Dec. 12, 1933. G. A. RICHTER 1,938,722

RIPENING OF VISCOSE SIRUP

Filed Dec. 8, 1930

Fig. 1.

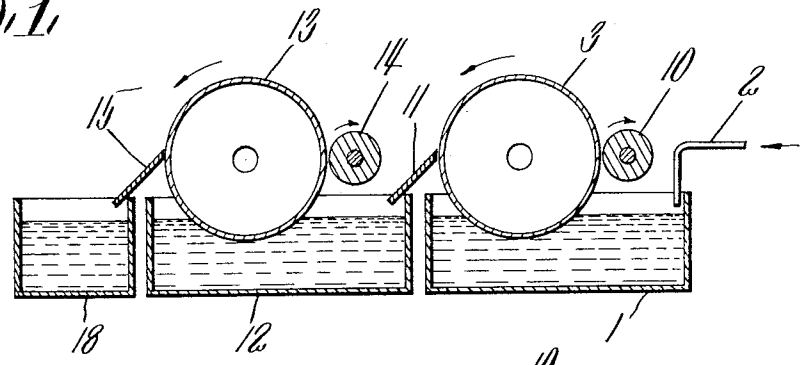


Fig. 2.

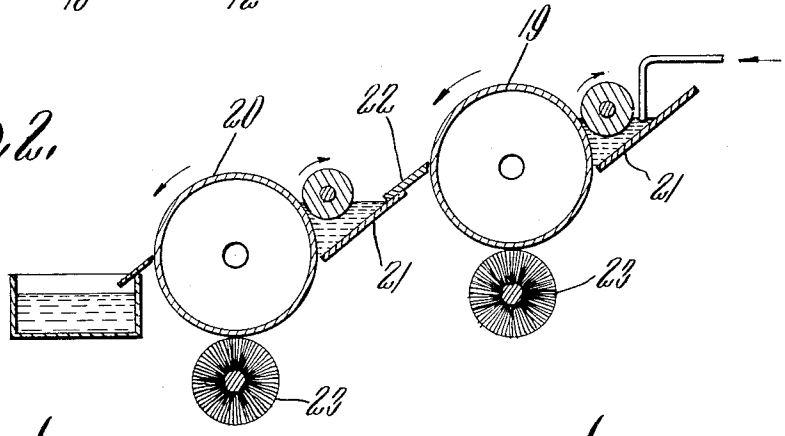


Fig. 4.

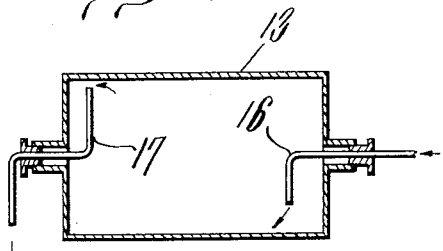
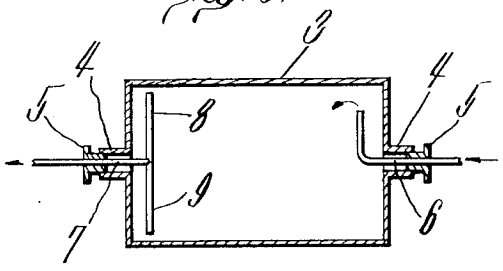


Fig. 3.



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RIPENING OF VISCOSE SIRUP

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4 Claims. (Cl. 260—100)

This invention relates to a process of ripening or maturing viscose sirup, more particularly from the standpoint of attaining that physical and chemical condition in the sirup at which it is ready and satisfactory for manufacture into products of the type of artificial silk filaments.

