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METHOD OF MANUFACTURING PAPER PULP FROM BAGASSE AND OTHER GRAMINEÆ PLANTS

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This invention relates to the method of manufacturing paper pulp from bagasse and other gramineæ plants, which consists in digesting fibrous material of bagasse and other gramineæ plants with solution consisting principally of magnesium bisulphite with a small quantity of magnesium sulphite added so as to absolutely evade the presence of free sulphurous acid and thus in separating fibre easily. The object thereof is to considerably economize the digesting agent and obtain strong paper material of superior quality very easily and economically.

According to the soda process generally known as the best method for manufacturing paper pulp from gramineæ plants such as bagasse reed, and straw, caustic soda of the quantity about 40–80% of the product is required for dissolving the non-fibrous material. Also, for the bleaching 10–25% of bleaching powder is wanted. As regards cooking by the sulphite process which employs mixed solution of calcium bisulphite and free sulphurous acid, the cost of the manufacture may be small, but the non-fibrous material is not lixiviated perfectly or rather the fibre is injured very much. The paper pulp thus produced has yellowish brown color and in spite of 40–80% of bleaching powder required for its bleaching the fibre has none the less yellowish brown color and is too fragile for practical use.

After careful study, I have discovered the best method of separating fibre from bagasse and other gramineæ plants. Firstly, I studied the reason why the sulphite process is not satisfactory for separating fibre from these sorts of fibrous materials and have ascertained that the greater the concentration of hydrogen ion in cooking liquor is, the worse it will be for the product, unlike the case where wood is digested. To give the outline of its result, the combined state of the non-fibrous material contained in the above raw materials, namely lignine, differs from that in wood. If digesting liquor containing hydrogen ion of big concentration is employed as in the digestion of wood, the fibre will have its color turned into brown and become fragile. Therefore, it is necessary to use liquor of the smallest possible concentration. That is to say, a small quantity of hydrogen ion such as found in the solution employed in the present invention which consists of magnesium bisulphite and small quantity of magnesium sulphite ($XMg(HSO_3)_2+YMgSO_3$) enables the non-fibrous material to be separated easily from the fibre and to be placed in a condition for dissolution as lignine-sulphonic acid. Particularly, the carbohydrates (hexosans and pentosans) contained in bagasse are easily dissolved, if it is digested with the liquor employed in this invention. Moveover, the concentration of hydrogen ion being very small, there is no fear of the dissolved carbohydrates being further decomposed and thus of making the bleaching of the fibre difficult. Further, there is the advantage of magnesium sulphite present in the cooking liquor neutralizing the acidic substance produced during the digestion and thus preventing the increase of hydrogen ion. However, liquor containing free sulphurous acid such as that ($Ca(HSO_3)_2+H_2SO_3$) employed in the known sulphite process, is not particularly effective for dissolving the non-fibrous material. It will only injure the fibre owing to the greatness of the concentration of hydrogen ion. Moreover, it will act upon the dissolved non-fibrous material and form the material difficult to bleach. This fact has made me invent the present method.

There have been many researches made and many patents granted for methods of separating fibre from gramineæ plants, but all of them describe that a good result is obtained if digestion is performed by increasing the amount of the combined sulphurous acid. However, unlike the present invention, none of them mentions the harm caused by the presence of hydrogen ion in the cooking liquor used for the separation of fibre from this kind of raw material. Also, in the known methods cooking liquors have considerable concentration, say about twice as great as that in this invention. Indeed, some even contain free sulphurous acid which is never allowed to be present in this invention. Further, some are used as a mixture with calcium salt, but calcium bisulphite has its solubility

decreased by the rising of a temperature due to the digestion and consequently free sulphurous acid is separated, which increases the concentration of hydrogen ion still more. This is fundamentally different from the subject matter of the present invention. The following is an outline of the preparation of the cooking liquor and of the operation according to this invention:—

Firstly, to prepare the cooking liquor, fill a wooden vat of the inner capacity of about 320 cubic feet with nearly 250 cubic feet of fresh water, into which the gas from the sulphur burner is introduced, at the same time throwing thereinto industrial magnesium oxide slowly from the upper part of the vat. Then, the liquor will be stirred up completely by the burner gas jetting in the water, so that magnesite without sinking down acts easily upon sulphurous acid and gradually is converted from sulphite into bisulphite and is dissolved in the liquor as follows:—

$$\underset{\text{I}}{MgO} \rightarrow \underset{\text{II}}{MgSO_3} \rightarrow \underset{\text{III}}{Mg(HSO_3)_2} \rightarrow Mg(HSO_3)_2 + H_2SO_3$$

