

Jan. 15, 1963

J. A. MITCHELL

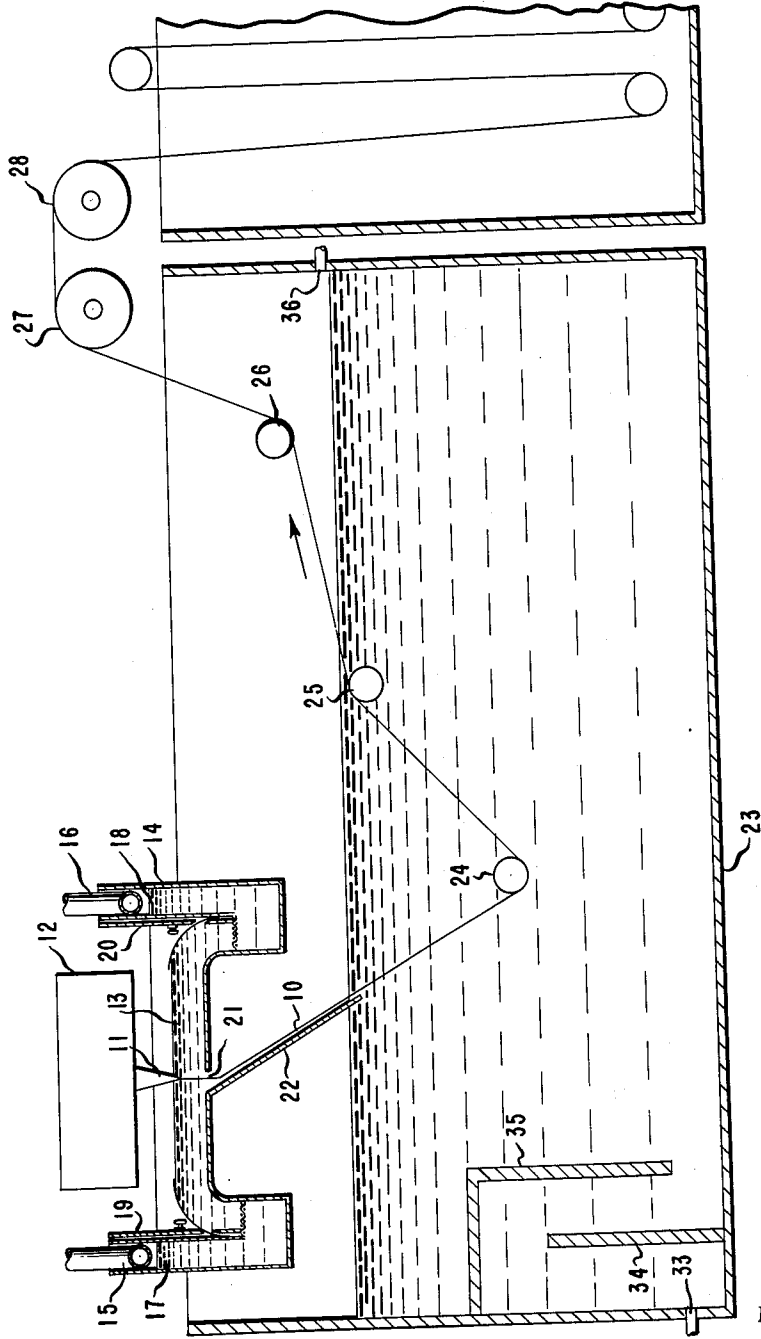
3,073,733

FILM AND METHOD OF CASTING FILM

Filed May 31, 1960

4 Sheets-Sheet 1

Fig. 1



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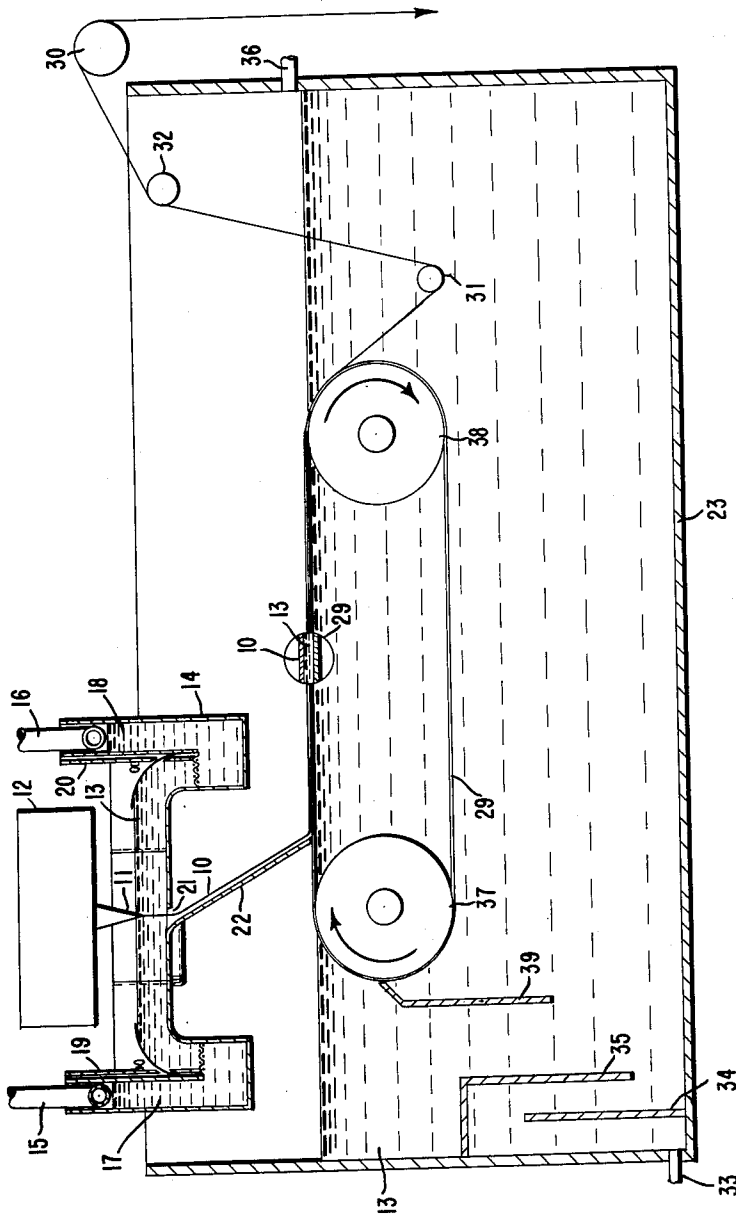
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FILM AND METHOD OF CASTING FILM

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4 Sheets-Sheet 2

