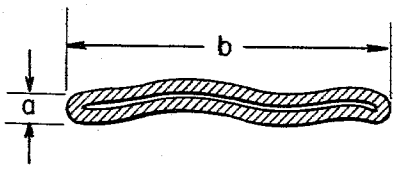
March 16, 1965 H. F. WISE ETAL 3,173,830

PAPER COMPRISING COLLAPSED REGENERATED CELLULOSE FIBERS

Filed June 16, 1959



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United States Patent Office 3,173,830 Patented Mar. 16, 1965

3,173,830

PAPER COMPRISING COLLAPSED REGENERATED CELLULOSE FIBERS

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Filed June 16, 1959, Ser. No. 820,602
11 Claims. (Cl. 162—146)

This invention relates to a new type of paper and to a new paper-making process.

Many proposals have been made over the years for introducing regenerated cellulose fibers into paper. In general, it has been felt that if regenerated cellulose fiber could be incorporated into paper, advantages could be obtained because the fiber could be tailor made as to staple length, denier and the like according to the properties desired in the paper.

In conventional paper made from natural cellulose, i.e. wood pulp, rags, and the like, the paper is bonded by the interlocking of fibrils which are produced when the stock is beaten. Unfortunately, regerenated cellulose fibers cannot be fibrillated by beating in the same way that natural cellulose can be, and so with regenerated cellulose fibers, some other source of bonding has to be employed.

It has been proposed to add various adhesives to the paper stock; such techniques, however, add considerably to the cost of the paper. It has also been proposed to treat a newly laid down paper sheet with caustic soda to cause softening and coalescence of the fibers. Treatments of this nature are, however, not well suited to incorporation in paper-making processes on conventional paper-making machines.

It has now been discovered that certain types of regenerated cellulose fibers have sufficient natural cohesion to form a strong bond without the addition of any external adhesive. Such fibers are characterized by a cross-section (taken in a direction substantially perpendicular to their axes) in which one dimension is from about five to about fifteen times the other. Preferably, they contain between about 1.5 and about 4.0% by weight of a finely divided inert particulate material. Such fibers can be used by themselves in conventional paper-making processes, without added adhesive, to give a smooth, strong sheet.

The invention, therefore, comprises a paper containing between about 10% and 100% by weight of regenerated cellulose fiber, each of said fibers having a cross-section in a plane perpendicular to its axis such that one dimension is from 5 to 15 times the other.

The invention further comprises in a method for making paper wherein a slurry of fibers is deposited on a surface and liquid is moved therefrom to form a paper sheet, the improvement which comprises forming said slurry at least in part from generated cellulose fibers having a cross-section in a plane perpendicular to its axis such that one dimension is between about 5 and about 15 times the other.

The drawing is a cross sectional view, greatly enlarged, of a regenerated cellulose fiber as used in the present invention, taken in a plane perpendicular to its axis.

The fibers which are used in the present invention are preferably collapsed hollow fibers made by extruding a viscose containing a suitable gas-forming agent and preferably also containing an inert particulate material, into a conventional spinning bath. As they are removed from the spinning bath the fibers are of tubular shape, but upon subsequent washing and drying, they collapse to give flat filaments.

More specifically, filaments having the dimensions required for the present invention may be made by extruding a conventional viscose containing 10–40% on the weight of the cellulose, of an alkali or alkaline earth metal carbonate or bicarbonate into an acid spin bath. Thus, for example, suitable fibers can be made by extruding a viscose containing 6–10% by weight cellulose, 5–7% NaOH, 1–4% sodium carbonate, having a total sulfur content of say 1.5 to 3 and a salt number of 5.0 to 7.0 into a spinning containing 11.5–12.5% by weight H_2SO_4, 0.5 to 2% $ZnSO_4$ and 18–25% Na_2SO_4. Preferably the viscose will also contain from about 0.15% to about 0.40% of an inert particulate material.

After emerging from the spinning bath, the fibers may be cut up to form a blanket of staple fibers and then subjected in the form of a blanket to the various washing treatments customarily given regenerated cellulose fibers, such as a regenerating treatment with dilute acid, a desulfurizing treatment with Na_2S and a souring treatment, again with dilute acid. Following this the blanket may be dried and stored; or it may be sent directly to a paper-making plant, where it can be opened by jets of water to form a suitably slurry.

Instead of being treated as a blanket of cut staple, the fibers may be treated in the form of a tow with the various washes outlined above; and cut up into staple at some later time.

