Nov. 21, 1933. G. JUER 1,935,637

MANUFACTURING ARTIFICIAL SILK

Filed Jan. 21, 1930 3 Sheets-Sheet 1

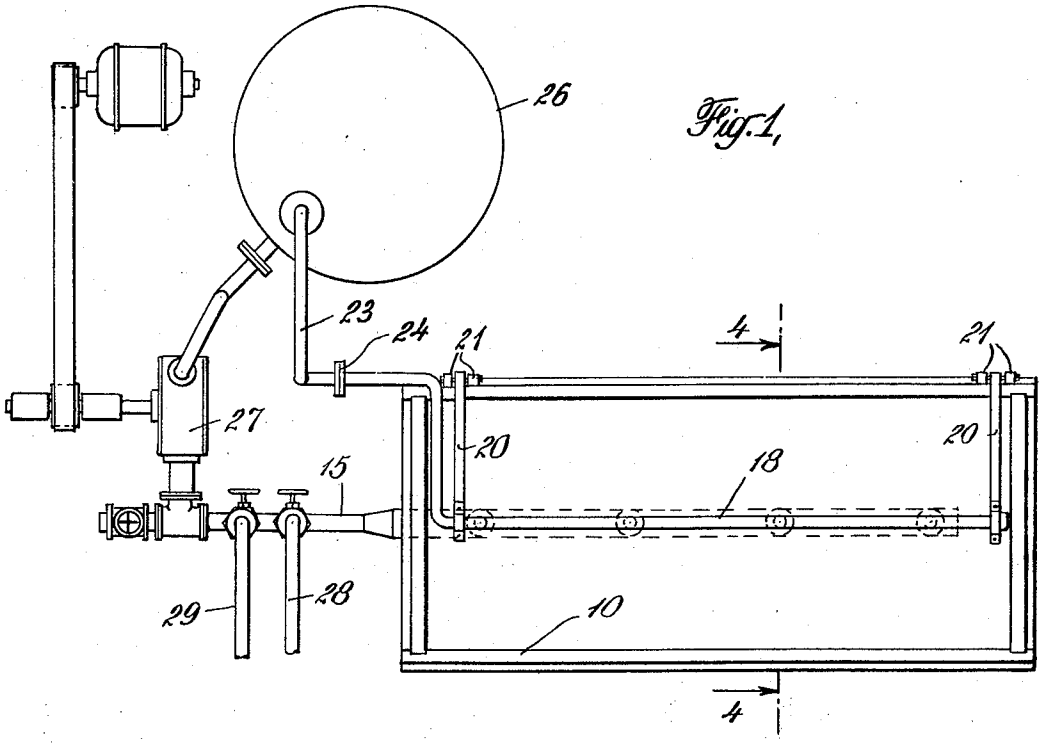


Fig. 1.

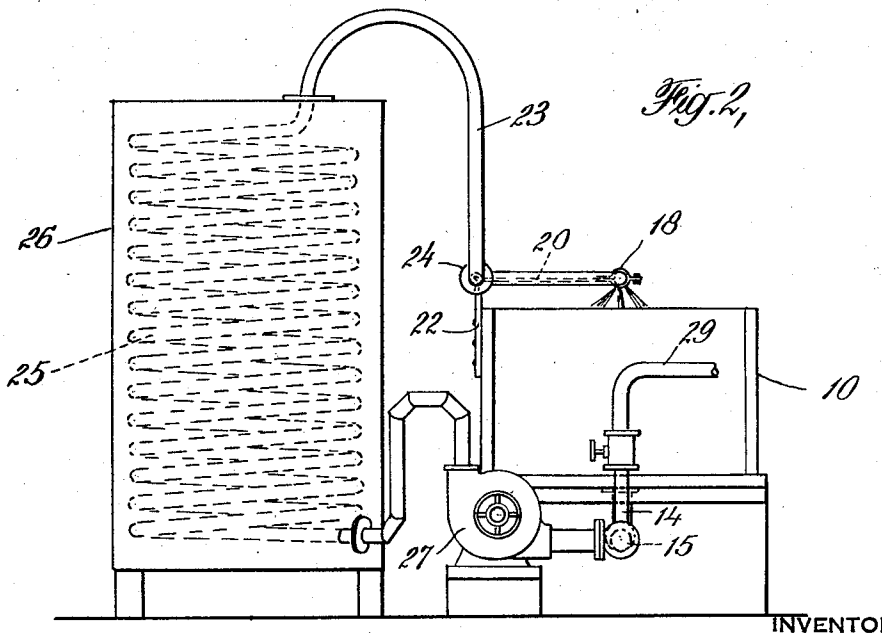


Fig. 2.