Fig. 2



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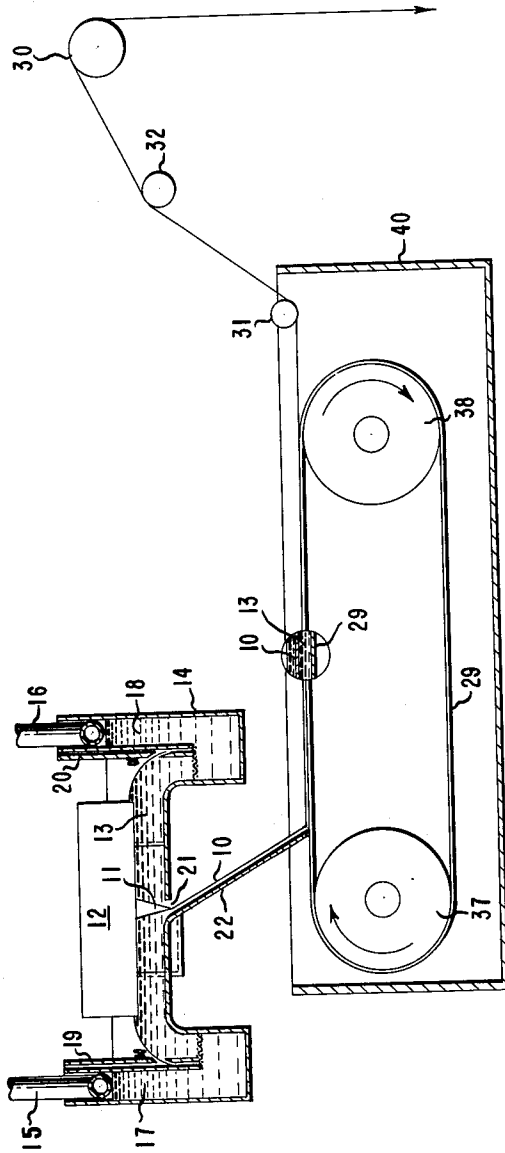
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FILM AND METHOD OF CASTING FILM

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4 Sheets-Sheet 3

FIG. 3



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FILM AND METHOD OF CASTING FILM