Thus, the fiber on coming from the spinning bath may be laid down as a tow in a plaited pattern on a moving belt, and subjected to the various washes in that form. After treatment the tow may be dried and stored. It may be furnished to the paper-making plant as tow; or it may be cut up into staple fiber beforehand.

The fiber as used preferably has a denier of about 1.5 to about 8 and is from say ⅛″ to 1½″, preferably from ¼″ to ½″ long.

In the drawing a fiber of the type used in the invention is seen in cross section. It will be observed that its width, i.e. the dimension "*b*," is from 5 to 15 times its thickness, i.e. the dimension "*a*."

The inert particulate material used in the present invention may be virtually any solid material which is inert with respect to the viscose and the spinning bath. Titanium dioxide is preferred, but alumina, silica, carbon black, and various pigments may also be used. The role of the inert material is twofold. In the first place it aids in forming fibers which are evenly inflated or tubular along their length by providing nuclei for the formation of gas bubbles. This function of the solid particles has been used hitherto in making inflated fibers. However, were the solid used only as an aid in gas formation, a much smaller amount, say 0.1% on the weight of cellulose, would be sufficient. The second function of the solid material is to raise fiber to fiber friction. The means by which this is accomplished is not known, but at least 1% on the weight of cellulose appears to be necessary for an appreciable effect to be obtained. There is no great advantage in using more than about 4%. The average particle size of the material should be not greater than about 1 micron and, preferably, particles above 5 microns in size should be absent.

Paper according to the present invention may consist

wholly of regenerated cellulose fibers or it may consist partly of regenerated cellulose fibers and partly of conventional natural cellulose fibers, e.g. paper pulp.

Besides simple blends of the special regenerated cellulose fibers referred to above with conventional cellulose fibers, other materials, cellulosic and non-cellulosic, may be used in papers according to the invention. Thus, for example, the invention includes blends of the special regenerated cellulose fibers with nylons, acrylic fibers, polyester fibers; cellulose acetate and other cellulose organic ester fibers. Such blends may or may not include conventional cellulose paper-making fibers. Normally at least 10% regenerated cellulose fibers are required to appreciably affect the characteristics of the final product.

In making paper according to the invention, the regenerated cellulose staple fiber need not be subjected to the prolonged beating that is required for conventional, natural paper-making fibers, since the cohesion of the novel paper does not depend on fibrillation, which is one of the more important effects of beating conventional fibers. It is desirable, however, to create an even slurry of the fibers for use in paper-making machines and one way to achieve this effect is to run the fibers through a refiner or Jordan engine, as a slurry containing from say .1 to 5.0% by weight fibers. Other mixing devices may be used; for example, in making hand sheets for test purposes, a Waring Blendor and a Tappi Standard Disintegrator have both been used successfully.

In many instances blends of the regenerated cellulose fiber and conventional paper-making fibers are desirable. In preparing such blends, the conventional fibers may be beaten in a beater to the desired freeness (say 500 Canadian) and then the regenerated cellulose component added. After circulating through the beater just enough to disperse large concentrations of regenerated cellulose fiber, the mixture may be sent to a Jordan for further mixing and for further refining of the natural paper. The fiber concentration in the beater (after addition of regenerated cellulose) is about 1.0 to 6.0%. In the Jordan it is about one-half that.

When the fiber has been dispersed as a slurry, it is formed into a paper by any conventional method. In the laboratory, test sheets are made from a slurry having a fiber concentration of say 0.05 to 0.2% on a Standard British Sheet Mold. Usually, following Tappi Standard T205 M–53, a concentration of 0.15% is used. For large scale production, any conventional machine of the Fourdrinier type may be used. In such machines an aqueous slurry containing say 0.1 to 1.0% fiber is formed in the head box of the machine and is then poured onto a moving screen where the water is sucked off to form the paper. The paper may be pressed and dried as desired in accordance with conventional practice.

If desired, conventional sizes may be added to the fiber at any stage in the process, from the beater to the head box of the paper-making machine. Alternatively, sizes may be applied to the paper after it has been formed. The character and quantity of the size depends on the use to which the paper is to be put, as will be understood by those skilled in the art.

Paper made according to the invention is in general less dense and more porous than comparable paper made from paper pulp. It has, in general, lower tensile strength but greater tear strength, greater fold strength and greater bursting strength than similar papers made from wood pulp.

