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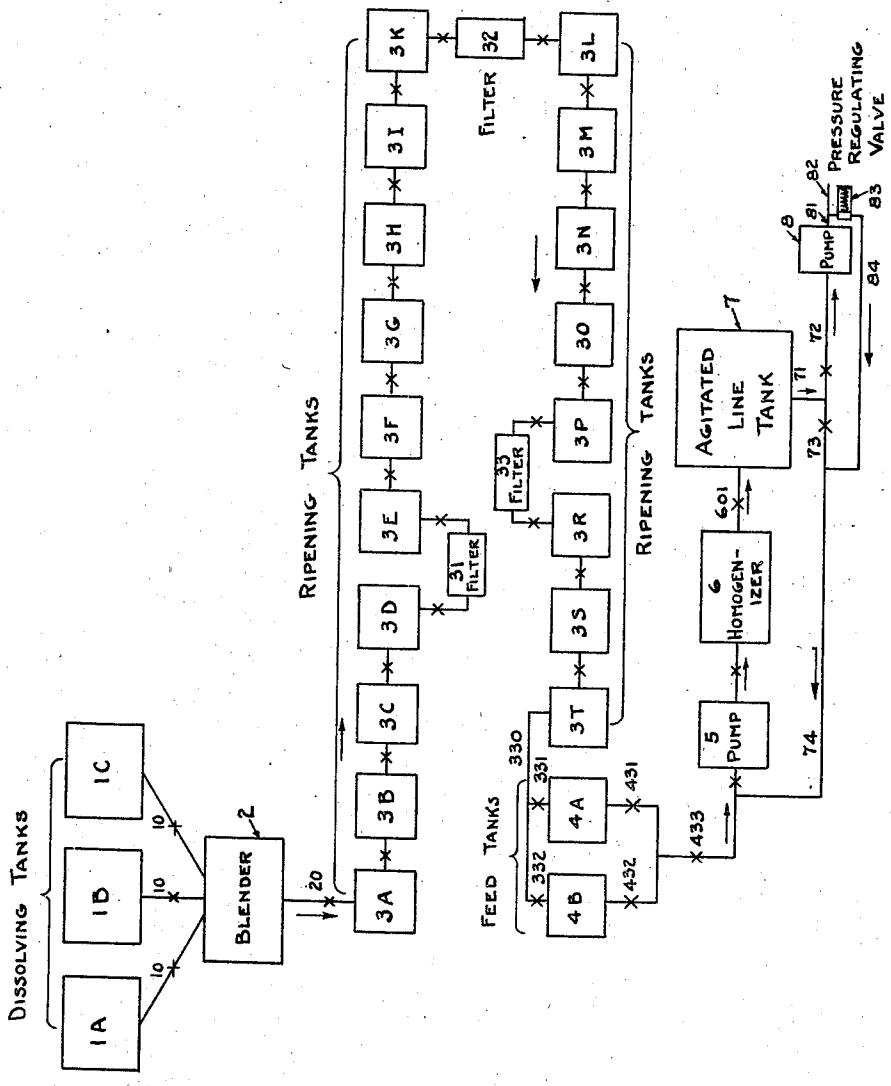

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VISCOSE PROCESS AND APPARATUS

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This invention relates to a viscose process. More particularly, the invention has to do with the treatment of substantially ripened viscose and the feeding thereof to the product-forming room. The invention will be described for convenience in its application to a process of making rayon, but the process is equally applicable to the manufacture of regenerated cellulose sheeting, or to any other process in which viscose is used.

In the manufacture of rayon by the viscose process, cellulose xanthate is formed in a manner which will be understood by those skilled in the art, is dissolved, is ripened, is filtered, is deaerated, and is extruded through a spinneret into a coagulating bath. The relation between the viscose solution and the coagulating bath is adjusted to produce yarn of the desired properties. The maintenance of this relation is essential in the production of a uniform product. It is relatively easy to maintain the coagulating bath at a constant value, but is difficult to maintain the viscose at a constant value because it changes with time and at different rates depending on the temperature. This change in the viscose is called ripening and the riper the viscose the more easily it coagulates. Viscose coagulability or its degree of ripeness is commonly measured in terms of "salt index". The salt index referred to in this specification is that defined by Reinthaler and Rowe in "Artificial silk", page 68, second full paragraph, published in 1929.* In the prior art the salt index of the viscose delivered to the spinning room has been controlled to within plus or minus 0.5°, but this gives rise to a non-uniform product.

