Aug. 10, 1943. R. D. MacLAURIN ET AL 2,326,150

MANUFACTURE OF THREAD OR THE LIKE

Filed July 29, 1940 3 Sheets-Sheet 1

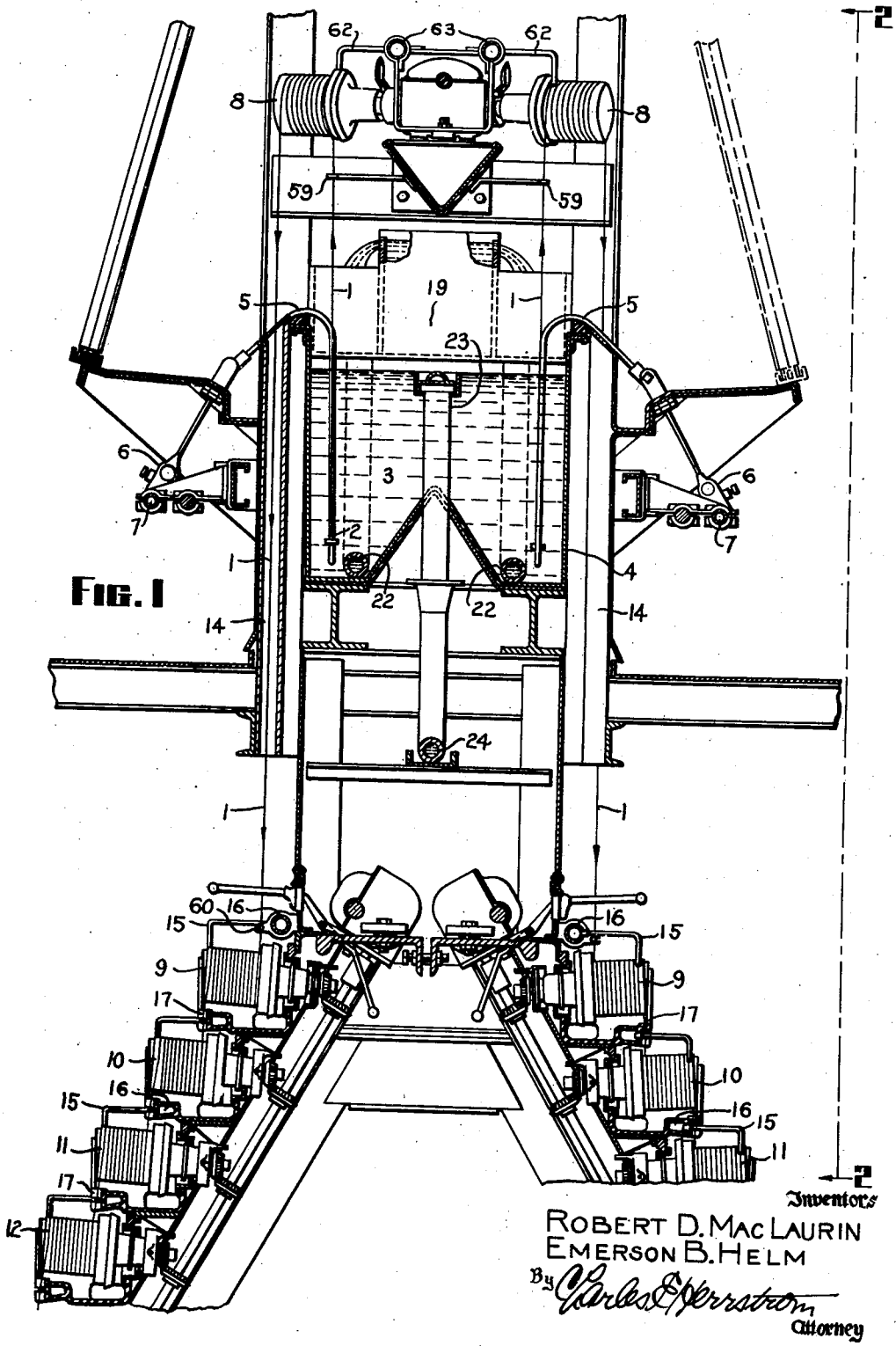


FIG. 1

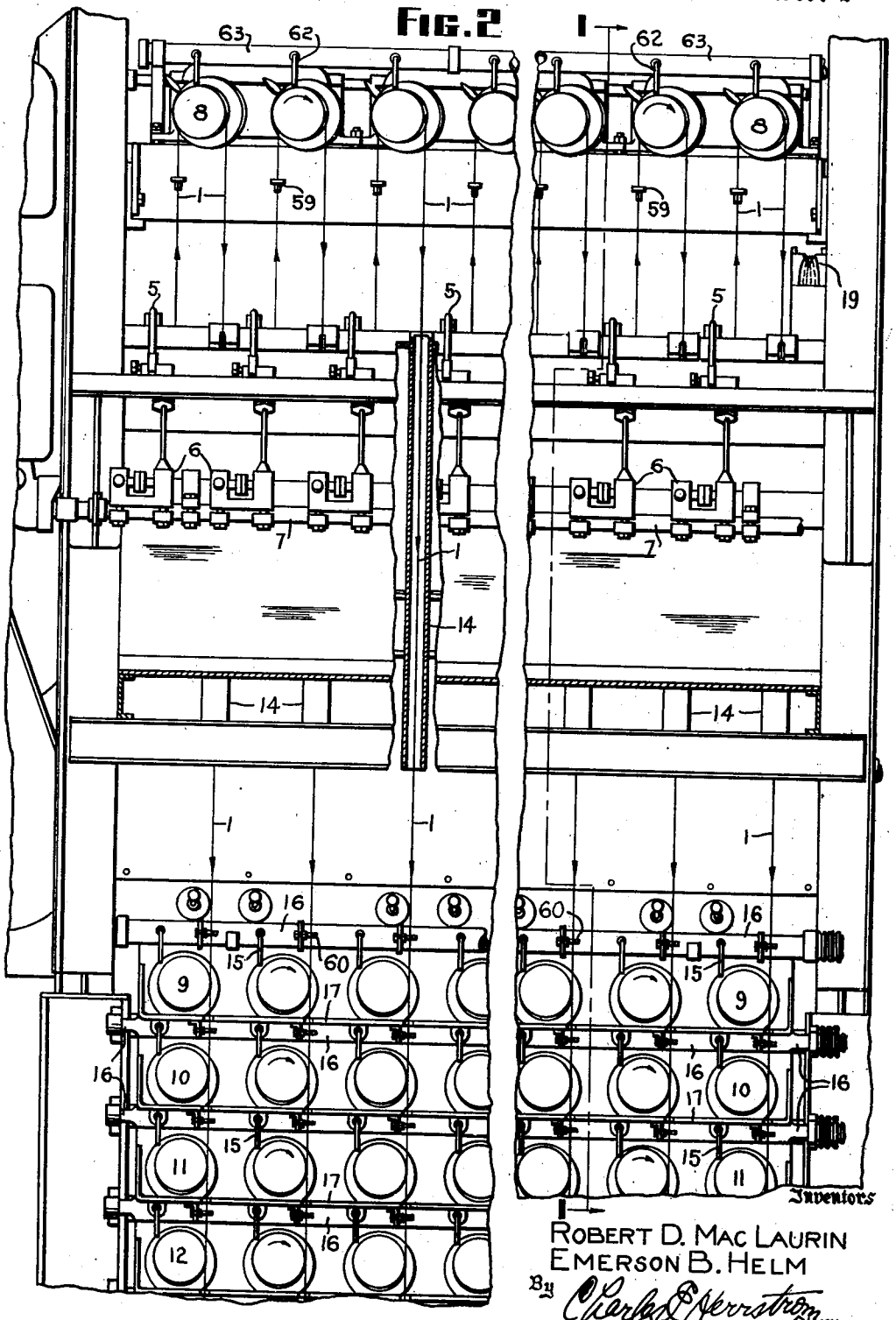
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MANUFACTURE OF THREAD OR THE LIKE

