July 11, 1950 J. A. CALHOUN 2,514,471

MANUFACTURE OF CELLULOSE ARTICLES FROM VISCOSE

Original Filed Aug. 14, 1946 2 Sheets-Sheet 1

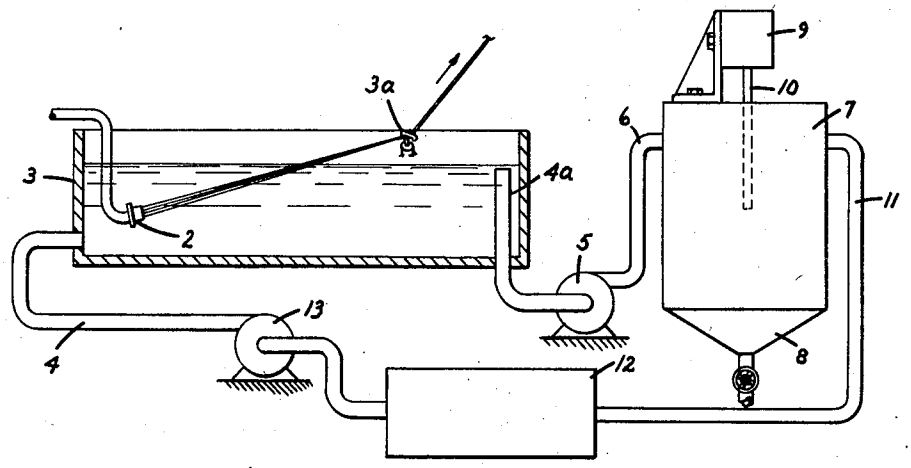

Fig. 1

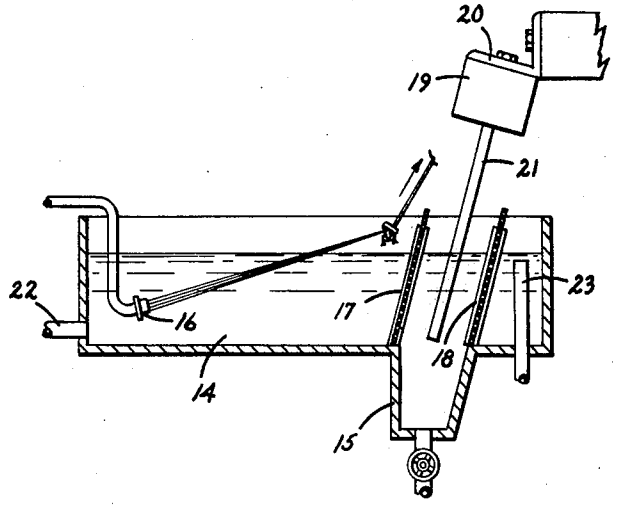

Fig. 2

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MANUFACTURE OF CELLULOSE ARTICLES FROM VISCOSE

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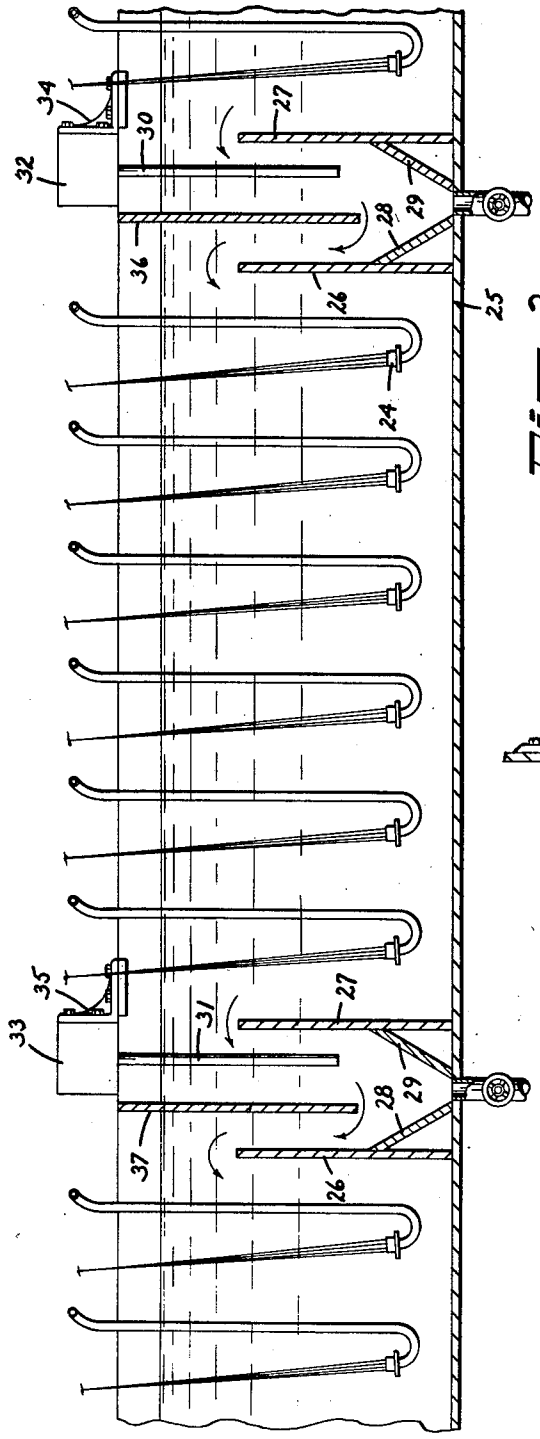
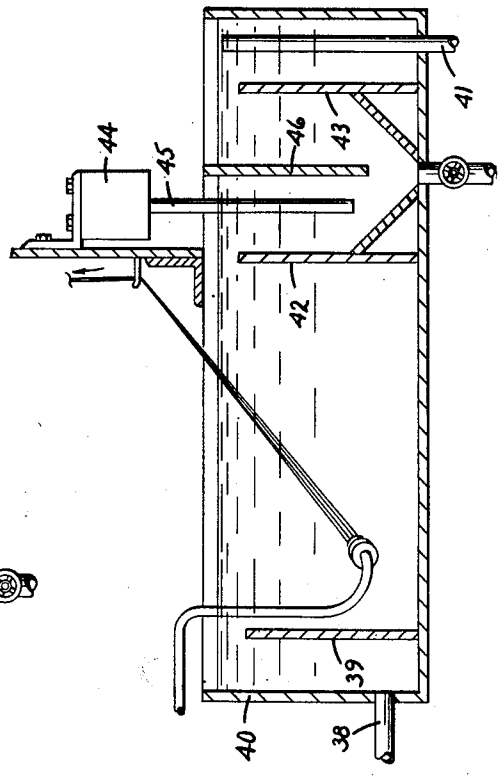


