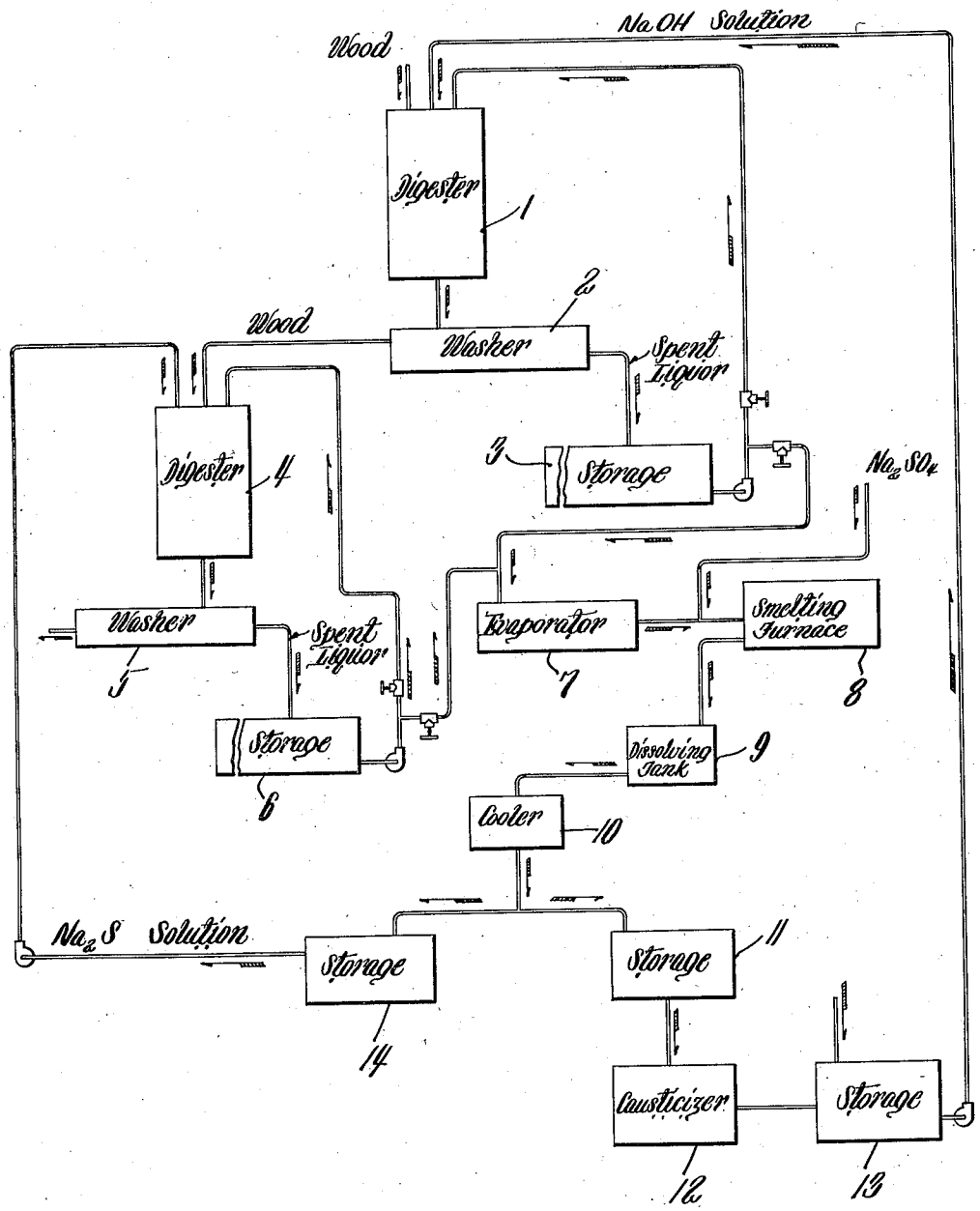
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PRODUCTION OF CHEMICAL WOOD PULPS

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PRODUCTION OF CHEMICAL WOOD PULPS

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This invention relates to the production of chemical wood pulps by cooking in liquors of an alkaline type, its object being the production of a high grade pulp of the general character of kraft pulp, so called, by a process which may be practised in an economical cycle of steps.

In the production of kraft pulp, it is usual practice to cook wood in the form of chips at elevated temperature and under pressure in a liquor containing caustic soda and sodium sulphide in solution, both these chemicals reacting upon and dissolving the cementitious material which binds the wood fibers together. It is known that the caustic soda is more drastic than sodium sulphide in its action on cellulose fiber, and accordingly that the higher the sulphidity of the cooking liquor, the better the quality of the resulting pulp. I have observed that degradation of the cellulose fiber occurs during the last stages of cooking, as at this time most of the cementitious material present in the original wood has been dissolved, so that the caustic soda no longer reacts with a high degree of selectivity upon such material, but reacts appreciably upon and degrades the fiber.