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FIG. 2



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MANUFACTURE OF THREAD OR THE LIKE

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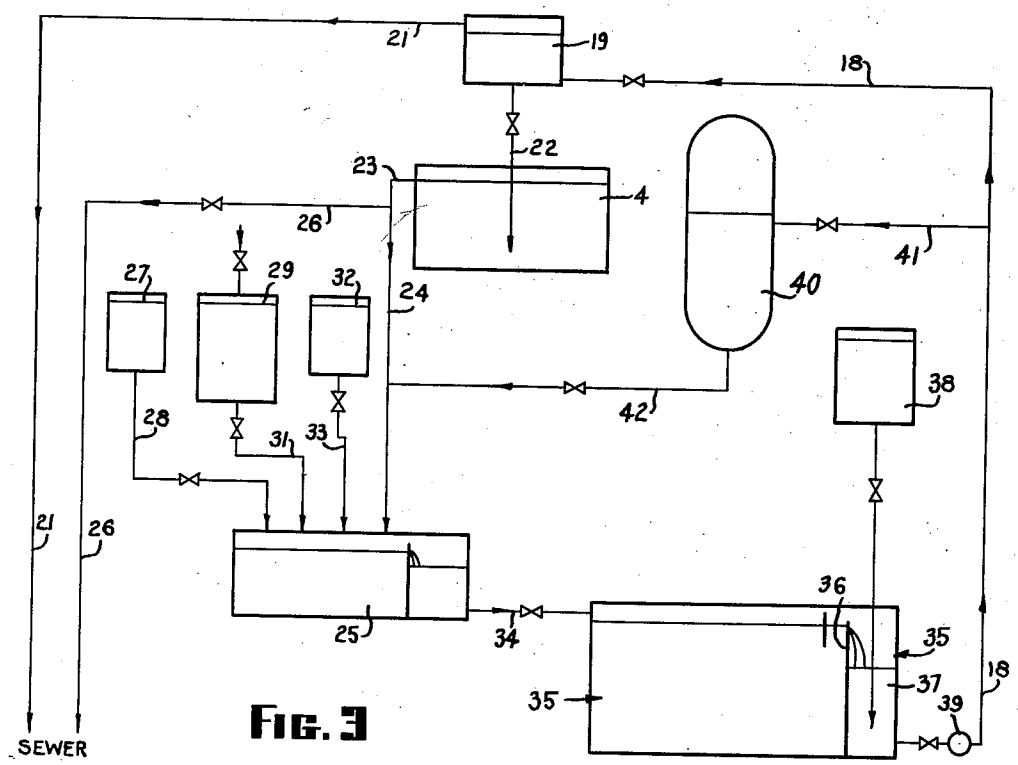


FIG. 3

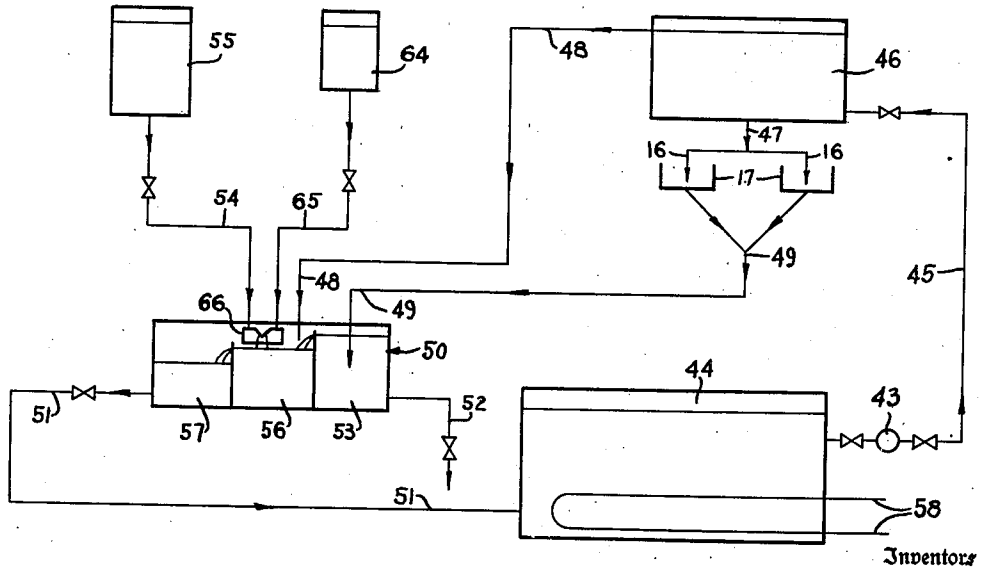


FIG. 4

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UNITED STATES PATENT OFFICE

2,326,150

MANUFACTURE OF THREAD OR THE LIKE

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Application July 29, 1940, Serial No. 348,082

