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MANUFACTURE OF CELLULOSE ARTICLES FROM VISCOSE

Filed Aug. 14, 1946

2 Sheets-Sheet 1

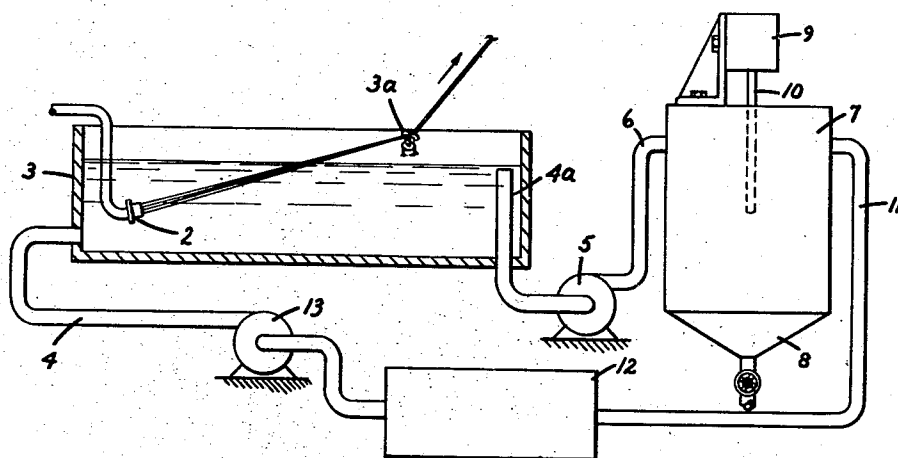


Fig. 1

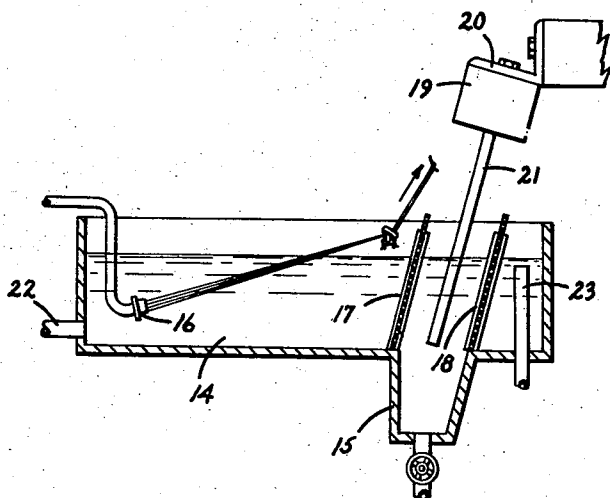


Fig. 2

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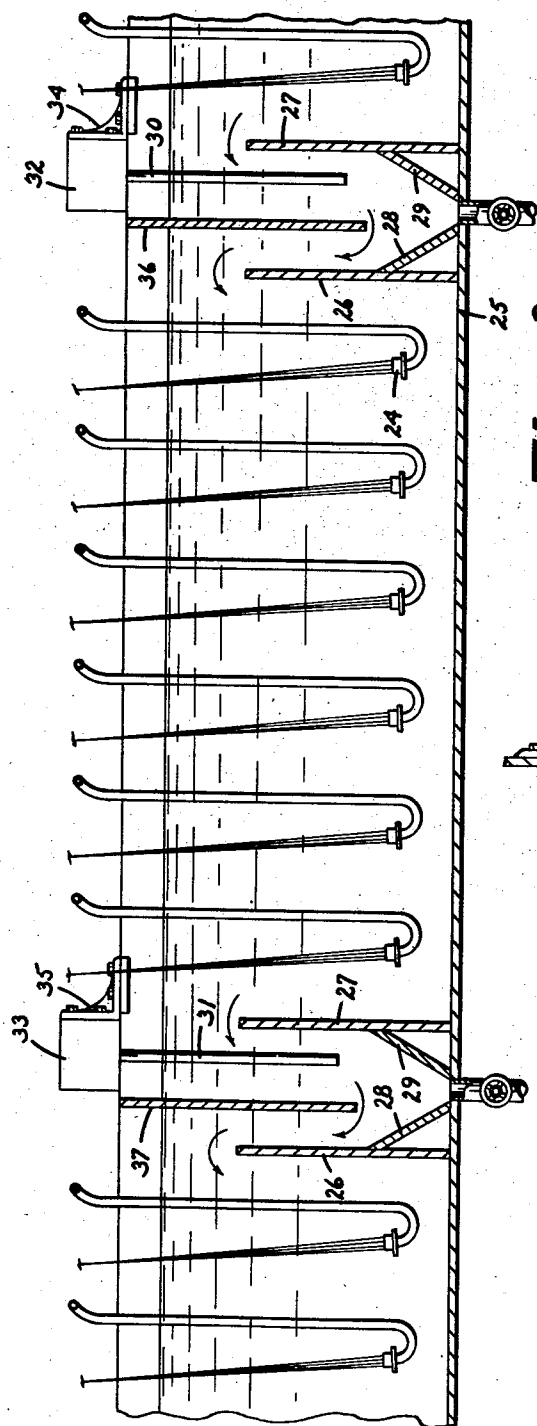


FIG. 3

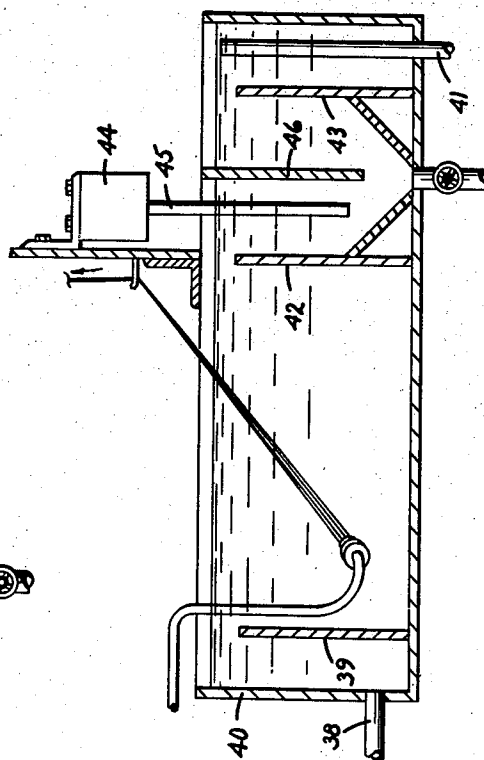


FIG. 4

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MANUFACTURE OF CELLULOSE ARTICLES FROM VISCOSE

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6 Claims. (Cl. 18—8)

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This invention relates to the manufacture of cellulose articles from viscose and to the treatment of acid setting baths used in the manufacture of such articles from viscose by wet spinning or casting methods.

In accordance with common practice, regenerated cellulose articles are manufactured by extruding the viscose into an acid setting bath, usually comprising an aqueous solution of sodium sulfate, sulfuric acid, and zinc sulfate or its equivalent. During the spinning or casting operations, the bath is fouled by impurities in the form of dispersed particles resulting from decomposition of the viscose, which impurities gradually accumulate in the bath and remain suspended therein. Eventually, the bath must be discarded or treated in some manner to remove the dispersed particulate impurities, to avoid discoloration and contamination of the regenerated cellulose articles being formed in the bath. Since large volumes of the bath are required, it is usual, for reasons of economy, to withdraw the fouled bath from the spinning tank or other vessel, clarify it by removal of the impurities arising from the viscose decomposition, condition it for re-use (as by the removal of excess sodium sulfate built up therein during the spinning operation and the addition of sulfuric acid thereto), and then return the bath to the spinning tank.