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Fig. 3.

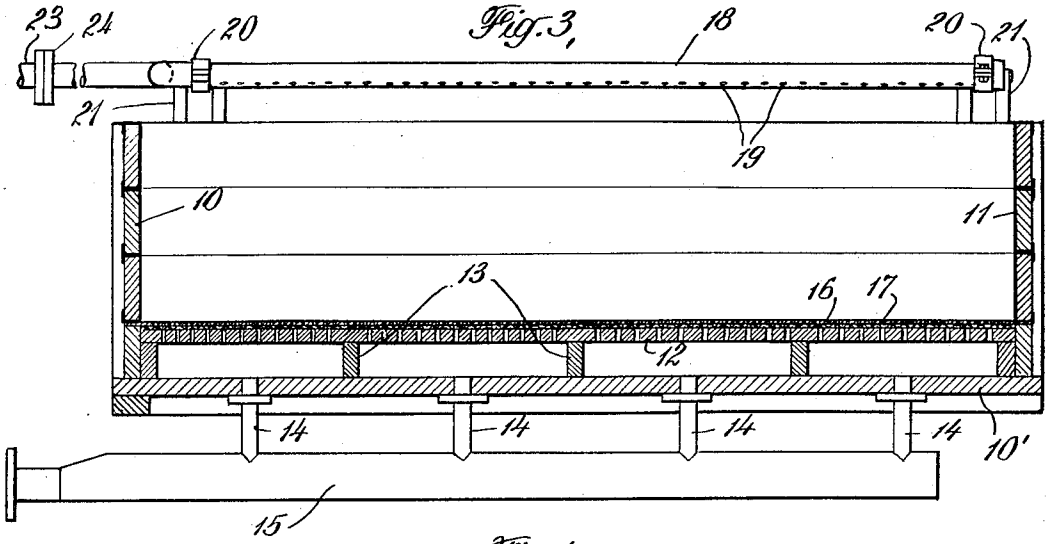
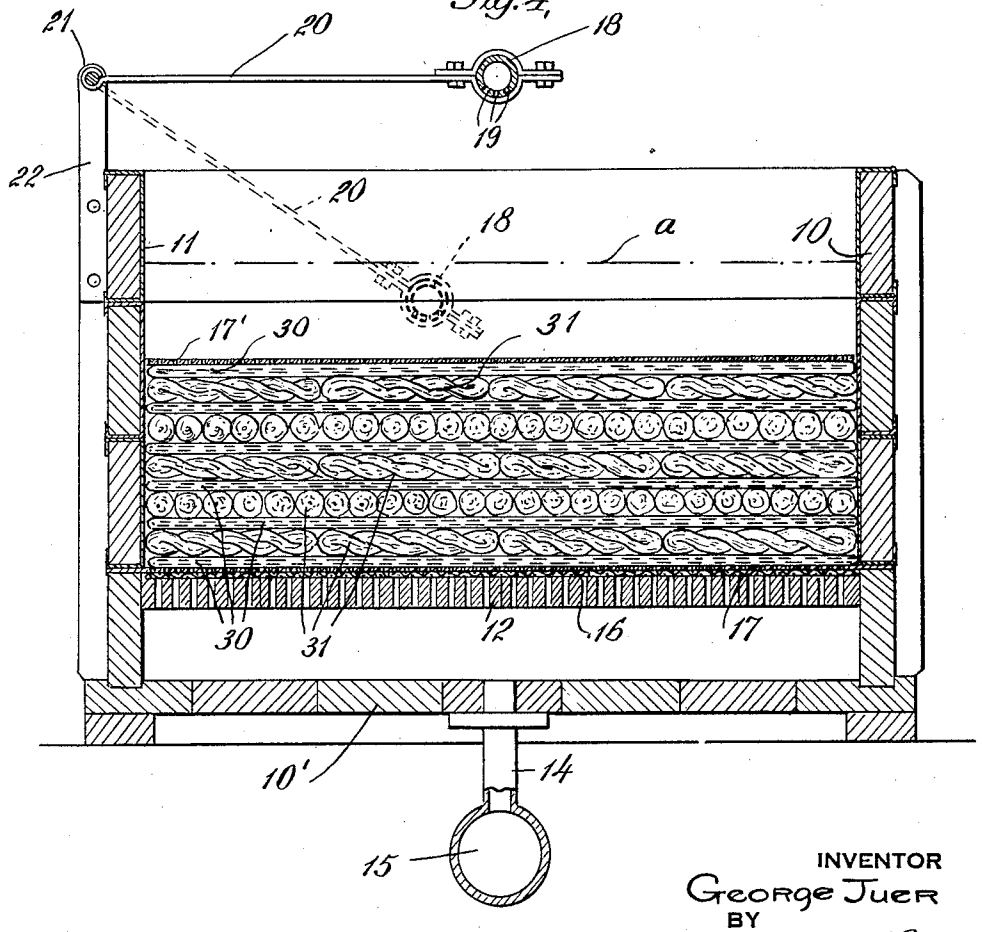