11 Claims. (Cl. 18—54)

This invention relates to the manufacture of filaments, threads, cords, ribbons and other forms of strand material, all of which are referred to hereinafter by the generic term "thread." For purposes of illustration, the features of novelty characterizing the invention will be described in connection with the manufacture of viscose artificial silk thread. It will, however, be understood that the invention may be employed, at least in certain of its aspects, in connection with the manufacture of other products formed by the extrusion of a coagulable mass into a coagulating liquid.

Viscose artificial slik thread is formed by extruding an aqueous solution of sodium cellulose xanthate (viscose) into an acid coagulating liquid which ordinarily includes sulfuric acid, sodium sulfate, a small amount of zinc sulfate and, if desired, other substances having beneficial effects on the thread. After its extrusion, the liquid viscose first solidifles without undergoing any important chemical reaction; that is, the material of which the thread is formed consists at first of only solidified sodium cellulose xanthate. Subsequently, the cellulose content of the viscose is regenerated with the formation of by-products such as sodium sulfate, carbon disulfide, hydrogen sulfide and free sulfur. The sulfur is insoluble, as are also sundry other substances, such as compounds of silicon, lead, zinc, and often nickel, which are present as impurities in the coagulating bath.

Regeneration of the cellulose does not occur instantaneously but requires an appreciable period of time; indeed, a very considerable part of the regeneration takes place only after the thread leaves the coagulating bath, during which time by-products such as sulfur are formed in and upon the thread. Such by-products, as well as impurities carried by the thread from the coagulating bath, tend to deposit on guides and other parts of the thread handling apparatus, with which the thread later comes into contact, forming incrustations which tend to damage the thread. Similarly, salts present in the coagulating liquid carried by the thread tend to crystallize out in the form of deposits on parts of the apparatus with which the thread comes into contact.

The problem is particularly acute in the manufacture of thread by a continuous process. In such a process, thread proceeding from the spinneret to the collecting device is subjected to processing treatment on a series of rotating thread-advancing reels each of which continuously but temporarily stores a long length of thread for an appreciable period of time in a large number of helical turns. Conditions are therefore such as to favor the deposition of the above-indicated substances on the reels, particularly if the cellulose is being regenerated while the thread is stored thereon. The deposition of such substances may, if not inhibited, result in the formation of incrustations which damage the thread severely, largely because wet thread is particularly delicate.

Such difficulties are eliminated according to the present invention by the application of a substance which prevents such deposition on the parts of the apparatus which are in contact with the thread and on which such incrustations therefore tend to form. Such substance is preferably an oleaginous material which operates by wetting the insoluble substances which tend to form such incrustation and/or by forming a film over the parts of the apparatus with which the thread comes into contact, thus preventing such substance, whether an insoluble substance or a crystallized salt, from adhering to the apparatus. The added substance may be applied directly to the part of the apparatus with which the thread comes into contact, incorporated in a processing liquid applied to the thread, or carried to such part by the thread itself, as hereinafter explained.

For the purposes of illustration, the invention will be described as employed in the manufacture of multiple filament viscose artificial silk thread on apparatus generally of the type disclosed in Knebusch and Burkholder Patent 2,225.642.

In the drawings, Figure 1 represents a cross section from line 1—1 of Figure 2 of a portion of a machine for the manufacture of viscose artificial silk thread similar to that disclosed in said patent. Figure 2 is a side elevation, from line 2—2 of Figure 1, of the portion of the machine illustrated in Figure 1. Figure 3 is a flow diagram of the circulation system for the coagulating liquid employed in said machine. Figure 4 is a flow diagram of the circulation system for a processing liquid applied to the thread after its coagulation in the usual coagulating trough.

As indicated in Figures 1 and 2, each thread 1 is formed by the extrusion of viscose through a spinneret 2 into a coagulating bath 3 contained in trough 4. The viscose is supplied to each spinneret 2 by a mass tube 5 through which the viscose is forced by suitable means, such as

a gear pump 6 associated with a viscose supply pipe 7 common to all pumps 6 on the same side of the machine. When the viscose comes into contact with the coagulating bath, a solidified thread 1 is formed which may be and preferably is drawn vertically upward to and by a first thread-advancing reel 8 of a descending series of several such reels. Five thread-advancing reels bearing reference numbers 8 to 12, inclusive, are shown in each series, although more or less may be employed.

Each of said thread-advancing reels 8 to 12, inclusive, is supported from one end only and is adapted, during rotation, to store thread continuously but temporarily in a plurality of closely spaced, generally helical turns which are advanced progressively from the supported to the unsupported end of the reel. The thread-supporting surface of each reel consists of a plurality of circumferentially spaced, axially extending bars which cooperate to advance the thread in the manner stated, the illustrated reel being in general of the type described in said Patent 2,225,642. The reels shown in the drawings of the present application are in semi-diagrammatic representation, the indicated spacing of thread turns being considerably greater than the spacing of thread turns actually employed in the apparatus.

All of the reels of each descending series are disposed in stepped relation with their unsupported ends extending in the same direction; i. e., toward the operating face of the machine on the side thereof on which such reels are located. The unsupported or thread discharge end of each preceding reel is in apposite relation to the supported or thread-receiving end of each succeeding reel; accordingly, the thread can pass downward from the unsupported end of each reel to the supported end of the succeeding reel. In such an arrangement, not only are the reels readily accessible for operating purposes but the thread can pass directly from reel to reel without undergoing detrimental bending or guide stresses.

In the apparatus illustrated, a plurality of such descending series of reels is disposed on each side of a common longitudinally extending coagulating trough 4. As shown in Figure 1, the banks of reels, when viewed in transverse cross section, resemble an inverted V. Corresponding reels of all downwardly extending series on each side of the machine are disposed in rows extending lengthwise of the machine, which makes possible the supply and removal of processing liquids by means of common longitudinally extending conduits on each side of the apparatus. It also provides various other practical advantages, such as construction economies.