The impurities or contaminants resulting from decomposition of the viscose occur in the form of extremely fine solid particles comprising sulfur and sulfur compounds including higher thionic acids apparently produced by reaction between the sulfur dioxide and hydrogen sulfide liberated during regeneration of the cellulose as well as compounds formed by reaction of the thionic acids with the metal commonly lead, comprising the vessel containing the bath and in which the casting or spinning operation is performed. Removal of the solid suspended impurities from the bath to the extent required to obtain a bath of the necessary high degree of clarity is greatly complicated by the extremely finely divided condition of the impurities in the bath. Filters of ordinary type cannot be used for clarifying the bath because the particles are so fine that they readily pass through the filter with the solution. Although the setting bath may be clarified satisfactorily by passing it through special filters, such as charcoal filters, which absorb or adsorb impurities from acid media, such filters are relatively expensive, and, furthermore, require quite frequent removal from service for back-washing.

It is possible, of course, to permit the bath to

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stand in a settling tank or the like until the impurities settle out of suspension and collect as sediment at the bottom of the tank, but the impurities are so fine and settle out so gradually under ordinary conditions, that the time required to obtain a clarified bath of the requisite high degree of clarity is too long to render that method of clarification feasible for use in conjunction with continuous processes for the manufacture of cellulose articles from viscose on a commercial basis.

It is one object of this invention to provide an improved method for the manufacture of cellulosic articles from viscose, by the wet spinning method, in which the setting is continuously clarified, conditioned for re-use, and recirculated to the spinning or casting vessel. Another object is to provide an improved method of clarifying contaminated acid setting baths resulting from the decomposition of viscose to form regenerated cellulose articles. Another object is to increase the rate at which the solid contaminants present in such baths settle out or precipitate from the solution. A further object is to provide an improved method of clarifying the baths which is adaptable to use in conjunction with and as a stage in continuous spinning or casting operations.

The objects of the invention are accomplished by propagating in the contaminated baths high frequency sound waves having a frequency in the range of about 1000 to a million or more cycles per second, herein called high frequency sound waves, until the dispersed particulate contaminants suspended therein have settled out and a substantially clear solution which is free of such contaminants is obtained. Under the influence of the high frequency sound wave radiations, the rate of settling out of the impurities is greatly accelerated and, usually, the sediment is produced and the desired clear solution is obtained in a relatively short time, seconds or minutes, as compared with the hours required for normal settling. The high frequency sound wave radiations have the effect of aggregating the original particles, which are usually microscopic, into larger aggregates which may be macroscopic. Baths containing the aggregated particles may be clarified by passing the bath through conventional acid proof filters or centrifuges which will retain the aggregated impurities.

The high frequency sound wave radiations may be generated by means of any suitable sound generating device, such as a suitably insulated piezoelectric sound generator, a magnetostriction

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sound generator, or an electromagnetic sound generator. The waves may be propagated in the contaminated bath after it has been withdrawn from the extrusion zone, and intermediate the tank and a regenerating or reconditioning system in which the relative proportions of the constituents of the bath are readjusted to condition it for re-use. Again, a sound wave irradiation zone may be established in the path of the bath as it flows through the spinning vessel or tank, beyond the point of extrusion of the viscose into the bath, and the bath may be passed at a controlled rate through the irradiation zone as it flows toward the exit end of the vessel to effect rapid settling out of the suspended impurities present therein as a result of the viscose decomposition. The sediment thus produced at the bottom of the vessel or tank may be withdrawn without interrupting the spinning operation. Within the scope of the invention, the fouled bath may be simultaneously or successively subjected to irradiation by sound waves having different frequencies within the range stated, and of different magnitudes.

The spinning bath may be degassed in any known or appropriate manner, prior to propagating the sound waves therein, to prevent dissipation of the vibrational energy due to the presence of gas bubbles, such as bubbles of hydrogen sulfide gas, which may occur in the bath.

