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3,414,469

TREATMENT OF FLASH DRIED PULP TO REDUCE NODULES THEREIN

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ABSTRACT OF THE DISCLOSURE

A method reducing nodule formation in flash dried pulp wherein the pH of alkaline digested pulp slurry is adjusted to between 5 and 8 and certain inorganic sulfur containing compounds are added to the pulp slurry while such slurry is at a pulp consistency above 25 percent and the thus treated pulp is then dried.

This invention relates to methods of preparing easily dispersible flash-dried alkaline-digested pulp and more particularly relates to methods of greatly reducing the quantity of difficultly dispersible fiber "nodules" from such pulp.

In the "flash dry" method of producing market pulp, the fibers produced by chemical digestion of lignocellulose materials are washed, treated so as to remove non-digested bundles of fibers, subjected to mechanical means to express water and produce a stock having a consistency greater than 25%, and dried by passing small particles of the high consistency stock through a flash dryer wherein the stock is contacted with hot gasses to cause very rapid removal of the water. The thus produced dried pulp which contains about 5 to 15% moisture may be compressed and baled in suitable form for shipment to distant locations where it will be redispersed in water and used in the making of paper, paper board or the like.

The flash dry method of producing pulp has very definite cost advantages over the older methods of producing pulp wherein the pulp stock after washing and deshriving is diluted to a low consistency of 1 to 5%, formed into a sheet of pulp on a cylinder or Fourdrinier type machine, squeezed to reduce the water content of the sheet of pulp, and finally dried by evaporating the remaining water in the web. Not only does the flash drying method achieve substantial savings in the heat required to dry the pulp but most important is able to accomplish this at substantially lower investment cost for equipment.

The flash drying process does suffer from one serious drawback, however. This is the tendency of individual fibers in the dried pulp to bond together thereby forming difficultly dispersible bundles variously referred to as "knots" or "nodules." As the term "knots" is generally employed in the paper field in reference to bundles of fibers which have been incompletely separated during the pulping operation, the term "nodules" will be employed herein to avoid confusion.

These nodules can be formed in the fibers whenever they are at high consistency, i.e. above about 20%. However, the formation of the nodules is dependent upon both time and temperature. If the dried pulp is immediately repulped after drying, few if any nodules will be found. If the pulp is stored for a few days the number of nodules will be materially increased. Continued storage will result in continuing increases in the number of nodules until about 90 days has elapsed. After this period of time, the nodule formation is essentially complete and few additional nodules are formed.

The rate at which the nodules are formed is greatly

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accelerated by increasing the temperature at which the pulp is stored. It has been found, for instance, that storage at 60° C. for 14 hours results in approximately the same formation of nodules as storage at 70° F. for 90 days.

Considerable effort has been expended toward finding ways of reducing or eliminating the formation of nodules. As a result, it has been found that the method by which the pulp is obtained from the lignocellulose greatly influences the results obtained by various treatments. For example, it has been found that nodules in flash dried groundwood pulp can be greatly reduced by the addition of lime to the pulp stock shortly before drying. The use of lime, however, has been found to be ineffective on alkali-digested pulps, i.e., those obtained by the soda and kraft or sulfate processes. With alkali-digested pulps, the most effective agent currently known is sulfur dioxide which is applied to low consistency pulp prior to mechanical expression of water and drying. While substantially reducing the number of nodules, the treatment with sulfur dioxide does not reduce the number of nodules formed to a wholly satisfactory level.

It is the principal object of this invention therefore to provide a process will produce a flash-dried alkali-digested pulp which will remain substantially free of nodules.

Other objects will be evident from the following disclosure.

In its most basic form our invention comprises the addition of from 0.03 to 0.5 molar equivalents of certain inorganic sulfur containing materials to 1000 parts by weight of air dried alkali digested pulp while such pulp is at a consistency above 25% and drying of the thus treated pulp, without intervening reduction in consistency, by contacting small particles of the pulp with heated gases. The inorganic sulfur containing materials suitable for this use are sodium, calcium, magnesium, potassium or zinc bisulfites and sodium or zinc dithionites.

