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PROCESS FOR IMPROVING THE DIMENSIONAL STABILITY OF REGENERATED CELLULOSE FILMS

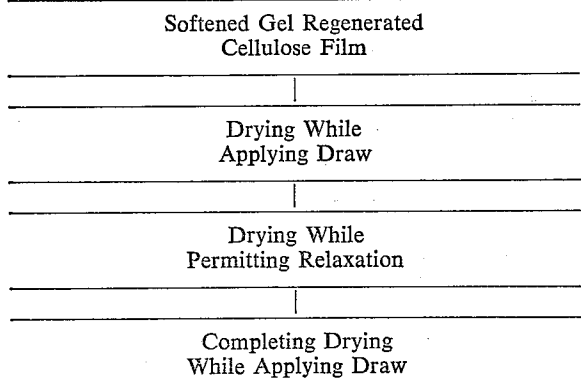
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No Drawing. Filed Sept. 20, 1960, Ser. No. 57,133

2 Claims. (Cl. 18-48)

This invention relates to the manufacture of regenerated cellulose film. More particularly, this invention relates to a method for improving the dimensional stability and appearance of regenerated cellulose films.

The following block diagram illustrates the process of the present invention:



It has been found that overwrapped packages exposed to high humidity develop a pattern of corrugations or ripples running in the machine direction of coated regenerated cellulose film. This ripple pattern detracts so seriously from the appearance of the package that some potential customers question the premium quality of these films. The term "rippling" has been given to the pattern of corrugation which develops in cellophane used for overwrap on a package wrapped at low or normal humidity and subsequently exposed to high humidity. Rippling is to be distinguished from "puckering" which is a localized sheet distortion usually associated with defects or nonuniformities in moisture-proof coatings. A puckered film will eventually flatten out as moisture equilibrium is reached in all parts of the film; rippling, however, persists as long as the film is exposed to the high humidity. Rippling has been a particularly serious problem on those films which have been promoted as a premium quality film giving improved appearance on the package.

Rippling of cellophane at high humidity is caused by simultaneous machine direction shrinkage and transverse direction expansion of the film on an overwrap package when the moisture in the film is increased. There are variations in the degree of rippling and practically all cellophane films ripple to some extent.

It has been found that tension in the cast film during drying is the most important single factor affecting rippling. Large variations in the high humidity distortion have been seen among different casting machines with different dryer types and different tension patterns.

While it is apparent that zero distortion in both directions would be ideal, if the differential distortion between the distortion in the machine direction and that in

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the transverse direction was less than 1.5%, substantially ripple-free films would be obtained. In any case, the distortion in either the machine direction or transverse direction alone must be less than 2%.

It is, therefore, an object of this invention to improve the dimensional stability of regenerated cellulose films. A further object of this invention is to provide a method for producing substantially ripple-free regenerated cellulose films. A still further object is to provide a method for producing such films while maintaining good film flatness and good appearance.

These and other objects are accomplished by the present invention which comprises, in a process for improving the dimensional stability of regenerated cellulose film, the improvement which consists in: (1) drying softened gel regenerated cellulose film to about 100 to 120% moisture while applying 2 to 4% draw, (2) further drying the said film to about 25 to 60% moisture while permitting about 1.5 to 2.5% relaxation, and (3) completing said drying to about 3.0 to 7.5% moisture while applying from about 0.5 to 1.0% draw.

In a preferred embodiment, the drying in step (2) will be conducted to about 25 to 35% moisture.

The term "percent moisture" as used herein refers to the percentage of water present based on the dry weight of cellulose over and above the dry weight of the cellulose itself (e.g., 50% moisture means for every 150 grams of wet cellulose, 50 grams of water and 100 grams of dry cellulose are present).

The term "percent draw" as used herein means the percentage extension of length of the film caused by stretching in the machine direction. The term "applying . . . draw" means the step of extending the length of the film in the machine direction by a given percentage of its original length.

