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H. DREYFUS

2,210,116

MANUFACTURE OF ARTIFICIAL MATERIALS

Filed June 18, 1937

FIG. 1

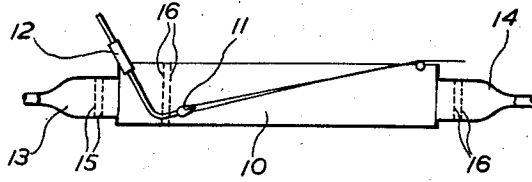


FIG. 2

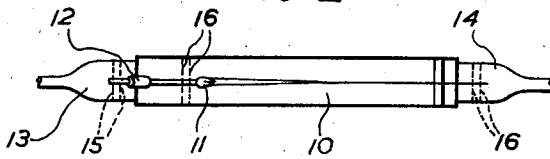


FIG. 3

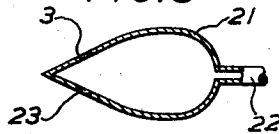


FIG. 4

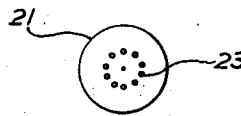


FIG. 5

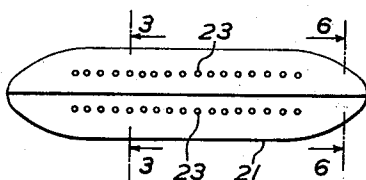


FIG. 6

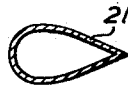
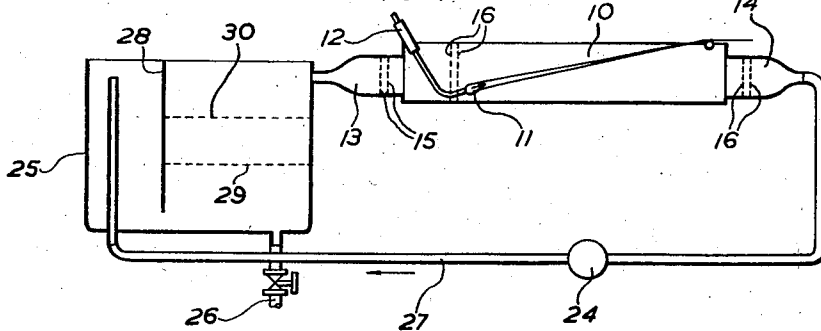


FIG. 7



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MANUFACTURE OF ARTIFICIAL MATERIALS

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6 Claims. (Cl. 18—8)

This invention relates to improvements in the manufacture of artificial materials, and particularly to improvements in the manufacture of filaments, yarns and similar materials, especially such materials having a basis of cellulose acetate or other organic derivative of cellulose, by wet spinning processes.

When artificial filaments and other products are manufactured by extruding solutions into liquid coagulating media the drag on the materials due to their motion through the liquid may be considerable, and it of course increases very rapidly as their velocity increases. It has been discovered that this drag may have a deleterious effect upon the properties of the artificial products, particularly upon their tenacity and extensibility, and that improved products may be obtained by carrying out the spinning operation so that the drag is reduced or eliminated.

The relative velocity of the materials with respect to the coagulating medium may be reduced by circulating the coagulating medium so that it travels in the same direction as the materials, but it has been found that unless precautions are taken turbulent motion may be set up in the coagulating medium and this turbulent motion may cause difficulties in spinning and may also be detrimental to the properties of the products obtained.

According to the present invention artificial filaments and other materials are produced, particularly at high spinning speeds, for example spinning speeds of 150 or 200 to 300 metres per minute or even 400 metres per minute or more, by wet spinning processes in which the coagulating media move with a substantially streamline flow in the direction of travel of the materials. The invention is also concerned with apparatus whereby this streamline flow may be achieved.

Turbulence in the coagulating medium may be reduced or eliminated by means of sheets of gauze or similar fine mesh material extending across the coagulating vessel. One or more of such sheets, for example 2, 3 or more, may be inserted behind the jet and others at the end of the coagulating vessel after the point at which the artificial materials emerge. The presence of such sheets assists in breaking up any turbulence due, for example, to the flow of liquid. Pulsation due to the pump or other circulating device may be reduced by having a vessel of large capacity in relation to the rate of flow of liquid in between the circulating device and the jet, e. g., a capacity such that the liquid circulated requires at least

30 secs. or 1 minute to pass through it. Preferably it has a large cross-sectional area so that the linear velocity of the liquid through it is low, e. g., 3-4 yards/minute. It may contain sheets of fine mesh material extending across it to assist in damping out pulsation or turbulence, and/or baffles to prevent the liquor from taking a direct path from the inlet to the outlet.

