips by the sodium sulphite liquor, it is possible to produce a high grade pulp from such chips by cooking in such alkaline liquor.

The spent liquors resulting from both cooking steps may be mixed, then concentrated as in multiple-effect evaporators, and their organic content burned and their inorganic content smelted in a reducing atmosphere to produce a smelt of sodium carbonate and sodium sulphide. The hot smelt may be dissolved in water and the resulting hot smelt solution may then be cooled to cause crystallization of sodium carbonate to take place, whereupon the crystallized sodium carbonate may be removed and converted to sodium sulphite for the preparation of a fresh liquor for the first cooking step, while the mother liquor, after causticization if desired, may be used for the preparation of a fresh liquor for the second cooking step.

The process of the present invention may find utility when raw cellulosic materials other than wood, e. g., cornstalks, bagasse and straw are treated thereby. Owing to the lack of a better generic expression, by the use of the expression "non-acid sulphite solution," I mean a neutral or alkaline sulphite liquor, in contradistinction to the acid liquors used in the acid sulphite process.

The process of the present invention and its application in practice having thus been described, it should be evident to those skilled

in the art that various changes or modifications might be made therein without departing from the spirit or scope of the appended claims.

I claim:

1. A cyclic process which comprises cooking raw cellulosic material in a non-acid solution of sodium sulphite without completing fiber liberation, cooking in an alkaline liquor containing sodium compounds to complete fiber liberation, mixing the spent liquors resulting from both cooking steps, smelting the inorganic content of the mixture in a reducing atmosphere to produce sodium carbonate and sodium sulphide crystallizing out sodium carbonate from the smelt, treating it with sulphur dioxide and water to produce a non-acid sulphite liquor for subsequent first-mentioned cooking, and using the rest of the smelt to produce an alkaline liquor for subsequent second-mentioned cooking.

2. A cyclic process which comprises cooking raw cellulosic material in a non-acid solution of sodium sulphite without completing fiber liberation, cooking in an alkaline liquor containing sodium sulphide to complete fiber liberation, mixing the spent liquors resulting from both cooking steps, smelting the inorganic content of the mixture in a reducing atmosphere to produce sodium carbonate and sodium sulphide, crystallizing out sodium carbonate from the smelt, treating it with sulphur dioxide and water to produce a non-acid sulphite liquor for subsequent first-mentioned cooking, and using the rest of the smelt to produce an alkaline liquor for subsequent second-mentioned cooking.

3. A cyclic process which comprises cooking raw cellulosic material in a non-acid solution of sodium sulphite without completing fiber liberation, cooking in an alkaline liquor containing caustic soda and sodium sulphide to complete fiber liberation, mixing the spent liquors resulting from both cooking steps, smelting the inorganic content of the mixture in a reducing atmosphere to produce sodium carbonate and sodium sulphide, crystallizing out sodium carbonate from the smelt, treating it with sulphur dioxide and water to produce a non-acid sulphite liquor for subsequent first-mentioned cooking, causticizing the sodium carbonate content of the rest of the smelt, and using it for subsequent second-mentioned cooking.

4. A process which comprises cooking raw cellulosic material considerably short of pulping in an alkali-containing sulphite liquor in which the sulphite preponderates in amount over the alkali, and then cooking in an alkaline liquor containing caustic soda and sodium sulphide to complete pulping.

5. A process which comprises cooking raw cellulosic material in a non-acid sulphite cooking liquor considerably short of converting such material into pulp, and then cooking in another liquor of distinct alkalinity to complete pulping.

6. A process which comprises cooking raw cellulosic material in a non-acid solution of sodium sulphite considerably short of converting such material into pulp, and then cooking in an alkaline liquor containing caustic soda and sodium sulphide to complete fiber liberation.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.