Patented July 16, 1929. 1,721,376

UNITED STATES PATENT OFFICE.

JOAQUIN JULIO DE LA ROZA, SR., OF BROOKLYN, NEW YORK, ASSIGNOR TO BAGASSE PRODUCTS CORPORATION, OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

METHOD OF MANUFACTURING CELLULOSE PULP.

No Drawing. Application filed June 11, 1927. Serial No. 198,310.

This invention relates to a method of manufacturing cellulose pulp for the production of very strong snow-white paper, and has for an object to provide for the production of fibrous material adapted for the manufacture of a paper having a strength which has heretofore been attained only in dark papers.

Another object of the invention consists in providing certain improvements in the method whereby advantageous results are obtained.

The method is applicable to various vegetable substances containing cellulose, including trees, cornstalks, etc., but is particularly applicable to sugar cane fiber such as bagasse.

There has been on the market for a considerable period of time a kind of paper, commonly known as kraft, which possesses exceptional strength, but is of a dark color. This has been produced by a method that includes undercooking various woody material in a so-called sulphate process. It has been understood that, by undercooking the material in this process, the inherent strength of the fiber is preserved, and the product is characterized, as stated above, by extraordinary strength. However, the pulp obtained by this process or method has not been bleachable without sacrificing its strength. Therefore it has been found necessary to chose one or the other of the two most desired properties, to wit, strength or color.

There has been an additional disadvantage in connection with the production of this pulp because the sulphate process causes such objectionable odors to emanate from the digesters during the cooking step that it has been regarded as so obnoxious as to be permitted in but few localities.

As a result of my invention, there is obtained pulp from which is produced paper having the strength of kraft combined with a snow-white color; while the method is conducted without the generation of obnoxious odors.

My method, which forms the subject matter of this invention, comprehends the treatment of the material in two main steps, the first of which is carried out in a hot aqueous solution including sulphur, and the second in a hot alkaline solution. The first step is also preferably of a slightly acid nature. In the preferred form of carrying it out, the method also includes a third step of separating the fibous material by suitable refining mechanism; and a fourth step of washing.

Broadly speaking, and assuming bagasse is the material to be treated, the invention comprehends subjecting the bagasse or fibrous elements of sugar cane to a first treatment with hot water and sulphur at a temperature of approximately 160° to 173° centigrade, which treatment generates an acid condition resulting from the decomposition of the sugars into organic acids, and which acid condition is utilized for changing the incrusting substances so as to render them readily soluble in dilute alkali as, for instance, by changing xylan and araban into xylose and arabinose, while the sulphur present has a whitening effect due probably to its capacity for combining with coloring matter in the bagasse to form soluble compounds; following which first treatment there is a second treatment of the material with a dilute alkali; and subsequent refining and washing.

In carrying out my invention, the bagasse is conveyed, by means of blowers or other suitable arrangement, into the interior of a vessel which is generally similar to the standard sulphite pulp digester. This vessel, however, is preferably composed of a ferric material, such as steel or iron, lined with brick adapted to resist the alternating action of dilute acid and dilute alkali. This character of brick is known to the chemical industry although it differs sharply from the brick used in the standard sulphite pulp digesters in that the latter are specially designed to withstand acid action only. The arrangement of valves, vents and the like may differ to a certain extent from the standard arrangement of the sulphite digesters, but such changes are well within the skill of the calling and merely for the purpose of better adapting the apparatus to the specific conditions in hand.

After the bagasse has been passed into this vessel, water is added. I prefer to use hot water if it can be economically obtained and, indeed, the hotter the water is the more the process will be expedited.

After the vessel has been substantially filled with water, I add a quantity of commercially pure sulphur. This may vary from one-half percent to ten percent of the weight of the bone dry material being treated, this weight being previously deter-

mined by a sample in the usual way. The amount of sulphur added will vary according to desired result, a lower percentage being well adapted for the manufacture of a thoroughly cooked snow-white cellulose pulp of considerable strength, and a higher percentage being better suited for an undercooked pulp adapted for the production of paper having the great strength of kraft, although white in color.

The valves, vents, openings and the like are then closed and steam is injected so that the contents are heated quite rapidly up to a temperature of approximately 130° centigrade. At this point a vent is opened so as to relieve substantially all of the entrapped air. This vent is then closed, or substantially closed, so that no appreciable amount of water or steam can escape from the interior.

Additional steam is then injected, although somewhat more slowly than before, until a temperature of from 160° to 173° centigrade is reached.