After being withdrawn from the coagulating bath, each thread 1 is advanced from the supported to the unsupported end of one of the reels 8, being discharged from the reel beyond the lateral edge of trough 4. No thread-processing liquid is applied to the thread on reel 8; instead, there is provided a period for furthering the solidification and regeneration of the thread. This is accomplished by storing a relatively long length of thread on said reel before the thread is subjected to processing liquid on succeeding reels 9 to 12, inclusive. As the thread 1 passes from reel 8 to the supported end of reel 9, it proceeds vertically downward through a channel 14 associated with the longitudinally extending side of coagulating trough 4.

On the reels subsequent to reel 8, liquid is applied to the thread by means of a reagent distributor 15 communicating with a conduit 16 which extends longitudinally of the machine. Said conduit 16 supplies all corresponding processing reels on one side of the apparatus. A longitudinally extending collecting trough 17 which communicates with the sewer or with suitable recirculation means extends lengthwise of the machine beneath each such horizontal series of reels. In the embodiment of the invention illustrated, conduits 16 for reels 10 to 12, inclusive, are formed integrally with the collecting troughs 17 above such reels.

It is desirable that each of the reels on which liquid is applied to the thread shell be so operated that a film of processing liquid extending entirely around and for substantially the length of its thread-bearing periphery is formed upon the reel. In the illustrated apparatus, the turns of thread on each of reels 9 to 12, inclusive, are spaced sufficiently close together to permit the processing liquid which is applied to the thread to form such a film. The reel is preferably rotated at a speed such that the centrifugal force which is developed helps to maintain the film.

Liquid is applied to the unsupported end of the reel, which is elevated slightly above the supported end so that countercurrent flow is obtained: under such conditions, the formation upon the reel of the film of processing liquid is promoted.

As previously mentioned, each thread 1 is formed by extrusion of viscose into a bath containing dilute sulfuric acid, after which it is stored on reel 8 in order to permit coagulation of the thread to proceed. To further the regeneration of the thread, the thread is subjected on reel 9 to a somewhat more dilute sulfuric acid solution, preferably heated, and on reel 10 to a still more dilute sulfuric acid solution. The latter need not be heated, but is preferably heated to a temperature somewhat lower than that of the liquid applied on reel 9. Preferably, the acidity of the liquid applied on reel 10 is so low that it does not cause deleterious effects when the thread is subsequently subjected on reel 11 to an alkaline desulfurizing medium such as dilute sodium sulfide. The thread may be washed on reel 12, after which it may be subjected to other processing operations on subsequent reels.

In Figure 3 is shown the coagulating liquid circulation system in and by which coagulating liquid is pumped through pipe 18 to a header box 19 disposed above the level of the coagulating liquid in trough 4. From header box 19, which is provided with an emergency overflow pipe 21, the coagulating liquid flows into the coagulating trough 4 through pipes 22 extending longitudinally of the trough (Figure 1). The coagulating liquid is drained from said trough 4 through a plurality of upright pipes 23 communicating with a common drain pipe 24 which pipes 23 are disposed at spaced intervals lengthwise of the trough: the inlets to such pipes 23 are disposed at a predetermined distance above the bottom of trough 4 in order to maintain the coagulating bath 3 at the desired level.

The coagulating liquid draining from trough 4 through pipes 23 and 24 passes to make-up tank 25, but only after a portion of the circulating liquid has been bled off through pipe 26 in order to prevent the volume of liquid in the system from increasing unduly.

The reagents needed to rejuvenate the coagulating liquid are added in make-up tank 25. Thus, sulfuric acid from tank 27 may be added through pipe 28, water from tank 29 through pipe 31, and zinc sulfate from tank 32 through pipe 33. The replenished liquid is shown as passing through conduit 34 into a storage tank 35 in which a substantial part of the impurities present in the coagulating bath are caused to agglomerate to form a floating sludge which can be separated without a great deal of difficulty from the coagulating liquid. The accumulated sludge is removed from time to time by any convenient method; e. g., by a skimming operation.

Coagulating liquid which is relatively free from sludge overflows a partition 36 in tank 35 into a compartment 37 in which an oleaginous material such as mineral oil, emulsified by means of a suitable emulsifying agent; e. g., the sodium salt of a sulfonated higher hydrocarbon, is added from a suitable source, such as tank 38. The purposes of adding the emulsified mineral oil to the coagulating liquid are to render innocuous, as, for example, by preventing deposition on the spinneret, the relatively large quantity of insoluble impurities in the coagulating liquid, to prevent the formation of rings of deposited material in the spinneret orifices, and to make possible the removal of such impurities from the coagulating liquid by forming the sludge to which reference has been made. The liquid in compartment 37 is forced by pump 39 through pipe 18 into coagulating trough 4, as indicated above.

In order to obviate the necessity of adding sodium sulfate to the system and for the purpose of reducing the amount of other substances, such as sulfuric acid, which it is necessary to add, some of the coagulating liquid is concentrated by passing it through an evaporator. To that end, a coagulating liquid evaporator 40 of the usual type is employed, its inlet being connected through pipe 41 with supply pipe 18. Its outlet is connected through pipe 42 with discharge pipe 24 from the coagulating trough 4. By means of such a system, a portion of the water in the coagulating liquid can be continuously removed; consequently, the concentration of sodium sulfate and other substances in the portion of the coagulating liquid returned to the system from the evaporator can be increased and the sodium sulfate concentration in the coagulating liquid maintained at the desired point without additions.

The circulation system for the coagulating liquid and the methods of and materials for dispersing the oleaginous substance in the coagulating liquid form no part of the present invention, but are claimed in copending application Serial No. 348,232, filed July 29, 1940, by Hayden B. Kline et al.

