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PREPARATION OF CELLULOSE PELLETS

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Fig. 2.

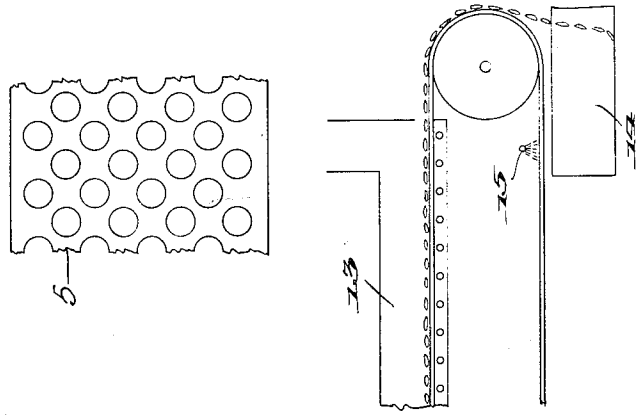
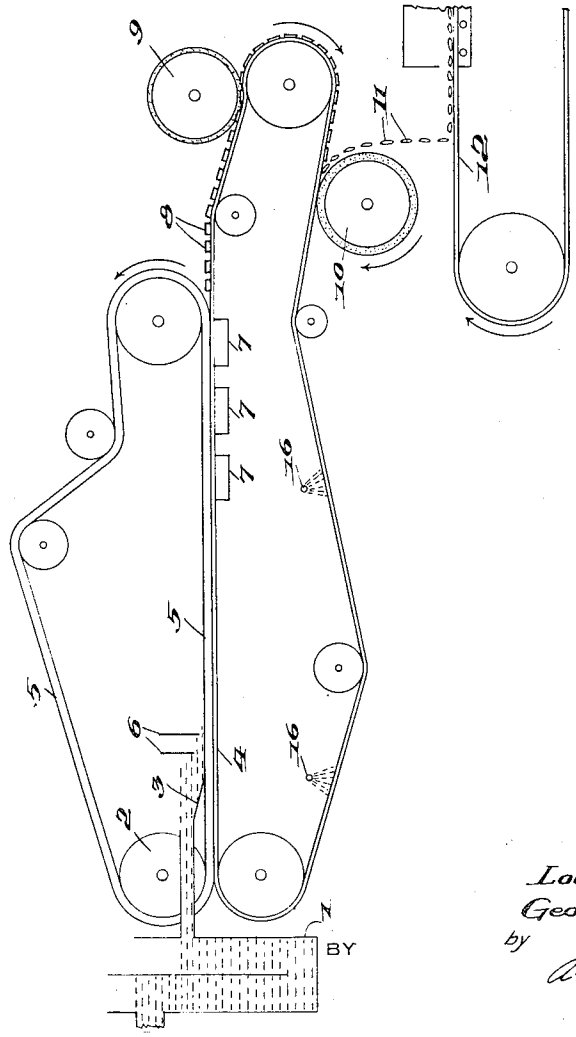


Fig. 1.



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PREPARATION OF CELLULOSE PELLETS

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2 Claims. (Cl. 92—54)

This invention relates to the preparation of cellulose fibers in suitable aggregates for conversion into cellulose derivatives, and more particularly to the preparation of cellulose pellets directly from cellulose pulp.

The present sources of cellulose for the preparation of cellulose derivatives are largely from cotton lint, wood fibers, and regenerated cellulose. The cotton lint or wood fibers are used in the form of a loose mass, in thick absorbent sheets, thin tissue, non-uniform aggregates of fibers mixed with free fibers or narrow strips cut into various lengths from dense or loosely compacted sheets. The particles of regenerated cellulose most effective for chemical treatment are small crumbled pieces made by cutting regenerated sheets and screening to uniform size. In all cases a great deal of manipulation is required to effect the aggregation into the final uniform particle size required. For example, lint or fibers from wood pulp must be beaten and then made into tissue paper which must then be shredded into small flakes. Or these fibers after sufficient beating are made into loose or dense thick sheets and then cut in two directions to make rectangular pieces. In one method, the moist fibers are passed between rotating discs which bruise some of them sufficiently to cause most of them to gather into lumps and short strings of various sizes. In each method the fibers require a great deal of mechanical treatment which cuts part of them into such short lengths that they are wasted in the subsequent processes.

