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PROCESS OF MANUFACTURING SULPHITE PULP

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4 Claims. (Cl. 92—11)

This invention relates to the manufacturing of sulphite pulp, a process which, in comparison with known methods, produces substantial improvements from the point of view of economy and the quality of the finished pulp, and which more particularly permits of better adjustment and control of cooking conditions than any of the processes now in use.

In the manufacture of sulphite pulp, the process as generally applied at present involves cooking of chipped wood under pressure and at elevated temperature with a solution containing calcium bisulphite or a mixture of both calcium and magnesium bisulphite and a comparatively large excess of free sulphurous acid. Cooking by means of direct or indirect heating is continued until fibre liberation is attained. This procedure has many disadvantages from a chemical point of view as well as from the viewpoint of economy on raw materials and heat. In cooking, the first step of the many reactions occurring and finally resulting in fibre liberation, is penetration and impregnation of the chips by the cooking liquor. The next step is characterized as the sulphonation of the lignin by means of the bisulphite ions of the cooking liquor and the formation of insoluble lime or magnesia salts of ligno-sulphonic acid. The third step is the hydrolysis of these compounds by the hydrogen ions of the cooking liquor and their decomposition into compounds of smaller molecular size, which are soluble in the cooking liquor. Thus, the incrusting material of the wood is removed and the cellulose fibres are liberated. The speed of all these reactions depends upon the concentration of the cooking liquor, the temperature prevailing and upon the actual acidity, the latter, in turn, largely depending upon the temperature and the cooking degree already reached.

Unfortunately, the three main reactions finally leading to fibre liberation, do not proceed properly one after the other, but to a certain extent occur simultaneously, and, consequently, a certain quantity of organic matter is already dissolved before both penetration and sulphonation are completed. The concentration of this organic matter, of course, increases steadily with further progress of cooking, and finally, during the third period, i. e., hydrolysis of the sulphonated incrusting matter, the cooking liquor is heavily laden with dissolved organic material.

This high concentration of soluble organic compounds is the reason that, at the high temperature which during this period has to be maintained, side reactions occur, of which the net result is an undue increase of acidity of the liquor, which in this stage of cooking shows only very small buffer action. Strong acids are formed, which hydrolyze the hemicelluloses and even part of the cellulose of the fibres and finally polymerize the lignin left in the fibres, so that the bleach consumption of the pulp obtained remains relatively high. Furthermore, such an undue increase of acidity is connected with untimely decomposition of the cooking liquor, this reaction raising the consumption of sulphur per unit weight of pulp produced. Another result of these conditions is the fact that the degree of purification of the pulp attainable is insufficient for many technical uses, since substantial removal of the hemicelluloses is always accompanied by cellulose attack and a considerable loss in both alpha-cellulose and pulp yield.

Within the scope of the sulphite cooking process, as carried out in commercial scale at present, it is possible to reduce both speed and magnitude of the side reactions by maintaining a lower temperature during the last period of cooking. This, however, also reduces the speed of fibre liberation and lengthens the cooking time to such an extent that additional digester space and far higher investment costs are required for a given production. On the other hand, it is possible to regulate and decrease the acidity of the cooking liquor during the last period by starting with a cooking liquor of high combined SO_2. This, however, again lengthens the cooking time, and, in addition, increases the sulphur consumption and necessitates the use of costly sodium as base of the cooking liquor instead of lime, since the latter may result in heavy precipitations of calcium monosulphite.

The object of this invention is to provide a process which will enable the manufacturer of sulphite pulp to overcome the difficulties outlined above, to reduce the speed and magnitude of side reactions and to adjust the acidity of the last period of cooking without lengthening the cooking time, increasing sulphur consumption or requiring the use of costly alkali salts. A further object of the invention is to provide a process which requires lower sulphur consumption, which permits the removal of dissolved organic substances so troublesome in the last period of cooking and which provides for control of acidity in the last cooking period in order to develop desirable characteristics in the pulp which requires a smaller consumption of bleaching agent.

In accordance with the invention, chipped wood or like cellulosic material, is cooked in two stages, in the first of which fibre liberation is avoided

and in which the waste and harmful reaction products dissolved are removed to avoid interference with the desired reactions in the final cooking period. In the first stage the chips or the like are cooked with a sulphite cooking liquor of the usual composition and cooking with this liquor is discontinued before fibre liberation is attained. This liquor is then drained off without substantial lowering of the temperature or pressure. This is accomplished by introducing direct steam into the top portion of the digester. Ordinarily drainage of the liquor in this way is sufficient to remove the bulk of the dissolved reaction products but it is sometimes preferable to wash the contents of the digester, at this point, with a hot liquor, such as water, SO_2 solution, dilute cooking liquor or the like.

