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CONTINUOUS XANTHATION

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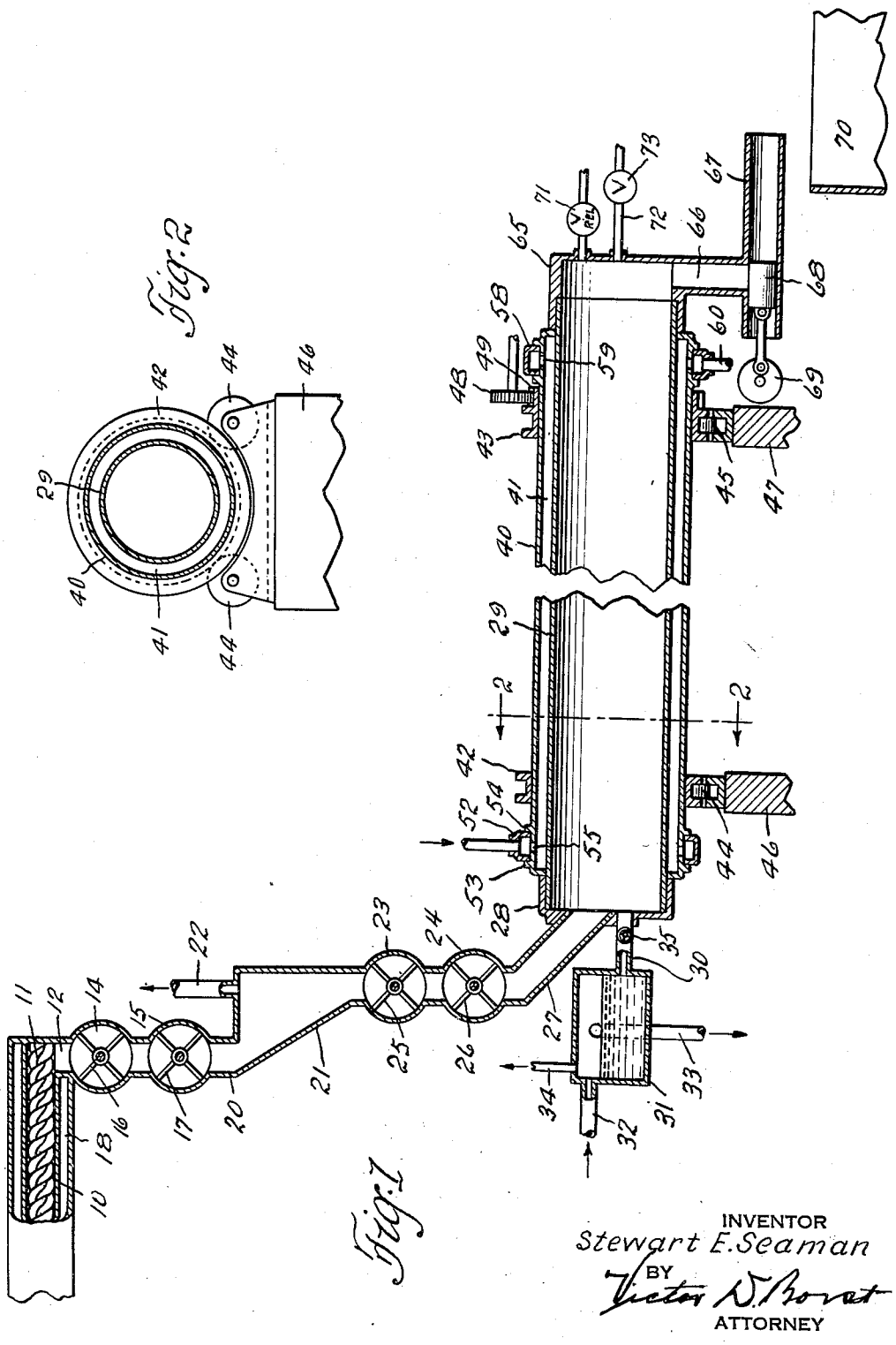


Fig. 2

Fig. 1

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CONTINUOUS XANTHATION

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7 Claims. (Cl. 260—216)

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This invention relates to the continuous xanthation of alkali cellulose in the manufacture of cellulose xanthates for viscose spinning solutions and has for an object to provide a novel and improved method and apparatus for carrying out the above steps on a commercial scale.

