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PULP MANUFACTURE

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This invention relates to the manufacture of paper pulp and has for an object the more efficient and economical manufacture of a superior grade of pulp, suitable for use in the paper, nitrocellulose, and other industries by the processing of wood and other vegetable fibrous materials.

It is a further object of the invention to provide an improved process for the manufacture of cellulose pulp from highly resinous wood.

These and other objects and advantages of the invention, which will more fully hereinafter appear, are attained by preliminarily treating the fibrous material with an aqueous solution of ammonia followed by digestion with an ammonium bisulfite solution.

According to this invention I am able to secure a high grade of pulp from a variety of raw cellulosic materials including those woods high in resin content, such as, for example, jack pine, pitch pine, scrub pine, Douglas fir, etc., which have been difficult, if not impossible, to process by the ordinary sulphite method. Because of the milder action achieved according to my invention by a preliminary treatment with an ammoniacal solution for the extraction of resinous, gummy, and other cementitious and binding material, followed by ammonium bisulfite cooking, to complete the defibering of the cellulose, economies in the usual bleaching operation are possible as well as the production of a light colored pulp of better than ordinary quality and relatively free from degraded cellulose. Furthermore, due to the milder loosening and defibering action of the chemicals employed according to my invention, a higher yield of pure cellulose is obtained. The high strength of the pulp fibers renders them particularly valuable for paper manufacture and like uses.

Although the use of wood in the form of chips is more specifically described in the following description of operating conditions, it will be understood that the references thereto are simply by way of illustration and the invention should be construed as broadly applicable to cellulosic materials of any form or variety. Furthermore, the specific conditions of operation employed, including, for instance, the temperature and length of preliminary treatment with ammoniacal solutions, the temperature and length of cook with ammonium bisulfite, and the strength of the solutions or liquors, may be varied depending upon the particular raw material under treatment and the desired quality of the final product.

It is well known that under ordinary sulfite pulping conditions the fiber is seriously degraded at the inception of the chemical operation due to the fact that the acid radical of the sulfite cooking liquor penetrates the chips more rapidly than does the basic ion of the chemical. This basic ion is necessary, however, to prevent excessive hydrolysis and degradation of the cellulose.

It is possible, according to my invention, to avoid the undesirable degradation above outlined by maintaining a definite quantity of gaseous or dissolved ammonia in the digester, diffused therein thruout the chips, after the chips have been subjected to preliminary extraction, by washing the chips with a weak solution of aqua ammonia and leaving a portion of the washing solution in the digester. The ammonium bisulfite solution (with or without sulfurous acid solution, depending upon the amount of ammonia in the wash water left in the digester) is thereafter introduced into the digester and ammonium bisulfite formed therein. In this way the base is diffused thruout the chips before digestion and as the acid subsequently penetrates, when introduced for purposes of digestion, it is neutralized in part by the diffused base and as a consequence no excessive nor harmful degradation of the cellulosic constituents occurs.

It is also possible by carrying out the process as outlined above to shorten the ordinarily long penetration period of the usual sulfite cooking, because there is quickly formed thruout the body of the chips a solution of sulphite acid containing ammonium salt, and temperatures which would usually be excessive at this period of the digestion are not harmful to the fiber.

In certain cases, such as in pulping resin-

ous woods particularly difficult to digest, as, for example, the heart wood of slash pine, highly resinous yellow pine, etc., it may not be desirable to carry out the process in the specific manner heretofore indicated. In such a case the ammonia may be thoroughly washed out of the chips after the preliminary treatment and (if the washing is not thorough the residual ammonia does no harm and serves as part of the base content of the acid) ammonium bisulfite liquor introduced into the digester and the cooking carried out in the usual manner save that a shorter penetration period and milder cooking treatment are possible. In all cases, however, the maximum temperature of the cook can be less than that usually required to complete the delignification of the cellulose, and there is less consumption of the chemical than is usual in this part of the process because some of the binding materials of the wood have been removed by the preliminary treatment. This is especially true of the difficultly pulpable resinous woods because the troublesome gums and coloring matter are removed by the preliminary extraction. These gums and other like materials prohibit the use of certain resinous woods for ordinary sulfite pulping; but by combining the advantageous effects of the preliminary ammonium hydroxide extraction treatment and the extraordinary solving action of ammonium bisulfite cook liquor for these resinous and gummy materials a higher grade pulp can be produced than is usual by ordinary methods.

The extracting operations above referred to are carried on by vigorously circulating a dilute ammonium hydroxide solution over the chips to be treated for a period of from 30 minutes to 3 hours and at a temperature of from about 90–180° C. although the preferable temperature range is 100–110° C.