Fig. 4.



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Fig. 5.

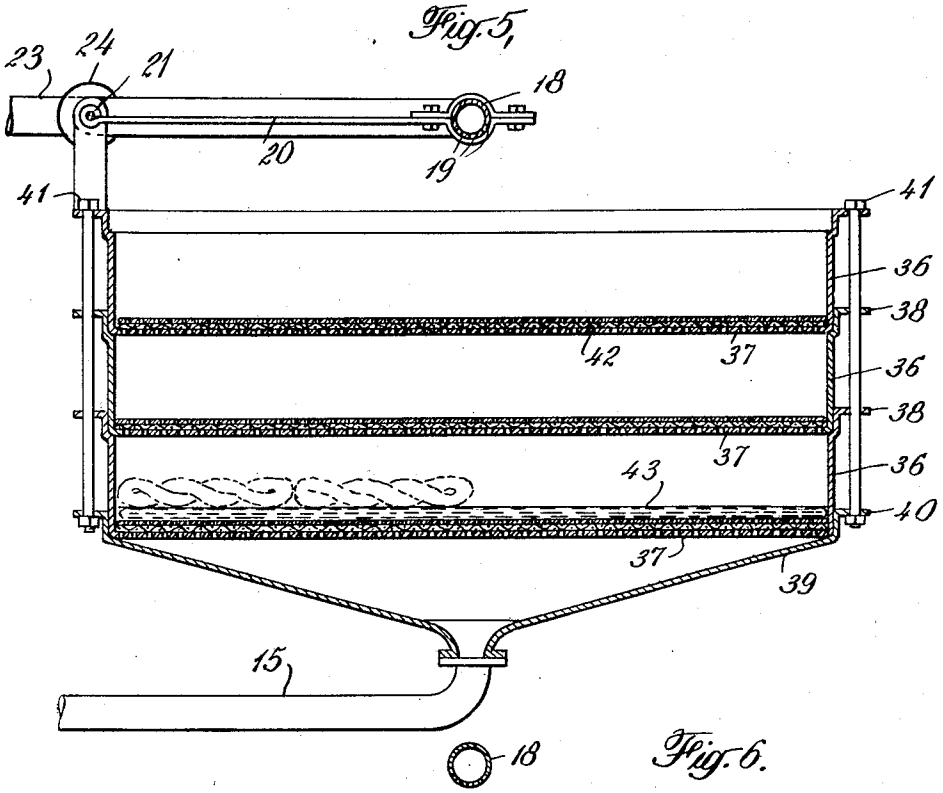
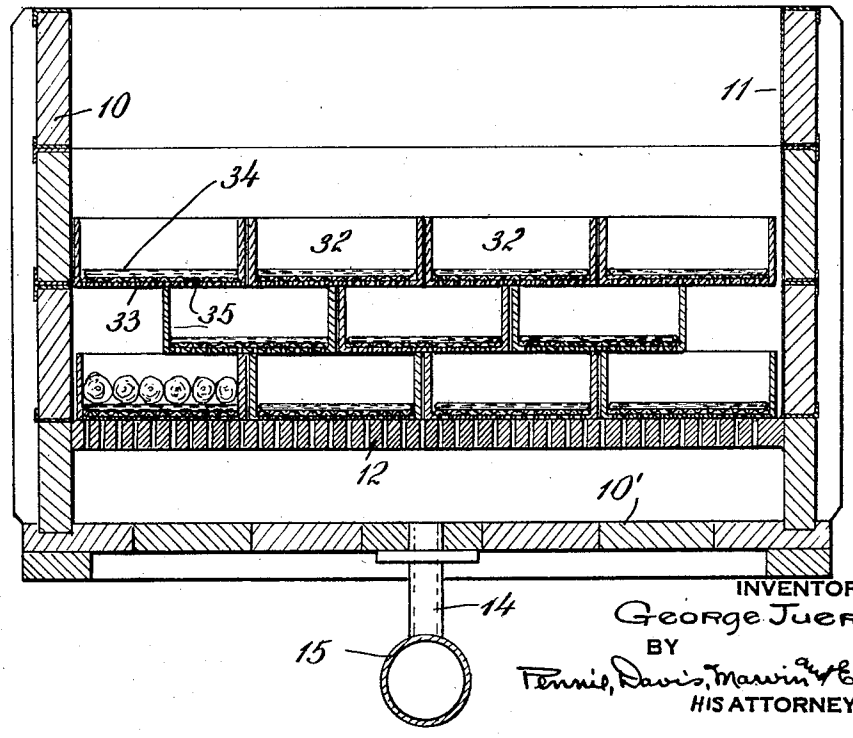