Another object is to provide a system for continuously reacting alkali cellulose with carbon bisulfide vapor to obtain a uniform product in a minimum reaction time.

Another object is to provide a system of the above type which may be so controlled as to substantially prevent the escape of carbon bisulfide vapor into the atmosphere.

Another object is to provide a system of the above type which may be operated continuously on a commercial scale with a minimum of manual control.

Various other objects and advantages will be apparent as the nature of the invention is more fully disclosed.

In accordance with the present invention, the shredded alkali cellulose in the form of a crumb or slurry is fed through a vacuum zone in which a reduced pressure is maintained so as to remove as much as possible of the air from the cellulose. The cellulose crumb or slurry is then fed continuously to a rotating reaction drum to which a controlled amount of carbon bisulfide liquid is supplied. The amount of carbon bisulfide is controlled with respect to temperature and pressure so that it vaporizes immediately in the reaction drum to maintain an atmosphere of carbon bisulfide vapor therein through which the cellulose crumb is tumbled as it is fed along in the rotating drum. When the crumb reaches the end of the drum the reaction is complete and the cellulose xanthate is removed through a vapor seal which prevents escape of the carbon bisulfide vapor. The cellulose xanthate thus formed is discharged into a dilute caustic bath or may be fed to a mixer or disintegrator or to a chamber for the evacuation of the carbon bisulfide from which it may be supplied to the mixer or disintegrator and is thereafter treated in the usual manner for the formation of the viscose spinning solution.

Although the novel features which are believed to be characteristic of this invention are pointed out more particularly in the claims appended hereto, the nature of the invention will be better understood by referring to the following description taken in connection with the accompanying drawing in which a specific embodiment thereof has been shown for purposes of illustration.

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In the drawing:

Fig. 1 is a diagrammatic representation of an apparatus for carrying out the process of the present invention; and

Fig. 2 is a transverse section through the rotating reaction drum taken on the line 2—2 of Fig. 1.

Referring to the drawing more in detail, the alkali cellulose which is received from a cellulose treating zone in the form of a comparatively dry crumb is fed through a pipe 10 by a rotating screw conveyor 11 and is discharged through a duct 12 through a pair of bell valves 14 and 15 in series. The bell valves 14 and 15 comprise rotating vanes 16 and 17 respectively which are continuously driven by any suitable means so as to feed the cellulose crumb downwardly while maintaining a vapor seal. The duct 10 is provided with a fluid jacket 18 through which a suitable fluid, such as water or gas, may be circulated for controlling the temperature of the cellulose crumb as it is advanced by the conveyor 11.

The bell valve 15 is shown as discharging the loosely descending crumb through a duct 20 to a chamber 21 in which a reduced pressure is maintained by suitable means indicated as an exhaust pipe 22 which may be connected to any suitable suction source such as a vacuum pump not shown.

The chamber 21 discharges the cellulose crumb through a pair of bell valves 23 and 24 containing rotating vanes 25 and 26 respectively, which are adapted to feed the crumb continuously while maintaining a vapor seal so as to hold the chamber 21 under a partial vacuum. The bell valve 24 discharges through a duct 27 to a stationary head 28 in which one end of a rotating reaction drum 29 is journalled.

Liquid carbon bisulfide is fed through the head 28 by means of a pipe 30 from a receiver 31 in which the carbon bisulfide is maintained at a fixed head by means of an inlet pipe 32 and a return pipe 33. A suitable vent pipe 34 may be provided for removing the carbon bisulfide vapor. The rate of feed of the liquid carbon bisulfide is controlled by a valve 35.

