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CELLULOSIC PELLICLE AND METHOD OF PREPARATION

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This invention relates to cellulosic structures, and more particularly, to a method for softening cellulosic structures by incorporating a softening agent and the product resulting from such treatment.

In the manufacture of cellulosic pellicles of the type prepared from aqueous solutions, for example; regenerated cellulose pellicles, such as sheets or films, caps, bands, continuous tubing and the like, it has long been customary to incorporate into the pellicle a softening agent in order to impart flexibility. The cellulosic pellicle without a softening agent is known to be quite brittle, and while such softener-free pellicle may find limited use, a softened structure has much wider use.

The most important compound used heretofore as a softening agent for producing flexible, transparent and durable cellulosic pellicles has been glycerol. Despite the numerous advances which have been made in the production and development of cellulosic pellicles, no substitute had been found for glycerol which did not seriously restrict the utility of the product. However, it is well known that cellulosic pellicles, particularly regenerated cellulose film softened with glycerol, undergo considerable dimensional changes when subjected to varying humidities. When using glycerol as a softening agent, the regenerated cellulose film displays an increase in all three dimensions with rising humidity and a substantial decrease with falling humidity. This dimensional instability, especially in the thickness of the regenerated cellulose film, causes telescoping of rolls of pressure-sensitive tapes made from these films upon exposure to high humidities. The handling, storage and use of such rolls leave quite a bit to be desired in a commercial operation.

It is therefore an object of this invention to provide a new and useful method for producing softened cellulosic materials possessing improved dimensional stability.

It is a further object to provide such improvement in dimensional stability without adversely affecting the desirable properties of the cellulosic material.

Other objects will appear hereinafter.

The objects of this invention are obtained by incorporating hexylene glycol (2-4 pentanediol, 2-methyl) into the cellulosic pellicle. Preferably, the objects are obtained by treating the pellicle so as to incorporate 8% to 25% of hexylene glycol (based on combined weight of cellulose plus hexylene glycol), removing the excess hexylene glycol softening agent, and drying the pellicle so treated. The most preferred range of hexylene glycol in the cellulosic pellicle is from 12% to 16%.

The invention contemplates the use of any cellulosic structure, particularly of pellicular nature such as a sheet or film, artificial straw, cap, band, or continuous tube, such as may be obtained by the coagulation and/or regeneration from an aqueous cellulosic dispersion in accordance with the procedure customary to the art. Thus, the invention comprehends the use of cellulose pellicles such as may be obtained from the viscose or cuprammonium processes or the process of U.S.P. 2,737,437 to

M. F. Bechtold, glycol cellulose, cellulose glycollic acid, lowly esterified or lowly etherified cellulose derivatives where there is only one ether or ester group associated with several cellulose units, and other cellulosic structures of similar chaarcter. Similarly, various types of paper, especially of the "glassine" type, may be used as base materials. Sheets of albuminous materials including gelatin, casein, or the like, may also be used. As a matter of convenience, the invention will be described in terms of its application to the sofening of regenerated cellulose pellicles such as those sheets or films of regenerated cellulose which are suitable for use as wrapping tissues, it being understood that the scope of the invention includes any of the other bases set forth above and that the invention is equally applicable thereto.

In the manufacture of regenerated cellulose sheets or films, as for example by the viscose process, the cellulosic dispersion is cast through suitable apparatus into coagulating and/or regenerating baths from which it is finally obtained in the form of a continuous cellulosic sheet which is customarily led in a continuous manner through a series of purifying, bleaching and washing operations. Just prior to the drying operation which is also usually continuous and coordinated with the casting operation, the film is passed through a bath containing a softening agent in such concentration that after the excess liquid has been removed from the surface of the sheet by suitable squeeze rolls and the excess moisture removed by passage through the drier, the final sheet can be wound up in a continuous fashion and will contain an appropriate, predetermined amount of soften agent. The final regenerated cellulose film contains approximately 4% to 10% moisture and anywhere from 8% to 25%, based on the combined weight of the cellulose and softening agent in the sheet, of the softening agent which formerly had been glycerol, and now in accordance with applicant's invention is hexylene glycol. It has been discovered that over 25% hexylene glycol in the regenerated cellulose film provides an objectionable haze or blush to the film, whereas below 8% hexylene glycol provides a film that is not sufficiently pliable.

