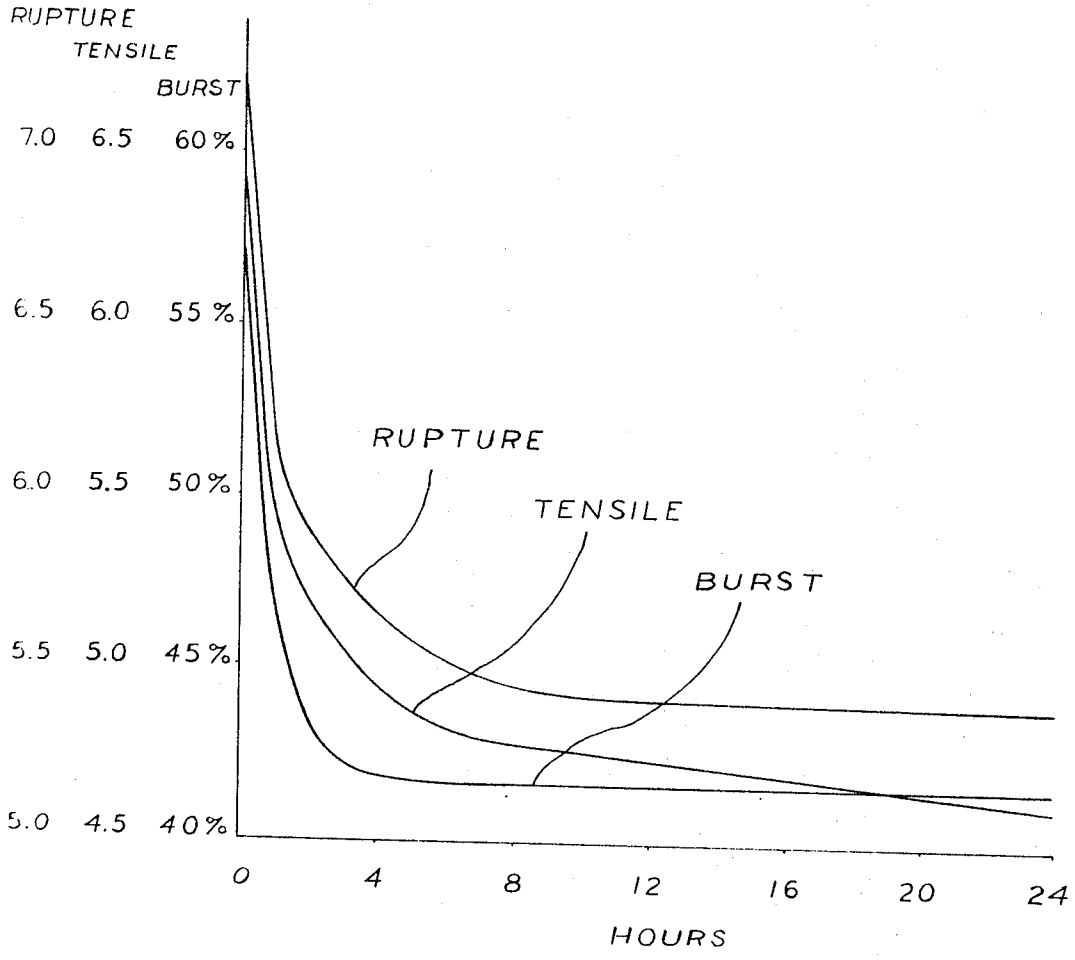
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STORAGE OF HIGH CONSISTENCY REFINED PULP

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STORAGE OF HIGH CONSISTENCY REFINED PULP

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ABSTRACT OF THE DISCLOSURE

A process for preparing a fibrillated pulp from defibered cellulosic fibers suitable for storage, wherein already defibered pulp of separated cellulosic fibers is processed to effect fibrillation of the separated fibers. The fibrillation is such that fibrils are formed in a fiber along its surface which are partially free to the extent that they separate and project from the fiber in hair-like strands while substantially maintaining the length of the fiber. Such fibrillated pulp is prepared for storage by introducing cool water to the pulp immediately after fibrillation thus to produce hydration of the separated fibrils, with water molecules attaching to hydroxyl groups in the fibrils through hydrogen bonding. Cooling of the fibrillated pulp with the addition of water promotes hydration of the fiber fibrils.