During the operation it is desirable to examine the concentration of magnesia and sulphurous acid radicle by analyzing the solution in the vat from time to time and to add magnesia or fresh water to dilute the solution. Before the whole of magnesia completes the conversion of the second stage, that is to say, when part of it still remains as sulphite, the introduction of the gas is stopped and the operation is finished. As liquor thus obtained not containing free sulphurous acid yet, does not give out the odor peculiar to the latter. However, the liquor which has reached the composition of the third stage even at the slightest, contains free sulphurous acid and consequently gives out peculiar sharp smell, which makes it unsuitable as the cooking liquor of this invention. Such being the case, before the completion of the change of the second stage the liquor consists chiefly of magnesium bisulphite with a small quantity of magnesium sulphite. That is to say, it has the constitution of $XMg(HSO_3)_2 + YMgSO_3$. The following is an example of the estimation of analyzing the concentration of the liquor thus obtained:—

First, 21.34 c. c. of $\frac{1}{10}$ standard iodine sol was put in a Mayer flask and the above-mentioned liquor diluted ten times was titrated thereto from a burette. In this case, 24.60 c. c. of the liquor was required to estimate the total sulphurous acid. Next, when the quantity of free sulphurous acid contained in the sample, after iodine was titrated, was examined with $\frac{1}{10}$ standard caustic soda solution using methyl orange as indicater, 31.79 c. c. of caustic soda solution was required. Now, upon calculating the concentration of the liquor with these three numerical values,

$Mg(HSO_3)_2 = 0.212$ mol/lit. $MgSO_3 = 0.010$ mol/lit.

The liquor of such concentration is employed to manufacture paper pulp according to this invention.

The following is an example of cooking methods:—

After filling a digester of the inner capacity of about 240 cubic feet with 1,400 lbs. of air-dried bagasse and closing the digester tightly, introduce high-pressure steam from the upper part of the digester and exhaust air, regulating the supply of the steam properly. Maintain the temperature of the interior of the digester at 100–115° C. for nearly 30 to 40 minutes and then stop the introduction of the steam. The object of this operation is to drive out air from the pore of the raw material, vaporize part of the water contained in the raw material and thus fill the pore with the steam. Soon after the above operation, the cooking liquor is introduced. Then, the raw material heated over 100° C. will contact with the liquor and be cooled. The steam in the pore will be condensed and form vacuum. Consequently, the liquor will be sucked into the raw material and permeate it uniformly. The average capacity of the liquor thus poured in is 200–210 cubic feet and its weight is about 13,000–13,650 lbs. Next, after closing the digester tightly, heat it with an indirect-heating apparatus in such a manner that the temperature reaches 150° C. in two hours. In this case, however rapidly the temperature may be raised, the fibre is not injured because of the absence of free sulphurous acid in the liquor. Upon estimating the concentration of the liquor in the digester after this temperature was maintained for five hours, when the digestion finished, almost the whole of useful ingredient, viz. bisulphite radicle (HSO_3) was consumed. Then, to lower the pressure the vapor was exhausted, after which the paper pulp was taken out from the digester and washed with water. The paper pulp thus obtained is of very superior quality with the appearance of light grey color, requiring only 7% of bleaching agent for perfect bleaching.

As the liquor used in this invention, namely magnesium salt, has great solubility in water, it can be prepared easily even in the tropical countries where bagasse is found. Although its concentration varies according to the raw material and heating method, it is small for such material as bagasse which is big in volume, but is small in weight. For the raw material which requires large quantity to fill the digester, it is usual to use liquor of considerable concentration such as contains 0.0150–0.250 mol. of magnesium bisulphite and 0.020 mol. or less of magnesium sulphite per litre. When a heating method in the cooking stage introduces steam directly, the cooking liquor is diluted by the condensed water of the

steam. Therefore, it is necessary to choose liquor having the concentration twice as big as the above-mentioned one. The proper quantity of the liquor is about 9–10 times as heavy as the raw material, so far as bagasse is concerned.

As stated before, the cooking liquor prepared according to the present invention never contains free sulphurous acid, it cannot only be prepared easily, but there is no fear of offensive odor produced by sulphur dioxide or damage done by the smoke gift to the neighborhood of the factory as is seen in the known sulphite process. Also, in cooking the paper pulp the fibre is not changed into brown color owing to the smallness of concentration of hydrogen ion. Consequently, no particular attention need be paid even in the technical direction. Thus, according to this invention paper pulp of excellent quality can be obtained quite easily from the above-mentioned various materials. Such a thing has never been done by the sulphite process.

Claim.

Method of manufacturing paper pulp from bagasse and other gramineæ plants, characterized by digesting fibrous material of bagasse and other gramineæ plants rapidly under pressure at a temperature of 100° to 115° C. for between 30 and 40 hours with solution consisting mainly of magnesium bisulphite ($Mg.(HSO_3)_2$) with a small quantity of magnesium sulphite ($MgSO_3$) added so as to absolutely evade the presence of free sulphurous acid.

In testimony whereof I have affixed my signature.

TETSUDO HAGIWARA.