In the particular embodiment shown, the reaction drum 29 is shown as provided with an outer housing 40 which forms a fluid jacket 41 therearound for controlling the temperature of the reaction. The housing 40 is provided with spaced tracks 42 and 43 which are supported by rollers 44 and 45 respectively mounted on supports 46 and 47. The supports 46 and 47 are so

arranged that the reaction drum 29 rotates about an inclined axis so that material is fed along the drum by gravity as the reaction proceeds and may be made adjustable for varying the pitch of the drum as required for obtaining the desired rate of feed of the material. The drum is driven by suitable means, shown as a gear 48 engaging a rack 49 on the drum at a rate to effect the proper feed of the material. Fluid is supplied to the jacket 41 for heat control purposes through an inlet annulus 52 which is stationary and is sealed to the rotating drum by suitable means indicated as flanges 53 and 54. A plurality of apertures 55 communicate from the annulus 52 to the chamber 41. A similar annulus 58 communicating with discharge passages 59 in the shell 40 receives the fluid from the jacket 41 for discharge to a duct 60.

The lower end of the drum 29 is journalled for rotation in a head 65 with which a discharge duct 66 communicates. Suitable packing glands may be provided for making a fluid seal between the drum and the heads 26 and 65. The duct 66 feeds the reacted material to a cylinder 67 in which a piston 68 is mounted to be reciprocated by suitable means shown as a rotating crank 69. The piston 68 may be gas pervious to allow the carbon bisulfide vapors to escape from the compacted mass into the duct 66. If desired the piston may be designed to stop just short of closing the duct 66 so as to avoid sealing off the vapors. The arrangement of the piston 68 is such that at each reciprocation it pushes along the cylinder 67 the material which has fallen into the space ahead of the piston from the passage 66. This material is compacted in the cylinder 67 to provide a seal which prevents escape of the carbon bisulfide vapor from the end of the cylinder. The material is discharged from the end of the cylinder 67 into a suitable receiver 70. A relief valve 71 may be included to prevent excessive pressure from being built up for any reason. The drum 29 may be evacuated by suitable means shown as a pipe 72 which may be connected to a suction device and is closed by a valve 73.

In the operation of this device the alkali cellulose crumb or slurry is fed continuously by the screw conveyor 11 into the vacuum chamber 21 which serves to remove the air from the alkali cellulose to a point such that the reaction with carbon bisulfide in the rotating drum 29 does not proceed with sufficient violence to produce combustion.

The drum 29 is also evacuated and the valve 73 closed. The carbon bisulfide is fed into the reaction drum in the liquid form through the pipe 30 as above described. However, the amount so fed is so controlled by the valve 35 that it immediately vaporizes upon entering the drum. The temperature and pressure within the drum are suitably controlled for this purpose by varying the rate of introduction of the carbon bisulfide and the flow of heating or cooling fluid through the jacket 41. The rate of feed of the carbon bisulfide is preferably so adjusted that there will be no excess carbon bisulfide to be removed from the drum at the end of the reaction period. The carbon bisulfide can be introduced at various points, for example, to flow countercurrent to the crumb if desired.

During the reaction time within the drum the alkali cellulose crumb is agitated and thoroughly mixed with the carbon bisulfide vapor so that all particles of the crumb are brought into contact with the vapor and a uniform and complete reaction takes place. The length of the drum and the rate of feed of the crumb are so controlled, as by varying the pitch or speed of rotation, that the material reaches the lower end of the drum at the time the reaction is completed to form cellulose xanthate. If desired the rate of flow of the material through the drum 29 may be more positively controlled by a screw feed means either within the drum or formed as helical vanes on the inner wall thereof. This xanthate then falls through the duct 66 into the cylinder 67 and is pushed along and compacted within the cylinder in the form of a comparatively hard mass which forms a vapor seal to prevent the escape of the carbon bisulfide vapor. The piston 68 also squeezes out any carbon bisulfide liquor which may be present in the material.

The cellulose xanthate is then discharged from the end of the cylinder 67 into the receiver 70 wherein it may be dissolved in a caustic solution to form the usual viscose spinning solution for the manufacture of rayon fibres, or it may be fed to mixers or disintegrators if desired or to a suitable zone for the removal of excess carbon bisulfide before being applied to the caustic tank.