This invention relates to the preparation of pulp such as may be used in the manufacture of paper and related products. More particularly, the invention concerns a method of processing pulps subjected to a high consistency refining treatment, to inhibit regression in the pulp on storage for any amount of time after preparation and to inhibit resulting loss in strength properties in products produced from the pulp.

A development in the paper-making art comprises frictionally refining a pulp, which previously has been subjected to defibering with the physical separation from each other of the cellulosic fibers in the pulp, to produce a pulp mixture wherein discrete fibrils in the cellulosic fibers are in a partially freed state. As is recognized in the art, a cellulosic fiber is formed of multiple layers of fibrils bound together and surrounded by an outer lignin lamella. In the production of paper it is common to separate the cellulosic fibers in the chips or other fiber agglomerates, either mechanically, or chemically by dissolving the lamella and this physical separation of the fibers is known as defibering. To obtain improved properties in paper, more recently it has been proposed to subject an already defibered pulp to a process wherein the cellulosic fibers are rubbed against each other under pressure, in such a manner that the fibrils in a fiber are partially freed whereby they project out in fine hair-like strands from the fiber body without substantial cutting or fracturing in these fibrils. This freeing of fibrils in the fibers is known as fibrillation, and the process has resulted in signicant improvements in certain properties of the pulp and of paper formed therefrom, such as the freeness of the pulp, and such strength properties as burst, tensile, tear, stretch and toughness. Such improvements apparently can at least partially be explained by the greater interfiber adhesion resulting in papers produced from the pulp, made possible by the presence of the projecting nonfractured fibrils of the cellulosic fibers.

The production of pulps and the fibrillation of the cellulosic fibers in such pulps forms the subject matter of an application entitled "High Strength Papermaking Pulp, Paper and Process of Producing the Same," filed Feb. 25, 1964, having Ser. No. 347,231, abandoned in favor of Ser. No. 606,446 now U.S. Patent 3,382,140.

A pulp with fibrillated fibers is commonly prepared by

the steps of first producing a relatively high consistency defibered pulp, i.e., pulp containing separated cellulosic fibers. Ordinarily the consistency of the pulp is such that the pulp is best described as nonfluid or semisolid, and generally of a nonpumpable character. Such pulp, after its preparation, is introduced into a working space formed between two opposed surfaces which are in relatively high motion relative to each other. While between these surfaces the pulp is compressed by the surfaces and because of the pressure conditions and the relative motion by the surfaces, a considerable amount of energy is expended on the pulp. The pulp on passing between these surfaces has a motion imparted to it whereby the fibers of the defibered pulp are rubbed between themselves with freeing of the fibrils in a fiber, or fibrillation resulting. The pulp after fibrillation has approximately the same consistency and usually a temperature ranging upwardly from about 150° F. because of the work performed thereon.

While pulps of this description have been found to produce superior types of paper products when prepared and converted into paper without storage, difficulties have been encountered in obtaining comparable products where the pulp has been stored and used at some later date. This essentially precludes most advantageous use of the pulp when it is to be converted into paper at a mill site different from where the pulp was prepared.

A general object of this invention is to provide a novel process of preparing a defibered pulp having fibrillated fibers, which pulp may be stored for a period of time.

More specifically, an object of the invention is to provide a process for preparing a pulp with fibrillated fibers, wherein the apparent fibrillated state of the fibers may be maintained for a period of time after preparation.

Yet another object of the invention is to prepare a pulp with fibrillated fibers particularly adapted for shipping. In this regard, a pulp is contemplated having a relatively high consistency which by reason of reduced water content best lends itself for economical transport.

A still further object is to provide a novel process for making paper products with fibrillated cellulosic fibers, wherein such fibers are stored and shipped between their initial preparation and the making of the final paper product.

These and other objects and advantages are attained by the invention, which is described hereinbelow in conjunction with the accompanying drawing, which illustrates graphically certain properties of pulps and how such properties are affected with storage time.