Fig. 6.



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UNITED STATES PATENT OFFICE

1,935,637

MANUFACTURING ARTIFICIAL SILK

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Application January 21, 1930. Serial No. 422,289

10 Claims. (Cl. 18—8)

This invention relates to the manufacture of artificial silk and has for its object the provision of certain improvements in the denitration step of the cellulose nitrate process for manufacturing artificial silk. More particularly, the invention contemplates an improved method of and apparatus for mechanically conducting the denitration step.

Denitration of cellulose nitrate (collodion silk) is effected by treatment with an appropriate denitrating liquor of a reducing character, such as an aqueous solution of sodium sulfhydrate (NaSH), in the course of which treatment the nitrate radical is removed from the cellulose nitrate and cellulose is regenerated. It has heretofore been customary, in commercial practice, to immerse the cellulose nitrate (usually in the form of skeins or hanks) in the denitrating liquor and to manually move the skeins through the liquor in an attempt to obtain uniform and complete contact thereof with the liquor as well as to wash off any precipitated sulfur. In such a manually conducted operation, the human element inevitably prevents the obtainment of constant uniform results, and consequently the denitrated product is rarely if ever of uniform quanity.

The present invention is particularly concerned with the provision of a mechanical method of denitration, as distinguished from the heretofore customary manual practices. In accordance with the method of the invention, the cellulose nitrate is supported on an appropriate porous medium while completely and continuously immersed in the denitrating liquor, and the denitrating liquor is passed through the cellulose nitrate and its porous support with appropriate control of the temperature of the liquor. The cellulose nitrate should preferably be so supported that every particle is completely immersed in and in contact with the denitrating liquor, and, according to one aspect of the invention, this is accomplished by supporting the cellulose nitrate directly on an absorbent porous medium, such as a layer of canvas, filter-press cloth, or similar loosely woven fabric. It is my preferred practice to circulate the denitrating liquor alternately in opposite directions through the cellulose nitrate and its porous supporting medium. Denitration raises the temperature of the denitrating liquor, and I have found that temperature control of the liquor is of particular importance. Furthermore I have found, using the heretofore customary sodium sulfhydrate liquor, that substantially more concentrated liquors may be used if the denitrating liquor is maintained at a temperature not exceeding about 35° C.