I may say, at this point, that the matter of temperature is one of substantial importance because the desired action upon the incrusting materials is not at all effective at a temperature substantially less than 160° centigrade while, at a temperature higher than approximately 173° centigrade, there is liability of a serious injury to the fiber, and the production of so-called caramel and humic compounds. It should also be kept in mind that the temperature cannot certainly be determined from the pressure alone, because pressure indications are apt to be augmented as a result of gas pressure automatically developed during process. Therefore it is important to observe the temperature requirements and to use a thermometer for directly indicating the same.

After the temperature above named has been reached, the charge is allowed to remain, while the temperature is maintained, for a period of from, say, twenty minutes to three hours. This time period depends mainly on the condition, age, etc., of the bagasse. I prefer to use fresh bagasse whenever possible or, at least, bagasse which has not been very long exposed to conditions leading to rotting or oxidizing. However, the process is adaptable to the use of old, baled bagasse, or wet bagasse or fermented bagasse or any other form of bagasse that may be encountered in sugar manufacture, without greatly altering the high quality of the product. The limitations above named will ordinarily cover the varying characteristics of the material being treated, and it is desirable, whenever possible, to avoid a treatment of over two hours at the temperature range of 160° to 173° centigrade because, at such elevated temperature, some of the acids automatically generated will attack and injure the cellulose if the treatment is prolonged. This may result in the bagasse becoming appreciably dark and having the appearance and characteristics of humic compounds, while the fibers may be appreciably weakened. It may be thought that such effects may facilitate the subsequent treatment with the dilute alkali but, even if this be so, the formation of the humic compounds and the weakening of the fibers has a tendency to offset any such gain and to prejudice the attainment of a fiber of the highest quality.

This treatment at 160° to 173° centigrade automatically generates an acid condition owing, probably, to the decomposition of certain sugars into organic acids and this acid bath directly dissolves some of the incrusting substances and has a hydrolytic action upon others whereby they are changed so as to be readily soluble in dilute alkali. For instance, such incrusting substances as xylan and araban are changed into xylose and arabinose which are readily dissolved by dilute alkali. Because of this automatic generation of an acid condition, I call this first treatment an acid treatment, and it does, in fact, react acid to litmus. The sulphur present in this treatment promotes the obtaining of a white color in the product, and this probably results from its action in combining with various coloring matters to render them soluble in the first or second treatment.

After the charge has been subjected to this treatment for the determined period of time, the pressure in the vessel is relieved to a certain extent by the opening of a vent, and the acidified liquid, containing such substances as may have been dissolved, is allowed to blow from or is otherwise removed from the vessel. In preparing to carry out the remainder of the process it may sometimes be advisable to remove the bagasse from this first vessel to another one, which may be located beneath it or at its side; but, because of the likelihood of matting the material and the expense of the step of removal, I prefer to carry out further treatment in the same vessel.

After the acidified liquid has been removed, the vessel is refilled with warm or hot water, and the latter then allowed to drain from the vessel for from fifteen to thirty minutes. In this step I may use ordinary neutral water, but I prefer to use the slightly alkaline water which has been drawn off after the dilute alkaline treatment which constitutes a subsequent step in the process. It will be understood that, when the process is in use, there will be numerous batches going through, so that the silghtly alkaline water which has been drawn off after the dilute alkali treatment given to one batch, may be employed in washing or treating another batch immediately after it has had its acid treatment just described. This procedure is of considerable

practical importance because the alkaline water drawn off after the dilute alkali treatment will substantially neutralize the batch coming from the acid treatment, and thereby enable the fresh alkali solution used in the second treatment to better dissolve or transform the incrusting substances. The use of the drawn off alkaline water in this way also eliminates the necessity of systems for recovering the alkali from the said water, which systems are costly.

After this water is drained, or has been otherwise removed from the vessel, the drain or other outlet is closed and fresh dilute alkali is added. This dilute alkali may consist of potassium or sodium hydroxide dissolved in water in the proportion of from say twenty to one hundred grams of sodium per litre of water, depending on the extent of purification desired.

Steam is then injected into the vessel, as in the first treatment, until a temperature of approximately 130° centigrade has been reached. At this point, a vent is opened, as before, in order to relieve the entrapped air. The injection of steam is then continued until a temperature of from 160° to 173° centigrade has been attained, and the charge is then maintained at said temperature for a period of from two and one-half to five hours or more. This period of time depends on the extent of the first treatment in the acid sulphur bath, upon the nature and condition of the material, and also upon the strength of the alkaline solution. Naturally, a more concentrated alkali will act more rapidly. However, it should be kept in mind that an increase in the alkali promotes a tendency to injury of the cellulose at an elevated temperature such as 160° to 173° centigrade; so that it is more desirable to extend the period of this treatment than to unduly increase the strength of the solution.

