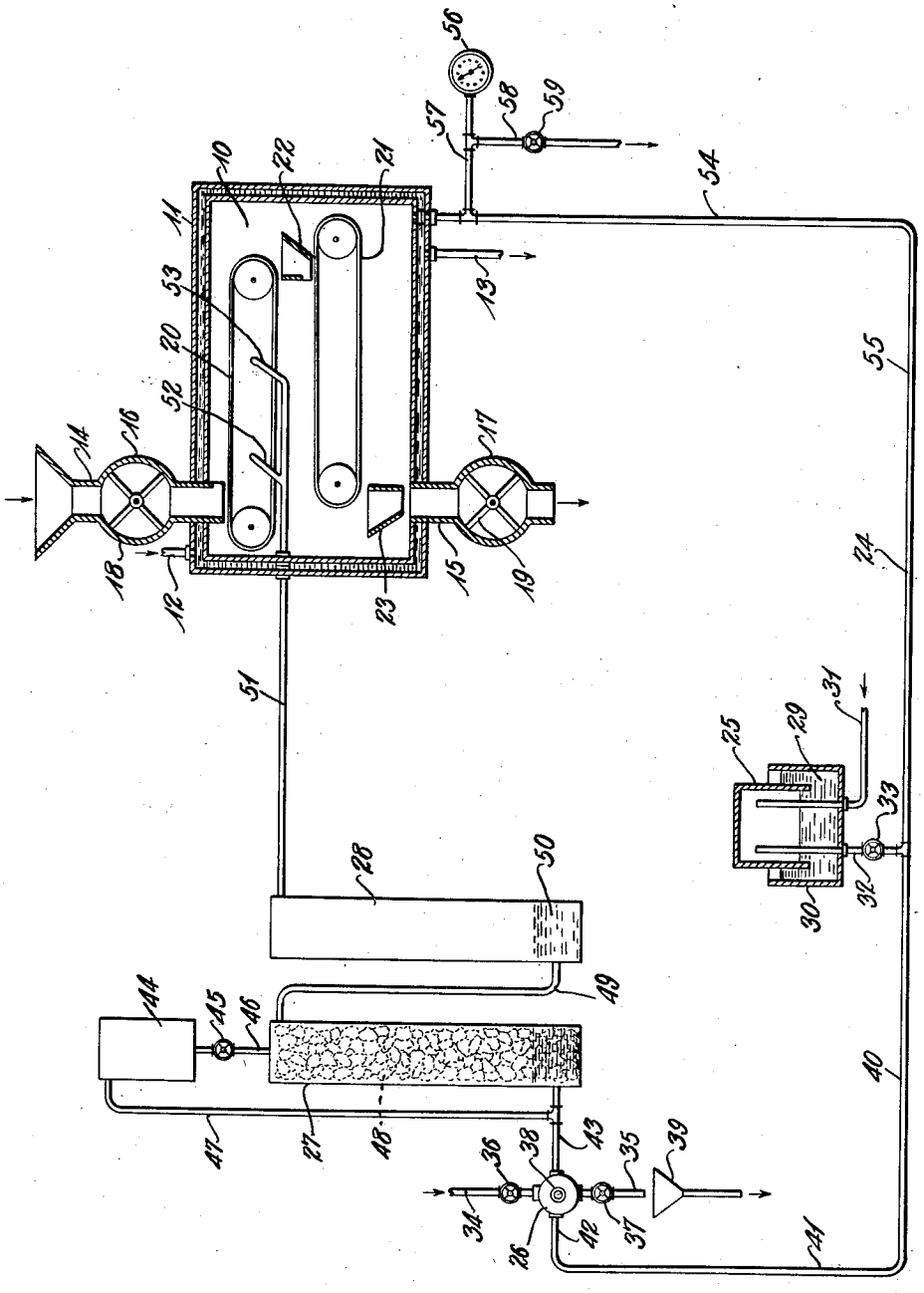
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APPARATUS FOR MANUFACTURE OF CELLULOSE XANTHATE

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APPARATUS FOR MANUFACTURE OF CELLULOSE XANTHATE

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1 Claim. (Cl. 23—260)

This application is a division of my application Serial Number 98,769, filed August 31, 1936, now Patent No. 2,122,519.