Full details of our invention, including various improvements of the basic process, are set forth in the following description of the manufacture of flash-dried pulp starting with a washed, screened alkali-digested pulp. This washed and screened pulp may be obtained by any of the many well-known processes known to the pulping art. As indicated hereinabove, the pulping process may be either soda or kraft cook. The degree of delignification during the cook may vary quite widely. Our process has been successfully employed with pulps ranging from a high yield pulp having a KAPPA No. of 65 to 75 to a bleachable grade of kraft pulp having KAPPA No. of 37 to 45. The pulp may be obtained from either softwood or hardwood. Due to the pronounced tendency of the longer fibered softwoods to form nodules, our process is especially effective with this type fiber.

After washing and screening, the pulp is desirably subjected to an impact type refining operation to break up any shives of non-digested pulp which may be present. This is most easily accomplished by passing the pulp at washer consistency of about 10 to 18% through a device such as a Bauer double disc refiner or Jones Vertifiner with a relatively wide opening so that there is little or no work done on the individual fibers.

After the shive removing operation, the pulp is preferably diluted to a consistency of about 1 to 6%. While in this diluted state, acid is added to the pulp, which due to residual alkali from the pulping operation, has a pH of about 8.5 to 10.5. Sufficient acid to reduce the pH of the pulp slurry to between 5 and 8 is used. The preferred acid is sulfuric because of its low cost, however, any other acid, such as, hydrochloric, sulfurous, dilute nitric or acetic may be employed.

While this acid adjustment step is a part of our preferred process, it may be omitted. If omitted, the dilution step may also be eliminated from the process and the pulp passed from the shive removing operation directly to the next step which is the expression of water to form a high consistency pulp of from 25 to 65% solids.

The principal purpose of the acid addition is to reduce the requirements for bisulfite or dithionite in later operations. If the pH of the pulp is not reduced to the 5 to 8 range in this step, it must be reduced to within this range using the mildly acidic bisulfite or dithionites, thus requiring the use of about 2 to 5 times as much of these more expensive chemicals as would be required if acid is employed.

Additionally, however, the use of acid to reduce the pH results in a substantial improvement in the mechanical operation used to express water from the pulp and increase the consistency.

After the acid addition, the water is expressed from the pulp to increase the consistency to 25 to 65%. This can easily be accomplished by mechanically squeezing the pulp to remove free water present by numerous types of equipment well known to the papermaking art such as a screw press, a canted disc press, a double roll press, or a grooved roll press.

From the standpoint of drying alone, the higher the consistency produced during this step, the lower will be the heat requirements for drying. However, considerable work must be done on the pulp to increase the consistency much above 45% and such work appears to favor the formation of nodules. Consequently, it is preferred that the consistency during this step be raised to between 30 and 45%.

While the pulp is at this high consistency, between 0.03 and 0.5 equivalents of the bisulfite or dithionite are applied per 1000 parts by weight of air dried pulp. The quantity of bisulfite or dithionite employed here is primarily dependent upon whether the acid addition had previously been employed to reduce the pH. Where the pH has been previously adjusted, from 0.03 to 0.15 equivalents of bisulfite or dithionite should essentially eliminate the formation of nodules. Where such adjustment has not been made, much larger quantities of the bisulfite or dithionite will be required to reduce the pH within the specified limits. The exact amount required will in large measure be dependent upon the extent to which the alkaline pulping chemicals have been removed during washing and preceding operations, which in turn will determine the pH of the pulp. With highly alkaline pulps, up to about 0.5 equivalent may be required to obtain the necessary pH.

The choice of which bisulfite or dithionite to employ is largely dictated by economic considerations. As sodium bisulfite is currently the least expensive of the usable materials, its use is preferred.