An object of this invention is to prepare short cellulose fibers of a purity suitable for conversion into cellulose derivatives, into small aggregates of uniform sizes, degrees of density and tenacity within the requirement of the cellulose art in which they are to be used. Another object is to prepare these aggregates (pellets) without cutting, crushing or abrading individual fibers more than the requirement for the specific use. A further object is to effect this pellet formation directly from the slush pulp stage without going through the costly steps of sheet formation, drying and cutting. These and other objects will more clearly appear hereinafter.

We have found that cellulose fibers in the form of a slurry ordinarily obtained in a pulp mill, such as a slurry of 5–10% consistency, may be formed into pellets, after diluting the slurry to paper making consistency (0.5–1.0%), by running this slurry onto a wire forming screen covered by a suitable perforated belt, removing water from the slurry by suction, pressing out excess water with a resilient surface couch roll, rolling the patches of fibers into pellets with a resilient-surfaced roll, dropping the pellets onto a belt conveyor and drying them by passing them through a heated chamber.

As the starting material for our process we use the purified lint or wood pulp fibers as they come from the pulp mill after their last washing step, at which stage they are usually at a consistency of about 5–10%. At these consistencies they may be readily pumped from one process to another. We take pulp at this consistency and give it a beater treatment to obtain a smooth dispersion of the degree of hydration to give pellets of the bulk density required. If fairly bulky pellets are required, we merely add water to the dispersed pulp to bring it to the consistency required for good sheet formation, which in general is about 0.5% to about 1.0%. If dense pellets are required, we beat the pulp with the roll set to avoid cutting but to produce a certain degree of fibrillation and then dilute to paper making consistency. The result of this beating treatment is termed "hydration" in the paper industry. When it is advantageous to use pulp from some source in which it must be transported to the pelleting mechanism, the fibers may be obtained from "wet lap" or dry pulp. These pulps are converted to a slurry by beaters.

For forming the fibers into little patches on a forming wire, we may use any type of paper machine forming device such as a fourdrinier or cylinder. An apparatus suitable for carrying out our process is illustrated in the accompanying drawing wherein:

Fig. 1 is a side elevation showing the wet end of a Fourdrinier machine with special attachments and dryer system; and

Fig. 2 is a detail of a portion of the belt showing one pattern of perforations therein.

Referring to the drawing, Fig. 1, a preferred procedure is as follows: the pulp slurry from a beater or other source, as described in the examples below, at a consistency of about 5% is diluted by the recirculation of water from the Fourdrinier system to a consistency of about 0.5%. From the flow box 1 it passes through troughs or other suitable conduits around the end of the roll 2 where it is distributed by the apron 3 into the perforations of belt 5 and on the face of the wire forming screen 4. Belt 5 is made of flexible material such as rubber, it is from 1/16 to 1/4 inch thick and is perforated with holes of any size and shape. A suitable pattern of perforations, by way of example, is illus-