The present invention relates to the preparation of viscose spinning solutions for the manufacture of artificial silk filaments, and has special reference to that portion of the process wherein alkali cellulose is converted into cellulose xanthate.

In the manufacture of viscose spinning solutions it is common practice to first soak sheets of wood pulp in a caustic alkali solution until the sheets are thoroughly saturated. The major part of the lye solution is then pressed out, leaving the sheets in the form of alkali cellulose. Subsequently, the alkali cellulose sheets are thoroughly macerated into fine crumbs and introduced into a xanthating drum together with a measured quantity of liquid carbon bisulfide. The drum is then revolved and the materials tumbled about therein, preferably at a temperature of about 25° C., whereupon the crumbs of alkali cellulose react with the carbon bisulfide to form yellow crumbs of cellulose xanthate, which are soluble in dilute sodium hydroxide to form viscose. After completing the xanthating operation the drum is emptied, cleaned, and refilled with another batch of alkali cellulose crumbs.

This method of manufacture, however, has presented numerous difficulties, particularly in connection with the conversion of the alkali cellulose into cellulose xanthate. For example, the alkali cellulose crumbs tend to form lumps of varying size and shape during the mixing operation, which is obviously a great disadvantage. These lumps not only fail to become thoroughly saturated with the liquid carbon bisulfide, thus enclosing alkali cellulose particles which entirely escape reaction therewith, but also tend to dissolve very slowly and imperfectly, which results in a spinning solution lacking in uniformity, i. e., having undissolved particles therein. These undissolved particles cause clogging of the spinning solution filters, necessitating frequent cleaning thereof as well as frequent interruption of the spinning procedure. Moreover, any fine particles of undissolved fibres which escape the filters and find their way into the finished product weaken the filaments and destroy their uniformity.

In addition to these disadvantages it has been found that a certain amount of the alkali cellulose tends to stick to the walls of the drums, thus escaping the reaction within the bulk of the mass. Moreover, it is obvious that by operating in separate batches costly interruptions become necessary for such purposes as discharging the completed batch, cleaning the drum or refilling with a new batch. In addition considerable difficulty is encountered in the handling of liquid carbon bisulfide owing to its highly volatile character and consequent tendency to cause dangerous explosions.

Accordingly, one of the principal objects of the present invention is the production of a cellulose xanthate by a continuous and substantially safe process which permits the production of predetermined quantities of the xanthate per unit of time and yields a product which is substantially free of insoluble particles of alkali cellulose and is capable of being uniformly dissolved in dilute alkaline hydroxide to form a viscose spinning solution suitable for the manufacture of artificial silk filaments. Other objects and advantages will become apparent upon studying the following detailed description as well as the accompanying drawing which consists of a diagrammatic view of a suitable apparatus for carrying out the invention, with parts shown in section.

The invention generally relates to a continuous method of manufacturing cellulose xanthate which comprises continuously treating thin layers of alkali cellulose with any desired quantity of a gaseous mixture comprising carbon bisulfide vapors and an inert gas, such as nitrogen, which simultaneously acts as a carrier and diluent for the bisulfide vapors, and not only causes the vapors to be uniformly dispersed, but renders heating unnecessary and permits accurate control of the quantity of bisulfide vapors supplied to the reaction chamber. Moreover, it is very advantageous to continuously move the alkali cellulose back and forth on traveling platforms or similar devices located within the reaction chamber to cause it to be repeatedly exposed to fresh portions of carbon bisulfide vapor. The reaction preferably takes place in a closed chamber to which the starting materials may be continuously and uniformly supplied and from which the reaction product may be continuously removed without substantial admission of external air or escape of enclosed gases.