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4 Sheets-Sheet 4

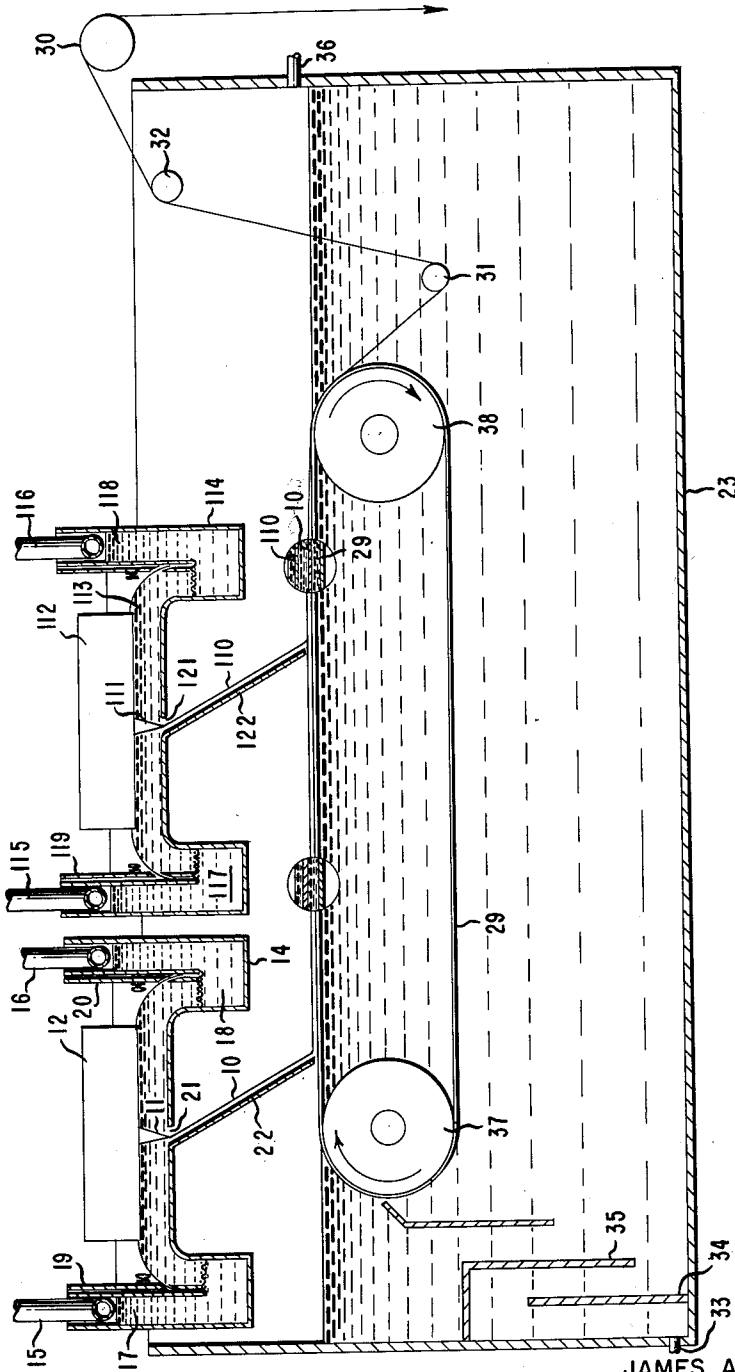


FIG. 4

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3,073,733

FILM AND METHOD OF CASTING FILM

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6 Claims. (Cl. 154-46)

This invention relates to the casting of a novel regenerated cellulose film and, more particularly, to a process and an apparatus for casting regenerated cellulose film at relatively high velocities (as high as 200 yards per minute or higher).

This application is a continuation-in-part of my copending application Serial No. 689,522, filed October 11, 1957, now abandoned.

In casting regenerated cellulose film, a viscous film-forming solution is extruded through the lips or orifice of a hopper into an acid-sulfate coagulating liquid. When drawn rapidly through the liquid bath, the film tends to draw liquid along with it causing a rapid flow of bath in the region of the extrusion lips. This flow is augmented by increased circulation of bath in an effort to keep the bath composition and temperature constant at the higher film rate. The rapid movement of bath in the region of extrusion provides turbulence which tends to affect the quality of the film adversely. The turbulence strains the freshly-formed film, often causing breaks and generally resulting in a streaky, narrowed, physically weakened regenerated cellulose film. Additional strain on the film is provided by the drag of the liquid bath on the film as the film moves through the bath. This strain on the film, due to bath drag, tends to reduce the durability of the film.

The objects of the present invention are an apparatus and process which will minimize the tension on the freshly-extruded film, minimize turbulence in the liquid bath and produce a novel, high quality regenerated cellulose film even at relatively high velocities. Other objects will appear hereinafter.

The objects are accomplished by mounting a smaller, specially-designed chamber above the conventional coagulating vessel, the chamber adapted to receive liquid coagulant, and extruding the film-forming solution below the surface of the liquid coagulant contained in the smaller chamber. The design of the smaller chamber comprises a slot in the bottom of the chamber, the dimensions of the slot being at least equal to the thickness and the width of the extruded film, and integral with the slot a downwardly declined tray adapted to convey liquid from the smaller chamber to the vessel below and to direct the extruded film sheet into the vessel.

