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D. J. KRIDEL

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PREPARATION OF CHEMICAL CELLULOSE FOR ESTERIFICATION

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FIG. 1.

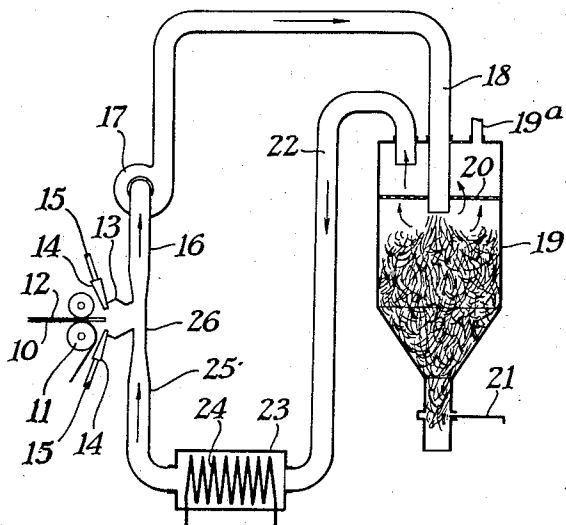
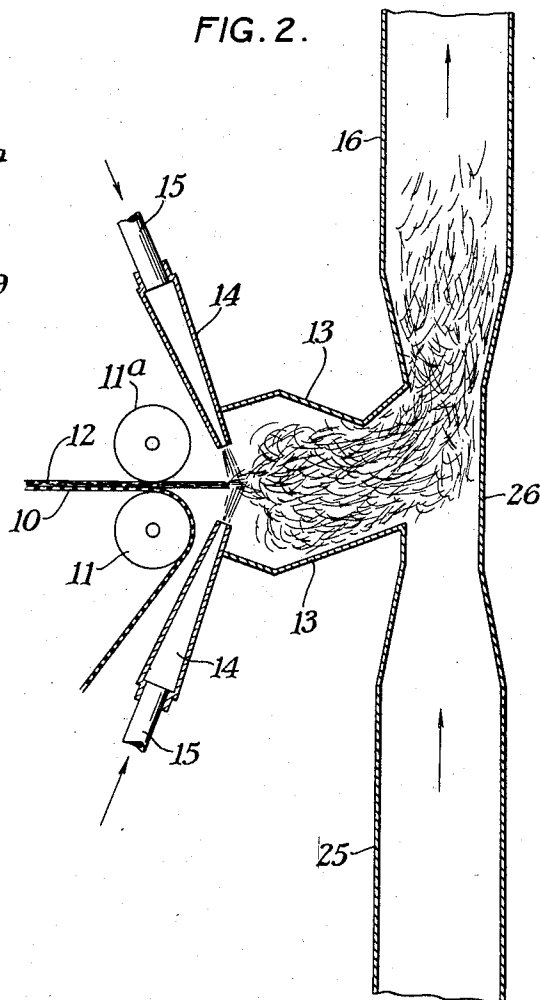


FIG. 2.



DONALD J. KRIDEL

INVENTOR

BY *H. M. Perrino*
Daniel J. Mayne
ATTORNEYS

UNITED STATES PATENT OFFICE

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PREPARATION OF CHEMICAL CELLULOSE
FOR ESTERIFICATION

Donald J. Kridel, Rochester, N. Y., assignor to
Eastman Kodak Company, Rochester, N. Y., a
corporation of New Jersey

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2 Claims. (Cl. 241-1)

This invention relates to the fluffing and drying of cellulose prior to its nitration by subjecting the cellulose in wet form to a violent blast of compressed gas, such as compressed air, followed by subjecting to a current of drying gases to remove the moisture therefrom.

Ordinarily in the manufacture of chemical cellulose, for instance, in the making of wood pulp, the material is dried while in a compact web form. The material is ordinarily produced in sheet or board form, the fibers being in contact with one another while in a highly plastic condition during the drying operation, consequently the cellulose fibers are firmly bonded together in the final product. In the preparation of refined cotton linters, the linters are obtained in a wet condition and are squeezed to the lowest possible water content and then dried. By either method lumps or aggregates are obtained which usually necessitates extensive picking or shredding operations to assure satisfactory yield and uniformity upon esterification.