When cellulose xanthate is dissolved in caustic soda solution to form viscose sirup, so called, the fresh or unripened sirup cannot successfully be spun into the usual acid-setting baths, for the reason that cellulose is not regenerated from the xanthate to a sufficient extent and at the proper rate to give rise to filaments of the desired tenacity, strength, elasticity, and other characteristics. In order to realize these desiderata, it has been found necessary to subject the viscose sirup to what is known as a ripening operation, during which operation it is generally understood that combined sulphur splits off and the cellulose complex becomes progressively poorer in sulphur. The ripening is carried to a point short of gelling of the sirup, for were gelling to take place, it would be impossible to handle the gel in the usual spinning machines without serious gumming and plugging of the pipe lines, pumps, spinnerets, and other parts. The customary practice in producing satisfactorily spinnable viscose sirups is to form a cellulose xanthate solution in caustic soda containing 7% cellulose, calculated as cellulose but in the form of xanthate, and 6½% caustic soda. This sirup, which is of a thick consistency not unlike molasses, is then filtered to remove undissolved fibers and other solid impurities which would tend to interfere with spinning by plugging the orifices of the spinnerets. The filtered sirup is then delivered to a storage tank, wherein it is stored or allowed to ripen under controlled low temperature conditions, usually 20° C., until it acquires the composition requisite for successful spinning. Depending upon the nature of the fresh or unripened sirup, it usually takes from about 72 to 96 hours standing at 20° C. to produce a fully and satisfactorily ripened sirup.

There are many disadvantages inherent in a viscose-ripening procedure such as hereinbefore described. Thus, the equipment required is expensive and occupies much space, including, as it does, a special thermo-insulated room, an elaborate system of refrigeration, control of the atmosphere of the room, a large number of storage tanks for the viscose sirup, and pumps for circulating the sirup. Again, a large inventory or stock of sirup must always be on hand; and if the sirup is damaged or completely spoiled, a delay of three to four days must be contended with before the inventory is adjusted to catch up with the requirements of the spinning machines, assuming that the plant is an efficient one equipped with the number of spinning machines to consume the entire output of the ripening apparatus.

I have discovered that the time necessary for ripening viscose sirup may be reduced to a comparatively insignificant period, if the fresh or unripened sirup is heated to a temperature materially above normal room temperature. The particular temperature to which the sirup is heated and the time period to be allotted for heating depends on various factors, especially upon the composition and characteristics of the sirup, which determine its tendency to split off combined sulphur and thereby to regenerate cellulose. In my application Serial No. 484,292, filed September 25, 1930, I have disclosed and claimed the principles of my discovery, as well as one specific method and apparatus embodying these principles. I have now found that the desired results may be realized by depositing an unripened viscose sirup as a film on a smooth, non-absorbent, heated surface, and keeping the sirup film in contact with the heated surface only for a sufficient time interval to effect the desired ripening, that is, without causing gelling to take place. I have further found that the time interval for heating in such case need be so short that the sirup may be progressively deposited on a heated carrier moving circuitously at the appropriate rate of speed, and then be progressively removed in a ripened but ungelled condition only an exceedingly short time after its deposition and before the carrier has completed a circuit. Such a process is advantageous, as it not only makes possible quick, high-capacity production of ripened viscose sirup, but also lends itself to easy control to produce ripened sirup of the proper quality. Thus, such factors as the temperature of the surface on which the sirup is deposited may be controlled thermostatically when the surface is heated either by steam, electrical units, or the like; and the station for removing the sirup film from the carrier may be shifted to ensure a removal when the sirup has reached the desired condition. A convenient and available type of carrier is a steel drum whose periphery is nicely finished and which is heated internally by steam. When the drum is slowly revolved so that a portion of its periphery comes in contact with a supply of viscose sirup, a film of sirup forms over the

periphery of the drum, owing to the adhesiveness and viscosity of the sirup. The sirup carried by the drum periphery may be readily removed as by a rubber or metal doctor blade. The use of a smooth, non-absorbent surface for receiving the viscose sirup makes possible the removal of practically all the sirup, but traces of residual sirup may be removed by sprays and/or brushes before the cleared surface again comes in contact with the supply of unripened sirup.