The accompanying drawing is illustrative of apparatus suitable for practicing the invention. In the drawing,

Figure 1 is an elevation view, partly in section of apparatus suitable for carrying out one embodiment of the invention;

Figure 2 is an elevation view, partly in section, of apparatus suitable for carrying out another embodiment of the invention.

Figure 3 is an elevation view, partly in section of another modification of the invention; and

Figure 4 is a side elevation, partly in section, of a still further modification.

Referring to Figure 1, the viscose is continuously extruded through a spinneret 2 positioned in vessel 3 to extrude the viscose in a generally horizontal direction into the acid regenerating bath which is continuously introduced into the vessel, at a controlled constant rate of speed, through pipe 4 to form fibers of regenerated cellulose which are continuously withdrawn from the bath through a guide 3a. The bath in vessel 3 is maintained at a constant level, an overflow pipe 4a being provided for this purpose. The overflow is continuously withdrawn by the action of pump 5, and circulated through pipe 6 to a settling tank 7 having a cone-shaped bottom 8. A sound generator 9 is supported above the settling tank 7 and has associated with it a tube 10 which projects into the tank to transmit the vibrational energy to the bath entering the tank to effect rapid settling out of the dispersed particulate impurities. The impurities collect in the cone-shaped bottom of the tank to produce a sediment which may be withdrawn either continuously or intermittently to a sludge collector or the like. The clarified supernatant liquid eventually overflows the tank and is circulated through pipe 11, to a regenerating or reconditioning system 12, in which it is conditioned for re-use, after which it is recirculated, by the action of pump 13, through pipe 4 to the spinning vessel 3. The rate at which the bath is delivered to vessel 3, and the rate at which the spent bath is withdrawn from the vessel and discharged to and from the settling tank 7, is controlled to

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insure viscous non-turbulent flow of the bath through the sound wave field, so that the contaminated solution is exposed to the action of the waves for a time sufficient to permit deposition of the aggregated impurities.

Instead of continuously circulating the bath through a field of high frequency sound waves, the clarification may be effected on a batch basis, that is, the spent bath may be withdrawn from the spinning vessel and stored for a predetermined period of time in a settler similar to tank 7 in which it is subjected to the high frequency sound wave radiations, to produce a sediment consisting of the dispersed particulate impurities, after which the clarified bath may be conditioned for re-use.

Figure 2 is illustrative of apparatus which may be used when it is desired to effect clarification of the bath while the bath is present in the spinning vessel.

As shown in Figure 2, the bottom of vessel 14, in which the spinning operation is performed, is provided with a depression forming a trap 15, at a point removed from the spinneret 16 positioned therein to spin the viscose in a generally horizontal direction into the setting bath. A pair of acid proof filters or screens 17 and 18 are fitted into grooves in the inner walls of vessel 14, so that they extend across the vessel in inclined relation to the inner walls thereof, and on either side of trap 15. A sound generator 19 is secured in a bracket 20, mounted on the face of the spinning machine above the level of vessel 14, and the transmission tube 21 associated with the generator projects into the vessel, between the elements 17 and 18 and above trap 15, to transmit the vibrational energy to the bath. The bath, which is continuously introduced into vessel 14 through pipe 22 at a constant controlled rate of speed, and withdrawn therefrom at a constant controlled rate to insure viscous non-turbulent flow thereof, passes through element 17 into the sound wave field. The impurities which accumulate in the bath as the viscose is decomposed, are sufficiently fine to be carried by the bath through filter or screen 17, but under the action of the sound waves upon the original fine particles suspended in the non-turbulent bath, the latter are brought into closer contact with one another and are quickly aggregated to a size favoring rapid sedimentation. The aggregated impurities do not pass through the filter 18. The particles settle out into trap 15, from which the sediment may be withdrawn, either continuously or periodically. The clarified supernatant liquid passes through screen 18 and is withdrawn from the vessel over overflow pipe 23, being forwarded to a regenerating or reconditioning system and recirculated to vessel 14 in the same manner as described in connection with Figure 1.