FIG. 3

FIG. 4



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MANUFACTURE OF CELLULOSE ARTICLES FROM VISCOSE

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7 Claims. (Cl. 18—8)

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This invention relates to the manufacture of cellulose articles from viscose.

This is a division of my application Serial No. 690,546 filed August 14, 1946, now Patent No. 2,484,013.

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 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 bath 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 clarified 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 piezo-electric sound generator, a magneto-stric-

tion 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 reconditioning or regenerating 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 degassified 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 **3***a*. The bath in vessel **3** is maintained at a constant level, an overflow pipe **4***a* 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 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 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 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 extruding viscose into an acid setting bath to effect decomposition of the viscose and regeneration of cellulose hydrate by the action of the bath, withdrawing the bath containing dispersed particulate impurities resulting from decomposition of the viscose from the zone of extrusion of the viscose, propagating high frequency sound waves in the fouled bath to effect rapid sedimentation of the impurities and clarify the bath under the influence of the waves, and separating the clarified supernatant bath from the sediment thus produced.

2. Method comprising extruding viscose into an acid setting bath to effect decomposition of the viscose and regeneration of cellulose hydrate by the action of the bath, withdrawing the bath containing dispersed particulate impurities resulting from decomposition of the viscose from the zone of extrusion of the viscose, propagating high frequency sound waves in the fouled bath to effect rapid sedimentation of the impurities and clarify the bath under the influence of the waves, separating the clarified supernatant bath from the sediment thus produced, conditioning the bath for re-use in the regeneration of cellulose hydrate from viscose, and returning the bath to the zone of extrusion of the viscose.

3. Method comprising continuously extruding viscose into an acid setting bath to effect decomposition of the viscose and regeneration of cellulose hydrate by the action of the bath, continuously withdrawing the bath containing dispersed particulate impurities resulting from decomposition of the viscose from the zone of extrusion of the viscose, continuously passing the fouled bath through a field of high frequency sound waves at a controlled rate to effect rapid sedimentation of the impurities in the field, continuously circulating the clarified bath to a reconditioning zone for reconditioning for re-use in the regeneration of cellulose hydrate from viscose, and continuously circulating the clarified reconditioned acid setting bath to the zone of extrusion of the viscose.

4. Method comprising continuously extruding viscose into an acid setting bath to effect decomposition of the viscose and regeneration of cellulose hydrate by the action of the bath, continuously withdrawing the bath containing dispersed particulate impurities resulting from decomposition of the viscose from the zone of extrusion of the viscose and circulating it to a reconditioning zone, and intermediate of the extrusion and reconditioning zones, subjecting the fouled bath to high frequency sound wave radiations to effect rapid sedimentation of the impurities and produce a clarified solution under the influence of the waves.

5. Apparatus for use in the manufacture of regenerated cellulose articles from viscose comprising a vessel, means for introducing an acid setting bath into the vessel, at one end thereof, means for withdrawing the bath at a controlled rate from the vessel, means for extruding the viscose into the bath, and means associated with the vessel for subjecting the bath, after contamination thereof with 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 under the influence of such radiations.

6. Apparatus for use in the manufacture of regenerated cellulose articles from viscose comprising, in combination, a vessel, means for introducing an acid setting bath into the vessel, at one end thereof, means positioned in the vessel for extruding the viscose into the acid bath, means for withdrawing the spent bath containing dispersed particulate impurities resulting from decomposition of the viscose from the vessel, at the other end thereof, a second vessel, means for circulating the spent bath to the second vessel, and means associated with the second vessel for subjecting the spent bath to high frequency sound wave radiations in said vessel to effect sedimentation of the impurities and clarify the bath.

7. Apparatus for use in the manufacture of regenerated cellulose articles from viscose comprising, in combination, a vessel, means for continuously introducing an acid setting bath into the vessel, at one end thereof, means positioned in the vessel for continuously extruding the viscose into the acid bath, a second vessel, means for continuously withdrawing the spent bath containing dispersed particulate impurities resulting from decomposition of the viscose from the first vessel, and for circulating the spent bath to the second vessel at a controlled rate, means associated with the second vessel for continuously subjecting the bath contained therein to high frequency sound wave radiations to effect sedimentation of the impurities in said vessel and clarify the bath, and means for continuously withdrawing the clarified bath from the second vessel.

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No references cited.