The shape of the jet employed may also have a considerable effect on the nature of the liquid flow, and by suitably designing the jet turbulence may be reduced. For example, an improved flow may be obtained by using, instead of the usual type of jet, a jet having streamline contours. Thus a line jet may be employed having a cross-section of streamline contours in the direction of flow, the orifices through which the solution is extruded being formed in the end of the tapered portion. Preferably the lateral extremities of the jet are also streamlined. Another form of jet which may be employed is one having the shape of the solid formed by the revolution of the cross-section described above about its longitudinal axis. In this case the jet orifices may be formed in a plane normal to the longitudinal axis and passing through the tapered portion, and their axes should be substantially parallel to the longitudinal axis of the jet.

When artificial filaments or other materials are stretched in the coagulating bath their velocity increases along the length of the bath, and in order to increase the velocity of the coagulating medium at the same rate the cross-section of the bath may diminish at the necessary rate in the direction of travel of the materials.

The invention is particularly concerned with wet spinning processes in which the coagulating medium flows at substantially the same rate as the rate of travel of the artificial materials, though, if desired, there may be a difference in the velocities of the materials and the coagulating liquid. For example, the coagulating liquid may flow at a higher speed than the materials so that it exerts a drag upon them which assists in stretching them, provided that the drag is not so great that the properties of the materials are detrimentally affected. Processes of this character are described in U. S. Patent No. 2,027,419, which describes the use of coagulating media of high viscosities so that they are capable of exerting considerable drag. The present invention, however, is particularly concerned with avoiding any drag on the materials due to the coagulating medium, and therefore, in general, no additions are made to the coagulating media for the pur-

pose of increasing their viscosities. Thus the coagulating media employed may have a viscosity less than, or at least not substantially greater than, that of water.

5 The coagulating vessels employed according to the present invention may be arranged so that the artificial materials travel in a substantially horizontal direction, or they may be vertical in which case the materials may be extruded either
10 upwards or downwards.

A further method of reducing the drag on the artificial materials comprises spinning under such conditions that the materials are sufficiently set after only a relatively short run through the coagulating medium, for example a run of only 6
15 inches or 4 inches or even less. For example, instead of using an aqueous coagulating bath containing 60% of diacetone alcohol as is described in Example 1 of British Patent No. 340,324
20 and immersing the filaments in the bath for a length of 8 to 10 inches, an aqueous bath containing only 50% or 40% of diacetone alcohol may be employed and the length of immersion may thus be reduced to 6 inches or 4 inches or
25 less. The composition of the bath and the length of immersion necessary to obtain the desired degree of coagulation will depend upon the rate of spinning and, in general, as the rate of spinning increases the coagulating power of the coagulating medium should be increased for any given
30 length of immersion. Thus when aqueous coagulating media containing substantial proportions of relatively non-volatile solvents, such as are described in British Patent No. 340,324 are employed, the proportion of solvent may be de-
35 creased with increasing rates of spinning. For example if for a given length of immersion a coagulating medium containing 50% of diacetone alcohol or glycol monoacetate is satisfactory at
40 a spinning speed of 100 metres per minute, when the spinning speed is increased, for example to 200 metres per minute, the concentration may be decreased to 45 or 40% or even less according to the degree of coagulation and the draw down
45 required.

Preferably spinning solutions comprising cellulose acetate or other organic derivative of cellulose dissolved in relatively volatile solvents, e. g. acetone, methylene ethylene ether or dioxane, are
50 employed according to the process of the present invention and such solutions may contain higher boiling solvents, for example glycol monoacetate, methyl glycol monoacetate, diacetone alcohol and ethyl lactate. Coagulating media containing
55 substantial proportions of relatively non-volatile solvents, such as those mentioned above and described in British Patent No. 340,324, are particularly suitable, though other coagulating media, for example solutions of salts, may be
60 employed.

Examples of apparatus according to the present invention are illustrated diagrammatically in the accompanying drawing, wherein

65 Figures 1 and 2 show respectively a sectional elevation and a plan view of a coagulating vessel containing sheets of gauze;

Figures 3 and 4 show a cross-section and an end view of a streamline jet in which the orifices are arranged in a circle;

70 Figure 5 shows an end view of a streamline jet in which the orifices are arranged in two lines, and Figure 6 a cross-section of the jet on the line 6—6 of Figure 5, the cross-section on the line 3—3 being similar to that shown in Figure 3; and
75 Figure 7 shows a wet spinning apparatus which

comprises a vessel of large capacity to assist in reducing turbulence.