The essential process of the invention involves extruding viscose into an acid-sulfate bath in the form of a film, conveying a thin layer of acid-sulfate liquid at a controlled speed, controlled by the inclination of a downwardly declined flume such as a tray, chute, trough, channel or the like, and continuously drawing the freshly-extruded film independent of the acid-sulfate liquid layer over and in contact with the layer of liquid, the liquid in contact with the film being only enough to substantially complete coagulation of the film in contact therewith. It will be recognized at the outset that the film itself will inherently carry a liquid layer on its surface. Thus, for the purpose of this invention, it is the total liquid in contact with the film, above and below the film, that must be only enough to substantially complete coagulation of the film. It will also be recognized that to minimize bath drag, it is preferred to extrude the viscose as close as possible to the point at which the liquid layer has formed and is being conveyed at the controlled speed.

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While the previously described "declined" flume or tray may be used alone, it is not the most satisfactory arrangement from a practical standpoint. Such a flume, if used alone, would have to be extremely long. Hence, the most satisfactory results are obtained when the "declined" flume is used in combination with a "moving" flume. The latter is the subject of a copending application U.S. Serial No. 689,735 to B. L. Hinkle and F. C. Stults, now Patent No. 2,962,766. The moving flume tends to extend the protection of the gel film to a point where the film is completely coagulated and to preserve the limitations expressed above that the film be continuously drawn independent of the acid-sulfate liquid on both flumes and that the total liquid in contact with the film be only enough to substantially complete coagulation of the film by the time the film leaves the moving flume.

The novel product produced by this invention is a highly durable film, the uniplanar orientation at each surface of the film as measured by the optical retardation of a 30-micron thick section of the film being at least 60 millimicrons lower than the uniplanar orientation at the center of the film.

The operation and the advantages of the invention will be more readily apparent by referring to the following detailed description in conjunction with the accompanying drawing, in which:

FIGURE 1 is a sectional diagrammatic side elevation of one embodiment of the invention;

FIGURE 2 is a sectional diagrammatic side elevation of another embodiment of the invention;

FIGURE 3 is a sectional diagrammatic side elevation of another embodiment of the invention; and

FIGURE 4 is a sectional diagrammatic side elevation of another embodiment of the invention.

In FIGURES 1 and 2 a viscose solution, which may contain 4%–15% cellulose and 4%–15% sodium hydroxide, is extruded downwardly in the form of a sheet of gel film 10 through the lips 11 of hopper 12 into the coagulating liquid or bath 13 disposed in the chamber 14 and preferably as close to the opening 21 as possible. The liquid coagulant is usually an acid-sulfate bath containing 4%–15% sulfuric acid and 5%–20% sodium sulfate. The liquid is introduced into the chamber 14 from pipe inlets 15 and 16 through two reservoirs 17 and 18. The reservoirs are separated from the hopper section of the chamber by two baffles 19 and 20. The liquid is forced to pass under the baffles to dissipate turbulence near the region of extrusion. In the bottom of chamber 14 is a slot 21, the dimensions of which are at least equal to the thickness and width of the extruded film. Integral with the slot is a downwardly declined tray 22. The slot and tray are adapted to convey the coagulating liquid and to direct the film to the vessel 23 below.

The liquid 13, which may be substantially the same composition as in chamber 14, is fed into vessel 23. This liquid, which serves to complete regeneration of the gel film, enters through conduit 33. Baffles 34 and 35 disposed in vessel 23 serve to minimize any turbulence that might be caused by the flow of incoming bath liquid. Spent liquid may leave the vessel 23 in any convenient manner such as an outlet 36.

By means of guide rolls 24, 25 and 26 and drive rolls 27 and 28 in FIGURE 1, the gel film 10 is drawn from vessel 23 to the subsequent purification, bleaching and softening treatments. The endless moving belt 29 in combination with guide rolls 31 and 32 and drive roll 30 serve a similar purpose in FIGURE 2. It should be noted that in both embodiments the film is drawn independently of the liquid. The film velocity does not depend on the liquid velocity down the declined tray nor the liquid velocity on the moving endless belt, but depends substantially

on the speed of the drive rolls 27 and 28 in FIGURE 1 and drive roll 30 in FIGURE 2. The driving sources for these power-driven rolls 27, 28 and 30 are not shown but may be conventional variable speed motors.