In the practice of today, batches of cellulose xanthate are dissolved in an aqueous caustic solution and three or four such charges blended to form a final viscose charge. These charges are sent to the ripening room, are confined in tanks, filtered, confined in other tanks, and the processes of filtering and confining repeated until the viscose has reached the stage of ripeness suitable for delivery to the room wherein it is to be formed into the desired products. When this stage has been reached, the viscose charge is flowed into one of two "feed" tanks from which it is delivered to the product-forming room. The viscose is forced from one feed tank, usually by air pressure, until the tank is substantially exhausted, then viscose is taken from the other feed tank, and so on. A "heel" of viscose must be left in each of these tanks to prevent air from blowing through and getting in the viscose pipe lines, otherwise continuous filaments or sheets could not be formed in an orderly manner and without excessive difficulty. When a feed tank is refilled with fresh viscose, the "heel" from the charge previously in that tank has quite a different salt index than the salt index of the new charge and since the two charges do not intermingle appreciably but remain substantially in distinct layers, it is impossible to deliver to the product-forming room uniform viscose or one having anywhere near a constant salt index. In addition, since the viscose must travel different distances through pipes in the relatively warm product-forming room to various machines, there is further non-uniformity of the product arising from the varying amount of ripening of the viscose that takes place in this travel.

It is an object of this invention to improve the process of preparing viscose. Another object of the invention is to prepare viscose having a substantially uniform salt index. Still another object is to deliver to the product-forming room a viscose having substantially constant salt index and to maintain that salt index substantially constant at all the extrusion devices or product-forming machines in that room. Other objects of the invention will be in part apparent and in part set forth hereinafter.

These objects are accomplished by the improved means and method herein set forth and claimed.

The drawing is a diagrammatic flow sheet representing one mode of practicing the invention.

Referring to the drawing, numerals 1A, 1B and 1C refer to dissolving tanks which perform the function of dissolving the cellulose xanthate in an aqueous caustic solution. These dissolvers may be three in number, as shown, or more or less. The batches of viscose from the several dissolvers pass through valve-controlled pipelines 10 leading from the dissolvers to a blender 2 in which the batches of viscose are mixed together in order to improve the homogeneity of the product. The blender is of any satisfactory type known to the art. The blended viscose passes

*The degree of ripeness of the viscose is determined by introducing small samples into common salt solutions of different concentrations. The fresher and less ripe the viscose, the stronger the solution of common salt necessary to precipitate the sodium cellulose xanthate. The "salting point" of a viscose signifies the concentration of a common salt solution that just suffices to coagulate a drop of viscose which is allowed to fall into it. The degree of ripeness most favourable for spinning is dependent upon various factors, e. g., the nature of the preparation of the viscose, the composition and temperature of the coagulating bath, etc. In the case of a viscose prepared as already described, for example, the correct degree of ripeness has been attained when it is coagulatd by 3–4% common salt solution. Such viscose, therefore, has a salting point of 3°–4°.

from the blender through valve-controlled line 20 to the first of a number of ripening tanks, 3A, 3B, 3C, etc., to which the charge from the blender is transferred. These ripening tanks are located in a room kept at constant temperature. The charges of viscose are advanced from one ripening tank to another through pipelines, which may be valve-controlled as shown, as the older viscose is consumed and during this advance, they are filtered through successively finer filtering media. For instance, the relatively green viscose is passed through a filter press 31, connected between tanks 3D and 3E by means of pipelines which may be valve-controlled. Between tanks 3K and 3L there may be another filter similarly located, having somewhat finer filter cloths, and further on, between tanks 3P and 3R, a third filter press may be similarly located, having a still finer filtering medium. After the last filtration, the viscose is stored under high vacuum, in order that the entrapped air be removed, in partly filled tanks 3R, 3S and 3T, connected to each other and to the filter press 33 by means of valve-controlled pipelines as shown. The filtered, air-free viscose is then flowed from tank 3T by means of valve-controlled lines 330, 331 and 332 alternately to "feed" tanks 4A and 4B.