trated in Fig. 2. The slurry fills these perforations and passes beneath the slices or doctors 6 and over the suction boxes 7 which serve to remove the free water. After passing the last suction box the belt 5 rises from the wire forming screen 4 leaving on the screen platelets or patches of fibers, indicated at 8, as little discs of fiber felted into sheet form and separated from each other so that subsequent pressure does not cause their edges to coalesce. These pass under a couch roll 9 which should be padded with material such as felt or porous rubber, to remove excess water. After pressing they remain upon the wire 4 until it has reversed its direction and the patches are upside down, at which point they are gently pressed by a soft rubber padded roll 10 which is rotating in a direction reverse to the travel of the screen. This rubs the patches off the wire and at the same time rolls them into little cigar-shaped pellets 11. These pellets fall upon the moving conveyor belt 12 which carries them through the heated drying chamber 13 where they are dried to the required degree of dryness. From the drier the pellets are carried by the conveyor 12 to the collecting receptacle 14 and any adhering pellets are removed from the screen by air jet 15. The screen 4 is cleaned by means of the usual showers 16 which strike it after it passes the pelleting roll 10. The belt conveyor 12 should be made of canvas or wire screen in order to facilitate passage of heated air upward through the belt. The pellets may be dried to any degree required up to 95% dry fiber weight by simple passage through the heated chamber 13. However, if dried to only 50% dry fiber weight, they are strong enough for handling without undue coalescence. These pellets when dried to 95% dry fiber weight may be packed in containers by simple vibration settling to give compact masses.

The following examples further illustrate the invention.

Example I

A bleached sulfite pulp containing 95% alpha-cellulose was dispersed in a Hollander type beater with enough water to form a 5% consistency. The roll was set so there was neither cutting nor fibrillation. When all lumps of fiber had disappeared the freeness was 124 seconds on the freeness tester described in U. S. Patent 1,857,100. The pulp was diluted with water at 25° C. to a 0.5% consistency. It was then poured into a sheet mold equipped with a rubber belt perforated with ¼ inch circular holes spaced ½ inch center to center. After suction to remove free water the perforated belt was removed and the patches were rolled into pellets with a piece of porous rubber. The pellets were dried in an oven to 95% dry fiber weight. They were from $\frac{4}{128}$ to $\frac{5}{128}$ inch in diameter and from $\frac{32}{128}$ to $\frac{40}{128}$ inch in length. These pellets were packed in a container by vibration and under these conditions they weighed 6.8 pounds per cubic foot.

Example II

Using the same pulp as in Example I the beating was continued with the roll in a position to produce some fibrillation but with a minimum of cutting. When the freeness was 260 seconds, the pulp was diluted with water and formed under conditions as in Example I so that the weight per pellet and moisture content would be the same. These pellets were from $\frac{9}{128}$ inch to $\frac{8}{128}$ inch in diameter and from $\frac{33}{128}$ to $\frac{41}{128}$ inch long. These pellets, packed in a container by vibration, weighed 11.0 pounds per cubic foot.

Example III

A high alpha-cellulose mercerized sulfite pulp was treated in the beater as in Example I in order to obtain the same weight per pellet and the same moisture content in the dried product, but the beater treatment was stopped when the pulp had a freeness of 46 seconds. The pellets were from $\frac{9}{128}$ inch to $\frac{12}{128}$ inch in diameter and from $\frac{33}{128}$ to $\frac{41}{128}$ inch long.

Cellulose fiber pellets prepared according to our process may be used for the manufacture of cellulose nitrate, cellulose acetate and propionate, cellulose ethers, viscose, etc. Nitration, acetylation and etherification of cellulose, for instance, using the pellets of our invention can be effected as follows:

Example IV

The pellets described in the above examples may be nitrated by immersing them in a nitrating mixture containing from 30 to 40% nitric acid content for a period from thirty to sixty minutes at a temperature from 40° to 50° C. As can be seen from the above examples, the size and bulk density of these pellets will vary with the conditions of preparation as well as with the type of pulp being processed. In order to use a nitrating acid mixture containing a low nitric acid content, e. g. 30%, pellets of low bulk density should be used, while with denser pellets it may be necessary to use a nitrating acid containing 40% or more nitric acid content, the requirement being that the nitric concentration be high enough to avoid excessive gelatinization of the fiber of the pellet used. The nitrogen content of the resulting nitrated pellets can be controlled, as is well known in the art, by controlling the sulfuric to water ratio in the nitrating acid mixture. It is desirable to agitate the pellets in the nitrating acid mixture throughout the nitration period. The nitration ratio, that is, the ratio of nitrating mixture to pellets, may be approximately 40 to 1; but if the density of the pellets is comparatively high it may be possible to obtain satisfactory nitration at a lower ratio, while if the density is low it may be desirable to resort to higher ratios in order to obtain satisfactory nitration.