To control the velocity of liquid coagulant flowing down the declined tray 22, the angle or slope of the tray may be varied. The speed of the moving belt 29 in FIGURE 2 is adjusted to convey liquid at a velocity substantially equal to the velocity of the liquid on the tray. The belt 29 in FIGURE 2 must be at least as wide as the film and should be constructed of a suitable, flexible, non-corrosive material. Preferred materials are "Mylar"¹ polyester film and a neoprene-covered fabric approximately one-quarter of an inch thick. The belt 29 is fitted around rolls 37 and 38, which rolls are driven by a variable speed drive not shown. The belt may be placed so that it picks up supplemental liquid from bath 13. However, it is preferred that the only liquid in contact with the film is the liquid from vessel 14. A squeegee-type baffle 39 serves to adjust the amount of supplemental liquid on the belt and to minimize any turbulence that might be caused by the pumping action of the belt. The level of the bath in vessel 23 is preferably about one inch below the top section of the belt 29. However, the height of the bath in vessel 23 is adjustable and may vary anywhere from slightly above to three inches below the level of the belt. The important consideration is that the bath level be adjusted so that the belt will retain a coagulating liquid layer that is only enough to substantially complete coagulation of the film and partially regenerate the cellulosic film by the time the film leaves vessel 23. Thereafter, regeneration of the cellulosic film is completed either in air or in a subsequent vessel; the film is washed, desulfured, bleached and softened in the conventional manner. FIGURE 3 illustrates an apparatus wherein a drip pan 40 containing no bath is used instead of vessel 23; the only liquid in contact with the film being the liquid from vessel 14.

FIGURE 4 illustrates a process wherein two viscose films, each of which is extruded in the manner described for FIGURE 2, are being processed simultaneously in accordance with the present invention. It should be understood that two films may be cast in an apparatus similar to FIGURE 3 in which a drip pan is substituted for vessel 23. The second viscose film 110 is extruded through the lips 111 of hopper 112 into the liquid 113 in chamber 114. The film 110 is drawn and the liquid flows through the slot 121 and down the tray 122 to the belt 29. Since the film 10 is already on the belt 29, the film 110 passes over film 10 with a liquid layer between the two films. The two films are then processed together in the manner described for the single film in FIGURE 2. It should be noted that the films are drawn independently of the liquid. The film velocity does not depend upon the liquid velocities down the declined trays nor the liquid velocity on the moving endless belt, but depends only on the speed of the drive roll 30.

In the present invention it is preferred to adjust the velocity of the liquid coagulant in the declined tray in FIGURE 1 and in the trays and on the endless belts of FIGURES 2, 3 and 4 so that the liquid flows at a speed substantially equal to the speed at which the film is drawn from the vessel by the drive rolls. However, for optimum results, the process may be operated so that the speed of the liquid in the declined tray and, in the embodiments shown in FIGURES 2, 3 and 4, on the endless belt as well, is from 80% to 125% of the film speed.

The invention, particularly the novel product of the invention, will be more clearly understood by referring to the examples which follow. These examples, although illustrating preferred embodiments of the invention, should not be considered limitative in any way.

¹ Registered trademark of E. I. du Pont de Nemours and Company.

EXAMPLE 1

In this example, a viscose containing 9% cellulose, 5.2% sodium hydroxide and 25.5% carbon disulfide, based on the weight of cellulose, having a viscosity of 55 poises, was cast in the apparatus illustrated in FIGURE 2. The coagulating liquid contained 12% sulfuric acid and 18% sodium sulfate. The angle of the downwardly declined tray and the speed of the moving belt were adjusted to convey the liquid coagulant at approximately the same speed at which the gel film was drawn, a speed of 135 yards per minute.

As a control, the identical viscose was extruded into the identical coagulating liquid using the conventional apparatus, a description of which may be found in U.S. Patent 1,548,864 to Brandenberger. Both films were washed, desulfured, bleached, softened to contain 17% glycerol and dried to a moisture content of 7% in the conventional manner.

The uniplanar orientation difference and the physical properties for the film of this example and the control film are presented in the following table:

Table I

	Example 1	Control
Uniplanar Orientation Difference (millimicrons) ..	-105	+25
Orientation at Surface	315	445
Orientation at Center	420	420
Impact Strength (kilogram-centimeters)	6.6	3.8
Percent Elongation:		
(Transverse Direction)	48.8	31.4
(Machine Direction)	23.8	12.0
Tear Strength (grams/mil):		
(Transverse Direction)	6.2	3.6
(Machine Direction)	4.2	3.5
Stress-Flex (cycles) ¹ :		
(75° F.)	56	24
(32° F.)	11	1-2

¹ Film was coated with 3 grams per square meter of moistureproof coating as described in Example I of U.S. Patent 2,236,546.

Uniplanar Orientation Difference is the difference obtained by subtracting the optical retardation at the center of a cross section of the film from the optical retardation at the surface. To measure optical retardation, film is selected that is free of wrinkles and folds. The humidity in the vicinity of the film used for these measurements must be minimized. Otherwise, the structure of the film may be altered and provide misleading results.