As indicated briefly above, in a pulp process where there has been fibrillation of the cellulosic fibers, with the fibrils in a cellulosic body through a rubbing action and pressure being caused to separate so as to project from the body without cutting or fracturing in the fibrils, certain strength properties are significantly improved. The improvement in strength properties apparently comes about by reason of the closer knitting and interaction of the cellulosic fibers possible in final paper products. While such has been recognized, the desirable properties have not been produced in papers produced from pulps which have been stored for any period of time. Studies indicate that the pulp in some respects tends to regress, so that while certain properties may show permanent improvement by such fibrillation processing, many strength advantages soon are lost with storage.

Processed pulp with fibrillated fibers on leaving the refiner where fibrillation has been produced exists at a relatively high temperature because of the energy expended on the pulp by the process. Large volumes of pulp are processed in the usual paper mill, and in conventional storage facilities such pulp after fibrillation may retain the heat generated in the refiner for some period of time. This invention is bottomed on the discovery that several phe-

nomena occur during ordinary storage of fibrillated cellulosic fibers producing regression in the usual strength characteristics of paper produced from the fibers.

For one thing, at an elevated temperature, certain degradation occurs in the respective cellulosic fibers with deterioration of their fibrous structures. This degradation is particularly apparent in cellulosic pulps containing fibrillated fibers, since the fibrils in the cellulosic bodies are in a partially separated state which increases the vulnerability of the cellulosic bodies to deterioration.

The usual pulp becomes nonflowable or nonpumpable at a consistency of 6%. In pulps processed to obtain fibrillation of the fibers, pulps of a nonflowable consistency are used. Relatively high consistency pulps are refined, in order to obtain the necessary intimate contact between fibers as they are rubbed together. While fibrillation of aqueous pulp mixtures ranging in consistency from as low as 10% to as much as 60% has been indicated, best results are obtained with pulp consistencies ranging from about 20–35%. (As used herein, consistency percentages refer to percent on a dry weight basis of fiber in a pulp.) With such consistency and the temperatures produced by fibrillation, apparently two factors are involved which inhibit hydration of the cellulose fibers. With hydration inhibited, an environment results wherein the fibrils separated out by the fibrillation tend to collapse and reorient in bundle form in the respective cellulosic bodies.

Further explaining, the fibrils present in a cellulosic fiber have a molecular structure including hydroxyl groups carried in relatively exposed positions. Hydration of a cellulose fiber occurs by reason of water molecules attaching to such hydroxyl groups through hydrogen bonding. In a fiber which has been subjected to fibrillation, apparently it is important to obtain as much hydration of the cellulose fibers as possible, since attachment of such hydrogen bonded water molecules to the separated fibrils tends to inhibit the tendency of the fibrils to fall back on the cellulosic bodies rather than remain as strand-like projections extending from the bodies. With pulp refined to obtain fibrillation, the hydration of the fibrils is affected by the relatively high consistency at which the pulp persists, since with the reduced amount of water present there is less availability of water molecules for bonding to the hydroxyl groups. Further, hydration is a function of temperature and decreases as temperature increases. The relatively hot condition of the pulp after refining thus further inhibits the amount of hydration that can take place.

The above-identified application explains how a pulp may be refined to obtain fiber fibrillation. In general, pulp is introduced into a working space formed between two opposed roughened refiner surfaces which are in relatively high motion relative to each other, and which are spaced and maintained apart a distance not less and desirably greater than the thickness of individual fibers being treated. This spacing is maintained because in high consistency refining it is important that the roughened surfaces do not materially fracture, chop or cut the fibers. The surfaces act primarily to impart motion to the fibers where they lodge between the surfaces, with rubbing occurring between individual fibers whereby the fibrils are freed.

The refiner surfaces are operated at sufficient relative tangential velocity and sufficient power input to maintain the interfiber friction desired. The pulp moves rapidly and continuously in a single pass through the space between the refining surfaces, in a direction away from the point of their introduction toward the point of their discharge.