The novel features of the invention will be best understood from the following description taken in conjunction with the accompanying drawings, in which

Fig. 1 is a plan view of an apparatus adapted for the practice of the invention,

Fig. 2 is an end elevation of the apparatus,

Fig. 3 is a longitudinal sectional elevation of the denitrating tank.

Fig. 4 is a transverse sectional elevation of the tank on the section line 4—4 of Fig. 1, and

Figs. 5 and 6 are transverse sectional elevations of modified tank arrangements.

The apparatus illustrated in Figs. 1, 2, 3 and 4 of the drawings comprises a rectangular tank 10, preferably built up of planks having their inner or liquor-contacting surfaces covered with sheet zinc 11. The tank has a perforated false bottom 12, built of wood or the like, appropriately spaced above the effective bottom 10'. The space between the perforated and effective bottoms (12 and 10') is divided into a plurality of compartments by transverse partitions 13. A pipe 14 connects each compartment beneath the false bottom to a manifold or main pipe 15.

A wire screen 16 or equivalent foraminous material is positioned on top of the false bottom 12, and a perforated zinc plate 17 is positioned on top of the screen. The perforations in the plate 17 are of considerably smaller diameter than the perforations in the false bottom 12, and the screen 16 serves to provide suitable channels intermediate the two perforated elements for the uniform flow of liquor through the perforations of both elements.

A liquor distribution pipe 18 is movably positioned above the tank 10. The distribution pipe is longitudinally and centrally disposed above the tank and is provided along its underside with three longitudinal rows of perforations 19 adapted, in one operating position, to deliver liquor to the tank in relatively fine streams distributed over the surface of the liquor in the tank. The pipe 18 is connected by a pipe 23 including a flexible joint 24 to the top of a cooling coil 25 operatively positioned in a cooler 26.

The pipe 18 is supported at the end of two arms 20 hinged at 21 to uprights 22 appropriately secured to one side wall of the tank. During loading and unloading of the tank, the pipe 18 is swung upwardly on its hinges 21 to one side of the tank. When operating to supply the denitrating liquor to the tank, the pipe 18 is swung

on its hinges 21 to a centrally disposed position just above the tank, as illustrated in full lines in Fig. 4. When operating to withdraw liquor from the tank, the pipe 18 is swung downwardly until it is beneath the liquor level (*a*) in the tank, as illustrated in dotted lines in Fig. 4.

The action of the cooler 26 and hence the control of the temperature of the denitrating liquor are preferably effected automatically in ways well understood in the art. The lower end of the coil 25 is connected to one side of a reversible pump 27, and the other side of the pump is connected to the pipe 15. Water and denitrating liquor may be introduced into the apparatus through the valved supply pipes 28 and 29 respectively.

In practicing the invention in the apparatus just described, the perforated plate 17 is covered with a layer of coarsely woven fabric 30 such, for example, as filter press cloth. A layer 31 of twisted skeins of cellulose nitrate is then laid on the cloth 30, preferably with all the skeins extending in one direction. The layer of cellulose nitrate 31 is then covered with another layer of woven fabric 30 and this in turn is covered with another layer of twisted skeins of cellulose nitrate extending at right angles to the adjacent lower layer. In this manner several superposed layers 31 of cellulose nitrate separated from one another by layers of cloth 30 are loaded into the tank. The top layer of cellulose nitrate is covered with a layer of cloth 30 on which rests a perforated plate 17'.

After the loading of the tank and during the subsequent denitrating operation, the cellulose nitrate is completely and continuously immersed in the denitrating liquor, the liquor level (*a*) in the tank being preferably maintained several inches above the top of the topmost layer of cellulose nitrate. The denitrating liquor is circulated through the tank alternately in opposite directions, that is downwardly and then upwardly through the layers of cellulose nitrate. During the circulation downwardly, the denitrating liquor is drawn through the layers of cellulose nitrate principally by the action of gravity due to the head of liquor constantly maintained in the tank and the withdrawal of liquor through the pipe 15 by the pump 27. The pump 27 forces the liquor withdrawn from the tank through the cooling coil 25, where its temperature is appropriately regulated by the cooler 26, and thence into the distribution pipe 18 and back into the tank. When downward circulation has been maintained for the desired time interval, the action of the pump 27 is reversed and the pipe 18 is swung down beneath the liquor level in the tank. Liquor is then withdrawn from the top of the tank through the pipe 18 into the cooling coil 25 and forced through the pipes 15 and 14 back into the bottom of the tank. The alternating time intervals of downward and upward circulation may be varied to suit operating conditions, from 10 to 30 minute intervals being generally satisfactory. Instead of a reversible pump 27, suitably valved by-passes may be arranged about a non-reversible pump for alternating the direction of liquor circulation through the tank.