The second stage cooking liquor is now introduced into the digester. This is a sulphite liquor of higher relative acidity than the first liquor and is preheated before introduction to the digester. The actual acidity of this liquor is adjusted in accordance with the characteristics desired in the finished pulp in a manner to be later described. This cooking operation is continued until fibre liberation is complete. The dissolved waste reaction products having been earlier removed, the desired reactions are not interfered with in this stage of cooking.

This process gives a higher degree of both purification of the pulp and hydrolysis of hemicelluloses than is possible by the usual cooking processes and without degradation of the cellulose and, therefore, without loss of alkali-resistant alpha cellulose. Moreover, the pulp produced requires less bleaching agent than pulps produced by the usual sulphite cooking process.

As illustrative of a specific operation of the process of the invention, the following is given: Cooking of the wood chips or the like is started in the usual manner with respect to temperature, pressure, relief of gas pressure, etc., and the first stage cooking liquor is of the usual bisulphite type, containing, for example, combined SO_2 in the proportion of 20 to 25% of the total SO_2, with, for example, 5.20% of total SO_2 and 1.20% combined SO_2. This cook is carried on for 5 to 11 hours until a temperature of 120 to 155° C. is reached. This liquor is then drained off without substantial lowering of the temperature or pressure, as above described, and the second stage cooking liquor, preheated to the transfer temperature, is forced into the digester in any desired known way. This liquor has a higher relative acidity than the first stage liquor and may contain combined SO_2 in the proportion of 5 to 15% of the total SO_2, and preferably has a lower concentration of total SO_2, the total SO_2, for example, being 1.5–4.5% and the combined being 0.08–0.67%. This cook is continued until fibre liberation is complete and the desired degree of purification of the pulp is reached. The temperature and pressure of this stage as well as the dump colour or dump test has to be adjusted according to the quality of pulp desired. The pressure is kept constant at, for example, 75 lbs. and is not lowered until the blow back at the end of the cook. The temperature is raised gradually from the transfer temperature to 145–155° C. which is reached in 3 to 6 hours after introduction of this liquor. The two stage cook may take 10 to 14 hours in all.

This process may be operated without any higher heat requirements than those of the usual one stage cooking process and with a substantial saving in sulphur consumption. The drain liquor from the first stage cook with its content of useful chemicals and heat may be returned to the cycle after making up the volume required and strengthening by known methods, whereas the dump liquor from the second stage cook may be discarded after recovery of its content of heat and sulphur dioxide, or it may be used in the same manner as any other sulphite waste liquor.

The higher relative acidity of the second stage liquor causes a lowering of the viscosity of the pulp which is desirable for certain important uses of the pulp as is well known. This may be illustrated by two pulps cooked to the same degree, the one cooked with a second stage liquor in which the proportion of combined SO_2 is 40% less than that of the second stage liquor in which the other is cooked, has a solution viscosity one-half that of the other.

Furthermore, the use of a second stage liquor having a concentration of SO_2 of the order given results in reducing the amount of bleaching medium required to produce the desired bright white colour. Higher SO_2 concentration may be used but it is unnecessary and unecomonical. A saving in bleach consumption as high as 60% may be made by thus controlling the concentration of the second stage liquor.

Pulp produced by this process, in comparison with similar pulps produced by the usual sulphite process, has 2 to 3% higher alpha cellulose content and a solution viscosity of substantially one-fifth to one-tenth that of the normal sulphite pulps, whereas its resin content is only substantially 50% or less of that of the normal sulphite pulps. It will thus be apparent that the pulp produced by this invention has substantial improvements in these most important characteristics of sulphite pulp.

What I claim is:

1. A two stage process for manufacturing sulphite pulp which comprises cooking cellulosic material in a digester with a sulphite liquor in which the combined SO_2 is 20 to 25% of the total SO_2, discontinuing this cook before liberating the fibres, removing the liquor without substantially lowering the temperature or pressure, introducing a second heated sulphite liquor of higher acidity in which 5 to 15% of the SO_2 is combined and continuing the cooking operation until the fibre liberation and purification of the pulp is complete.

2. A two stage process for manufacturing sulphite pulp which comprises cooking cellulosic material with a sulphite liquor containing substantially 5.20% total SO_2 and 1.20% combined SO_2, removing this liquor before liberating the fibres and without substantial lowering of the temperature or pressure, and completing the cook with a sulphite liquor containing 1.5–4.5% total SO_2 and 0.08–0.67% combined SO_2.

3. A cellulose pulp produced by a two stage sulphite cooking process, said pulp having an alpha cellulose content of substantially 90%, a resin content not higher than 0.7% and a solution viscosity not more than one-fifth that or normal sulphite pulp.

4. A cellulose pulp produced by a two stage sulphite cooking process characterized by its low consumption of bleach, an alpha cellulose content of substantially 90% and having a solution viscosity substantially one-fifth to one-tenth that of normal sulphite pulp.

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