The relative movement between the two surfaces will vary depending upon the types of apparatus employed. The tangential velocity of the surfaces should desirably be at least 5,000 feet per minute, and in practising this invention preferably is 9,000 feet per minute or more. In the case where both surfaces are moving in opposite directions, the relative tangential velocity of at least 15,000 feet per minute is preferred. Under all circumstances the velocity should be sufficiently great for any given spacing between the surfaces as to impart sufficient energy to the fibers to effect attrition therebetween and the fibrillation essentially by the interfiber friction developed.

The power input used in fibrillation will vary in accordance with the type of pulp being processed and with the refined pulp qualities desired. For example, with the pulp under compression in the working space between the refiner surfaces of a refining machine, in the case of unbleached kraft pulp, the amount of power applied for a single passage of pulp between the refining surfaces should be in the range of between 10 and 60 horsepower days/air dry ton pulp, namely, the daily total horsepower required to produce one ton of pulp. Ground wood pulp requires a power input also in the range of between 10 and 60 horsepower days/air dry ton of pulp. Sulfite pulp requires a lower power input, between 3 and 30 horsepower days/air dry ton of pulp, because of being comprised of a softer fiber and requiring less energy input. Chemical pulps derived from fibrous nonwoody plants, such as bagasse, require still relatively lower power inputs, in the range of 1 to 20 horsepower days/air dry ton of pulp.

In performing the refining operation a double disc refiner may be employed such as the one known in the industry as the Bauer 411 refiner. This refiner is essentially the same in principle as the one disclosed in U.S. Patents Nos. 2,214,707 and 2,568,783. A particular type of Bauer 411 refiner, such as may be employed in this invention and the way the same may be operated in producing fibrillation of fibers in a defibered pulp, is described in the aforementioned patent application.

Although the operating pressure of the refining surfaces on the pulp fibers may vary, in general, in the Bauer 411, a pressure of between 5 to 20 pounds per square inch will be sufficient to produce a pulp of desired properties.

The effect of storage on the strength properties of paper produced from pulp processed to obtain fiber fibrillation is demonstrated by the following example.

It will be noted that all tests on pulps produced herein were performed using standard TAPPI methods, except that pulp hand sheets were air dried between blotters without being contacted with the conventional polished metal plates in order to simulate more closely the drying conditions of a paper machine where some shrinkage of paper webs occurs.

EXAMPLE I

An unbleached kraft pulp produced by the conventional kraft process from Douglas fir chips was prepared. This pulp was then dewatered in a conventional screw press to obtain a consistency of 33%. The pulp after dewatering was refined to produce fibrillation of its fibers, by metering it into the center or eye of a double disc Bauer 411 refiner. The refining discs providing the opposed refiner surfaces in the refiner were mounted to have an initial clearance of about 0.007 inch. The relative tangential velocity (peripheral velocity) of the two discs was 24,000 feet per minute. The compression exerted by the discs or plates on the pulp was about 10 pounds per square inch. The power usage for a single pass of the pulp between the disc surfaces of the refiner was about 20 horsepower days/air dry ton of pulp. After a pass through the refiner the pulp had a consistency which was essentially the consistency of the pulp before processing.

A 250 pound batch of pulp was processed in this manner. The initial temperature of the pulp before being processed in the Bauer 411 was 75° F., and after processing, by reason of the work performed on the pulp, the temperature of the pulp had risen to 182° F.

As the result of the refining, and because of interfiber

friction produced by rubbing of the fibers, fibrillation of the fibers as described above was produced. A sample of this pulp coming from the refiner was diluted with water to a usual papermaking consistency of 1.5%, and made into paper. The following table sets forth certain strength properties found to be possessed by such paper.

Table I

Bursting strength, percent (Mullen)	58.4
Tensile strength, lbs. per ½ inch, (Instron tester)	6.4
Rupture energy, ft. lb./ft.^{2}	7.2

Pulp coming from the refiner was stored for a period of 24 hours. At intervals during this storage time, the temperature of the pulp was recorded, and samples removed and converted into paper. The paper produced was tested for bursting strength, tensile strength and rupture. The following table summarizes the results obtained in these tests for the samples obtained.

