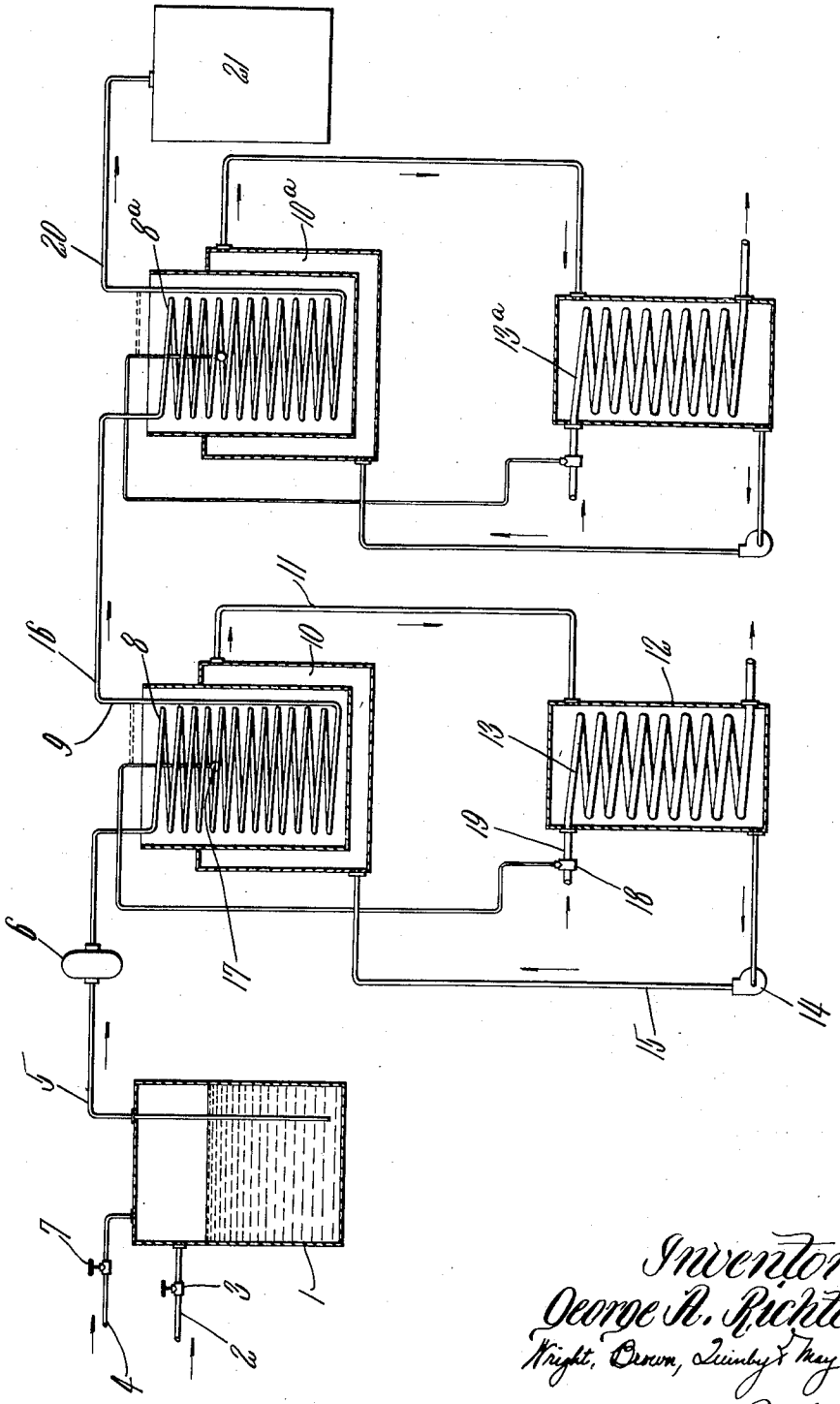
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RIPENING OF VISCOSE SIRUP

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RIPENING OF VISCOSE SIRUP

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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 spinning into 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 mechanical 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 to 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 thermal-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 should the sirup be damaged or completely spoiled, a delay of three to four days is experienced before the inventory is adjusted to catch up with the demand of the spinning machines, assuming that the plant is 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 negligible period, if the fresh or unripened sirup is heated to a temperature materially above normal room temperature, say, to about 50° to 90° C. The particular temperature to which the sirup is heated and the time period allotted for heating depend on various factors, especially upon the composition and those characteristics of the sirup which determine its tendency to split off combined sulphur and thereby to regenerate cellulose. For example, if the causticity of the sirup is below normal, i. e., below 6½% caustic soda content, the instability of the sirup is increased so that an elevated temperature at the low end of the range given coupled with a period of heating of even one minute may suffice to produce the desired ripening effect. On the other hand, other kinds of sirup may have to be heated for 15 minutes or more at the low end of the temperature range given before they reach the desired spinnable condition; and still other kinds of sirup may have to be heated to temperatures as high as 90° C., or even higher, at which temperatures, however, it is usually the case that the desired ripening action takes place quite rapidly. In any event, however, it is essential that the heat-ripened sirup either be chilled quickly to temperatures downwards of 20° C. or be spun practically as quickly as it is ripened in order to avoid the gelling attendant upon keeping the heated sirup in ripened condition for a substantial period of time. In actual commercial operation, it may be more expedient to chill the sirup to downwards of 20° C., as at such temperature it may be allowed to stand as a batch for some hours without danger of gelling. 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 have undergone 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 distinctly advantageous to spin the heat-ripened