Example V

One hundred (100) parts of air-dry cellulose pellets or agglomerates as prepared by the process of Example II, are treated with 500 parts of glacial acetic acid at a temperature of 20° C. for a period of two hours with constant shaking. Two (2) to 4 parts of sulfuric acid are added in an additional quantity of 40 parts of acetic acid and the agitation continued at a temperature of 20° C. for an additional period of two hours. A total of 225 parts of acetic anhydride are then added in small quantities over a period of one hour and the resulting mixture tumbled until a clear triacetate solution is obtained. Increasing the temperature to 40°–50° C. during the latter stages of acetylation accelerates greatly the rate of esterification. In order to prepare the acetone-soluble type of cellulose acetate, sufficient aqueous acetic acid of approximately 60–70% strength is added to the triacetate gum to destroy the excess anhydride and reduce the acetic acid content of the solution to approximately 95%. The resulting solution is then

allowed to stand until the desired degree of hydrolysis is obtained. The rate of hydrolysis can be accelerated by the addition of more sulfuric acid (8 to 10 parts) and by carrying out the reaction at higher temperatures (40° to 50° C.). The hydrolyzed cellulose acetate is precipitated by pouring into a large quantity of water, filtered, washed neutral, and dried. The resulting product prepared in this manner can be employed in any of the commercial uses for cellulose acetate such as in molded plastics, fibers, films, etc.

In addition to the above homogeneous procedure for preparing cellulose acetate, cellulose in the form of the above-described agglomerates is also particularly suited for acetylation by the heterogeneous process in which the reaction is carried out in a non-solvent type diluent such as benzene. It is also to be understood that cellulose in the above physical form can be employed in the preparation of other cellulose esters.

Example VI

One hundred (100) parts of cellulose pulp pellets or agglomerates, as by Example II above, 240 parts of sodium hydroxide, 120 parts of water, 300 parts of ethyl chloride, and 500 parts of benzene are charged into an autoclave, agitated and heated gradually to 150° C. over a period of two to three hours, and maintained at this temperature while continuing mixing for seven to eight hours. The reaction mixture consists of a solution of ethyl cellulose in benzene, ethyl alcohol and diethyl ether (the last two components are by-products), in which are suspended crystals of sodium chloride and excess caustic solution. The mixture is treated with water to dissolve the salt and then steam-distilled during agitation to remove the volatile solvents. The ethyl cellulose remains as an amorphous, granular mass suspended in the alkaline salt solution. It is purified by washing with water steeping in dilute acid to neutralize any trace of alkali and then washed to remove all traces of acid. The product after drying dissolves in benzene, toluene-alcohol, ethyl acetate and a number of other solvents. Films produced from the material show a high degree of clarity and toughness. The product is especially suited for the manufacture of molded plastics, film, transparent foils, lacquers, artificial leather, etc.

By varying the etherifying agent and the reaction conditions, other cellulose ethers may be produced. Using the cellulose pulp agglomerates, cellulose ethers may be produced by other modifications of the general etherification process. For example, alkali cellulose suitable for etherification or for xanthation may be produced by steeping the agglomerates in caustic solution and pressing to remove the excess liquor, and then etherified by treatment with etherifying agent. Also, alkali cellulose produced as described may be treated first with solid alkali and then, after mixing, with the etherifying agent.