The addition of the bisulfite or dithionite is easily accomplished by spraying an aqueous solution of from 5 to 15% solids on the fibers while the fibers are being mechanically agitated thus ensuring uniform application. Our preferred method of accomplishing this is to add the bisulfite or dithionite to the pulp as it is fed to a "fluffer" preceding the dryer. The "fluffer" is an impact type of commutating device designed to break the mass of high consistency pulp into small particles or fiber balls which allows intimate contact between fibers and gases and results in rapid water removal during drying. Suitable equipment to accomplish fluffing are the single disc refiner equipped with the so-called devils tooth plates, a hammer mill or even a rapidly rotating fan blade. Desirably the median diameter of the particles produced in the fluffer will range from about 1 to 5 millimeters.

Without further addition of water, the treated, high consistency particles are passed to a hot gas dryer where the remaining water is removed very rapidly. The rapidity at which the water is removed, generally on the order of

about 5 to 20 seconds, has resulted in terming the process as flash drying. There are a number of standard dryers usable, such as the rotating drum, either single or multiple pass type, the tower type and even the fluidized bed type. All these dryers are characterized by the use of hot gases within the range of 400-1200° F., as the drying medium, and concurrent flow of gases and pulp through the dryer. Regulation of the moisture content of the pulp leaving the dryer is easily obtained by control of the gas temperature and gas flow through the dryer.

After drying, the fiber particles can be compressed and baled as desired for shipment.

The exact practice of this invention may best be seen in the following examples.

EXAMPLE 1

In a standard flash drying process, a Kraft pine pulp which had been washed, screened and lightly refined to break up any non-digested shives was fed to a Zenith screw press at a consistency of 3%. The screw press by mechanical expression of water raised the consistency of the pulp to 35%. This 35% consistency pulp was fed to a Bauer single disc refiner equipped with devils tooth plates. This refiner acted as a "fluffer" breaking the pulp into small fiber balls having a mean diameter of about 2 mm. These fiber balls were then fed to a triple pass Heil hot gas rotary dryer where they were dried to approximately 12% moisture using a gas temperature of about 1200° F. The dried pulp was conveyed to a baler where it was compressed into 500 pound bales having a density of 50 pounds per cubic foot.

Samples of this pulp were subjected to accelerated aging by placing the samples in an air tight polyethylene bag and heating in an oven at 60° C. for 14 hours (approximately equivalent to 90 days at ambient temperature). Nodule count determinations were then made by placing 140 grams (O.D.) pulp in a container and adding sufficient water to make 4200 mls. of stock. This stock was allowed to stand for one hour after which it was placed in a Dazey Churn disintegrator and churned for 7 minutes. Eight ml. of pheno-fast red dye (10 grams of pheno-fast red diluted to 100 ml. with water) was then added and the stock churned for an additional 8 minutes. 1000 ml. of the resultant stock was removed and diluted with water to make 11,00 ml. 575 ml. of the dilute stock was employed in making each of 4 separate handsheets by standard practices. After drying of the handsheets, areas of 10 cm. x 10 cm. were marked off near the center of the handsheets and the number of non-disintegrated fiber nodules appearing in this area counted. An average of the count for the 4 handsheets was finally computed.

Without the use of any additives the nodule count in nodules per 100 cm.² amounted to 134.

Utilizing the same type pulp the process was repeated except that the 3% consistency pulp was acidified to pH 7 with sulfuric acid prior to entering the Zenith screw press. Additionally, sodium bisulfite in the form of a one pound per gallon aqueous solution was sprayed on the high consistency pulp immediately preceding the Bauer refiner buffer. The rate of application was ten pounds or 0.096 equivalent sodium bisulfite per 1000 pounds of air dried pulp.

The nodules count obtained on the pulp thus treated was 8 nodules/100 cm.², or a reduction in the number of nodules of approximately 94%.