In order to make possible the keeping of the ripened sirup in ungelled condition for a substantial period of time, it is cooled promptly after ripening, preferably in a manner similar to that of its heating, to temperatures downwards of 20° C., as at such temperatures it may be allowed to stand as a batch for some hours, without danger of gelling. In other words, the hot, ripened sirup may be cooled by the use of apparatus similar to that employed in its heating, except that the drum periphery on which it is deposited is cooled, rather than being heated.

With the foregoing and other features and objects in view, my invention will now be further described in conjunction with the accompanying drawing, wherein

Figures 1 and 2 show diagrammatically and conventionally different forms of apparatus involving the use of both heating and cooling drums.

Figures 3 and 4 represent sections through a heating and cooling drum, respectively.

In Figure 1, I have shown a vat 1, in which is maintained a bath of unripened viscose sirup. The sirup may be continuously delivered into the bath from a suitable source of supply, through a pipe 2, at a rate corresponding to the rate of removal of sirup from the bath. Rotating partially submerged in the bath of sirup is a steam-heated drum 3, which continuously picks up a film of sirup and carries it out of the bath. As shown in Figure 3, the ends of the drum have trunnions 4, in which are fitted glands 5. Through one of these glands a steam-supply pipe 6 passes upwardly into the hollow of the drum. Through the other of these glands, a steam outlet pipe 7 enters into the hollow of the drum, this latter pipe preferably terminating as a T having an upwardly extending portion 8 to carry out the steam, and a downwardly extending portion 9 to carry out the condensate. Uniformity of the film of sirup clinging to the periphery of the drum may be ensured by a roll 10 arranged somewhat above the surface of the bath and rotating in contact with the film in a direction opposite to the rotational direction of the drum. The roll 10 is preferably adjustable to vary the clearance between it and the drum, and thus to determine the thickness of the film remaining on the drum. The film of sirup is carried by the drum periphery for a while, but immediately before the line of dipping into the bath it is scraped from the drum periphery by a doctor blade 11, which also serves to direct the ripened sirup into a second vat 12. The bath of hot, ripened sirup thus formed in the bath 12 is cooled by employing instrumentalities similar to those used in heating the unripened sirup delivered into the vat 1. That is to say, I use a drum 13, a distributing roll 14, and a doctor blade 15, but the periphery of the drum 13 is cooled, rather than being heated. The cooling drum 13, as shown in Figure 4, may be of a construction similar to the heating drum 3. When a liquid cooling medium such as chilled brine is circulated through the cooling drum, it is preferable that the brine be fed downwardly into the drum hollow from a supply pipe 16, and that a discharge pipe 17 project upwardly into the drum hollow, as in such case the warmest brine tends to rise to the top of the pool of brine maintained in the drum and flow into the discharge pipe. The cooled sirup removed from the drum 13 may be directed by the doctor blade 15 into a tank 18, from which it may be withdrawn for use as desired.

The apparatus shown in Figure 2 is similar to that shown in Figure 1, except that the vats have been dispensed with and in their stead I have made provision to maintain banks of sirup in contact with the drums. To this end, I have arranged a heating drum 19 at a level above that of a cooling drum 20, each of said drums rotating in contact with the lower edge of an inclined platform or shelf 21 having end-pieces (not shown) and which serves to form, with one side of the drum, a reservoir for holding a bank of sirup. As the heated drum rotates, it picks up a film of sirup from the bank constantly maintained on the upper platform, and carries it to the other side, where it is removed by a doctor blade 22 onto the lower platform. Such an apparatus has the advantage that considerable periphery surface of the drum is exposed for the action of cleaning means, such as brushes 23, between the doctor blade and the station for supplying the viscose sirup.