First, a square approximately 5" x 5" to 6" x 6" is cut from the center of a mill roll width sheet. From this square, a strip of film is cut with a razor blade or sharp scissors about 1/4" wide and about 3" long, the long edge being parallel to the machine direction of the film. A similar strip is cut with the long edge parallel to the transverse direction but overlapping the same machine direction lane as the first strip. Each strip is then fastened into a trough-shaped mold with slotted end plates, the ends passing through the slots being fastened to the outer surfaces of the end plates with pressure-sensitive tape. The strip is embedded in wax especially formulated for this purpose (e.g. "Tissue-mat" supplied by Fisher Scientific Co.) by melting the wax and heating it about 5° C.-10° C. above its melting point, and pouring it into the mold until the regenerated cellulose film strip is submerged by at least 1/8". Sufficient wax should be poured into the mold so that upon cooling, the film strips are completely covered. Prior to pouring the wax, the mold should be lubricated with a silicone release agent or with glycerol for easy removal of the wax block after solidification.

After the wax block cools to room temperature, the block is removed from the mold and is shaped by carving with a surgical scalpel to a rectangle of wax surrounding the film strip by at least 1/16" at the edges and 1/8" on each side. Too little supporting wax will cause the film to become distorted during slicing. Sections are then cut with a microtome for mounting on a microscope slide.

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The thickness of the sections is standardized at 30 microns for convenient reading of optical retardation using a graduated quartz wedge.

Each section is mounted on a microscope slide by means of an adhesive such as a 10% solution of "Bakelite" BRL-1100 phenol-formaldehyde resin in acetone. Precuring of the resin by heating to a syrupy consistency is desirable before dissolving in acetone. A few drops of the solution are spread on the slide with the side of a glass rod. After drying the slide for a few seconds, the section is placed on the slide and the slide warmed on the spreading table at about 45° C. The section is lightly flattened with a metal roller, the wax is dissolved away in two xylene washes. The xylene is allowed to evaporate and the section mounted on the wedge is ready for examination in the polarizing microscope.

The section which presents a cross section of the film is examined in a polarizing microscope, e.g. Spencer Polarizing Microscope No. 41 manufactured by the American Optical Company, using a 57× dry objective lens and replacing the standard eye piece by a graduated quartz wedge compensator. Optionally, the film section may be immersed in xylene or other non-swelling liquid to eliminate surface irregularities on the film. When the section is brought into focus it is seen that the zero order shadow is displaced from the zero line of the wedge an amount which is read directly in millimicrons as the optical retardation. A line may be drawn from one surface to the other through points representing the densest portions of successive layers of the zero order shadow. The surface readings are taken at the intersection of this line with the film surfaces, and the center reading is taken half-way through the film section. A relative measure of the degree of uniplanar orientation in a given layer is obtained by adding together the readings of optical retardation taken in the machine direction and transverse direction for that layer. The number so obtained depends on the polarization anisotropy of the individual molecules and on the thickness of the section as well as on orientation. In the measurements described herein, these quantities are constant for a given film composition.

As an illustration of the method, the method for obtaining the uniplanar orientation difference for Example 1 will be described. First, the optical retardation of a 30 micron section of the regenerated cellulose film cut in the machine direction is read. It was observed that the optical retardation values for the top and bottom surfaces were substantially the same. Let the value at each surface be "a", and the value in the center be "b". Then values from a similar section cut in the transverse direction are read. It was noted that these values for top and bottom surfaces were also substantially the same. Let the value at each surface be "a'" and the value at the center be "b'". The uniplanar orientation difference "Q" is then

$$Q = (a + a') - (b + b')$$

For Example 1, the values in millimicrons were: a, 275; b, 250; a', 40; and b', 170. Substituting in the equation above:

$$Q = (275 + 40) - (250 + 170) \\ Q = 315 - 420 = -105$$

A negative value indicates that the surface has lower uniplanar orientation than the interior; a positive value indicates that the surface has a higher uniplanar orientation than the interior. Ordinarily, optical retardation measurements are made for each surface and the difference between center and each surface recorded. For films of the present invention, each difference must be at least 60 millimicrons. However, if the values for the top and bottom surfaces are substantially the same, as in most cases, only one calculation of the uniplanar orientation difference need be presented.

Impact Strength or Pneumatic Impact Strength is the energy required to rupture a film. It is reported in kilograms-centimeters/mil of thickness of the film sample and

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is determined by measuring the velocity of a ball mechanically accelerated by air pressure, first in free flight and then in flight immediately after being impeded by rupturing the test film sample. In this test, the film sample is 1 3/4" x 1 3/4". The projectiles are steel balls 1/2" in diameter and weighing 8.3 grams. The velocity of the ball in free flight is 40 ± 2 meters/second. The velocities are measured by timing photoelectrically the passage of the steel balls between two light beams set a definite distance apart. The pneumatic impact strength is measured by the loss in kinetic energy of the ball due to the rupturing of the film sample and is calculated by multiplying a constant by the difference between the square of velocity in free flight and the square of velocity in impeded flight. The constant is directly proportional to the weight of the projectile and inversely proportional to the acceleration due to gravity. The test is carried out at 23° C. at 50% relative humidity and the test samples are conditioned for 24 hours at 23° C. and 50% relative humidity prior to the test.