In accordance with the process of the present invention, therefore, wood or other raw cellulosic material is first cooked in a solution of caustic soda to effect a removal of a portion of the cementitious material, but without effecting fiber liberation, and is then cooked in a solution of sodium sulphide to effect fiber liberation, as it has been found that with this process the resulting pulp is of much better than usual quality. Such a process permits a removal of most of those cementitious materials which are reacted upon and dissolved readily by caustic soda solution, but without degrading the fiber, as the caustic soda solution reacts with a high degree of selectivity upon the cementitious material of the wood before so much of it has been removed as to result in fiber liberation. After the initial cooking in caustic soda solution, the wood is in condition to be readily converted into pulp and when cooked in the milder sodium sulphide solution, the sodium sulphide solution reacts upon and dissolves residual cementitious material, binding the wood fibers together but without injuring the cellulose fiber. Such a two-step cooking process may be economically practised, as the spent liquors resulting from both cooks may be treated together for the recovery of their valuable constituents, the spent caustic soda solution and the spent sodium sulphide solution being mixed and their valuable inorganic content smelted under reducing conditions to produce a smelt of sodium carbonate and sodium sulphide. The sodium carbonate may be separated from the smelt as by selective crystallization, and then causticized for use in a subsequent first cooking step, while the rest of the smelt may be used in a subsequent second cooking step. The raw material from which the chemicals are prepared for carrying out the process of the present invention may be similar to those used in the kraft process, losses taking place in the cycle being made up by adding sodium and sulphur constituent as in the form of sodium sulphate to the mixed spent liquors before delivery into the smelting furnace, or, if desired, by adding caustic soda to the fresh caustic soda cooking liquor and sulphur to the mixed spent liquors. In lieu of causticizing the smelted mixture of sodium carbonate and sodium sulphide, however, the sodium carbonate may be separated from the sodium sulphide as by selective crystallization, so that it may be independently causticized and the resulting caustic soda used in carrying out a subsequent independent initial cook of fresh wood, whereupon the wood may then be cooked in a solution of sodium sulphide prepared from the remainder of the smelt.

The process of the present invention may best be understood from the following more complete description thereof when considered in connection with the accompanying drawing, which is a flow sheet depicting the various steps of the process and indicating the flow of the materials therein.

Referring to the drawing, 1 represents a digester into which the chipped wood used as a raw material may be delivered, together

with sufficient caustic soda solution to maintain the wood immersed therein. The solution may vary in its alkalinity, containing, say, from about .5 to 5 pounds of caustic soda calculated as Na_2O per cubic foot, weak solutions preferably being employed when cooking is to be effected under super-atmospheric pressure, and strong solutions preferably being employed when cooking is to be effected under atmospheric pressure conditions. The charge may be cooked as by injecting steam directly thereinto until a substantial portion of the cementitious content of the wood has been reacted upon and dissolved, but, as previously stated, the concentration of alkali in solution, the temperature, pressure, and time employed in any cook should be such as will not complete fiber liberation. After cooking has been completed, the charge may be delivered into a washer 2, wherein the wood may be separated from the spent liquor with or without washing. The spent liquor from the washer may be passed to a storage tank 3 from which a portion may be withdrawn for use together with fresh caustic soda solution in subsequent cooking of fresh wood. The rest of the spent liquor may be subjected to treatment for the recovery of its valuable inorganic content, as will hereinafter be described. The wood from the washer 2 may be delivered into a digester 4 of the type customarily employed for effecting fiber liberation in alkaline liquors under heat and pressure. To this wood may then be added a solution of sodium sulphide in amount sufficient to cover it. Inasmuch as a substantial proportion of the resinous and ligneous matter present in the original wood has been removed, fiber liberation may be effected in a liquor of lower alkalinity than that usually employed when the usual one-step cooking process is employed. If desired, however, the solution may be of an alkali content similar to that of the usual kraft cooking liquors, in which case fiber liberation may be effected in a shorter time than ordinarily. Using a sodium sulphide solution of an alkali concentration of 2 to 2.5 pounds per cubic foot calculated as Na_2O and cooking at a temperature of about 300° to 335° F., it is possible to produce a high grade pulp in a period of about three hours. After fiber liberation has been effected, the digester contents may be discharged into a washer 5, wherein a separation of pulp from spent liquor may be effected. The spent liquor may be passed to a storage tank 6, from which a portion may be withdrawn for use together with fresh sodium sulphide solution in subsequent cooking of other batches of wood coming from the washer 2. The rest of the spent liquor may be mixed with the excess spent caustic soda liquor accumulating in the storage tank 3, and the mixture delivered to an evaporator 7 serving to remove a portion of the aqueous content of the mixture and thus make possible an economical recovery of its valuable inorganic content. The concentrated mixture may be fed into a furnace 8 maintained under reducing conditions, wherein the combustible content of the liquor may be burned and its valuable inorganic content smelted to produce a smelt consisting essentially of a mixture of sodium carbonate and sodium sulphide. The hot smelt may be delivered into a body of water maintained in a dissolving tank 9, the hot aqueous solution produced preferably being of a concentration permitting selective crystallization of sodium carbonate when cooled. Crystallization may be accelerated as by passing the hot smelt solution through a cooler 10, the resulting sodium carbonate crystals being removed from the mother liquor in which the more soluble sodium sulphide remains in solution. The sodium carbonate crystals associated with but little sodium sulphide may be dissolved in water in a tank 11, to produce a sodium carbonate solution which may then be causticized in a tank 12 to form a caustic soda solution. If desired, however, the sodium carbonate solution may be removed along with the desired amount of sodium sulphide, then dissolved in water and causticized, so that the first cook, which is carried short of fiber liberation, may be effected in a liquor containing much more caustic soda than sodium sulphide, and the second cook, which is carried to complete fiber liberation, may be effected in a liquor containing much more sodium sulphide than caustic soda. The causticized solution may be diluted in a tank 13 with water to form a fresh liquor of alkali concentration desired for use in cooking fresh wood in the digester 1. The mother liquor, containing sodium sulphide and but a relatively small amount of sodium carbonate, may be diluted in a tank 14 to the strength desired for use in the digester 4, its sodium carbonate content being causticized, if desired, before such use. Losses occurring in the cycle may be made up by adding sodium and sulphur constituent as in the form of sodium sulphate to the concentrated mixture of spent liquors being delivered to the furnace 8. Or, if desired, sodium losses may be made up by adding fresh caustic soda to the solution in the tank 13, and sulphur losses may be made up by adding sulphur to the mixture of spent liquors.