TABLE II

Sample	1	2	3	4	5
Pulp temperature (° F.) when sample removed	179	169	162	166	145
Storage time when sample removed (hr.)	1	3	5	7.5	24
Bursting strength of paper, percent (Mullen)	46.5	42.3	41.8	41.7	41.7
Tensile strength of paper, lbs. per ½ inch, (Instron tester)	5.4	5.1	5.0	4.8	4.6
Rupture energy of paper, ft. lb./ft.^{2}	6.1	5.5	5.5	5.7	5.4

From the above it will be seen that paper produced from pulp subjected to fiber fibrillation exhibits certain desirable strength properties. Under ordinary storage conditions, these properties are not maintained when the pulp is stored for any appreciable length of time. In fact, after a period of about three hours a loss in pulp strength of from 20–30% occurred. This is not to say that certain other properties, such as freeness in the pulp will be severely affected by a long storage time. It does demonstrate, however, that where the strengths indicated are desired, without special treatment, paper manufacture should commence without storage. This of course is a disadvantage if pulp is to be prepared at one location and then transported to another plant for ultimate processing into paper.

In the drawing, the data set forth in Tables I and II has been charted more clearly to present the deleterious effects resulting from ordinary storage.

The inventors have discovered that storage time has a minimal affect on strength properties if after treatment to produce fibrillation of the cellulose fibers the pulp is processed to inhibit degradation of the now more highly vulnerable separated fibrils, and most important, to produce conditions promoting hydration of the cellulose fibers by the attachment of water molecules to the hydroxyl groups contained in the molecular structure of the fiber fibrils, through hydrogen bonding. It has been discovered that these ends may be attained by introducing to the hot pulp mixture after fibrillation sufficient quantities of water to lower the consistency of the pulp preferably at least some 50%, i.e., from the preferred consistency at which fibrillation is performed (20–35%) to a consistency ranging downwardly from about 17%. Desirably a final diluted consistency of from 2–10% should be produced with the water addition, and best results are obtained when papermaking consistencies ranging downwardly from 6% are produced. The water used in the dilution should be cool water, at temperatures ranging downwardly from about 90° F. With such addition of cool water, hydration of the fibrillated fibers is promoted, and deterioration of loosened fibrils is inhibited.

During the addition of water, it is preferred that mixing take place accompanied with agitation of the pulp water mixture. This effects faster mixing of the cool water and faster cooling. More important, the agitation during dilution frees fibers from clotting into small fiber clumps. This clotting is preferably avoided, as when such occurs over a period of time some hardening of the clotted fibers is apparent which introduces difficulties in ultimately separating the clotted fibers before the preparation of paper.

With cool water added to the pulp, accompanied with agitation, pulp mixtures having initial temperatures in the range of 165–190° F. typically may have their temperatures reduced at least about 35° F., and usually considerably more.

The dilution of the high consistency pulp with water should best be done within about an hour after the refining of the pulp, and preferably immediately in order to minimize loss of strength properties in paper. The addition of the water may be done and preferably is performed continuously by introducing a stream of water to the pulp as such emerges as a continuous stream from the refiner. After the addition of water, however, and the completion of the refining, agitation of the water and pulp mixture preferably is carried on for a period ranging up to about four hours.

The following example illustrates the advantages to be obtained in immediately reducing after refining the consistency of a high consistency cellulosic pulp after fibrillation of the fibers, when such is to be stored for a period of time prior to making paper.

EXAMPLE II

A bleached kraft was prepared and dewatered in a screw press to 39% consistency. The pulp was passed in a single pass through a Bauer 411 refiner to produce fiber fibrillation, in the manner similar to that set forth in Example I. The temperature of the pulp on leaving the refiner was about 185° F. The power usage during refining was 34 horsepower days/air ton of pulp. The pulp on leaving the refiner at about 36% consistency was immediately mixed with cool water, at a temperature of about 60° F., in quantities sufficient to reduce the consistency of the pulp to about 1.5%. Water was introduced as a stream to the stream of pulp leaving the refiner, at a rate sufficient to produce continuously approximately the final consistency desired. With water dlution, a final temperature in the pulp of about 75° F. was produced. The pulp and water mixture was collected in a vessel, and subjected to agitation by circulating the contents of the vessel using a propeller stirrer. Two-hundred pounds of pulp were processed, and a flow rate of pulp through the refiner of some 2,400 pounds per hour maintained. The mechanical agitation (propeller stirring) was continued for about one hour after the termination of the refining step. Over a period of 76 hours after the preparation of the diluted pulp product, the pulp was made into paper. This paper was tested for different strength properties, and the results obtained are tabulated below. This data shows that storage of pulp at low consistency did not significantly alter pulp and paper properties.