Viscose is pumped or otherwise flowed alternately from the tanks 4A and 4B, through valve-controlled pipelines 431, 432 and 433 to a pump 5 which may be of any suitable type but preferably is a constant delivery pump driven by an electric motor. The pump discharges the viscose into a homogenizer 6, which may be any suitable blending means. The homogenizer is so constructed that the two viscoses (the second of which will be described hereinafter) fed thereinto will be thoroughly blended together to form a homogeneous mixture. The viscose passes on from the homogenizer tank 6 through valve-controlled pipe 601 and into the agitated line tank 7. This agitated line tank contains agitating means which preferably is a relatively slowly revolving paddle which will not violently disturb the surface of the viscose and permit air to be entrapped therein. It should be understood, however, that thorough mixing of the viscose leaving the homogenizer with that in the agitated line tank must be provided to prevent pocketing or by-passing of unblended viscose. The volume of the agitated line tank should be sufficiently large to suppress fluctuations in index but not so large as to cause an excessive salt index drop from the feed tank to it. In general, the capacity of this tank should be about that of one of the feed tanks. This agitated line tank is jacketed preferably to about two-thirds the height of the tank or to about the normal viscose level. This jacketed space is connected with warm and cold water lines or with other temperature control media such as brine. The homogenizer may also be jacketed for temperature control.

The viscose flowing into tank 7 should preferably enter below the viscose level therein. A portion of the viscose is drawn from the bottom of this tank through valve-controlled line 71—72 by means of pump 8, which is set to deliver viscose through line 81 in excess of the requirements of the spinning machines or to other machines for forming shaped articles. A pressure control valve 83 maintains the pressure in the feed line 82 constant by permitting a certain quantity of viscose to return by means of lines 84 and 74 through pump 5 and homogenizer 6 to blend with the viscose coming from one of the feed tanks 4A or 4B. If it is found desirable, a filter press may be located between pump 8 and the extrusion devices in order to insure the cleanliness of the viscose.

In the practice of our invention, viscose is fed continuously into the homogenizer and blending system from one of the two feed tanks alternately. This viscose is passed through pump 5 to homogenizer 6 along with approximately 10 times its volume of viscose taken from line tank 7. After passing through homogenizer 6, it is fed into the line tank 7 immediately below the surface of the viscose therein. In passing down through line tank 7, it is blended with the viscose in the tank and emerges from the bottom of the tank, being then fed in part to the feed line 82. By this system of operation, even though the small heel of the previous charge in the feed tank may have an index so far below the balance of the charge that it would not produce a good product, it becomes so thoroughly and completely blended with the large volume of the material in line tank 7 that the index of the viscose leaving line tank 7 and going to feed line 82 is changed but very slightly.

In addition to correcting the variations that arise from the periodic passage of these small heels and other small amounts of viscose having similar, greatly different indices, the volume of viscose in this system and the close control of it permit to a very great degree the neutralizing of other types of index variation of comparatively short duration and is therefore a useful means of insuring a constant supply, to the extrusion room, of uniform viscose.

For example, if the index of the charge of viscose being drawn from the feed tank is abnormally high, viscose of the standard salt index can be delivered to the spinning room by temporarily reducing the volume of viscose fed into this system from the feed tanks and if necessary by increasing the temperature of the viscose in the homogenizer and in the line tanks.

Thus the operation of the plant is considerably facilitated and it is possible to produce a product far superior to that produced heretofore.

By the process just previously described, we are able to deliver viscose having a substantially constant salt index to the spinning room. However, since the viscose must be conducted various distances or for various lengths of time through pipes in the relatively warm spinning room, in order to reach all the machines and spinning positions therein, it becomes further and non-uniformly ripened. This variation in ripening as measured by salt index of viscose taken from various machines and various spinning positions may be as much as 0.3° or even more.

In order to diminish this variation and improve the uniformity of the products formed, we incorporate in the viscose an agent that inhibits or retards further ripening of the viscose. For instance, we may add a small amount of sodium sulfite, such as 0.1% to 5% or more and preferably about 1%, based on the cellulose content of the viscose. The addition may be made either as a solution or as a solid. When we add to the viscose 1% of sodium sulfite based on the cellulose content of the viscose, we find the uniformity of the viscose as measured by salt index is improved throughout the spinning room about three-fold. The addition of the inhibitor is preferably made to the thoroughly blended viscose described above as it leaves the system in line 82. It is to be understood that suitable precaution will be taken to

insure the uniformity of the resultant viscose. In lieu of adding sodium sulfate as such, it may be formed in situ by the addition of SO_2 gas to the viscose.

By virtue of our invention, it is possible to deliver to the product-forming room viscose of substantially constant salt index. The salt index of this viscose will vary only about plus or minus 0.1° which compares very favorably indeed to the viscose of the prior art processes wherein variations in salt index of plus or minus 0.5° were common. Furthermore, by the addition of a small amount of a ripening inhibitor such as sodium sulfite to the viscose, as previously described, we can not only deliver viscose having a substantially constant salt index to the product-forming room but we can retard further ripening of the viscose so that substantially uniformly ripened viscose is delivered to every product-forming device.