The system for circulating the dilute acid regenerating liquid to which the thread is subjected on reel 9 is shown diagrammatically in Figure 4. In said system, liquid is forced upward by means of pump 43 from storage tank 44 through pipe 45 to a header box 46, which is so constructed that the liquid level therein is maintained at a predetermined distance above the discharge orifices of the reagent tubes 15 for the reels 9 disposed on both sides of the apparatus, thus insuring a predetermined uniform discharge of liquid through each of reagent tubes 15. More liquid is supplied through pipe 45 than passes from header box 46 through pipe 47 and conduits 16: the excess passes out through overflow pipe 48. The overflow liquid passed through pipe 48 and the liquid collected in troughs 17 beneath reels 9 and removed by means of pipe 49 are intermingled in make-up tank 50, from which the mixture passes through pipe 51 to storage tank 44.

In the apparatus illustrated in the drawings, only chemicals not present in the coagulating bath need be added to the system. The amount of sulfuric acid, sodium sulfate and zinc sulfate carried over by the thread from the coagulating bath is more than sufficient to maintain the desired concentration of these substances in the precessing liquid employed on reels 9. In time, they tend to build up to a concentration considerably greater than that desired; accordingly, a portion of the processing liquid is continuously bled from the system through pipe 52. For reasons of economy, the liquid so bled off is liquid to which no new additions have been made: to that end, pipe 52 communicates with a first compartment 53 in make-up tank 50 containing only contaminated liquid passing to it from reels 9.

In order to replace the volume of liquid so bled off from the system, fresh water is added to make-up tank 50 through pipe 54 from any suitable source, such as tank 55. Water pipe 54 and overflow pipe 48 from header box 46 discharge into a second compartment 56 in make-up tank 50 in such manner that they do not intermix with the liquid bled off from the system. In this manner, both the acid concentration and the quantity of liquid in the system are maintained at the desired amount. At the same time, the quantity of sodium sulfate in the liquid is maintained at the desired value, which is approximately 5%.

The liquid overflowing from compartment 56 into still a third compartment 57 of make-up tank 50, from which latter compartment the liquid passes through pipe 51 to storage tank 44, creates sufficient turbulence to mix the materials added to the processing liquid. Preferably, the water added through pipe 54 is hot; however, in addition to the heat supplied by such added water, other means may be provided to heat the liquid to maintain it at the desired elevated temperature. Thus steam or hot water pipes 58 are shown as disposed in storage tank 44, but could, if desired, be located in compartment 56 or 57 of make-up tank 50.

The liquid applied to the thread on reel 10, which, as stated, may be a mildly acidic washing liquid, may be recirculated by a similar system in which the ingredients are supplied by the thread proceeding from reel 9 to reel 10. The liquids applied to the thread on the succeeding reels; e. g., reel 11, on which a dilute solution of sodium sulphide is applied to the thread for desulphurizing purposes, may be recirculated by similar systems including means for bleeding off contaminated liquids and adding fresh reagents.

As already indicated, in the manufacture of viscose artificial silk thread, regeneration of the cellulose of the sodium cellulose xanthate of which the viscose is composed does not occur instantaneously. The spinning solution apparently first solidifies without undergoing any important chemical reaction; that is, the material of which the thread is formed consists at first only of solidified sodium cellulose xanthate; thereafter, the cellulose content of the viscose is regenerated. Such sulfur as is not evolved as H_2S either goes into the coagulating bath or is carried away by the thread. Both the solidification and regeneration processes require an appreciable period of time, regeneration requiring more time than

solidification and occurring largely after the thread has left the coagulating bath.

In the illustrated embodiment of the invention, vertical upward withdrawal of the thread from the coagulating bath by means of reel 8 causes a column of coagulating liquid to rise with the thread, thus in effect adding to the time of immersion of the thread. The liquid is largely stripped from the thread by guide 59, but sufficient liquid is carried beyond said guide so that the thread stored on reel 8 is thoroughly wet with coagulating liquid. Solidification of the thread seemingly proceeds to completion on reel 8, but regeneration of the thread, although occurring to a large extent on said reel, is not completed thereon. The hot dilute acid to which the thread is subjected on reel 9 results in substantially complete regeneration of the thread on this reel, as evidenced by a decided hardening of the thread.

Regeneration of the cellulose content of the viscose thus occurs in part in the coagulating bath, in part while the thread is passed from the coagulating bath to reel 8, in part on reel 8, in part between reels 8 and 9, and in part on reel 9, on which the regeneration reaction is substantially if not entirely completed. During such regeneration reaction, sulfur and other insoluble by-products are formed in and upon the thread; in addition, others are carried up from the coagulating bath by the thread. Such substances tend to deposit upon the supporting means with which the thread comes into contact; i. e., guides 59, 60 (Figures 1 and 2), reels 3, 9, etc., upon which they form incrustations which, if not prevented, seriously damage the delicate filaments of which the thread is composed.

The problem is particularly serious with respect to the reels 8 and 9, inasmuch as the considerable length of thread stored thereon affords a particularly good opportunity for the formation of such incrustations.

Another source of possible trouble arises out of the crystallization of the salts used in the liquid in which the thread is formed. Dissolved salts in the coagulating liquid carried by the thread to guide 59 and reel 8 and, to a lesser extent, also to guide 60 and reel 9 tend to crystallize thereon, thus causing damage to the thread. In the illustrated embodiment of the invention, the possibility of such crystallization is greatest on reel 8, on which the thread is stored for a considerable period of time and on which is applied no processing liquid which would tend to wash such salts away.

These difficulties may, however, be virtually completely eliminated by applying to the parts of the apparatus with which the thread comes into contact and on which harmful incrustations tend to form a substance or substances, preferably oleaginous, without deleterious effects on the thread but capable of preventing the deposition of sulfur, salts, and insoluble impurities in places where they can do damage to the thread. Such oleaginous substances are advantageously those which tend to wet the sulfur or other insoluble material and at the same time form a film over the portion of the member contacting the thread on which the deposit otherwise builds up, thus effectively preventing the formation of incrustations. The oleaginous substances may be applied to the desired member by direct application thereof, by their incorporation in a processing liquid applied to the thread, or by being carried to such member by the thread itself.