The arrangement shown in Figure 2 may be modified to provide a plurality of depressions or traps 15 at spaced points along the length of a conventional spinning vessel and beyond each of the spinnerets positioned therein, and a plurality of pairs of filters or screens 17 and 18 may be supported in the vessel in inclined relation to the walls thereof and on either side of each of the traps, to define a series of vibration or irradiation zones into which a transmission tube associated with a sound generator projects, to subject the contaminated bath to the high frequency sound waves at different points along the path of viscous flow of the bath through the vessel.

Instead of providing an irradiation zone beyond

each of the individual spinnerets positioned in the spinning vessel, the irradiation zones may be spaced along the length of the vessel, between groups of spinnerets, as shown in Figure 3. As shown, the spinnerets 24 are positioned in vessel 25 to spin the viscose generally vertically upwardly into the spinning bath which is continuously introduced at one end of the vessel and withdrawn, at a constant controlled rate, at the opposite end. The bath flows longitudinally of the vessel and is forced to flow in the direction of the arrows, over a plurality of pairs of spaced weirs 26 and 27 (two pairs being shown), the weirs having sloped sides 28 and 29 defining traps in which the sediment produced under the vibrational action of transmission tubes 30 and 31 associated with the sound generators 32 and 33 mounted in brackets 34 and 35 on the face of the machine is collected. Baffle plates 36 and 37 are supported on the vessel and project into the irradiation zone adjacent the tubes 30 and 31. The baffle plates serve to deflect the bath downwardly and to retard the flow thereof through the vessel to afford ample opportunity for the impurities present in the bath to settle in the traps at the bottom of the vessel, from which the sediment may be withdrawn to a waste disposal system. Irradiation zones defined by pairs of spaced weirs similar to weirs 26 and 27 and into which the transmission tube associated with a sound generator projects, may be provided beyond a group of six spinnerets as shown, or beyond a group of spinnerets consisting of two spinnerets or more, or the irradiation zones may be provided beyond each individual spinneret, in the path of the flow of the bath from the inlet to the outlet end of the vessel, and may be provided at regular or irregular intervals along the length of the machine. That is, depending upon prevailing conditions, the acid setting bath may be passed into a sound wave irradiation zone as frequently as is necessary during the spinning operation to insure that the bath into which the viscose is extruded has the necessary high degree of clarity.

The arrangement for effecting clarification of the contaminated bath shown in Figure 4 is similar to that illustrated in Figure 3, but there the bath entering the spinning vessel through pipe 38 is forced over a weir 39 and then flows transversely of the vessel 40, and as shown, generally transversely of the direction of spinning, being withdrawn at the opposite end of the vessel through pipe 41. Although only one irradiation zone is shown in Figure 4, defined by the weirs 42 and 43, and only one sound generator 44 having a transmission tube 45, and a single baffle plate 46 are shown in the drawing, it will be readily understood that a plurality of irradiation zones may be provided along the length of vessel 40 and that such zones may be provided beyond each of the spinnerets positioned in the vessel, or beyond a group of spinnerets comprising two or more spinnerets. In Figure 4, the spinneret is shown positioned to spin the viscose generally horizontally into the bath. However, it will be apparent, that in any of the arrangements illustrated, the spinneret or spinnerets may be positioned for either horizontal or vertical spinning.

Various changes and modifications may be made in carrying out the method described herein without departing from the spirit and scope of the invention as defined in the appended claims.

I claim:

1. Method comprising introducing an acid set-

ting bath into a vessel, at one end thereof and withdrawing the bath from the vessel at the other end thereof, and, intermediate the entrance and exit ends of the vessel, passing the bath through an extrusion zone in which viscose is extruded into the bath to effect decomposition of the viscose and regeneration of cellulose hydrate by the action of the bath, and then through a zone of high frequency sound wave radiations at a controlled rate to effect rapid sedimentation of the dispersed particulate impurities introduced into the bath as a result of decomposition of the viscose and clarify the bath under the influence of the sound wave radiations.

