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METHOD OF HANDLING VISCOUS CELLULOSIC MASSES

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The present invention relates to a method of transferring viscous or plastic masses, containing cellulosic material, in which a compatible liquid is introduced as a cushioning layer to lubricate its movement especially through restricted passages.

The prior art has recognized the difficulty of pumping viscous cellulosic masses and in some cases has attempted to overcome this difficulty. For instance, the use of a large excess of concentrated acetic acid in the acetylation of cellulose, has been suggested to assure a somewhat dilute form of the completed reaction mixture and thereby facilitate its handling. The dilution of a cellulose acetate reaction mass, in which cellulose has been acetylated and the resulting ester hydrolyzed, with sufficient precipitant, such as water, to bring the mass fairly close to precipitation, to facilitate passage of the material through a pipe has been suggested. In each of these cases there is dilution of the entire mass rather than mere formation of a cushioning layer on the outside so that a relatively large quantity of liquid must be moved considering the weight of cellulose ester which is transferred.

An object of the present invention is to provide a method of facilitating the movement of a viscous cellulosic mass without appreciably diluting the material being moved. Another object of my invention is to provide a method of facilitating the movement of the viscous mass, which increases the delivery capacity of the system employed and yet eases the strain on the pumping means which moves the viscous mass. A further object of my invention is to provide for the more rapid movement of the solution of a cellulose ester in a hydrolyzing bath after the desired hydrolysis has occurred so that the uneven hydrolysis of the cellulose ester, which would occur with the slow passage of the solution, is avoided.

I have found that cellulose ester dopes, solutions or pastes may be more readily moved by cushioning the viscous cellulosic material with a small amount of liquid which is compatible with the solution which is being pumped. I have found that by so introducing a liquid into the intake of a pump employed to move cellulose ester dope for example, a cushioning coating is formed about the viscous material which lubricates its movement through the passage into which it is pumped. My invention is primarily directed to the moving of cellulose ester dopes prior to their precipitation from the reaction and hydrolysis reagents. My invention is particularly adapted to the movement of dopes of high viscosity esters of cellulose which are ordinarily resistant to movement. The

rate at which these dopes may be moved is greatly increased by my invention, thus decreasing the time in which the dope is in transit and thereby aiding the uniformity of the product. My invention is particularly adapted to moving dopes or solutions of cellulose acetate, cellulose acetate propionate or cellulose acetate butyrate. For instance, my invention is particularly applicable to dopes of the type prepared in accordance with the disclosure of U. S. Patent No. 2,026,986 of Carl J. Malm in which high viscosity esters are prepared. However, even with the lower viscosity types of cellulose esters, my invention is applicable to facilitate the moving of such dopes.

My invention is also concerned with facilitating the movement of pastes of cellulosic material which are relatively stiff or heavy due to the small percentage of solvent which is present therein. In the case of pastes it is preferred to add the liquid to the paste in a mixer so that the paste is broken up into lumps or chunks having a very slippery outer surface.

In the pumping of cellulose ester dopes, a positive displacement pump, such as a triple cylinder, direct driven pump, is usually employed. In employing such a pump for a viscous solution, such as met with in cellulose ester work, there is a terrific strain upon the gears, chains and various other parts of the pump. A considerable amount of power is necessary to move such a dope by means of the pump. As the viscosity of the dope is high and a great amount of power is necessary to move it, the delivery capacity of a system employed to move the dope may be rather slow. By my invention the delivery capacity of the system is materially increased.

The proportion of liquid to dope may be up to as much as 100% based on the weight of the dope. It is preferred that the liquid be at least 5% based on the weight of the dope. The amount of liquid desirable will obviously depend on various factors such as the viscosity of the dope to be transferred, the restriction of the passages, the rate of flow desired and the effectiveness of the pump which is employed. The diluent cushioning layer markedly reduces the frictional resistance to flow of the dope or paste. The amount of reduction of resistance desired depends upon the conditions under which the viscous mass is to be moved and obviously for economic reasons the minimum proportion of liquid will be employed which gives the desired speed of flow.

The following example illustrates the moving of a cellulose ester dope in accordance with my invention:

6000 lbs. of dope consisting of cellulose acetate propionate in the form of a 14% solution in a slightly aqueous lower fatty acid which solution had a first stage viscosity of approximately 200 seconds was pumped by means of a triple cylinder, direct driven pump during which 500 lbs. of 50% aqueous acetic acid was drawn into the pump uniformly along with the 6000 lbs. of dope. The dilute acetic acid formed a cushioning coating or layer between the pipe and the dope and thereby lubricated the passage of the dope through the pipe. The time of pumping was only 50-60 min., whereas, if the acid had not been introduced into the intake of the pump, it would have required 3½-4 hours to pump the dope from the container into the vessel where the ester is to be precipitated from solution. Also, as the amount of additional liquid to lubricate the passage of the dope was small, it was not diluted to a great extent. The dope which was moved consisted of the cellulose ester dissolved in its hydrolysis bath. Therefore, as the ester was being moved along it was being hydrolyzed. If the rate of hydrolysis is sufficiently fast and a long time is required for pumping the dope, the ester will vary in the amount of hydrolysis to which it was subjected depending upon whether it belongs to the first or the last portion of the dope pumped through.