sirup in a hot condition immediately as it is produced, as the hot sirup is of low solution viscosity and so lends itself to ready 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. Such heating promotes lowering of solution viscosity, which, as already indicated, is markedly affected by temperature change.

The principles of the present invention are applicable to a two-stage ripening process in which the first or second stage consists in permitting the sirup to ripen from, say, 12 to 24 hours, under the usual conditions. For instance, the fresh, unripened sirup may be first partially ripened in the usual manner at 20° C. over a 12 to 24-hour period, whereupon ripening may be completed with the application of heat in accordance with the present invention. Or the raw sirup may be first partially ripened with the application of heat in accordance with the present invention, whereupon ripening may be completed in the usual manner over a 12 to 24-hour period. During the preliminary ripening in the usual manner, the batch of sirup may be under vacuum to effect the removal of air or gas entrapped therein. A two-stage ripening process, such as described, lends itself to practice with a temperature of heating of the viscose sirup lower than that necessary when heating alone is relied upon to accomplish all the ripening of the sirup. Such a lower temperature of heating of the sirup is desirable in that it leads to a ripened sirup of better quality in those instances when the sirup being handled is sensitive to a higher temperature.

With these and other features and objects in view, the process of the present invention will now be described with reference to the accompanying drawing which illustrates more or less diagrammatically and conventionally one form of apparatus which may be employed for carrying out the process.

Referring now in detail to the drawing, 1 represents a closed tank into which the fresh or unripened sirup may be delivered by a pipe 2 from the filter press. The pipe 2 may be provided with a valve 3 so that after the batch of sirup has been discharged into the tank, the valve may be closed to cut off flow of sirup into the tank. Passing through the top of the tank is a pipe 4 which conducts compressed air from an air-compressor or compressed air chamber (not shown) to the air space above the batch of sirup in the tank. The compressed air serves to drive the sirup through a pipe 5 extending from the bottom of the tank upwardly through its top to a metering pump 6, for instance of the gear or piston type, which forces the sirup at predetermined controlled rate through a suitable heating system. The pipe 4 is preferably equipped with a valve 7 so that the air may be turned off when the tank is being supplied with a bath of sirup and turned on when the pump is put in operation to feed sirup through the heating system. The heating system may comprise a coil 8 within a jacketed tank 9, through the jacket 10 of which a suitable heating medium, such as water or oil, may be circulated at a rate designed to heat the sirup to the desired elevated temperature. The heating medium may be withdrawn from an upper side wall of the jacket through a pipe 11 into a heater 12 in which the medium receives heat from a steam coil 13, whereupon the heated medium may be withdrawn from the heater by a pump 14 and passed through a pipe 15 into the bottom of the jacket. In order to control the temperature of the heating medium so as to ensure heating of the sirup to the desired temperature, a thermostat 17 may be provided in the tank 9 or in the pipe 16, which conducts the heated sirup from the coil 8, the thermostat controlling the opening or closing of a valve 18 in the pipe 19 which supplies steam to the steam coil 13. A normal unripened sirup at an initial temperature of 20° C. need remain in the coil 8 for only about four minutes and be heated to a final temperature of about 75° C. to acquire a fully ripened condition for spinning. If desired, the hot, ripened sirup may be filtered, as in such condition it is very fluid and lends itself to flow through filtering media much more easily than sirups at room temperature or lower. The filter is preferably of a design permitting the presence of only a small volume of sirup in the filter at any time so as to avoid stagnation at any point in the filter, since, when these conditions are not fulfilled, there is liable to be gelling of the sirup.