TABLE III

	Storage time after refining hours				
	1.25	3.25	6	22	76
Bursting strength, percent (Mullen)	151	161	158	160	160
Tensile strength, lbs. per ½ inch (Instron tester)	11.7	12.1	12.0	11.5	12.6
Rupture energy, ft. lb./ft.^{2}	21.5	21.2	21.9	20.8	22.4

It has been further discovered that pulps processed to obtain fibrillation of the cellulose fibers, and then treated by the addition of water for the purpose above set forth, may subsequently be thickened to consistencies ranging up to 30%, and then stored or shipped, without severe impairment of the strength properties of paper produced from the pulp. This thickening step, of course, is advantageous with pulps to be shipped some distance, since it lowers the overall weight of the pulp for a given mass of cellulosic fiber. The ability of the pulp to be subsequently thickened after dilution, while papers made from

the pulp retain strength properties, apparently can be explained by the fact that the water removed in a subsequent thickening process is primarily free water, with minimum amounts of hydrogen bonded water and water contained within the fiber walls of the cellulosic fibers being removed. Thus, on removal of such water in the thickening step, the projecting fibrils of the cellulosic fibers are maintained in a condition wherein they project outwardly in hair-like strands from the cellulosic fiber bodies, in a condition promoting attachment and interaction with other fibrils to produce a strong final paper.

EXAMPLE III

Bleached kraft pulp was produced in a conventional kraft pulping and bleaching process from Douglas fir chips, and dewatered to a consistency of 40%. This pulp was then processed to produce fibrillation of the fibers therein by passing it through a Bauer 411 refiner at the rate of 3,000 pounds per hour, with the discs of said refiner having a clearance of about 0.005 inch. The relative tangential velocity of the two refining discs was 24,000 feet per minute. Power used was about 13 horsepower days/air dry ton of pulp. After passage through the refiner sufficient cool water at 70° F. was added to the pulp to lower the consistency thereof to 1.5% following the procedure of Example II. The temperature of the pulp after the addition of water in the completion of the refining step was approximately 75° F. Pulp was diverted from the refiner directly after refining and converted into paper. Paper prepared from such pulp when subjected to testing was found to exhibit the properties listed in the following table in the column labeled "Control."

TABLE IV

Property measured	Control	25% consistency	43% consistency
Bursting strength, percent (Mullen)	86	80	72
Tensile strength, lbs. per ½ inch, (Instron tester)	7.2	6.7	6.0
Rupture energy, ft. lb./ft.²	15.6	14.2	12.4

Samples of the pulp, after agitation in a collection vessel for a four hour period, were then thickened to consistencies of 25 and 43%, using a conventional screw press for dewatering purposes. After a storage period of 16 days the thickened pulp samples were diluted with water to a consistency of 0.5% and converted into paper of the same type as previously prepared. This paper was then tested to determine strength properties. Table IV shows that storage at high consistencies of 43% gave a 5–25% drop in sheet strength. However, lower storage consistencies of 25% gave minor strength losses of only 3–7%.

Thus, in thickening pulp after original dilution, it is preferable that the consistencies of the final pulp be not above about 30%, if strength properties are to be essentially maintained. With consistencies greater than this percentage, tight clumps ranging in size from approximately 1/32 to ¼ inch diameter tend to form, which gentle agitation after subsequent dilution will not break up. Such pulps, while having relatively good tear resistance when made into paper, do not retain maximum strength properties.

The process contemplated herein is applicable to all pulps treated mechanically for the purpose of producing fibrillation or freeing of the fiber fibrils. Such fibrillation of fibers is applicable to so-called mechanical types of pulps, wherein initial defibering is performed by mechanically grinding or otherwise treating cellulosic material such as wood chips. The process is also applicable to defibered pulps where defibering is a result of chemical action, as in the case of chemically treated pulp, or semichemical pulp.