Tear Strength is determined on a conventional Elmendorf tear tester which measures the force required to tear a two-inch strip of film, as described by D. W. Flierl, *Modern Packaging*, 52, 129 (1951).

Percent Elongation is determined by elongating the film sample on a Scott Serigraph tester. A 3" x 7" sample is placed in 1" wide jaws stationed 3" apart. The sample is then elongated at the rate of 12" per minute. The force applied at the break in lbs./sq. in. (p.s.i.) is the tensile strength. The elongation is the percent increase in the length of the sample at breakage.

Stress-Flex is a measure of the flexibility and durability of the film. A sample of film, 4" x 7", is placed between two rubber-face clamps 1" apart. One clamp is stationary, the other slides back and forth by gravity on two rods flexing the film as the whole assembly rotates, until the film sample breaks. The stress-flex value indicates the number of strokes or cycles of the movable clamp until the film sample breaks. For tests at 75° F., the sample consists of two sheets, previously conditioned at 75° F.; the sliding clamp has a weight of 4 pounds. For the test at 32° F., the sample consists of a single sheet, previously conditioned at 32° F.; the sliding clamp has a weight of 1 1/2 pounds.

Example 2

In the following example, a viscose containing 9% cellulose and 5.2% sodium hydroxide was cast in the apparatus shown in FIGURE 2 using a liquid coagulant containing 12% sulfuric acid and 18% sodium sulfate. The angle of the downwardly declined tray and the speed of the moving belt were adjusted to convey the liquid coagulant at approximately the same speed as the gel film, a speed of 150 yards per minute.

As a control, the identical viscose was extruded into the identical liquid coagulant using the conventional apparatus, a disclosure of which may be found in U.S. Patent 1,548,864 to Brandenberger. Both films were washed, desulfured, bleached, softened to contain 17% glycerol and dried to a moisture content of 7% in the conventional manner.

The film produced by the present invention was wider, stronger and less oriented than that produced in the control. The uniplanar orientation differences and the physical properties are compared in the following table:

Table II

	Example 2	Control
Width (inches).....	8.3	7.0
Impact Strength (kgms.-centimeters).....	6.0	3.3
Tear Strength (grams).....	5.6	4.0
Uniplanar Orientation Difference (millimicrons).....	-100	+20
Orientation at Surface.....	325	410
Orientation at Center.....	425	390

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EXAMPLES 3-4

In these examples, a viscose containing 9% cellulose, 5.2% sodium hydroxide and 25.5% carbon disulfide, based on the weight of cellulose, and having a viscosity of 55 poises, was cast in the apparatus illustrated in FIGURE 4. The coagulating liquid contained 12% sulfuric acid and 18% sodium sulfate. The angles of the downwardly declined trays and the speed of the moving belt were adjusted to convey the liquid coagulant at approximately the same speed at which the gel film was drawn. The two films were washed, desulfured, bleached, softened and dried in the conventional manner and wound on separate mill rolls. The dried films contained 13% glycerol and 7% moisture. Example 3 was performed at 90 yards per minute; Example 4, at 120 yards per minute. The properties and the uniplanar orientation differences of each of the four films produced are shown in Table III.

Table III

	Example 3 (Cast at 90 y.p.m.)		Example 4 (Cast at 120 y.p.m.)	
	Top Film	Bottom Film	Top Film	Bottom Film
Uniplanar Orientation Difference (millimeters).....	-70	-60	-125	-70
Orientation at Surface.....	360	380	390	430
Orientation at Center.....	430	440	515	500
Impact Strength (kilogram-centimeters).....	7.8	6.8	6.1	6.0
Tear Strength (grams per mil):				
Transverse Direction.....	14.9	11.0	13.5	12.1
Machine Direction.....	5.6	4.3	5.6	5.5
Stress-Flex (cycles) ¹ :				
75° F.....	56	48	58	50
32° F.....	30	25	32	26

¹ Film coated with 3 grams per square meter of moistureproof coating such as described in Example I of U.S. Patent 2,236,546.

It will be noted from the data in the examples that the films of this invention show uniplanar orientation values in the surface planes or layers that are considerably less than those in the interior of the film. It is believed that, since cellulose behaves like a network structure, a higher uniplanar alignment results in lowering the amount of extensibility left in the structure. And, conversely, a lower uniplanar alignment increases the residual extensibility. Thus, the lower uniplanar orientation of the surface layers, by permitting greater extensibility at the surfaces than in the interior, permits the film to withstand the bending and folding involved in the ordinary use of regenerated cellulose film for packaging and the like. This toughness and ability to absorb work before failure is reflected in the stress-flex test.