While the temperature of the periphery of the heating drum may be varied from, say, 50° to 120° C., nevertheless, even taking into consideration the wide variabilities possible in the nature of unripened viscose sirups, the operating temperature range need not fall outside of between 60° to 100° C. The particular temperature employed depends, among other factors, upon the alkali content of the sirup when it is prepared. The usual sirup contains 7% cellulose and 6½% caustic soda, but for some purposes one might prefer a sirup containing the same amount of cellulose, but a materially lower amount of alkali, say, 3% to 3½% caustic soda. Sirups of such reduced caustic soda content are ripened very quickly at temperatures not above 70° to 75° C. If desired, the sirup may be warmed somewhat, or partially ripened, before it is brought into contact with the heating drum, but when using a sirup which is thin or of low viscosity, it may be of advantage to increase its viscosity by cooling it somewhat, in order to enhance its adhesiveness to and distribution over the heating drum. The heating treatment of the sirup may be carried out in an atmosphere of sufficient humidity to avoid much evaporation of water therefrom, as evaporation of considerable water is conducive to gelling. The use of a humid atmosphere may be especially desirable if the heat treatment is carried out at temperatures above 90° C., at which there would be considerable tendency for the evaporation of water from a film of sirup unless such evaporation were inhibited by a suitably humid atmosphere. The heat-ripened sirup may be kept hot for a while, although, as already indicated, it is usually desirable to effect its cooling promptly to temperatures downwards of 20° C., unless the ripened sirup is to serve immediately for the manufacture of artificial

silk filaments or the like. In fact, it may be desirable to cool the heat-ripened sirup to as low as 10° C., at which temperature it may be preserved for days without appreciable change, whereas a batch of ripened sirup at 22° C. must be spun within about five to six hours, in order to avoid non-uniformity of the artificial silk spun therefrom, by reason of the fact that the last portions of the batch are liable to undergo spontaneous variation from the first portions of the same batch. When the sirup is of unusually high viscosity, so that it may be spun only with difficulty at temperatures downwards of 20° C., it is advantageous to spin the heat-ripened sirup in a hot condition immediately as it is produced, as the hot sirup is of low viscosity and so lends itself to good spinning. The normal sirups, however, even when at temperatures as low as 10° C., not only spin satisfactorily, but generally yield a better quality of silk at low temperatures, because regeneration of cellulose is accompanied by less violent liberation of hydrogen sulphide, which at high temperatures tends to occlude to the nascent silk filaments and to break them. If desired, however, the heat-ripened sirup may initially be chilled to the desired temperature whereat it may be kept stable, and the cold sirup then heated to the desired temperature and viscosity immediately before spinning.

The embodiment of my invention hereinbefore described may further be varied while still falling within the ambit of the invention. For instance, rather than carrying out a one-stage heating of the sirup, the heating step may be performed in stages by using a series of two or more heating drums, the temperature imparted to each drum in the series being different from and controlled independently of the temperatures of the other drums.

I claim:

1. A process which comprises progressively depositing an unripened viscose sirup as a film on a heated surface under atmospheric pressure; and progressively removing the heated film of sirup from such heated surface in a ripened but ungelled condition.

2. A process which comprises progressively depositing an unripened viscose sirup as a film on a heated surface under atmospheric pressure, progressively removing the heated film of sirup from such heated surface in a ripened but ungelled condition; progressively depositing the hot, ripened but ungelled sirup on a cooled surface; and progressively removing the cooled film of ungelled sirup from such cooled surface.

3. A process which comprises depositing an unripened viscose sirup as a film on a heated surface under atmospheric pressure and enveloped in a humidified atmosphere serving to inhibit the evaporation of water from said sirup; and removing the heated film of sirup from such heated surface in a ripened but ungelled and substantially unconcentrated condition.

4. A process which comprises progressively depositing an unripened viscose sirup as a film under atmospheric pressure on a surface heated to a temperature of about 50° to 120° C.; progressively removing the heated film of sirup from such heated surface in a ripened but ungelled condition; progressively depositing the hot, ripened but ungelled sirup on a surface cooled to a temperature downwards of 20° C.; and progressively removing the cooled film of ungelled sirup from such cooled surface.

GEORGE A. RICHTER.