2. Method comprising continuously introducing an acid setting bath into a vessel, continuously extruding viscose into the bath to effect decomposition of the viscose and regeneration of cellulose hydrate by the action of the bath, continuously propagating high frequency sound waves in the bath, at a point in the line of flow thereof through the vessel and beyond the point of extrusion of the viscose into the bath, to effect rapid sedimentation of the dispersed particulate impurities introduced into the bath as a result of decomposition of the viscose, continuously withdrawing the clarified bath from the vessel, at a rate relative to the rate of introduction of the bath into the vessel such that the flow of the bath through the vessel is non-turbulent and viscous, continuously conditioning the bath for re-use for regenerating cellulose hydrate from viscose, and continuously returning the conditioned clarified bath to the vessel.

3. Apparatus for use in the manufacture of regenerated cellulose articles from viscose comprising a vessel, means for continuously introducing an acid setting bath into the vessel, and for continuously withdrawing the bath from the vessel, at a controlled rate to insure non-turbulent viscous flow of the bath through the vessel, means positioned in the vessel for continuously extruding viscose into the bath, means within the vessel and at a distance beyond the spinneret in the path of flow of the bath through the vessel from the inlet to the outlet means for continuously subjecting the bath to high frequency sound wave irradiations to effect sedimentation of impurities resulting from decomposition of the viscose and clarify the bath under the influence of such radiations, and means for collecting the sediment thus produced.

4. Apparatus for use in the manufacture of regenerated cellulose articles from viscose comprising a vessel, means for continuously introducing an acid setting bath into the vessel, at one end thereof, and for continuously withdrawing the spinning bath from the vessel at the other end thereof at a controlled rate to insure non-turbulent viscous flow of the bath through the vessel, means positioned in the vessel for continuously extruding viscose into the bath, means within the vessel defining a relatively narrow compartment in the path of flow of the bath through the vessel from the inlet to the outlet means and beyond the extrusion means, and means projecting into the compartment for subjecting the bath flowing away from the extrusion means and containing dispersed particulate impurities resulting from decomposition of the viscose to high frequency sound wave radiations to effect sedimentation of the impurities and clarify the bath.

5. Apparatus for use in the manufacture of

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regenerated cellulose articles from viscose comprising a vessel, means for continuously introducing an acid setting bath into the vessel, and means for continuously withdrawing the bath from the vessel at a controlled rate to insure non-turbulent viscous flow of the bath through the vessel, a plurality of spinnerets positioned in the vessel for continuously extruding viscose into the bath, means positioned within the vessel at regularly spaced intervals beyond the first and last of any predetermined number of the spinnerets in the path of flow of the bath through the vessel from the inlet to the outlet means for continuously subjecting the bath to high frequency sound wave radiations to effect sedimentation of impurities resulting from decomposition of the viscose and clarify the bath under the influence of such radiations, means for collecting the sediment thus produced, and means for withdrawing the sediment from the bottom of the vessel.

6. Apparatus for use in the manufacture of regenerated cellulose articles from viscose comprising a vessel, means for continuously introducing an acid setting bath into the vessel, at one end thereof, and for continuously withdrawing the spinning bath from the vessel at the other end thereof at a controlled rate to insure non-turbulent viscous flow of the bath through the vessel, a plurality of spinnerets positioned in the vessel for continuously extruding viscose into the

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bath, means within the vessel defining relatively narrow compartments at regularly spaced intervals beyond the first and last of any predetermined number of the spinnerets in the path of flow of the bath through the vessel from the inlet to the outlet means, means projecting into the compartments for subjecting the bath flowing away from the spinnerets and containing dispersed particulate impurities resulting from decomposition of the viscose to high frequency sound wave radiations to effect sedimentation of the impurities and clarify the bath, and means for withdrawing the sediment from the bottom of the vessel.

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