After the extraction operation the ammonium hydroxide liquor is partly or wholly removed from the chips, as hereinbefore described, while at a temperature of from about 60–80° C. and the chips are washed with water or dilute ammonia within the same temperature range, i. e. 60–80° C.

Although the details and methods of carrying out the invention may be varied within wide limits without departing from the invention or sacrificing any of the advantages thereof, the following examples will serve to indicate the preferred embodiments and methods of procedure for executing the invention.

Example 1.—Into a digester of conventional design and of sufficient strength to withstand the conditions of temperature and pressure encountered in the pulping of wood and equipped with a suitable pump for circulation of extraction liquor, there are introduced 100 parts by weight of hemlock chips. Thereafter 500 parts of ammonium hydroxide of 2.5% strength are introduced and circulated through the chips for a period of 45 minutes at a temperature of 110° C. Following the circulation the materials in the digester are allowed to cool to a temperature of 70° C. when the ammonium hydroxide solution is drained off without release of pressure. The ammonia is recovered from the extraction liquor by distillation or other suitable means and the extracted material recovered if desired.

Two hundred parts of a 1% NH_4OH solution are then introduced into the digester and circulated thru the chips at 70° C. for about 15 minutes. The ammonia solution is drained off except for 65 parts which are left in the digester in immediate contact with the chips.

To the washed chips there are then added 435 parts of sulfurous acid solution of 5.5% concentration to form ammonium bisulfite in immediate contact with the chips and the cook is completed in the usual way, save that the temperature can be rapidly raised above 110° C., thus allowing a shorter than usual penetration period and a more rapid cook.

It will be found that the resulting pulp is of a white color and of exceptionally high quality, easily bleachable and of high alpha cellulose content.

Example 2.—Into a digester as described in Example 1, there are introduced 100 parts by weight of slash pine containing a high content of heart wood. Thereafter 500 parts of ammonium hydroxide of 3% strength are introduced into the digester and circulated through the chips for 2 hours at 100° C. The temperature is then allowed to drop to 70° C., the ammonium hydroxide containing solution drained off, and treated as described in Example 1, and the chips washed with water at 70° C. for about 15 minutes. The washing water is circulated thoroughly thruout the chips and drained off.

Thereafter 500 parts of ammonium bisulfite liquor of 5.5% total SO_2 and 1.15% combine SO_2, is introduced into the digester and the cook completed in the usual way. The resulting product is low in pitch content, free from shives and uncooked portions, of good color and strength, and easily bleachable.

I claim:

1. A process for the production of pulp which comprises subjecting cellulosic material to preliminary treatment with a dilute aqueous solution of ammonia and thereafter digesting with ammonium bisulfite.

2. A process for the production of pulp which comprises subjecting resinous wood to preliminary treatment with a dilute aqueous solution of ammonia and thereafter digesting with ammonium bisulfite.

3. A process for the production of pulp which comprises subjecting cellulosic material

to preliminary treatment with a dilute aqueous solution of ammonia, removing said solution of ammonia, and washing with a relatively more dilute aqueous solution of ammonia.

4. A process for the production of pulp which comprises subjecting cellulosic material to preliminary treatment with a dilute aqueous solution of ammonia, removing the greater part of said solution of ammonia, and washing with a relatively more dilute aqueous solution of ammonia.

5. A process for the production of pulp which comprises subjecting resinous wood to preliminary treatment with a dilute aqueous solution of ammonia, removing said solution of ammonia, and washing with a relatively more dilute aqueous solution of ammonia.

6. A process for the production of pulp which comprises subjecting cellulosic material to preliminary treatment with a dilute aqueous solution of ammonia at a temperature in excess of 90° C., and thereafter digesting with an ammonium bisulfite solution.

7. A process for the production of pulp which comprises subjecting cellulosic material to preliminary treatment with an aqueous solution of ammonia of from 2–4% strength at a temperature in excess of 90° C., and thereafter digesting with an ammonium bisulfite solution.

8. A process for the production of pulp which comprises circulating a dilute aqueous solution of ammonia over wood chips in the proportion of solution to chips of about 5:1 by weight, for about 45 minutes, at a temperature of 100–110° C., removing said solution of ammonia without reduction of pressure, circulating about two parts by weight of a relatively more dilute solution of aqueous ammonia per part of wood chips, for about 15 minutes at 70° C., removing all but about 0.65 parts of said solution, and thereafter cooking with about 4.4 parts of sulfurous acid solution of about 5.5% concentration.

In testimony whereof I affix my signature.

ROGER W. RICHARDSON.