The invention will be further described with reference to the following specific examples which, it will be understood, are given for purposes of illustration only and are not to be taken as in any way restricting the invention beyond the scope of the appended claims.

EXAMPLE I

A viscose is prepared having the following characteristics:

Cellulose	percent by weight	7.5
NaOH	do	6.0
Na_2CO_3	do	2.5
Total sulfur	do	2.1
Salt index	do	5.0

This is spun into a spinning bath having a temperature of about 48° C. and the following composition (Wt. %):

H_2SO_4	12.0
$ZnSO_4$	1.25
Na_2SO_4	21.25
Water	balance

Upon emerging from the bath the fibers are cut up to form a blanket, the size of the individual fibers ranging from 3/16 to 5/16 inch, and then subjected to conventional regenerating and desulfurizing treatments. Specifically, the fibers are first washed with 2% H_2SO_4 at 70° C. for regeneration, then given a neutral rinse, followed by a desulfurization wash with 0.2% Na_2S at about 60° C., and another neutral wash to remove sulfide liquors. After this the fibers are given a sour wash with 0.3% H_2SO_4 at room temperature with a final neutral rinse at 70° C. The blanket is then dried and stored.

Upon examination it is found that the individual fibers are flat, deflated tubes each having a cross-section in a plane perpendicular to its axis such that on the average one dimension is about ten times the other.

The fibers made as above are used to make paper test sheets 6¼" in diameter on a British Standard Sheet Mold. Four different types of sheets are made up. In trial A the sheets are made solely from pine paper pulp (500 freeness). In trial B the sheets contain 75% by weight pine paper pulp (500 freeness) and 25% of the regenerated cellulose fiber. In trial C the sheets consist of 50% regenerated cellulose fiber and 50% pine paper pulp (500 freeness). In trial D the sheets are 100% regenerated cellulose.

In making the sheets the regenerated cellulose fiber is opened by jets of water to form a slurry. This slurry is then put into a standard Tappi disintegrator with conventional pulp as appropriate and sufficient water to bring the fiber concentration to about 1.75% by weight. The disintegrator is run for 300 counts to form a smooth dispersion. This dispersion is diluted to 0.15% by weight and the sheets are made on a Standard British Sheet Mold. They are given a first pressing of 50 p.s.i. for five minutes. They are then reversed on a dry blotter and repressed at 50 p.s.i. for two minutes. Finally, they are dried at 50% relative humidity and 23° C. (73° F.) until equilibrium is reached with the conditioning atmosphere. This procedure is in accordance with Tappi Standard T220 M–53. The sheets are then tested for tear factor, porosity (air), tensile strength, breaking length, stretch and fold strength. The results appear in Table I below:

Table I

	Sample			
	A	B	C	D
Percent Rayon (by weight)	0	25	50	100
Percent Pulp (by weight)	100	75	50	0
Basis Weight Oven Dry (Weight in pounds of 500 sheets 24" x 40")	43.0	42.6	42.1	44.5
Caliper (Thickness in inches x 0.001)	3.7	3.8	4.0	5.4
Apparent Density (Basis Weight/Caliper	11.6	11.2	10.5	8.2
Tear Factor	123	142	189	265
Air Porosity (sec./100 cc.)	19	9	4	0
Tensile Strength (lbs./0.5" width)	14.3	12.2	9.5	4.7
Breaking Length (meters)	8,445	7,270	5,700	2,680
Stretch (percent)	3.1	3.7	3.7	1.4
Fold (MIT)	909	714	706	644

Details of the tear factor test are found in Tappi Standards T414 M–49; of the porosity test in Tappi Standards T460 M–49; of the test for tensile strength in Tappi Standards T404 M–50; of the breaking length test in Tappi Standards T404 M–50; of the stretch test in Tappi

Standards T457 M–46; and of the fold strength test in Tappi Standards T423 M–50.

EXAMPLE II

Example I is repeated except that the tow emerging from the spinning bath is subjected to the wash treatments in that form. After drying it is cut by hand into staple fibers exactly ¼" long. These fibers are then used to make up two types of test sheets using the procedure of Example I. Type A contains 25% regenerated cellulose and 75% pine paper pulp (500 freeness). Type B is a 50–50 blend of the same stocks. Test results are shown in Table II.