This invention is applicable to the pelleting of any kind of fibrous cellulosic material that can be made into a slurry which will operate on a paper machine. For the longer fibers, such as linters, flax, ramie, rayon waste, etc., the perforations in the pattern felt must be of suitable dimension and, in general, should be larger than ¼ inch in diameter. The density of the pellet can be controlled by the choice of fibers, degree of beating, temperature of the water in the forming stage, pressure of the top couch roll, and pressure of the roll which forms the patches into pellets. By varying these factors and especially by varying the degree and severity of the beating, we may vary within a fairly wide range both the density of the individual pellet and the bulk density as defined by weight per cubic foot, which may vary from about 6 pounds to about 20 pounds or even higher. As is known in the art, the end point of the beating operation may be controlled by the freeness test and examples have been given wherein the beating was stopped at a freeness as low as 46 seconds and as high as 260 seconds. However, pulp outside this range may be used satisfactorily where exceptionally low or high bulk density of product is desired. Preferably the pulp has a freeness ranging from about 40 to about 300 seconds measured on the freeness tester described in U. S. Patent 1,857,100. The mean weight of the individual pellets (best stated as number per gram) may be calculated from the consistency of the pulp and the volume of belt perforation, and conversely the required magnitude of any one of these three factors may be calculated if the other two are known.

The kind of forming surface may be that of a Fourdrinier or cylinder machine.

The pattern belt may be of any flexible material such as a wire screen having a pattern made by filling in the meshes with water-insoluble material to form the patches on the screen, such pattern belt may in this case be the forming screen. It may be made from sheet metal with suitable perforations. It may be of various thicknesses depending on the amount of fibers to be deposited on a given area. A rubber composition belt of ⅛ inch thickness is suitable for most purposes.

The rolling of patches into the individual rolls or pellets may be done by any rubbing-rolling device that does not injure the screen. A soft rubber-covered roll is satisfactory for most purposes.

The shape of the pellets may be varied by varying the shape of the perforations in the flexible belt, by the position in respect to the direction of the pelleting roll, and by the thickness of the original patches of fibers deposited on the forming wire. Thus, spherical pellets may be made by rubbing an elliptical patch off the wire lengthwise provided the thickness of the patch is correct.

The drying process may be accomplished in any known manner for drying discrete bundles of cellulose fibers. Force drying by passage through a heated chamber on a porous endless belt is satisfactory for most purposes.

The most suitable degree of drying depends upon the use to which the pellets are to be put. For purposes of nitration, acetylation, etc., dryness of 95% dry fiber weight and above is desirable. For the preparation of low-substituted cellulose ethers such as alkali-soluble methyl cellulose, the pellets may be used at about 60% dry fiber weight.

One advantage of this process for preparing pellets from cellulose fibers is to increase the yields of derivatives because the shorter fibers are held intact within the individual pellets during the process step required to make cellulose derivatives. Another advantage is that the size, shape and density of these pellets can be controlled so that wetting by reagent is rapid and uniform. These pellets may be handled more

easily than other forms of cellulose fibers with very low loses due to dusting. The method of pelleting does not cut the fibers. One of the most important advantages is in the directness of the process from the pulp purification process to the pellet stage which utilizes pulp in a form of lowest known cost (slush pulp) and avoids the sheeting and cutting steps required by previous processes.

The above description is for purposes of illustration only, it being understood that obvious variations and modifications falling within the spirit of the invention as defined in the appended claims are to be included within the scope thereof.

We claim:

1. A process of preparing directly from a cellulose-containing fibrous pulp slurry, small pellets of cellulose of uniform size and density, which comprises feeding a pulp slurry of paper making consistency onto a forming screen in the form of uniformly sized platelets or patches, removing free liquid from the pulp slurry by suction through said forming screen, removing the platelets or patches from the forming screen by a rubbing and rolling action exerted by a surface moving in a direction directly opposed to that of the screen whereby the platelets or patches are rolled into pellets, and drying the pellets.

2. A process of preparing directly from a cellulose-containing fibrous pulp slurry, small pellets of cellulose of uniform size and density, which comprises feeding a pulp slurry having a freeness within the range of about 40 seconds to about 300 seconds onto a forming screen in the form of uniformly sized platelets or patches, removing free liquid from the pulp slurry by suction through said forming screen, removing the platelets or patches from the forming screen by a rubbing and rolling action exerted by a surface moving in a direction directly opposed to that of the screen whereby the platelets or patches are rolled into pellets, and drying the pellets.

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