Specifically, toughness and durability at a surprisingly high level is only obtained when the uniplanar orientation or alignment of the surface layers of regenerated cellulose film, measured as described using a 30 micron thick section of film, is at least 60 millimicrons less than the uniplanar orientation at the center of the film. The durability of the film will increase as this difference between surface and center alignment increases. However, when this difference becomes greater than about 200 millimicrons, then the extremely high extensibility of the surface tends to detract from the film stiffness. Reduction in film stiffness makes it difficult to use the film in ordinary machinery for packaging and the like. If the great uniplanar orientation difference, above 200 millimicrons, is attributable to a very inextensible center (a high uniplanar orientation at the center of the film), then there may actually be a reduction in the durability of the film.

While the invention has been described for the casting of viscose into an acid-sulfate bath to form one or two regenerated cellulose films, the invention is applicable to the formation of a plurality of films. It is also ap-

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plicable to any system wherein a film may be produced from a coagulable film-forming dispersion or solution by extrusion into a solution of a chemical coagulant. In the case of cellulose film, the invention is applicable to the extrusion of viscose, cuprammonium solutions or solutions of cellulose in inorganic salts and organic acids and organic solvents into known chemical coagulants. The invention is particularly useful for high speed casting, at least 90 yards per minute to 200 yards per minute. However, the invention may be used for casting film at velocities from 40 yards per minute to 200 yards per minute or higher.

Having fully disclosed the invention, what is claimed is:

1. In a process for preparing regenerated cellulose film wherein viscose is extruded in the form of a gel film into a bath of acid-sulfate liquid and the gel film is subsequently removed from the bath and subjected to treatments that include washing and drying to form a regenerated cellulose film, the improvement which comprises establishing a stream of acid-sulfate liquid from said bath in a downwardly declined flume by flowing liquid from said bath directly to said flume; conveying the stream of acid-sulfate liquid in said flume at a speed controlled by the inclination of said flume; extruding said viscose in the form of a gel film below the surface of said bath and, immediately thereafter, passing said film into the stream established in said flume; and continuously drawing said film independent of the speed of said liquid stream, the liquid in contact with said film being only enough to substantially complete coagulation of the film in contact therewith.

2. A process as in claim 1 wherein the speed of the stream of acid-sulfate liquid in said flume is from 80% to 125% of the speed of drawing said film.

3. In a process for preparing regenerated cellulose film wherein viscose is extruded in the form of a gel film into a bath of acid-sulfate liquid and the gel film is subsequently removed from the bath and subjected to treatments that include washing and drying to form a regenerated cellulose film, the improvement which comprises establishing a stream of acid-sulfate liquid from said bath in a downwardly declined flume by flowing liquid from said bath directly to said flume; conveying the stream of acid-sulfate liquid in said flume at a speed controlled by the inclination of said flume to a moving endless belt and conveying the stream of liquid on said belt; extruding said viscose in the form of a gel film below the surface of said bath and, immediately thereafter, passing said film into the stream established in said flume and on said belt; and continuously drawing said film independent of the speed of said liquid stream, the liquid in contact with said film being only enough to substantially complete coagulation of the film in contact therewith.

4. A process as in claim 3 wherein the speed of the stream of acid-sulfate liquid in said flume and on the moving endless belt is from 80% to 125% of the speed of drawing said film.

5. In a process for preparing regenerated cellulose film wherein two viscose solutions are extruded in the form of two gel films into two baths of acid-sulfate liquid and the gel films are subsequently removed from the baths and subjected to treatments that include washing and drying to form two regenerated cellulose films, the improvement which comprises establishing a first stream of acid-sulfate liquid from said first bath in a first downwardly declined flume by flowing liquid from said first bath directly to said first flume; conveying said first stream of acid-sulfate liquid in said first flume at a speed controlled by the inclination of said flume to a moving endless belt and conveying the stream of liquid on said belt; extruding a first viscose solution in the form of a first gel film below the surface of said first bath and, immediately thereafter, passing said first film into the first stream established in said first flume and on said belt;

establishing a second stream of acid-sulfate liquid from said second bath in a second downwardly declined flume by flowing liquid from said second bath directly to said second flume; conveying said second stream of acid-sulfate liquid in said second flume at a speed controlled by the inclination of said second flume onto said first film; extruding a second viscose solution in the form of a second gel film below the surface of said second bath and, immediately thereafter, passing said second film into the second stream established in said second flume and on said first film; continuously drawing said films independent of the speeds of said liquid streams, the liquid in contact with said films being only enough to substantially complete coagulation of the films in contact therewith.

6. A highly durable regenerated cellulose film, the uniplanar orientation at each surface of said film, as measured by the optical retardation of a 30-micron thick

section of said film, being at least 60 millimicrons lower than the uniplanar orientation at the center of said film.

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