The apparatus illustrated in Figures 1 and 2 comprises a coagulating vessel 10 containing a streamline jet 11 provided with a candle filter 12.
5 The vessel comprises inlet and outlet connections 13 and 14 for the coagulating medium. In front of the orifice 13 is positioned a gauze 15, and gauzes 16 are fitted just behind the jet 11 and in the outlet 14.
10

The jet illustrated in Figures 3 and 4 comprises a hollow body portion 21 having an inlet 22 for spinning solution and provided with spinning orifices 23, the axes of these orifices being substantially parallel to the longitudinal axis of
15 the jet.

The jet illustrated in Figures 5 and 6 has a cross-section on the line 3—3 similar to that shown in Figure 3 and comprises two rows of orifices 23, the axes of which are substantially
20 parallel to the plane of symmetry passing between the two lines of orifices. The ends of the jet are streamlined as indicated by the cross-section shown in Figure 6 taken on the line 6—6
25 of Figure 5.

The apparatus illustrated in Figure 7 comprises a coagulating vessel 10 containing a streamline jet 11 of the type illustrated in Figures 3 and 4 which is provided with a candle filter 12 and
30 inlet and outlet pipes 13 and 14 for the coagulating medium. It also comprises gauzes 15 and 16. The outlet pipe 14 is connected with a pump 24 for circulating the coagulating medium, the outlet side of the pump being connected by a pipe
35 27 with a vessel 25 of large capacity from which the coagulating medium passes into the coagulating vessel through the pipe 13. The inlet pipe 27 is carried to near the top of the vessel which contains a partition 28 extending nearly to the
40 bottom and two horizontal gauzes 29 and 30. The coagulating medium introduced into the vessel is thus forced to flow down one side of the partition and then up the other side through the gauzes before reaching the outlet pipe 13. The
45 vessel 25 is provided with an outlet 26 for removing the coagulating medium if required.

The present invention is particularly concerned with the manufacture of artificial filaments and similar materials having a basis of cellulose acetate and other organic derivatives of cellulose,
50 for example cellulose formate, cellulose propionate, cellulose butyrate, cellulose nitroacetate and other cellulose esters and mixed esters, methyl, ethyl, propyl and butyl celluloses, and other cellulose ethers and mixed ethers, and mixed ether-
55 esters, for example ethyl cellulose acetate and oxy-ethyl cellulose acetate.

Having described my invention what I desire to secure by Letters Patent is:

1. Apparatus for the production of artificial
60 filaments, yarns and similar materials by wet spinning processes, which comprises a vessel for coagulating medium provided with an inlet and an outlet for said medium and with fine mesh material extending across the vessel and sub-
65 stantially normal to the direction of flow of the medium.

2. Apparatus for the production of artificial filaments, yarns and similar materials by wet spinning processes, which comprises a vessel for coagulating medium provided with an inlet and
70 an outlet for said medium and with sheets of gauze extending across the vessel and substantially normal to the direction of flow of the medium.

3. Apparatus for the production of artificial filaments, yarns and similar materials by wet spinning processes, which comprises a vessel for coagulating medium provided with an inlet and an outlet for said medium, with fine mesh material extending across the vessel substantially normal to the direction of flow of the medium, and with a spinning jet having streamlined contours and positioned in said vessel downstream of the fine mesh material.

4. Apparatus for the production of artificial filaments, yarns and similar materials by wet spinning processes, which comprises a vessel for coagulating medium provided with an inlet and an outlet for said medium, with sheets of gauze extending across the vessel substantially normal to the direction of flow of the medium, and with a spinning jet having streamlined contours and positioned in said vessel downstream of the sheets of gauze.

5. Process for the production of artificial filaments, yarns and similar materials, which comprises extruding a solution of a filament-forming base through a shaping orifice directly into a bath of coagulating liquid moving in substantially the same direction as the extruded materials and in a manner such that all the extruded materials are immediately contacted by the coagulating liquid of

the bath, and while the coagulating liquid is moving in the direction of the extruded materials subdividing said coagulating liquid, at a point prior to the point of contact of the same with the extruded materials, into a large number of parallel streams and allowing the streams to reunite so as to produce a substantial stream-line flow of the coagulating liquid in the direction of movement of said extruded materials.

6. Process for the production of artificial filaments, yarns and similar materials, which comprises extruding a solution of a filament-forming base through a shaping orifice directly into a bath of coagulating liquid moving in the same direction and at substantially the same velocity as the extruded materials and in a manner such that all the extruded materials are immediately contacted by the coagulating liquid of the bath, and while the coagulating liquid is moving in the direction of the extruded materials subdividing said coagulating liquid, at a point prior to the point of contact of the same with the extruded materials, into a large number of parallel streams and allowing the streams to reunite so as to produce a substantial stream-line flow of the coagulating liquid in the direction of movement of said extruded materials.

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