Table II

	Sample	
	A	B
Percent Rayon (by weight)	25	50
Percent Pulp (by weight)	75	50
Basis Weight Oven Dry	42.6	40.6
Caliper	4.7	5.5
Apparent density	9.1	7.4
Tear Factor	231	438
Air Porosity (Sec./100 cc.)	3	1
Tensile Strength (lbs./½" width)	10.2	6.5
Breaking Length (meters)	6,080	4,080
Stretch (percent)	3.0	2.3
Fold Strength (MIT)	548	1,047

EXAMPLE III

The procedure of Example I is repeated except that the viscose has the following composition:

Cellulose	percent by weight	7.5
NaOH	do	6.0
Na_2CO_3	do	2.5
TiO_2	do	0.19
Total sulfur	do	2.1
Salt index		6.0

The staple fibers after washing and drying are found to have a cross-section in which, on the average, one dimension is 12 times the other.

Hand sheets are made up as in Example I. Sheet A is 100% pine pulp (500 freeness), sheet B 25% regenerated cellulose and 75% pine pulp, sheet C 50% regenerated cellulose and 50% pine pulp, and sheet D 100% regenerated cellulose. The characteristics of these sheets are shown in Table III.

Table III

	Sample			
	A	B	C	D
Percent Rayon (by weight)	0	25	50	100
Percent Pulp (by weight)	100	75	50	0
Basis Weight Oven Dry	40.8	42.1	47.9	44.3
Caliper	3.9	4.2	4.6	4.3
Apparent Density	10.5	10.1	10.4	10.4
Tear Factor	137	123	153	348
Air Porosity (sec./100 cc.)	11	18	18	10
Tensile Strength (lbs./½" width	12.7	11.6	10.6	6.1
Breaking length (meters)	7,920	7,000	5,620	3,473
Stretch (percent)	3.1	3.8	3.5	2.0
Fold Strength (MIT)	403	713	685	762

EXAMPLE IV

To show the difference between fibers according to the invention and conventional rayon, sheets are made up with varying proportions of normal textile grade rayon staple fiber and pine pulp. Sample A has 10% rayon, sheet B 25% rayon and sheet C 50% rayon. Sheets made from 100% rayon are so weak that they cannot be tested. The results of tests on the other sheets are shown in Table IV.

Table IV

	Sample		
	A	B	C
Percent Rayon (by weight)	10	25	50
Percent Pulp (by weight)	90	75	50
Fold Strength (MIT)	699	333	50
Mullen Bursting Strength	111	87	50

Sheets made according to the process of Example I have Mullen factors as follows:

90% pulp/10% rayon	543
75% pulp/25% rayon	560
50% pulp/50% rayon	584
100% rayon	644
100% pulp	523

What is claimed is:

1. Paper comprising regenerated cellulose fibers, each of which has a cross-section in a plane perpendicular to its axis such that one dimension is between about 5 and about 15 times the other.

2. The paper claimed in claim 1 wherein the regenerated cellulose fiber comprises between about 10% and 100% of the weight of the paper.

3. Paper comprising regenerated cellulose fibers, each of said fibers having a cross-section in a plane perpendicular to its axis such that one dimension is between about 5 and about 15 times the other and containing between about 1.5% and about 4.0% by weight of an inert particulate material.

4. The paper claimed in claim 3 wherein the regenerated cellulose fiber comprises between about 10% and 100% of the weight of the paper.

5. Paper comprising regenerated cellulose staple fibers, each of said fibers being in the shape of a collapsed tube and having a cross-section in a plane perpendicular to its axis such that, on the average, one dimension is between about 5 and about 15 times the other.

6. Paper as claimed in claim 5 wherein the fiber contains from about 1.5% to about 4.0% by weight of an inert particulate material.

7. Paper as claimed in claim 6 wherein the inert particulate material is titanium dioxide.

8. Paper comprising wood pulp and not less than 10% by weight regenerated cellulose fibers, each of said regenerated cellulose fibers having a cross section in a plane perpendicular to its axis such that one dimension of said cross-section is from 5 to 15 times the other.

9. Paper containing as its sole fibrous component, regenerated cellulose fibers each of which is characterized in having a width from 5 to 15 times its thickness and in containing from about 1.5% to about 4.0% by weight titanium dioxide.

10. Paper comprising regenerated cellulose fibers wherein a predominate portion of said fibers is composed of fibers, each of which has a cross-section in a plane perpendicular to its axis such that one dimension is between about 5 and about 15 times the other.

11. Paper comprising regenerated cellulose fibers which have a ratio of width to thickness of from about 5:1 to about 15:1.

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