In the drawing, the numeral 10 designates a stationary xanthating tank or chamber having a double wall 11, which may be supplied with a suitable temperature regulating medium such as water, air, oil, or the like, by means of the pipes 12 and 13. Tank 10 is further provided with an inlet duct 14 for effecting an entrance of the alkali cellulose crumbs into the xanthating cham-

ber, and an outlet duct 15 for discharging the crumbs after they have been converted into cellulose xanthate, said ducts 14 and 15 having enlarged spherical portions 16 and 17 provided with paddle wheels 18 and 19 which may be rotated by any suitable means to regulate the entrance and discharge of the cellulosic material and to materially inhibit the entrance of excess air into the xanthating chamber. In addition the interior of the tank is provided with a plurality of endless traveling conveyor belts 20 and 21 arranged one above the other in staggered relation and motivated by any suitable means (not shown). A funnel or hopper such as shown at 22 and 23, is positioned at the delivery end of each belt, the funnel 22 serving to discharge the alkali cellulose coming from upper belt 20 onto lower belt 21 and the funnel 23 serving to convey the completely reacted cellulose xanthate into the outlet duct 15.

A circulating pipe system, generally indicated at 24, is connected with the xanthating chamber, and has associated therewith a gas supply tank 25, a pump 26 of the water circulation type, a vaporizing tank 27 and a moistening tank 28. Tank 25 rests or floats upon the liquid 29 contained in vat 30, and is adapted to supply inert gas, such as nitrogen, to the system, thus acting as a pressure regulator and serving to compensate for gas losses in the apparatus. The amount of gas in the tank, which may be replenished by means of the pipe 31 connecting with any suitable source of supply (not shown), is automatically indicated by the rise and fall of the tank in the vat, the discharge of gas into the system being made through pipe 32 and valve 33.

Pump 26 is of conventional construction, and consists of inflow and outflow pipes 34 and 35 controlled by the valves 36 and 37, the pump chamber 38 and the funnel 39 for discharging the used water. Any other suitable pump may, however, be substituted therefor. Upon activating the pump inert gas from tank 25 is conducted through the pipes 40, 41, 42 and 43 into the lower end of the evaporating tank 27, which serves as an evaporator for the liquid carbon bisulfide flowing down from the carbon bisulfide supply tank 44 positioned above tank 27. The downward flow may be controlled by the valve 45 as the liquid flows through the connecting pipe 46, the pressure in tank 27 being maintained constant by means of the by-pass pipe 47.

Upon entering the evaporating tank 27, the liquid carbon bisulfide drips downward over Raschig rings 48 or other similar devices placed within the evaporator to provide the greatest possible evaporating surface. At the same time the inert gas, rising within the evaporator, contacts the carbon bisulfide vapor formed therein and becomes saturated therewith, the degree of saturation depending upon the temperature of the evaporator, which may be regulated by any suitable means, not shown. The vapor-gas mixture thus created rises and passes out through the pipe 49 into the lower end of the moisture regulating tank 28 and is bubbled upward through a suitable liquid 50, such as a Glauber salt solution, which has a vapor tension equal to the vapor tension of the alkali cellulose to be treated and accordingly permits compensation for the loss of moisture in the alkali cellulose crumbs as they are deposited on the conveyor belts. After absorbing moisture in tank 28, the vapor-gas mixture passes out through the pipe 51 to the xanthating chamber 10 and is diffused therein by means of the pipes 52 and 53. The carbon bisulfide vapor contained in the gas mixture then reacts with the alkali cellulose to form cellulose xanthate, while the inert gas returns through pipes 54 and 55 to the supply tank 25 in the direction indicated by the arrow.

A pressure gauge 56 for measuring or recording the gas pressure in the apparatus is connected with the circulating pipe system by means of pipe 57, which also has connected thereto an additional pipe 58 provided with a valve 59 for removing the gas mixture, as may be necessary when first filling or in cleaning the apparatus. In addition, a condensor or absorption apparatus for the recovery of carbon bisulfide may be connected to pipe 58, if desired.