One of the important advantages of the process herein described is that it makes possible the production of strong and comparatively easily bleachable pulps. Experiments indicate that sodium sulphide is particularly effective as a delignifying agent. For instance, if raw cellulosic material, such as spruce wood chips, is cooked under the usual temperature, pressure, and time conditions

until fiber liberation is completed in two alkaline liquors of similar alkali content calculated as Na_2O, in one liquor all the alkali being present in the form of caustic soda and in the other liquor say about 40% of the alkali being present in the form of sodium sulphide and the rest in the form of caustic soda, it is found that the pulp produced in the sodium-sulphide-containing liquor is stronger and has a higher cooking stain (i. e., is more easily bleachable) than the pulp produced in the all-caustic soda liquor. In other words, sodium sulphide is apparently more effective and selective in its action upon lignin than caustic soda. With this in mind, it is thus seen that if a portion only of the lignin and resin of raw wood is removed by cooking in a liquor containing largely caustic soda, a substantially all-sodium sulphide liquor then being used to cook the wood to complete fiber liberation, the resulting pulp will be stronger and more easily bleachable than when fiber liberation is carried out to completion in an all-caustic soda liquor or in a liquor containing caustic soda in excess of sodium sulphide.

The example hereinbefore given deals with the use of wood as the raw cellulosic material, but the process of the present invention may be applied in the production of pulp from other raw cellulosic materials, such as straw, corn stalks, bagasse, and the like.

Having thus described the process of the present invention and certain of its practical applications, it should be evident to those skilled in the art that various changes and modifications might be made therein without departing from the spirit or scope of invention as set forth in the appended claims.

I claim:

1. A process which comprises cooking raw cellulosic material in a solution of caustic soda short of effecting fiber liberation, and then cooking in a solution of sodium sulphide to complete fiber liberation.

2. A process which comprises cooking raw cellulosic material in a solution of caustic soda short of effecting fiber liberation, then cooking in a solution of sodium sulphide to complete fiber liberation, mixing the spent liquors resulting from both cooking steps, and recovering the inorganic content of the mixture.

3. A process which comprises cooking raw cellulosic material short of effecting fiber liberation in a mixed caustic soda and sodium sulphide solution in which the caustic soda preponderates in amount, and then cooking to complete fiber liberation in a mixed caustic soda and sodium sulphide solution in which the sodium sulphide preponderates in amount.

4. A cyclic process which comprises cooking raw cellulosic material in a solution of caustic soda short of effecting fiber liberation, then cooking in a solution of 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, separating sodium carbonate from the smelt, causticizing the sodium carbonate for use in such subsequent first-mentioned cooking of wood, and using the rest of the smelt for such subsequent second-mentioned cooking.

5. A cyclic process which comprises cooking raw cellulosic material in a solution of caustic soda short of effecting fiber liberation, then cooking in a solution of sodium sulphide to complete the 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, dissolving the smelt in water to form an aqueous smelt solution, selectively crystallizing sodium carbonate from the smelt solution, causticizing the sodium carbonate for use for such subsequent first-mentioned cooking of wood, and using the rest of the smelt solution for such subsequent second-mentioned cooking of wood.

6. A cyclic process which comprises cooking raw cellulosic material in a solution of caustic soda short of effecting fiber liberation, then cooking in a solution of caustic soda 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, dissolving the smelt in water to form an aqueous smelt solution, selectively crystallizing sodium carbonate from the smelt solution, causticizing the sodium carbonate for use in such subsequent first-mentioned cooking of wood, and causticizing the rest of the smelt solution for such subsequent second-mentioned cooking of wood.

In testimony whereof I have affixed my signature.

GEORGE A. RICHTER.