The ripened sirup in heated condition may be delivered to the spinning machines and spun in such condition as previously described; and this mode of operation is of advantage when a sirup of high viscosity is employed or when it is highly viscous because of a higher than the usual 6½% cellulose concentraton. I do not, however, herein claim per se the step of spinning a hot viscose sirup as this is disclosed in my Patent #1,678,-354, issued July 24, 1928. According to the process of that patent, a highly viscous sirup, which has been ripened over a long period in accordance with the prior art practice, is heated while it is on its way to the spinnerets, in which case the viscosity of the sirup is vastly reduced, but the time during which the sirup is hot is so short as to have no appreciable ripening effect on the already ripened sirup. The process of the present invention is differentiated from that of my patent in that my present process involves the use of an unripened sirup as the raw material and the heating of the sirup to cause this ripening while at the same time permitting one to enjoy the benefit of the reduced viscosity of the heated sirup for spinning purposes, when such benefit is to be had. Ordinarily, however, it is preferable, as previously indicated, to cool the heat-ripened sirup to a temperature downwards of 20° C., say, to 10° C., before the sirup is spun. Accordingly, as depicted in the drawing, the heat-ripened sirup is conducted by the pipe 16 to a suitable cooling system which may be of a type similar to the sirup-heating system previously described, excepting that a cooling medium is passed through the jacket 10*a* and a refrigerating medium is circulated through the coil 13*a* which serves to maintain the cooling medium at the desired low temperature. The chilled sirup delivered from the cooling coil 8*a* through the pipe 20 may be discharged into a storage tank 21, from which it may be withdrawn as needed by the spinning machines.

Another advantage in chilling the sirup before spinning is that I am enabled by my process to ripen sirups which have a strong tendency toward gelling in ripened, hot condition, as is the case with viscous sirups having a lower than normal causticity. By chilling such heat-ripened sirups to low temperatures, it is possible to check the gelling tendency sufficiently so that there is no danger of sirup spoilage. It is thus possible when applying the principles of the present invention to economize in the use of caustic soda by producing unripened sirups of less than the normal 6½% caustic soda content and further to economize in the use of acid in the setting baths, as acid is consumed by reaction with the caustic soda. Still another advantage in chilling the heat-ripened sirup flows from the fact that ripening by heat may be overdone without fear of spoiling the sirup by gelling, as the quick chilling of an over-ripened sirup inhibits gelling tendencies sufficiently long to permit spinning. It is of advantage to over-ripen the sirup in certain instances, as I have found that by mixing or blending an unripened sirup with an over-ripened sirup, it is possible to spin silks having characteristics, difficultly, if at all, attainable otherwise. Thus it is possible to produce silks having higher strength than that of silk derived from a ripened or over-ripened sirup, and having softness far more pronounced than that of silk derived from an under-ripened or unripened sirup. This combination of high strength and softness is doubtless attributable to the fact that an over-ripened sirup usually yields silk filaments of low strength but of high softness, whereas an under-ripened sirup usually yields harsh silk filaments of high strength. By properly mixing or blending an over-ripened sirup with an under-ripened or unripened sirup, it is possible to realize a combination of both high strength and softness in the silk derived from the mixture.

What I claim is:

1. A process which comprises heating an unripened viscose sirup to a ripened condition short of gelling, and indirectly cooling the heat-ripened sirup quickly and artificially to a temperature at which the sirup may be kept in ungelled condition for a considerable period of time.

2. A process which comprises maintaining an unripened viscose sirup at temperatures of from 50° to 90° C. for a relatively short period of time to ripen it short of gelling, and indirectly and quickly cooling the heat-ripened sirup artificially to downwards of 20° C. to avoid gelling upon standing of the sirup.

3. A process which comprises progressively withdrawing unripened viscose sirup in controlled amount as a stream from a bulk supply, indirectly and quickly heating the stream to a ripened condition short of gelling, and then indirectly and quickly cooling the stream artificially to a temperature at which the ripened sirup may be kept in ungelled condition for a considerable period of time.

4. A process which comprises progressively withdrawing unripened viscose sirup in controlled amount as a stream from a bulk supply, indirectly and quickly heating the stream to a ripened condition short of gelling, and then indirectly and quickly cooling the stream artificially to downwards of 20° C. to check gelling tendencies therein.

5. A process which comprises progressively withdrawing unripened viscose sirup in controlled amount as a stream from a bulk supply, indirectly and quickly heating the stream to a temperature of about 50° to 90° C. and maintaining such temperature for about 1 to 15 minutes to ripen the sirup short of gelling, and then indirectly and quickly cooling the stream artificially to about 20° to 10° C. to check gelling tendencies therein.

6. A process which comprises progressively withdrawing unripened viscose sirup in controlled amount as a stream from a bulk supply, indirectly and quickly heating the stream to a ripened condition short of gelling, filtering the stream, and then indirectly and quickly cooling the stream artificially to a temperature at which the ripened sirup may be kept in ungelled condition for a considerable period of time.

7. A process which comprises indirectly heating an unripened viscose to a ripened condition short of gelling, and indirectly cooling the heat-ripened sirup quickly and artificially to a temperature at which the sirup may be kept in ungelled condition for a considerable period of time.

8. A process which comprises indirectly heating an unripened viscose sirup to a temperature of about 50° to 90° C. and maintaining it at such temperature for a short period of time to ripen it short of gelling, and indirectly and quickly cooling the heat-ripened sirup artificially to downwards of 20° C. to avoid gelling upon standing of the sirup.

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