The circulation of the denitrating liquor serves as a convenient means of controlling its temperature, since the artificial cooling means may advantageously operate upon the liquor exterior of the denitrating tank. Circulation furthermore inhibits irregular local reactions by maintaining the composition of the liquor substantially uniform throughout the tank. A further important function of the circulating liquor is to wash off any precipitated sulfur adhering to the cellulose nitrate.

I have found that with appropriate temperature control the method of the invention permits the use of substantially stronger or more concentrated denitrating liquor than heretofore used in the industry. The usual denitrating liquor contains 3½% by weight of sodium sulfhydrate. Using a liquor of double this concentration, that is 7% by weight of sodium sulfhydrate, with regulation of the temperature of the denitrating liquor in the tank so as not to exceed about 35° C. (preferably from 20 to 25° C.), I have obtained substantially improved denitration. In particular, the higher concentration of denitrating liquor and the lower operating temperatures result in advantageous physical and chemical properties of the denitrated product, such as an increase of 10 to 15% in tensile strength.

If desirable, a penetrating agent may be added to the circulating denitrating liquor. Any penetrating oil, alcohol, or other substance which tends to soften the artificial silk thereby permitting better penetration of the denitrating liquor may be used.

Instead of loading the cellulose nitrate directly into the denitrating tank, it may be placed in portable trays 32, and several layers of these trays piled in the tank 10, as shown in Fig. 6 of the drawings. Each of the trays 32 has a perforated bottom 33 covered with coarsely woven fabric 34, preferably with an intermediate layer of screen 35. While the bottom layer of trays are shown in Fig. 6 as resting directly on the false bottom 12, intermediate layers of screen and fabric (16 and 17) as shown in Fig. 4 may be found advantageous. On the other hand, the perforated false bottom 12 may be replaced by slats or other appropriate supporting means for the trays 32, in which case the perforated bottoms of the lowermost layer of trays serves as the equivalent of the false bottom 12.

Where the cellulose nitrate is packed in portable trays, the denitrating tank may be formed of a column of superposed trays suitably secured together to form a liquid-tight vessel or tank. Thus, in Fig. 5 of the drawings, the denitrating tank is built up of a column of superposed portable trays 36 having perforated bottoms 37 and circumferential rim flanges 38. The lowermost tray 36 fits into the top of a funnel or hopper 39 having a circumferential rim flange 40, and the superposed column of trays are secured together and to the funnel by bolts 41 extending through aligned holes in the flanges 38 and 40. The perforated bottom of each tray is covered first with a layer of screen 42 and then with a layer of fabric 43 and the cellulose nitrate laid flat on the fabric.

The topmost layer of cellulose nitrate is covered with a layer of fabric or equivalent absorbent porous medium on which rests a perforated metal plate of sufficient weight to hold the cellulose nitrate down during upward circulation of the denitrating liquor. It may even be found desirable to cover the cellulose nitrate in each tray with a layer of fabric to prevent contact with the bottoms of the trays above during upward circulation.

The layer of loosely woven fabric, or other absorbent porous medium, underneath (or above) each layer of cellulose nitrate serves as a distributor for the liquor as it is circulated there-

through. The fabric reduces the effective surfaces of contact between the cellulose nitrate and its support (or other contacting surface) to practically a negligible quantity, so that for all practical purposes every particle of the cellulose nitrate is completely and effectively immersed in the denitrating liquor. This results in uniformity of denitration and substantial absence of unacted upon material which might otherwise be protected from effective action by the liquor.