In the illustrated embodiment of the invention, a liquid oleaginous material, such as a mineral oil, is applied directly to reel 8: to that end, a tube 62 communicating with a common supply conduit 63 extending lengthwise of the apparatus on each side thereof serves to suply the desired amount of oil to the reel. The oil is maintained at the proper pressure in each of said conduits by a header box (not shown). The illustrated apparatus shows the preferred disposition of the tube 62 with respect to reels 8; that is, the discharge orifice of each tube is located at the rear of the reel so that the thread traveling toward the unsupported end of the reel effectively and economically distributes oil in a film over the thread-bearing portion of the reel bars. The tube 62 is so disposed that it drops the oil on the reel just ahead of the first turn of thread, wherefore no turn of thread comes into contact with an unoiled portion of the reel on which incrustations can form.

Experience in the operation of such apparatus has shown that only a few drops of oil per thread per minute are necessary to prevent the deposition on reel 8 of insoluble substances, such as sulfur, or the formation of salt crystals. For this reason, no special means are needed beneath reel 8 to collect any oil dripping therefrom. On the contrary, oil dripping from reel 8 is allowed to fall into the coagulating bath 3 therebelow, where it mixes with the emulsified oleaginous material in the coagulating liquid, with which it is entirely compatible.

Inasmuch as a processing liquid is applied to the thread on reel 9, it is not feasible to apply an oleaginous substance directly to said reel by means similar to that employed in conjunction with reel 8, more especially since the processing liquid might wash the added material away before it could form a film over the surface of the reel. Accordingly, the oleaginous substance is incorporated in the processing liquid applied to the thread. Such oleaginous substance may be a so-called soluble oil such as "Twitchell oil," which is essentially a sulfonated hydrocarbon oil, or an insoluble oil suitably emulsified in the processing liquid applied to the thread. Thus, for example, mineral oil emulsified by a sulfonated oil such as "Twitchell oil" may, if desired, be employed in the processing liquid.

As shown diagrammatically in Figure 4, means are provided for adding such oleaginous substance to the processing liquid being circulated to and from the reels 9. In the system in question, the oleaginous substance, either a soluble oil such as "Twitchell oil" or a previously prepared emulsion of an insoluble oil such as mineral oil, is supplied from tank 64 through pipe 65, which discharges into compartment 56 of make-up tank 50. In order to obtain an efficient dissemination of the oil in the liquid, means are provided for mixing the oil with hot make-up water supplied to the system from pipe 54, the mixing being effected in a container 66 in make-up tank 50 from which container the mixed liquid overflows into compartment 56. This provides a thorough intermixing of the oil and water before the mixture passes into the main body of processing liquid. The liquid overflowing from the compartment 56 into the third compartment 57 effects additional mixing.

The following is an example of the operation of the described apparatus according to the invention:

Viscose is extruded into a coagulating bath containing, by weight, approximately 10.75%

sulfuric acid, approximately 20% sodium sulfate, approximately .37% zinc in the form of zinc sulfate, and from .6% to .9% emulsified mineral oil, the coagulating liquid being maintained at a temperature of approximately 45° C. Threads of 150 denier, 40 filaments, are formed by extrusion of the viscose through the spinnerets, each such thread being drawn upward by a correspondingly disposed reel 8 located above the coagulating bath. No processing medium is applied on reel 8 other than mineral oil of 40 to 80 seconds Saybolt viscosity at 100° F., which is supplied to each thread at the rate of 30 drops per minute. Each reel 8 is so constructed that it advances the thread lengthwise thereof for a distance of approximately 4 inches in a helix 5 inches in diameter having 11 turns to the inch, the angular speed of the reel being 154 R. P. M.

From reel 8, the thread is passed downward to succeeding reel 9, which is likewise 5 inches in diameter but which rotates at a speed of 169 R. P. M. This increase in peripheral speed imparts a stretch of approximately 10% to the thread between reels 8 and 9, the thread being, as indicated, completely solidified but incompletely regenerated when so stretched. The thread is stored on reel 9 in a helix about 4 inches long having 19 turns per inch. The liquid applied to the thread on the reel contains approximately 2.5% sulfuric acid, approximately 5% of sodium sulfate, approximately .07% of zinc as zinc sulfate, and about .5% of a Twitchell oil composed of about 10% to 15% of the sodium salt of sulfonated mineral oil, together with plain mineral oil. The liquid is applied to each reel at the rate of 340 cc. per minute at a temperature of about 60° C.

The processing liquid for reel 9 is recirculated as already described by the system shown in Figure 4. The only liquids added to the system are fresh hot water and Twitchell oil, the latter being added to the system at the rate of from 3 to 5 grams per thread per hour to replace that removed from the system by the threads and in the discarded processing liquid. The remainder of the ingredients in the processing liquid are supplied by the liquid carried over by the thread from the coagulating bath to reel 9.

The thread then passes to reel 10, which is a 5-inch diameter reel rotating at the same speed as reel 9. The turns of thread on this reel are disposed in the helix the pitch of which is 23 turns per inch. Processing liquid containing from .615% to .025% of sulfuric acid, from .03% to .05% of sodium sulfate, traces of zinc in the form of zinc sulfate, and small quantities of Twitchell oil is applied to the thread on reels 10. The ingredients other than water will ordinarily be present in substantially the same ratio as in the liquid applied to the thread on reel 9, although in much smaller amounts, since such ingredients may be carried over by the thread. On reels 10, the liquid is applied to the thread at the rate of 240 cc. per minute at a temperature of 30° C.

On reel 11, a desulfurizing medium consisting of a dilute alkaline solution of sodium sulfide is applied to the thread, the reel being of the same dimensions, rotating at the same speed, and advancing the thread in a helix of substantially the same pitch as in the case of reel 10. Preferably, the solution contains from 0.3% to 0.5% sodium sulfide and, for the purpose of stabilizing the solution, about .05% sodium hydroxide. The solution is applied to the thread on the reel at a temperature of approximately 50° C. at a rate of 240 cc. per minute.

In the practice of the invention under the conditions above described, the oleaginous substance, whether carried by the thread, applied to it as such, or incorporated in the processing liquid, effectively prevents the deposition upon the apparatus of sulfur formed as by a by-product of the regeneration reaction, of other insoluble compounds resulting from the regeneration reaction or present as impurities in the coagulating bath; e. g., lead trithiocarbonate, and of salts crystallizing out of the coagulating liquid carried by the thread. Damage to the thread, which, as is well known, is particularly delicate when but incompletely regenerated, of a sort that would be occasioned by the passage of the thread over incrustations is entirely avoided; consequently, the invention makes it possible to operate continuous processing apparatus for long periods of time without the necessity of shutting down the apparatus to remove incrustations.