Various methods of disintegrating the dried cellulose have been proposed. Although cellulose has been fluffed by these methods, ordinarily they result in considerable fracture of the individual fiber. A non-uniform product is usually obtained and dustiness is encountered. Numerous tearing and shredding patents evidence the interest in the disintegration of dried cellulose which has been manifested in recent years.

Ordinarily cellulose nitration processes are made up of a combination of an esterification and a hydrolysis operation carried out simultaneously. Both of these operations are very dependent as to velocity and degree of completion upon the water content of the nitrating acid in the reaction zone. A change of as little as 0.1% in water content will noticeably change the nitrogen content of the ester and will also cause greater hydrolysis as evidenced by lower viscosity and yield of production.

Water is a by-product of the esterification reaction. If the water formed thereby is localized rather than diffusing from the reaction zone surrounding the cellulose aggregate or fiber into the main body of the acid, the increase in water content will cause excessive hydrolysis. This excessive hydrolysis may be avoided by one of two methods:

1. By increasing the nitric acid content of the nitrating acid, this hydrolysis effect may be alleviated. This is probably due to the cellulose nitrate being less susceptible to hydrolysis than is the unesterified cellulose. Apparently the increased nitric acid content of the acid speeds up the rate of esterification and the overall hydrolysis effect is diminished. This method is employed in nitrating chipped or cut wood cellulose sheets. The nitric acid employed in such nitrating

baths is often as high as 28% as compared to 16-22% ordinarily used in the nitration of cotton linters. The retained acid costs in these nitrations are consequently higher due to the relatively high value of nitric acid.

2. By separating the cellulose fibers from each other so as to eliminate mats and clusters, the cellulose is put into condition to facilitate the diffusion of the water of reaction out of the reaction zone into the main body of the acid. The ultimate point is that at which the cellulose exists entirely as separate fibers.

One object of my invention is to provide a method of forming a fluffed dry chemical cellulose which fits in well with present day pulp manufacture. Another object of my invention is to provide a method of forming a fluffed dry chemical cellulose which provides for drying of the pulp in separated condition avoiding matting or adherence of the fibers. A still further object of my invention is to provide cellulose in a form in which it can be nitrated uniformly and with nitrating acids of low nitric acid content. Other objects of my invention will appear herein.

I have found that if cellulose in a matted or aggregate form is made wet and then subjected to a violent blast of gas, such as a blast of compressed air, that the cellulose is blown up into separate fibers which pass through the highly plastic wet stage as individual fibers and cementing of the fibers does not occur during the drying operation. The wet fibers are in a form in which they can be carried along by a current of air such as in a drying operation and the resulting product is eminently suitable for use immediately in a nitrating process or it may be baled or compressed while still retaining its characteristics as a batch or mass of individual fibers and may be employed as desired.

As most pulp mills are set up to turn out the pulp in the form of a sheet or board, my process fits in well with present day practice in pulp manufacture. My fluffing process may be carried out directly upon the pulp board in the mill so that drying is unnecessary or it may be used upon the dry pulp sheet as supplied by the manufacturer, in the latter case wetting of the pulp sheet being necessary. Also if cotton linters are treated by my process either the linters may be fluffed in their wet conditions in the mill in which the linters are refined for use in esterification processes or the dried linters may be thoroughly wetted and run through a drying process in accordance with my invention.

The drawing illustrates apparatus which is very suitable for carrying out my invention. Fig. 1 is a flow sheet of a combination of apparatus in which the cellulose is fluffed and carried by drying gases into a bin or storage compartment. Fig. 2 is a representation in section of the portion