The term "percent relaxation" is the opposite of the term "percent draw" and refers to the percentage decrease in length of the film caused by shrinking in the machine direction. The term "permitting . . . relaxation" refers to the step of decreasing the length of the film in the machine direction by a given percentage of its length by taking advantage of the tendency of the moist film to shrink. Specific means for accomplishing draw and relaxation of the film will appear hereinafter.

The term "soften gel regenerated cellulose" refers to regenerated cellulose in a gel condition containing about 3.0 to about 3.5 times the weight of dry cellulose as water. Further clarification of this term will appear hereinafter.

The initial distortion of cellophane (increase or decrease in length) in passing from 35% humidity to 81% humidity has been the most important criteria yet devised for predicting rippling.

It has been found that when softened gel regenerated cellulose film was dried from about 100 to 120% moisture with 2 to 4% draw, and further dried to 25 to 60% moisture with 1.5 to 2.5% relaxation, and the drying completed to 3.0 to 7.5% moisture with 0.5 to 1.0% draw, the differential between the transverse direction distortion and machine direction distortion was not greater than about 1.5%. Films produced by the described process were ripple-free when exposed to high humidity (i.e., 81% relative humidity), and had good appearance and good film flatness.

The optimum increase or decrease in draw or relaxation, accomplished by differential peripheral speeds of dryer rolls, differ somewhat for different thickness of

film, and for films with different degrees of plasticization. For this reason it is desirable to be able to vary the difference between peripheral speeds before and after the points of critical film moisture content.

The process of the present invention is generally applicable to the drying of continuous webs of any wet cast cellulosic film. However, because of the commercial importance of regenerated cellulose film derived from viscose, such film constitutes the preferred material for treatment in accordance with the principles of the present invention.

The commercial scale method of continuously manufacturing regenerated cellulose film from an aqueous solution of sodium cellulose xanthate (viscose) is disclosed in U.S. Patents 1,548,864 and 1,601,289 to Brandenberger. In this process, viscose is forced through an elongated orifice in the form of a sheet into a coagulating bath to form a coherent web, which freshly coagulated film is then promptly regenerated, washed, desulfured, bleached, softened and dried. The dried film is wound into rolls for convenience in handling, storing, and further processing. Up through the softening stage the web of regenerated cellulose remains in a gel condition (i.e., completely saturated with aqueous solution). Since gel regenerated cellulose film contains about 3.0 to 3.5 times the weight of dry cellulose as water, a substantial amount of moisture must be removed in the drying operation. In losing this moisture, the film shrinks in length, width and thickness (thus diminishing the area of film obtained). To prevent excessive loss in area, the formation of surface irregularities, puckers, wrinkles and the like, the impairment of transparency, it is customary to dry the web by passing it through a series of heated rolls, such as described in Herdon, U.S. Patent 2,000,179; Chylinski, U.S. Patent 2,141,277; Stevens, U.S. Patent 2,746,166 and Britton et al., U.S. Patent 2,746,167. The present invention is concerned with the drying stage of the above-described process of manufacture.

Wet, softened, gel regenerated cellulose film, after leaving the softener tank, passes between squeeze rolls or scraping devices which remove excess softener bath and enters the first section of the dryer and contains approximately 300 to 350% water, based on the weight of dry cellulose. In the first stage of the dryer the film passes over a series of conventional dryer rolls operated at a peripheral speed greater than the rolls in the wet-end of the casting machine which will maintain the web under 2 to 4% draw. The dryer rolls throughout the dryer are uniformly heated and heated air is circulated across the web so as to maintain the web at substantially uniform drying temperature to ensure a symmetrical transverse moisture distribution profile. When the moisture content of the film has been reduced to approximately 100 to 120% moisture based on the weight of dry cellulose, the web is passed over a second series of conventional dryer rolls driven at a peripheral speed 1.5 to 2.5% lower than that of the preceding rolls to effect a decrease in, or a relaxation of, the tension of web. In this stage of the dryer, the moisture content of the film is reduced to 25 to 60% after which the film is passed through an additional series of conventional dryer rolls drawn at a peripheral speed of from 0.5 to 1.0% greater than that of the rolls in the previous stage to exert 0.5 to 1.0% draw on the film. In this stage, the moisture content of the film is reduced to about 3.0 to 7.5% moisture based on the weight of dry cellulose. Thus, with the drying completed, the film leaves the dryer and is collected as a mill roll.