In operation the alkali cellulose crumbs are fed continuously into the inlet duct 14 and the paddle wheel 18 is rotated by any suitable means. The pump 26 is then set in operation and the vapor-gas mixture injected into the xanthating chamber. The crumbs drop upon one end of the uppermost conveyor belt 20 in synchronism with the movement of the belt, thereby forming a thin, uniform layer thereon, and are simultaneously acted upon by the carbon bisulfide vapor contained in the vapor-gas mixture. Upon reaching the end of the uppermost belt assembly, the crumbs drop upon the belt assembly 21 next below, traveling in the opposite direction, and continue on in this manner, repeatedly passing from one end of the reaction chamber to the other. In the course of its travel the alkali cellulose is continuously acted upon by the carbon bisulfide vapor contained in the vapor-gas mixture, and upon leaving the lowermost belt assembly drops through the funnel 23 into the outlet duct 15 in the form of cellulose xanthate.

It has been found highly desirable to maintain a uniform concentration of carbon bisulfide vapor throughout all parts of the xanthating chamber between the entrance and outlet points for the gas. This is facilitated by causing the amount of inert gas in the circulation system to be comparatively high per unit of time in relation to the amount of carbon bisulfide vapor, so that the alkali cellulose in the xanthating chamber is uniformly contacted by the carbon bisulfide at all stages of the xanthating procedure. In addition, the total gas pressure in the vapor-gas mixture should be maintained at a constant, i. e., approximately atmospheric pressure, in order to avoid the entrance of oxygen into the chamber as a result of under pressure in the xanthating chamber, or the escape of gas as a result of excess pressure.

It is also essential to successful xanthation to maintain a constant temperature in the xanthating chamber, which in the present instance is assured by means of circulating tempered water, air, oil or similar agents within the double wall 11 of the xanthating chamber. Moreover, a temperature suitable for xanthating should be adhered to throughout the entire system, although the temperature may be varied within certain limits without harmful effects. For example, under certain conditions temperatures below 20° C., as well as temperatures above 30° C. may be used. It has been found, however, that best results are obtained if the temperature of the system is maintained at about 24° C., as at this temperature the partial vapor pressure of the carbon bisulfide is 34 cm. of mercury, or approximately equivalent to the partial pressure of the nitrogen or other inert gas used.

Any desired number of conveyor belts may be employed within the xanthating chamber, but it has been found preferable to employ two or more. They may be made of any suitable material that is resistant to the chemicals with which they came in contact and that inhibits the adhesion of the cellulosic material thereto. Leather, for example, has been found to provide an excellent carrying surface, since it is resistant to chemicals used and the cellulosic material does not cake thereon.

The method of vaporization employed in carrying out the present invention, wherein an inert gas is allowed to absorb the carbon bisulfide vapor, has proved to be exceedingly effective, as the vapor-gas mixture formed disseminates evenly throughout the xanthating chamber and the required amount of carbon bisulfide reacts with the alkali cellulose uniformly and completely, resulting in a xanthate which is easily and quickly soluble. Furthermore, such a vapor-gas mixture need not be heated, as is the case when the vapors are derived from liquid carbon bisulfide alone, and it is easily maintained at the desired temperature and degree of concentration.

It is to be understood of course, that the process and apparatus herein described may be varied within reasonable limits without departing from the spirit and scope of the invention, and that the drawing merely represents one form of apparatus which has been found suitable for carrying out the proposals described.

What I claim is:

An apparatus for producing cellulose xanthate from alkali cellulose and carbon disulphide comprising in combination, a closed reaction chamber surrounded by a double wall, said double wall containing a temperature regulating medium, substantially vertical inlet and outlet ducts suitably arranged to introduce and exhaust solid reactant materials and provided with rotatable paddle wheels adapted to maintain air-tight conditions within said tank, a plurality of endless traveling conveyors within the tank and positioned one above the other in staggered relationship and means for continuously supplying a gas-vapor mixture to and removing it from the reaction tank, which means comprising a circulating pipe system connected to the reaction chamber, said system having associated therewith an automatically regulatable gas supply tank, an evaporator tank for evaporating a liquid and serving to mix the resultant vapor with gas, a moisture regulating tank and a pressure gauge connected with the reaction chamber for determining the pressure therein.

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