EXAMPLE 2

The general procedure shown in Example 1 was repeated using a somewhat different pulp stock. Two runs were made in which sodium bisulfite was applied at the rate of five pounds or 0.048 equivalent per 1000 pounds of air dried pulp. In one run the pH of pulp slurry fed to the screw press was adjusted to 7.4 with sulfuric acid. In the other run, this pH adjustment was not made, the pH remaining at 10.1. In the first run the nodule count was 9 nodules/cm.² while in the second run the nodule

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count was 41 nodules/cm.². This well indicates the necessity of employing acid to reduce the pH of the pulp slurry or as indicated in the following example of employing sufficient bisulfite or dithionite to reduce the pH to within the range of 5 to 8.

EXAMPLE 3

Again using a somewhat different kraft pine pulp, a flash dried pulp was made without chemical addition following the procedures of Example 1. The nodule count of this pulp was 325 nodules/cm.². A dried pulp was also produced in which 30 pounds or 0.288 equivalent of sodium bisulfite were added to 1000 pounds (air dry basis) of pulp entering the Bauer refiner fluffer. The nodules count on this dried pulp was only 11 nodules/cm.².

It has surprisingly been found that the use of SO₂ is not nearly as effective as the materials of this invention.

EXAMPLE 4

Using a kraft pine pulp a control run was made following the procedures of Example 1 without additives. The nodules count of this dried pulp was a relatively low 75 nodules/cm.². Following this control, a run was made in which SO₂ was added to the low consistency pulp going to the screw press in sufficient amount to reduce the pH of 9.5 to 5.5. The pulp produced from this run had 20 nodules/cm.² or a reduction in nodules of only 73%.

We claim:

1. In the method of treating flash dried pulp wherein a slurry of alkaline digested pulp is mechanically treated to increase its consistency to between 25 to 65% and said high consistency pulp is thereafter dried by passing small particles of said high consistency pulp through a flash dryer wherein said high consistency pulp is contacted with

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heated gases the improvement which comprises adjusting the pH of the pulp slurry to a pH between 5 and 8 and adding to the high consistency pulp between 0.03 and 0.5 molar equivalents of an inorganic compound selected from the group consisting of sodium bisulfite, potassium bisulfite, calcium bisulfite, magnesium bisulfite, ammonium bisulfite, zinc bisulfite, sodium dithionite and zinc dithionite per 1000 parts by weight of air dried alkaline digested pulp.

2. The method of claim 1 wherein between 0.03 and 0.15 molar equivalent of said inorganic compound is employed.

3. The method of claim 1 wherein the inorganic compound added to the high consistency pulp is sodium bisulfite.

4. The method of claim 1 wherein the pH of the pulp slurry is adjusted by acidifying with an acid from the group consisting of sulfuric, hydrochloric, sulfurous, nitric or acetic.

5. The method of claim 1 wherein the pH of the pulp slurry is adjusted by acidifying with an inorganic compound from the group consisting of sodium bisulfite, potassium bisulfite, calcium bisulfite, magnesium bisulfite, ammonium bisulfite, zinc bisulfite, sodium dithionite and zinc dithionite.

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S. LEON BASHORE, *Primary Examiner*.

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

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James R. Brown et al.

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 4, line 75, "9 nodules/cm²" should read -- 9 nodules/100 cm² --. Column 5, line 1, "41 nodules/cm²" should read -- 41 nodules/100 cm² --; line 10, "325 nodules/cm²" should read -- 325 nodules/100 cm² --; line 14, "11 nodules/cm²" should read -- 11 nodules/100 cm² --; line 23, "nodules/cm²" should read -- nodules/100 cm² --; line 27, "nodules/cm²" should read -- nodules/100 cm² --. Column 6, line 8, "1,000 parts by weight" should read -- 1,000 pounds by weight --.

Signed and sealed this 10th day of March 1970.

(SEAL)

Attest:

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Commissioner of Patents