If desired, instead of introducing the cushioning material into the pump it may be introduced or bled into the passage thru which the dope passes preferably before movement thru a restricted passage is necessary. In my invention the cushioning material while compatible with the dope does not dissolve in the dope except around the outer surface thus forming a lubricating layer between the dope and the walls of the passage. In this way the movement of the dope is facilitated without increasing the amount of power used to move it.

As a modification of my invention, a solution of a cellulose ester or a paste thereof may be moved through pipes or through thin orifices by introducing into the paste a small amount of volatile solvent to form a cushioning layer on the surface of the mass. In cases involving extrusion processes in which heavy dopes or colloidized masses are extruded through thin orifices, a small amount of volatile solvent such as acetone, ethylene chloride, methyl ethyl ketone, ethyl or methyl alcohol, ethyl acetate or any of the solvents which are used in the preparation of the pastes may be applied to the surface of the mass just as it enters the restricted portion of the orifice. As a consequence, the paste or mass will be moved much more rapidly resulting in a higher rate of extrusion and economy in the amount of power necessary to move the mass along.

For example, a completely homogenized paste having the following composition:

100	parts of cellulose acetate
35	parts of plasticizer
25.8	parts of acetone
13	parts of ethyl acetate
9½	parts of ethyl alcohol
9½	parts of 1:4 dioxan

was mixed with 2-3% of acetone in a mixer resulting in breaking up of this very heavy viscous paste into lumps, about the size of an apple, having a very slippery outer surface caused by the addition of the acetone. These lumps were easily discharged from the mixer and allowed easy pas-

sage through screw conveyors, pipe lines, pumps, etc. The amount of diluent which it is desirable to use is limited up to about 50% of the paste only by the point at which precipitation of the cellulose ester occurs and by economic considerations. For instance if speed of movement is desired, the quantity of cushioning liquid employed may be relatively high.

Wherever the viscosity of a cellulose ester in its reaction mixture is referred to, it is the time in seconds in which a ½ inch glass bead will drop through 10 cm. of a solution at 25° C. of the reaction mixture in an equal amount of a mixture of glacial acetic acid and tetra-chloroethane.

I claim:—

1. The method of facilitating the pumping of a viscous mass comprising a solvent-soluble fatty acid ester of cellulose and a solvent therefor through a relatively restricted passage which comprises uniformly bleeding into the mass at a point near the beginning of the passage, 5-100% of a liquid of comparatively low viscosity, which is compatible with the solution of the cellulose ester, so as to form a lubricating layer between the mass and the walls of the passage, which will facilitate travel of the mass through the passage.

2. The method of facilitating the pumping of a viscous mass comprising a fatty acid ester of cellulose dissolved in its spent esterifying bath through a relatively restricted passage which comprises uniformly bleeding into the mass at a point near the beginning of the passage, 5-100% of a lower fatty acid, which is compatible with the solution of the cellulose ester, so as to form a lubricating layer between the solution and the walls of the passage, which will facilitate travel of the solution through the passage.

3. The method of facilitating the pumping of a viscous mass comprising a lower fatty acid ester of cellulose in solution in its spent reaction mixture through a relatively restricted passage which comprises uniformly bleeding into the mass at a point near the beginning of the passage, 5-100% of 50% aqueous acetic acid, which is compatible with the solution of the cellulose ester, so as to form a lubricating layer between the solution and the walls of the passage, which will facilitate travel of the mass through the passage.

4. The method of facilitating the pumping of a viscous mass comprising a cellulose acetate and a solvent therefor through a relatively restricted passage which comprises uniformly bleeding into the mass at a point near the beginning of the passage, 5-100% of a liquid of comparatively low viscosity, which is compatible with the solution of the cellulose ester, so as to form a lubricating layer between the mass and the walls of the passage, which will facilitate travel of the mass through the passage.

5. The method of facilitating the pumping of a viscous mass comprising cellulose acetate and a solvent therefor through a relatively restricted passage which comprises uniformly bleeding into the mass at a point near the beginning of the passage, 5-100% of a volatile solvent, which is compatible with the solution of the cellulose ester, so as to form a lubricating layer between the mass and the walls of the passage, which will facilitate travel of the mass through the passage.

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