In this apparatus from 15 to 30 minutes may be required for the complete xanthation in the drum 29 as distinguished from the two or three hours normally required for baratte batch operation. The process has the further advantage that the carbon bisulfide vapor is not discharged into the room nor is special evacuating apparatus required as the amount introduced may be accurately controlled so as to avoid excess.

In order to prevent building up the carbon bisulfide pressure in the drum when the crumb feed is interrupted, a suitable automatic control for the valve 35 may be provided such as an electric eye actuated by the crumb in the duct 27 or a high pressure shut-off responsive to the pressure in the drum 29. Suitable cleaning means such as a scraper blade may be provided if necessary to prevent the material from adhering to the inside of the drum.

While a specific embodiment of the invention has been shown for purposes of illustration, it is to be understood that the invention is capable of various uses and that changes and modifications may be made therein as will be readily apparent to a person skilled in the art. The invention is only to be restricted in accordance with the scope of the following claims.

What is claimed is:

1. An apparatus for the xanthation of alkali cellulose comprising a closed rotating drum positioned to rotate about an inclined axis suited to feed alkali cellulose crumb along said drum due to gravity in the course of its rotation, an evacuating chamber, means for maintaining said chamber at reduced pressure, vapor-seal means to feed alkali cellulose crumb to said chamber, and vapor-seal means to feed alkali cellulose from said chamber to said drum, means for introducing carbon bisulfide into said drum for reaction with said alkali cellulose, and means for removing the cellulose xanthate thus formed from the lower end of said drum, said removing means comprising means for compacting the cellulose xanthate to a comparatively hard mass which forms a vapor seal.

2. An apparatus for the xanthation of alkali cellulose comprising a closed rotating drum positioned to rotate about an inclined axis suited to feed alkali cellulose crumb along said drum due to gravity in the course of its rotation, a

closed chamber above the upper end of the drum, means for maintaining said chamber at reduced pressure, a downwardly feeding duct for introducing alkali cellulose crumb to the upper end of said chamber, at least one bell valve interposed in said duct, a duct connecting the bottom of the chamber and the upper end of the drum, at least one bell valve interposed in the duct between said chamber and said drum for discharging said material from said chamber to said rotating drum while maintaining a pressure seal for said chamber, and means for removing the cellulose xanthate formed from the lower end of said drum, said removing means comprising means for compacting the cellulose xanthate to a comparatively hard mass which forms a vapor seal.

3. In a continuous process for xanthating alkali cellulose, the steps which comprise substantially continuously passing alkali cellulose in the form of a loose crumb through a gas-evacuating zone, continuously evacuating gas from the loose crumb in the gas-evacuating zone, and then, without appreciably compacting the loose crumb, continuously introducing the loose crumb discharged from said zone into a previously evacuated xanthating zone in the presence of carbon bisulfide vapor, continuously agitating the crumb in the xanthating zone while advancing the crumb therethrough, substantially continuously removing the xanthated crumb from the xanthating zone, and substantially preventing the transfer of gas into and out of said zones with the crumb.

4. A process as defined in claim 3 in which the crumb descends loosely by gravity through the gas-evacuating zone.

5. A process as defined in claim 3 in which the carbon disulfide is passed countercurrent to the loose crumbs in the xanthating zone.

6. A process as defined in claim 4 in which the carbon disulfide is passed countercurrent to the loose crumbs in the xanthating zone.

7. In a continuous process for xanthating alkali cellulose, the steps which comprise substantially continuously passing alkali cellulose in the form of a loose crumb through a gas-evacuating zone, continuously evacuating gas from the loose crumb in the gas-evacuating zone, and then, without appreciably compacting the loose crumb, continuously introducing the loose crumb discharged from said zone into a previously evacuated xanthating zone in the presence of carbon bisulfide vapor, continuously agitating the crumb in the xanthating zone while advancing the crumb therethrough, substantially continuously removing the xanthated crumb from the xanthating zone, and substantially preventing the transfer of gas into and out of said zones with the crumb, the prevention of gas transfer at the discharge end of the xanthating zone being effected by compacting the xanthated crumb to a comparatively hard mass which forms a vapor seal.

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