The application of an oleaginous substance to the thread-contacting parts also serves to lubricate the thread, thereby reducing friction of the thread not only on reels 8 and 9 and their associated guides but also on succeeding reels and guides. In particular, when, as in the illustrated embodiment of the invention, the thread is spun into a lubricant-containing bath and then subjected to the action of an oleaginous substance on one or ore of the succeeding reels, extremely advantageous results are obtained. Throughout its length the thread is lubricated by oleaginous materials carried by the thread, including the portion thereof where it is but incompletely regenerated and hence most susceptible to damage.

Under the above-described conditions, the oleaginous materials form a film over the parts on which deposits tend to build up and in this manner inhibit the formation of incrustations; however, they also have the property of wetting the sulfur and other insoluble compounds, presumably by forming a film around the particles, thereby preventing them from sticking to each other or to the parts of the apparatus with which the thread comes into contact. Using in the processing liquid applied to the thread on reel 9 a substance like Twitchell oil which has dispersing properties, sulfur and other insoluble substances will be found in finely dispersed condition in the processing liquid collected under reels 9. Although these particles do not tend to settle out of the dispersion, the concentration of such substances in the processing liquid is effectively prevented from increasing beyond a very small amount by the fact that a considerable portion of such substances is continuously discarded in the liquid bled off from the circulation system.

It further appears that the application of a suitable substance, such as the Twitchell oil, to the thread at the point where regeneration of the cellulose is substantially but not entirely completed; i. e., on reel 9, not only prevents deposition of sulfur and other insoluble impurities on the reels but also facilitates their removal from the thread. It appears likely that the presence of a substance such as Twitchell oil in the processing liquid serves to loosen the sulfur from the thread, while the stream of processing liquid applied to the thread washes the sulfur away. This may be due to the detergent action of the added substance, to its surface-tension reducing properties, or to the fact that it has a greater

affinity for the sulfur than has the thread or reel.

The incorporation in processing liquid applied to the thread on a reel of an oleaginous substance having surface tension reducing properties, as has the Twitchell oil incorporated in the processing liquid applied to the thread on reel 9, is also extremely advantageous in that it serves to reduce the surface tension of the processing liquid. Thus the formation of the previously-mentioned film of processing liquid between adjacent turns of thread on reel 9 is greatly facilitated, wherefore the thread turns may be considerably more widely spaced than otherwise would be the case. A wide spacing of thread turns on reel 9 is desirable since it eliminates the possibility of damage to the delicate thread filaments of the kind which would tend to occur if adjacent turns of thread came into contact with each other. Moreover, reduction of the surface tension of the processing liquid operates to reduce the surface tension of the film which forms between the reel and the last turn of thread leaving the reel.

The above-described embodiment of the invention is of course susceptible of various modifications without departure from the spirit of the invention. For example, while an oil is shown as applied to the thread on reel 8, under certain conditions the apparatus may be successfully operated without the direct application of any oleaginous substance to said reel. It is, however, desirable in any case to apply to the thread on one or more of the reels succeeding reel 8 an oleaginous deposit-inhibiting substance.

While in the illustrated embodiment of the invention no means are shown for collecting the oil applied to reel 8, it is obvious that a suitable collecting trough could be provided beneath the reels 8 for the collection and recirculation of the oil. In the illustrated apparatus, however, the oil applied to said reel merely drips into the coagulating trough, where it does no harm in view of the fact that the coagulating liquid also contains an oil. Under such conditions, it is of course desirable that the oleaginous material applied to reel 8 be the same or at least not incompatible with the oleaginous material contained in the coagulating liquid.

Where an oleaginous substance is applied to the thread on reel 8, it is apparent that various types of oleaginous substances other than the mineral oil disclosed may be employed. For example, various other kinds of oils, whether insoluble or soluble, may be employed. Among the insoluble oils in addition to mineral oil may be mentioned the various vegetable oils, such as pine oil, coconut oil, etc., and insoluble animal oils. It is preferable that such oils be not of too great a viscosity, or else they will not form a film over the reel, nor not of too low a viscosity, or else the film formed will not have sufficient tenacity to cling to the reel.

Among the soluble oils which may be added are the sulfated, sulfonated or sodium salts of sulfonated mineral, vegetable, or animal oils such as oils of the above types. Indeed, substances may be employed which are not oils but which have oleaginous properties, such as higher mono- or poly-hydroxy alcohols, of which lauryl alcohol is an example, as well as esters of higher fatty acids, such as the sorbitol or mannitol esters of such acids. The amount of such oleaginous substance which may be added of course varies greatly, but in general, for the purpose of reducing the cost, should be as small as possible consistent with efficient results.

It is also apparent that where the oleaginous material is incorporated in a processing liquid applied to a thread-contacting part, such as reel 9, any one of various kinds of oleaginous materials other than the Twitchell oil referred to above may be employed. As examples of soluble oleaginous substances may be mentioned the sulfated, sulfonated, or sodium salts of sulfated or sulfonated mineral, animal or vegetable oils, of which sulfonated castor oil, sulfonated coconut oil, etc., are examples of oils which work well. Other types of soluble oleaginous substances may, of course, be employed.

As has been indicated, insoluble oleaginous substances may also be applied in a processing liquid applied to the thread, provided they are finely dispersed or emulsified. Indeed, any one of the insoluble oleaginous substances mentioned above may be suitably dispersed or emulsified in the processing liquid applied to the thread. As examples, pine oil emulsified by sulfonated castor oil may be employed to advantage. Mineral oil, preferably of 75 to 100 seconds Saybolt viscosity at 100° F., which has been emulsified by any one of several substances such as triethanolamine oleate, sulfonated castor oil, or the sodium salt of any one of many sulfonated higher hydrocarbons, may also be employed.