At the termination of this period of dilute alkali treatment, the pressure in the vessel is relieved by the opening of a vent, and the charge is blown or otherwise removed therefrom into a device for separation of the fibers to produce pulp, which device may be a ball refiner or rod mill, both of which are well known to the pulp industry.

After this treatment in the refining device, the material may be transferred to a pit or vat and therein washed, while being agitated, with hot water and thereafter with cold water until the alkalinity has been substantially eliminated. It will be understood that the water is drained off after each washing and that it carries with it the dissolved incrusting materials and the like. The greater the number of washings the cleaner will be the material, so that the extent to which this proceeding is carried out will depend greatly upon the quality of product desired. Ordinarily, I prefer to wash at least three times.

After washing, the material may be subjected to the various steps ordinarily employed in the production of pulp for the manufacture of strong paper such as kraft.

In the second step, or alkaline treatment, the temperature may, if desired, be maintained somewhat below 160° centigrade, which would require somewhat longer cooking period and yet give a somewhat stronger product; but it is not advisable to exceed the upper temperature of 173° centigrade.

In carrying out the method in connection with materials other than bagasse, the procedure will be the same, although some of the previous description relating to the substances present and their change of condition will be inappropriate because of difference in material.

It may also be noted that, when bagasse or corn stalks are adopted as the material, the resulting pulp will be snow-white without the use of any bleach. In the case of other cellulose-containing materials, such as wood and the like, the pulp should be given a mild bleaching treatment, varying in intensity according to the amount of the lignin content of the material.

The acid condition of the first treatment is preferably, as previously indicated, brought about by the automatic generation of acids during said treatment, but it may, if desired, be supplemented by the original addition of a small amount of acid to the contents of the vessel, or to the water before it is run into the vessel. It may also be noted that, in the case of certain materials, the first treatment may not be of an acid nature, because the said materials may not contain ingredients that generate acid when subjected to the hot aqueous sulphur first treatment. This, however, relates to a change in material and not to a difference in the steps of the method followed.

The inclusion of a quantity of sulphur, or its equivalent, in the first hot aqueous treatment is a matter of prime importance in carrying out this invention because, by the use of that ingredient, I am enabled to obtain fibrous pulp which is snow-white and yet has that extraordinary strength characteristic of the paper above referred to as kraft.

It will be understood that various changes may be resorted to in the steps of the process, in the materials, and in the apparatus without departing from the spirit and scope of my invention, and hence I do not intend to be limited to the details herein set forth except as they may be included in the claims.

What I claim is:

1. A method of producing pulp for the manufacture of snow-white paper of great strength, which includes treating cellulose-containing material in a hot aqueous solution including free sulphur, subsequently separating the material from the said solution and

treating it in a hot dilute alkaline solution, both said treatments being carried out at a temperature of approximately 160° to 173° centigrade, and then refining the material in suitable apparatus, and washing.

2. A method of producing pulp for the manufacture of snow-white paper of great strength, which includes treating cellulose-containing material in a hot aqueous solution containing free sulphur, and subsequently separating the material from the said solution and treating it in a hot alkaline solution of insufficient strength to produce a pure alpha cellulose, the first treatment being carried out at a temperature of approximately 160° to 173° centigrade, and the second treatment at over 100° centigrade.

3. A method of producing pulp for the manufacture of snow-white paper of great strength, which includes treating cellulose-containing material in a hot aqueous solution containing free sulphur, subsequently separating the material from said solution and treating it in a hot alkaline solution of insufficient strength to produce a pure alpha cellulose, the first treatment being carried out at a temperature of approximately 160° to 173° centigrade, and the second treatment at over 100° centigrade, and then refining in suitable apparatus, and washing.

4. A method of producing pulp for the manufacture of snow-white paper of great strength, which includes treating cellulose-containing material in an aqueous solution including sulphur and of no substantial alkalinity, and subsequently separating the material from the said solution and treating it in a dilute alkaline solution, both said treatments being carried out at a temperature above 100° centigrade.

5. A method of producing pulp for the manufacture of snow-white paper of great strength, which includes treating cellulose-containing material in an aqueous solution including free sulphur, subsequently separating the material from the said solution and treating it in a dilute alkaline solution, both said treatments being carried out at a temperature above 100° centigrade, and then refining the material in suitable apparatus, and washing.

In testimony, that I claim the foregoing as my invention, I have signed my name this 10th day of June, 1927.

JOAQUIN JULIO DE LA ROZA, SENIOR.