It also should be readily apparent that the cellulosic material employed as a source of cellulosic fiber may be derived from the various species of coniferous pulp woods such as hemlock, spruce, fir, pine and others, as well as deciduous pulp woods such as poplar, birch, cottonwood, alder and others, and from such materials as the fibrous nonwoody plants suitable for papermaking exemplified by the cereal straws, cornstalks, etc.

From the above it should be apparent that a novel method is contemplated wherein high consistency pulps processed mechanically to produce interfiber rubbing and fibril loosening, may be stored over periods of time without appreciable loss of strength properties. The invention further contemplates a process for treating pulps which facilitates the shipment of these pulps as high consistency mixtures. Pulps according to the invention include those usable for making a wide variety of paper products such as wrappings, newsprint, etc.

While certain specific examples have been set forth in this specification for the purpose of illustrating and defining the present invention, by including such examples it is by no way intended to limit the proportions, conditions or equipment utilizable in practicing the invention. It is intended to cover herein all modifications and variations of the invention as would be apparent to one skilled in the art.

It is claimed and desired to secure by Letters Patent:

1. A process for preparing a fibrillated pulp from a fiber pulp of defibered separated cellulosic fibers suitable for storage, comprising providing a defibered pulp having a consistency of at least about 10% and in the form of a nonflowable mass with fibers in intimate contact, effecting fibrillation of the separated fibers in the pulp along their surfaces and while substantially maintaining the length of the fibers by continuously feeding the defibered pulp into one region of a working space bounded by opposed working surfaces disposed in facing relation and in relative rotational motion with respect to each other while continuously effecting discharge of fibrillated pulp from another region of the working space, said feeding being done at a rate to maintain the working space packed with fibers contiunously in motion and under compression and in intimate contact to provide a continuous path of pulp between the working surfaces and while applying pressure to the continuous path by such working surfaces with the surfaces apart a distance obviating substantial fracturing of the fibers, fiber fibrillation being produced within the working space by interfiber friction with the production of partially freed fibrils in a fiber extending out from the fiber as a hair-like strand, adding water to the fibrillated pulp discharged from said other region of the working space prior to the fibrils in a fiber falling back on the fiber and by such water addition hydrating the fibrils with water molecules attaching by hydration to the fibrils, such molecules acting to stabilize the fibrils whereby they remain as hair-like strands projecting from the fibers in the pulp and the pulp in this way being stabilized for storage to the extent that the fibrillated character of the fibers remains over a period of time.

2. The process of claim **1**, wherein the temperature and the amount of the water added is sufficient to produce a reduction in temperature in the pulp of at least about 35° F. which reduction in temperature promotes hydration of the fibrils in the fibrillated pulp.

3. Paper and related products produced from pulp prepared by the process of claim **1**.

4. The process of claim **1**, wherein the defibered pulp is fibrillated by feeding the defibered pulp continuously through a refiner, and water is added to the fibrillated pulp by adding water substantially continuously to the fibrillated pulp upon its leaving the refiner.

5. The process of claim **4**, wherein the consistency of the fibrillated pulp is reduced at least about 50% by the addition of water.

6. The process of claim **1**, wherein the fibrillated pulp

after addition of water to effect hydration of the separated fibrils is thickened, and the pulp mixture after thickening is subjected to storage.

7. The process of claim 1, wherein the fibrillated pulp after addition of water to effect hydration of the separated fibrils is thickened to a consistency of not greater than 30%, and the pulp after thickening is subjected to storage.

8. The process of claim 1, wherein fibrillation is carried out on a defibered pulp having a consistency ranging from 20 to 40%, and addition of water to the fibrillated pulp to effect hydration produces a final consistency of less than 10%.

9. The process of claim 8, wherein the defibered pulp is fibrillated by feeding the defibered pulp continuously through a refiner, and water is added substantially continuously to the fibrillated pulp leaving the refiner, and

after addition of water the fibrillated pulp is subjected to gentle agitation for a period of at least one hour.

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