Of course, certain oleaginous substances may be readily dispersed in the processing liquid without the use of a dispersing or emulsifying agent. Examples of such substances are solubilized polyhydric alcohol esters of higher fatty acids, such as solubilized sorbitol laurate, and solubilized mannitol esters of higher fatty acids, such as solubilized mannitol monostearate. Such esters are solubilized by treatment with ethylene oxide and are obtainable from the Atlas Powder Company. While such substances are substantially soluble in water, they are not wholly soluble in the processing liquid applied on reel 9. They will, however, form dispersions of substantially insoluble particles which have the desired affinity for insoluble substances and will inhibit the formation of incrustations of these as well as other substances.

Where an insoluble oleaginous substance is dispersed by a suitable agent, various proportions of the dispersing agent to the oleaginous substance may be employed, from as low as .2% to as high as 16% or more. In general, however, satisfactory results are obtained when 2% or less of a dispersing agent is employed, based on the weight of the oleaginous substance. While various amounts of the oleaginous substance whether in the insoluble or soluble form may be employed in the processing liquid applied to the thread, in general satisfactory results are obtained when the oleaginous substance constitutes 1% or less of the processing liquid applied to the thread, although other percentages either less or more and even as high as 3% or more may be employed.

Other modifications may also be made in the illustrated embodiment. For example, while an oleaginous substance has been described as being applied only in the processing liquid applied to a thread on one reel, it is apparent that an oleaginous substance may be thus applied on several reels; furthermore, while, as has been stated, it is desirable to incorporate the oleaginous substance in the processing liquid applied to the thread in a stage on which the thread is sub-

jected to liquid processing treatment, it is apparent that the oleaginous substances, whether soluble or insoluble, may be applied directly to the reel from a source other than the reagent distributor through which the processing liquid is being applied. In such case the oleaginous substances will of course mix with the collected processing liquid.

Furthermore, while in the illustrated embodiment of the invention the oleaginous substances added to the circulation system are shown as mixed in the make-up tank with the other additions, it is apparent that such added oleaginous substance may be introduced at other points. For example, it may be added directly to the reel as described immediately above, or it may be added to or in the immediate vicinity of the reagent distributing tube for the reel. In such case, the fresh oleaginous substance comes into contact with the reel immediately after its addition to the system, which may be an advantage.

While the invention has been described in connection with the manufacture of thread by a continuous process, it is apparent that it is equally applicable to the manufacture of thread by other processes.

Moreover, the invention is not restricted to the manufacture of thread, but may, at least in certain of its aspects, be applied to the manufacture of other products; e. g., strips, ribbons, tubes, etc., by processes which involve the extrusion of coagulable masses. Besides thread per se, the term "thread" as employed in the appended claims is intended to embrace such articles. The term "thread-advancing thread store device" is of course intended to embrace other types of thread-advancing devices than the reel illustrated. The term "insoluble substances" is intended to include such insoluble substances as sulfur, colloidal or otherwise, produced as a result of the coagulating reaction of the viscose thread, as well as other insoluble substances produced during such reaction or in the coagulating bath and carried therefrom by the thread.

It is intended that the patent shall cover, by suitable expression in the appended claims, whatever features of patentable novelty reside in the invention.

What is claimed is:

1. In the process of manufacturing thread according to which thread is continuously but temporarily stored on a rotating thread-advancing device in the form of a helix characterized by a large number of turns under conditions such that substances carried by the thread tend to deposit on said thread-advancing device in the form of incrustations, the step of inhibiting the formation of such incrustations which comprises treating the thread on said thread-advancing device with an oleaginous substance.

2. The process as in claim 1 in which the thread on said thread-advancing device is treated with a water-soluble oleaginous substance.

3. The process as in claim 1 in which the thread on said thread-advancing device is treated with a water-soluble oil.

4. The process as in claim 1 in which the thread on said thread-advancing device is treated with a processing liquid having dissolved therein an oleaginous substance.

5. In the process of manufacturing viscose artificial silk thread according to which incompletely regenerated thread is continuously but temporarily stored on a rotating thread-advancing device in the form of a helix characterized by a large number of turns under conditions such that substances carried by the thread tend to deposit on said thread-advancing device in the form of incrustations, the step of inhibiting the formation of such incrustations which comprises treating the thread on said thread-advancing device with an aqueous liquid capable of furthering the regeneration reaction, said liquid having dissolved therein a water-soluble oleaginous substance.

6. The process as in claim 5 in which the aqueous liquid has dissolved therein a sulfonated hydrocarbon.

7. The process as in claim 1 in which the thread on said thread-advancing device is treated with a water-insoluble oleaginous substance.

8. The process as in claim 1 in which the thread on said thread-advancing device is treated with a water-insoluble oil.

9. The process as in claim 1 in which the thread on said thread-advancing device is treated with a processing liquid having dispersed therein a water-insoluble oleaginous substance.

10. In the process of manufacturing viscose artificial silk thread according to which incompletely regenerated thread is continuously but temporarily stored on a rotating thread-advancing device in the form of a helix characterized by a large number of turns under conditions such that substances carried by the thread tend to deposit on said thread-advancing device in the form of incrustations, the step of inhibiting the formation of such incrustations which comprises treating the thread on said thread-advancing device with an aqueous liquid capable of furthering the regeneration reaction, said liquid having dispersed therein a water-insoluble oleaginous substance.

11. The process as in claim 10 in which the aqueous liquid has a water-insoluble oil dispersed therein.

ROBERT D. MacLAURIN.
EMERSON B. HELM.

CERTIFICATE OF CORRECTION.

Patent No. 2,326,150. August 10, 1943.

ROBERT D. MacLAURIN, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, first column, line 14, for "slik" read --silk--; and second column, line 36, for "2,225.642" read --2,225,642--; page 2, second column, line 16, for "shell" read --shall--; page 3, second column, line 13, for "precessing" read --processing--; page 4, first column, line 75, for "lilustrated" read --illustrated--; and second column, line 5, for "suply" read --supply--; page 5, second column, line 32, for "ore" read --more--; page 6, first column, line 47, after the word "same" insert --as--; line 66, before "sulfonated" insert --sulfated or--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 19th day of October, A. D. 1943.

Henry Van Arsdale,
Acting Commissioner of Patents.

(Seal)