Viscose contains a small percentage of impurities even when a careful preparation and/or treatment reduces the impurities to a minimum. The process of this invention, in addition to insuring the delivery to the product-forming devices, of viscose having a substantially uniform salt index, effects a very uniform distribution of the impurities throughout the viscose, and a delivery to the product-forming devices, of viscose having at all times a uniform amount and distribution of impurities, thereby insuring the continued production of improved thread and the like having substantially uniform physical characteristics.

Any variation or modification which conforms to the spirit of the invention is intended to be included within the scope of the claims.

We claim:

1. In the process of improving the uniformity of ripeness of viscose, the steps which comprise delivering viscose from a feed tank to a line tank, withdrawing viscose from the line tank, delivering a portion thereof to a product-forming device, continuously and uninterruptedly recycling a portion thereof in excess of the amount delivered to said product-forming device, and commingling the same with the viscose issuing from the feed tank whereby to maintain the variation of the salt index of the viscose substantially less than plus or minus 0.5.

2. In the process of improving the uniformity of ripeness of viscose, the steps which comprise delivering viscose from a feed tank to a line tank, withdrawing viscose from the line tank, delivering a portion thereof to a product-forming device, recycling a portion thereof in excess of the portion delivered to said product-forming device and commingling the same with the viscose issuing from the feed tank and thoroughly blending the mixture.

3. In the process of improving the uniformity of ripeness of viscose, the steps which comprise delivering viscose from a feed tank to a line tank, agitating the viscose in the line tank, withdrawing viscose from the line tank, delivering a portion thereof to a product-forming device, recycling a portion thereof in excess of the portion delivered to said product-forming device and commingling the same with the viscose issuing from the feed tank and thoroughly blending the mixture.

4. In the process of improving the uniformity of ripeness of viscose, the steps which comprise delivering viscose of a predetermined degree of ripeness to a feed tank, feeding the viscose from the feed tank through a blending device to a line tank, withdrawing viscose from the line tank, delivering a portion thereof to a line which feeds a product-forming device, recycling another portion of the viscose coming from the line tank in excess of said first-named portion to the viscose being fed into the blending device, and blending the latter portion with the viscose being fed into the blending device.

5. In the process of improving the uniformity of ripeness of viscose, the steps which comprise subjecting green viscose to a period of ripening until a predetermined degree of ripeness is attained, delivering the viscose to alternate feed tanks, withdrawing a stream of viscose alternately from the feed tanks, delivering the viscose to a homogenizer and thence to a line tank, withdrawing a stream of viscose from the line tank and dividing it into two portions, delivering one portion to a line leading from the line tank to product-forming devices, recycling another portion greater than said first-named portion and introducing it into the homogenizer, and adding an agent which will retard ripening to the viscose prior to its delivery to the product-forming devices.

6. Apparatus adapted to control the uniformity of a cellulosic solution comprising a feed tank, a line leading from the feed tank, blending means in said line, a line tank connected with the blending means, a product-forming device, means for delivering a portion of solution from the line tank to the product-forming device, means for recycling another portion from the line tank to the blending means.

7. Apparatus adapted to control the uniformity of a cellulosic solution comprising a feed tank, a line leading from the feed tank, blending means in said line, a line tank connected with the blending means, a product-forming device, means for delivering a portion of solution from the line tank to the product-forming device, means for recycling another portion from the line tank to the blending means, and means for recycling to the blending means, a part of the solution being delivered to the product-forming device.

8. In the process of improving the uniformity of ripeness of viscose, the steps which comprise delivering viscose from a feed tank to a line tank, which is as large as the feed tank, withdrawing viscose from the line tank, delivering a portion thereof to a product-forming device, recycling a portion of the viscose in excess of the amount delivered to the product-forming device, commingling the same with the viscose issuing from the feed tank and thoroughly blending the mixture, and adding an agent which retards ripening to the viscose during its travel to the product-forming device.

9. In the process of improving the uniformity of ripeness of viscose, the steps which comprise flowing a viscose solution from a feed tank to a point from which a portion of said solution is passed to a product-forming device, delivering a portion of said solution to said device, continuously and uninterruptedly recycling another portion of said solution in excess of the portion delivered to said product-forming device, and commingling the same with the viscose solution flowing from the feed tank whereby to maintain the variation of the salt index of the viscose less than about plus or minus 0.5.

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