The lining of the wooden tank 10 with sheet zinc, galvanized iron or the like, is desirable in order to prevent injury to the silk through contact with a rough wooden surface. The circulatory liquor system may be made of aluminum, or any other material resistant to the action of the concentrated denitrating liquor. The portable trays, when used, may be made of zinc, aluminum, galvanized iron or other suitable material.

Artificial silk denitrated in accordance with the invention possesses materially improved general qualities as compared with artificial silk denitrated in accordance with the heretofore customary manual practices. In the first place, artificial silk denitrated in accordance with the invention is remarkably uniform in quality and a relatively high percent of first class silk is obtainable. Moreover, it possesses vastly improved winding qualities and its dyeing qualities are more even than manually denitrated artificial silk. A particularly outstanding improvement in artificial silk denitrated by the invention is its superior tensile strength, particularly evident with the more concentrated denitrating liquor and lower operating temperatures. The higher concentration of the waste denitrating liquor further permits a more economical treatment of this liquor for regeneration or for the recovery of valuable constituents therein. The invention effects a marked saving in labor and permits the use of considerably larger denitrating units than are possible in manual practices. The loading and unloading of the silk is readily accomplished, and during denitration the silk is neither touched nor moved. While denitration is preferably carried out with the silk in the form of skeins, it will be understood that the silk may be in any other physical form appropriate for the denitration method of the invention.

I claim:—

1. An apparatus of the character described comprising a tank, means for circulating a liquor through said tank, a porous medium for supporting material while immersed in the liquor in said tank, and means for artificially cooling the liquor in the course of its circulation.

2. An apparatus of the character described comprising a tank, means for circulating a liquor through said tank, an absorbent porous medium for supporting material while immersed in the liquor in said tank, and means for artificially cooling the liquor in the course of its circulation.

3. An apparatus of the character described comprising a tank, means for circulating a liquor through said tank, a plurality of superposed absorbent porous media for supporting material while immersed in the liquor in said tank, and means for artificially cooling the liquor in the course of its circulation.

4. An apparatus of the character described comprising a tank having a perforated bottom, a plate having smaller perforations than and appropriately spaced above said bottom, an absorbent porous medium covering said plate, means for circulating liquor alternately in opposite directions through the porous bottom of said tank, and means for cooling the liquor in the course of its circulation.

5. An apparatus of the character described comprising a tank having a perforated bottom, a liquor distribution pipe normally positioned above said tank and adapted to supply liquor to the tank in relatively fine streams, means whereby said distribution pipe may be moved to one side of the tank, and means for drawing liquor downwardly through the perforated bottom of the tank and delivering the withdrawn liquor to said distribution pipe.

6. An apparatus of the character described comprising a tank having a perforated bottom, a plate having smaller perforations than and appropriately spaced above said bottom, a liquor distribution pipe normally positioned above said tank and adapted to supply liquor to the tank, and means for drawing liquor downwardly through the perforated bottom of the tank and delivering the withdrawn liquor to said distribution pipe.

7. An apparatus of the character described comprising a tank having an absorbent porous supporting medium, a liquor distribution pipe normally positioned above said tank and adapted to supply liquor to the tank, and means for drawing liquor downwardly through the porous supporting medium of the tank and delivering the withdrawn liquor to said distribution pipe.

8. An apparatus of the character described comprising a tank having a perforated supporting member, an absorbent porous medium covering said member, a liquor distribution pipe normally positioned above said tank and adapted to supply liquor to the tank, means for drawing liquor downwardly through the supporting member and delivering the withdrawn liquor to said distribution pipe, and means for cooling the withdrawn liquor prior to its delivery to the distribution pipe.

9. An apparatus of the character described comprising a tank having a perforated supporting member, an absorbent porous medium covering said member, means for circulating liquor alternately in opposite directions through said supporting member and said porous medium, and means for cooling the liquor in the course of its circulation.

10. An apparatus of the character described comprising a tank having a perforated supporting member, an absorbent porous medium covering said member, a movable liquor distribution pipe mounted above said tank, means for circulating liquor alternately in opposite directions through said distribution pipe and the perforated supporting member of the tank, and means disposed between said pipe and said member exteriorly of the tank for cooling the liquor in the course of its circulation.

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