The table below indicates the various tension patterns used in eight different examples with the resulting distortions having been produced by subjecting the finally dried regenerated cellulose films to the distortion test which will be described below. In the following table "MD" indicates the distortion in millimeters in the machine direction, and "TD" indicates the distortion in

millimeters in the transverse direction after the samples have been subjected to the distortion test.

DISTORTION TEST

Samples of regenerated cellulose film (about 0.88 mil after drying) dried under the conditions indicated in Examples 1 to 8 inclusive, below, were coated on both sides with a vinylidene chloride copolymer from a solvent solution, properly dried to evolve solvent, and then conditioned at 75° F. and 35% relative humidity for 72 hours. (After coating, the films were about 1 mil in thickness.) After this conditioning, strips of film 400 millimeters in one direction by one inch in the other direction were cut approximately out of the center of a full cast width of the coated sheet of regenerated cellulose. Samples were cut such that some samples had the long direction parallel to the transverse direction and some parallel to the machine direction. Each of the film samples was supported against a frame having the approximate dimensions of each film sample, and the film sample was fixed at the top of the frame so that its long dimension was running vertically when the frame was hung in a conditioning chamber. A 20 gram weight was clamped at the bottom of each film. The frames to which each film sample was affixed at one end thereof were hung in a room maintained at 81% relative humidity and 85° F. The change in dimensions under the conditions of 81% relative humidity was recorded as change in length (in millimeters) after 72 hours. The corresponding values are recorded in the following table under "MD" and "TD."

The percent distortion in the machine direction (MD) and transverse direction (TD) is calculated by the following:

$$\frac{\text{MD (mm.)}}{400 \text{ mm.}} \times 100 = \text{Percent Distortion in MD}$$

$$\frac{\text{TD (mm.)}}{400 \text{ mm.}} \times 100 = \text{Percent Distortion in TD}$$

The differential distortion (expressed as percent) is calculated by the following:

$$\frac{\text{TD (mm.)} - \text{MD (mm.)}}{400 \text{ mm.}} \times 100$$

= Percent Differential Distortion

In the following tables when the sign in front of MD (mm.) and TD (mm.) is plus (+), it means that the film is expanded in the test. When the sign is minus (-), it means that the film shrank in the test. It should be emphasized, however, that even though the sign of TD (mm.) may be minus it is always used as a plus value in the above formulation when calculating percent differential distortion. The calculation is carried out in this manner because it is desired, if there is any change in dimension at all in the film during the test, that the change in dimension be an increase (plus) to avoid any semblance of rippling, it being understood, as mentioned before, that the percent distortion in either the MD or TD alone must be less than 2%.

Examples 1 and 2 below illustrate operation within the scope of this invention:

Ex.	Moisture content	Tension pattern	MD (mm.)	TD (mm.)	Differential distortion (percent)
1	Gel to 100%----- 100% to 30%----- 30% to 5%-----	2.5% draw----- 1.5% relax----- 0.7% draw-----	+2.0	+7.0	1.25
2	Gel to 100%----- 100% to 30%----- 30% to 5%-----	3.2% draw----- 1.5% relax----- 1.0% draw-----	+1.75	+6.0	1.05

For comparison, the following Examples 3 to 8 are given of operation outside the scope of this invention:

Ex.	Moisture content	Tension pattern	MD (mm.)	TD (mm.)	Differential distortion (percent)
3	Gel to 100%----- 100% to 30%----- 30% to 5%-----	2.0% draw----- 2.0% draw----- 2.0% draw-----	-7.1	<u>-2.1</u>	<u>2.3</u>
4	Gel to 100%----- 100% to 5%-----	2.0% draw----- 1.5% relax-----	+1.3	<u>+8.2</u>	<u>1.7</u>
5	Gel to 30%----- 30% to 5%-----	1.5% relax----- 2.0% draw-----	+1.1	+7.3	1.5
6	Gel to 100%----- 100% to 30%----- 30% to 5%-----	2.3% draw----- 4.6% relax----- 1.6% draw-----	+2.75	<u>+8.75</u>	1.5
7	Gel to 100%----- 100% to 30%----- 30% to 5%-----	3.9% draw----- 1.4% relax----- 0.29% draw-----	+1.2	<u>+8.65</u>	<u>1.86</u>
8	Gel to 100%----- 100% to 30%----- 30% to 5%-----	4.9% draw----- 4.7% relax----- 4.9% draw-----	+0.5	<u>+12.75</u>	<u>3.06</u>

NOTE.—The underlined figures indicate distortions and differential distortions (percent) above allowable limits.

Below are listed additional comments on the films processed in accordance with the above examples:

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| Ex. | Comments |
| (1) | The differential distortion was about 1.25%. The film did not ripple and had good film flatness and appearance. |
| (2) | The differential distortion was about 1.05%. The film did not ripple and had good film flatness and appearance. |
| (3) | The film had a differential distortion of about 2.3%. The film rippled and also had excessive edge droop and poor appearance. |
| (4) | The film had a transverse direction distortion greater than 2% and a differential distortion of about 1.7% but had excessive edge droop. |
| (5) | The film had good film flatness but poor appearance with watermarks and excessive striations. The differential distortion was about 1.5%. |
| (6) | The transverse direction distortion was greater than 2% (2.18%). The film rippled, had poor appearance and excessive edge droop. |
| (7) | The transverse direction distortion was greater than 2% (2.16%). The film rippled, had fair appearance and excessive edge droop. |
| (8) | The transverse direction distortion was greater than 2% (3.18%). The film rippled, had poor appearance and excessive edge droop. |

The above examples illustrate the criticality of the limitations in the invention by showing that operation outside the scope thereof yields films which are for one or more reasons unacceptable.

Flatness of a film is a characteristic of continuous films which is an important determining factor in their running abilities on converter and processing equipment. An absolutely flat sheet, having no droop, is the goal of all continuous film manufacturers. Flatness is determined by stretching a film longitudinally across two carefully aligned, horizontal parallel supporting rolls or bars 15 feet apart. The film ends are rigidly held and tension is applied longitudinally to the film over the unsupported span of 15 feet. The contour of a transverse section of the film at approximately the center of an unsupported span is observed and the major departures from the straight, horizontal line are measured in inches. Experience has shown that a lack of flatness and, particularly, the presence of a slack longitudinal lane between two supporting lanes, called center droop, is extremely detrimental to satisfactory operation in bag making machines, wrapping machinery, printing presses, etc. Drooping of the film edges, depending upon the severity, is also undesirable. Film flatness, as measured by droop, is considered unsatisfactory for use on such automatic equipment, if the edge droop exceeds one-quarter inch at 1.4 lbs. of tension per inch of film width. The films processed within the scope of this invention exhibited satisfactory flatness when measured by the above test.

This invention provides for the production of a trade acceptable film from the standpoint of rippling, while maintaining good appearance and the desired film flatness.

What is claimed is:

1. In a process for improving the dimensional stability of regenerated cellulose film, the improvement which consists in: (1) drying softened gel regenerated cellulose film to about 100 to 120% moisture while applying 2 to 4% draw, (2) further drying the said film to about 25 to 60% moisture while permitting about 1.5 to 2.5% relaxation and (3) completing said drying to about 3.0 to 7.5% moisture while applying about 0.5 to 1.0% draw.

2. Process according to claim 1 wherein the moisture in step (2) is from about 25 to 35% moisture.

References Cited in the file of this patent

UNITED STATES PATENTS

2,115,132	Alles et al. -----	Apr. 26, 1938
2,517,570	Irons -----	Aug. 8, 1950
2,955,345	Howe -----	Oct. 11, 1960