of the apparatus where the wet cellulose is subjected to a blast of air or other inert gas to blow it up or cause the fibers to separate from each other. The drawing illustrates the preferred manner of subjecting the wet cellulose to a blast of gas which involves subjecting the cellulose to the action of two impinging jets of air under high pressure. In the apparatus the cellulose such as in the form of a wet pulp sheet 12 is borne along by an endless conveyer 10 motivated by roll 11. A pressure roll 11a is also provided to assist in feeding the sheet into the apparatus. Pipes 15 supply jets 14 with compressed air or other compressed gas at high pressure, the jets being arranged so that they impinge with the wet cellulosic sheet introduced at the line of intersection. In the apparatus shown the fluffing occurs in chamber 13 and the air stream introduces the fibers from this chamber into a current of conveying and drying air at point 26 which, as shown in the apparatus, is a Venturi constriction in the duct. A circulating fan 17 is present in the line and the fibers are introduced at the suction side of this fan. The fibers are circulated through conduits 16 and 18 and are deposited in the bin 19. Bin 19 is provided with a screen arrangement 20 to allow the air to escape from the fibers, some of the air escaping through the vent 19a and some being introduced into passage 22. The bin is also provided with an outlet at the bottom so that the fibers may be withdrawn by means of discharge valve 21. The air which is withdrawn from the bin by conduit 22 passes through a preheater 23 in which the air is heated by means of a steam coil 24. The air then passes through conduit 25 to Venturi point 26 where it takes a fresh supply of fluffed cellulose. The apparatus may be operated continuously, the rate at which it is running depending upon various factors such as the rate at which the cellulose is fed into the apparatus, the pressure of the impinging blasts and the rate of circulation of the drying gases through the apparatus. It is desirable that the air at 19a be vented as rapidly as the compressed air is taken in through the jets 14. In this way the moisture evaporated from the fibers is being continuously moved from the apparatus and the air, therefore, retains its drying properties. If desired, a drying unit may be inserted in conduit 22 so that the air heated in preheater 23 has little or no moisture therein. This, however, is not ordinarily necessary unless high speed operation of the apparatus is desirable.

It is desirable that the cellulose which is introduced be sufficiently wet so that it is in a soggy condition in order to give the best results. Obviously, however, this is a matter for the judgment of the individual operator and in some cases it may be that a lower moisture content would be desirable than in other cases.

The following example illustrates the preparing of cellulose nitrate employing my invention:

A sheet of refined wood pulp cellulose of good whiteness and having an α -cellulose content of 90% was moistened so as to have a water content of 60%. This material was then subjected to the action of a blast of air of approximately 50 lbs. pressure. The fluffed material is then blown into a collector by drying air. When the material had dried satisfactorily, it was subjected to nitration

by introducing it into a nitrating acid containing 20% nitric acid, 2.5% Na_2O_4 and 60% H_2SO_4 . A cellulose nitrate was prepared having a nitrogen content of 12.01% and a viscosity of 33 seconds. The yield was 94% of theoretical. The resulting product, after stabilization, was found to dissolve up giving a brilliant solution completely free of undissolved fibers and chits.

Various advantages reside in the use of the product prepared in accordance with my invention for preparing cellulose nitrate. These include

1. Lower nitric acid content nitrating agents may be used effecting a lower cost of retained acid costs and a saving in total acid usage. For instance, for nitrating the fluffed cellulose in accordance with my invention, nitrating acids may be employed having a nitric acid content of 16-22%. Any of the nitrating acids specified in Talbot Patent No. 2,118,275 may be employed for nitrating the cellulose material obtained as described.

2. A more uniform nitration is accomplished probably resulting in greater product strength. This appears to be due, among other things, to the uniform distribution of the nitrating acid throughout the cellulose.

3. Higher yields of nitrate are obtained than upon other cellulose starting materials with a nitrating acid having the same nitric acid content.

4. The resulting cellulose nitrate exhibits less viscosity lowering at a given temperature than is found to be true with other cellulose starting materials. This characteristic is an advantage in a process in which a high viscosity cellulose nitrate is desired such as in cellulose nitrate for munitions. Although the drying process described and illustrated herein fits in very well with the fluffed cellulose as it is easily carried along by a current of drying gas, other drying processes may be employed, particularly where the cellulose is permitted to remain in fluffed condition. Although my invention is particularly concerned with preparing cellulose for nitration processes, the resulting product is also opened up so that it may be employed in processes in which organic acid esters of cellulose are prepared.

I claim:

1. A method of converting wood pulp board to a form suitable for esterification which comprises subjecting the wood pulp board while containing at least 60% water directly to a violent blast of compressed air so as to cause blowing apart of the fibers of the cellulose pulp and while in the resulting highly plastic, blown-apart condition, conveying the pulp by means of drying air so as to remove the moisture therefrom and thereby assure the absence of compact lumps therein.

2. A method of converting chemical cellulose board to a form suitable for esterification which comprises subjecting the cellulose board while containing at least 60% water directly to a violent blast of inert gas so as to cause blowing apart of the fibers of the cellulose board and while in the resulting highly plastic, blown-apart condition, drying the fibers by means of drying gases so as to remove the moisture therefrom and thereby assure the absence of compact lumps therein.

DONALD J. KRIDEL.