The invention will be more clearly understood by referring to the following examples, Example I representing the best mode contemplated for performing the invention. These examples, although illustrating specific embodiments, should not be considered limitative of the principles of the invention.

In the examples, the dimensional stability of the regenerated cellulose films was determined by testing the films for thickness deformation under different conditions of humidity and temperature. More specifically, thickness deformation was measured as follows: Five strips, 1" x 3" each, were clipped together and conditioned in an oven at 35% relative humidity and 24° C. for 4 hours. Their combined thickness, T_1, was then measured. These same strips were then conditioned in an oven at 95% relative humidity and 35° C. for 4 hours and their combined thickness, T_2, was again measured. The dimensional stability was then determined by calculating the percent thickness deformation from the formula:

$$\frac{T_2 - T_1}{T_1} \times 100$$

A low percent thickness deformation indicates a relatively dimensionally stable product.

EXAMPLE I

A sheet or film of gel regenerated cellulose, the final thickness of which was approximately 0.0016" was drawn through an aqueous bath containing 20.6% hexylene glycol. The excess liquid was removed from the

surfaces of the film by passing the film through suitable squeeze rolls and the film was dried in the usual manner down to a moisture content between 4% and 10%.

As a control, a gel regenerated cellulose film was passed through an aqueous bath containing approximately 20% of glycerol, the remaining steps being identical to those indicated above. The two films, one containing 14.5% hexylene glycol, the other containing 15.0% glycerol, were substantially identical insofar as flexibility, durability, transparency, etc., were concerned. However, the thickness deformation of the hexylene glycol-containing film was 11.3%; whereas the glycerol-containing film displayed a thickness deformation of 41.6%.

EXAMPLES II–XIV

In the following table, Table 1, are presented the results of 13 examples in which the gel regenerated cellulose film was treated in the manner described in Example I, using, however, a treating bath containing various amounts of hexylene glycol from 6% to 31.5% as given in the table:

Table 1.—Dimensional stability of hexylene glycol-treated films

Example	Percent Hexylene Glycol in Bath	Percent Hexylene Glycol in Film	Thickness Deformation (Percent)
II	6	6.5	14.9
III	10	7.8	14.9
IV	9.0	8.7	13.7
V	10.1	10.8	14.0
VI	12.0	11.0	14.3
VII	14.3	12.5	13.5
VIII	15.1	13.9	14.0
IX	24	15.3	12.0
X	26.1	16.6	12.9
XI	25.0	17.5	11.1
XII	25.5	18.5	11.5
XIII	29.3	19.3	13.7
XIV	31.5	21.4	11.2

As controls, regenerated cellulose film was softened with various amounts of glycerol in the manner previously described. The results are given in the following table, Table 2:

Table 2.—Dimensional stability of glycerol-treated film

Percent Glycerol in Film	Thickness Deformation (Percent)
9.6	31.0
12.2	34.6
15.0	41.6
17.6	48.2
19.6	56.9

The hexylene glycol softening agent may be incorporated into the regenerated cellulose film at any stage during the manufacture of the film. However, it is preferred to incorporate the softening agent just prior to the final drying operation. The softening agent may be incorporated by passing the film through a bath containing the agent in a continuous manner or in a batch manner. The solution of softener may also sprayed into the film or applied manually by brushing or the like.

The films of this invention are used advantageously as packaging materials for foods, cigarettes and the like and in pressure-sensitive tapes. They are particularly useful in the latter application where dimensional stability of the rolls of tape is a practical necessity.

As many widely different embodiments can be made without departing from the scope of my invention, it is understood that the invention is not limited except as defined in the appended claims.

I claim:

1. A flexible, durable, transparent cellulosic pellicle selected from the group consisting of regenerated cellulose, glycol cellulose, cellulose glycolic acid, lowly esterified cellulose derivatives and lowly etherified cellulose derivatives containing 8–25% of 2-4 pentanediol, 2-methyl based on the combined weight of the cellulosic pellicle and 2-4 pentanediol, 2-methyl.

2. A regenerated cellulose pellicle containing 8–25% of 2-4 pentanediol, 2-methyl based on the combined weight of cellulose and 2-4 pentanediol, 2-methyl.

3. A regenerated cellulose pellicle containing 12–16% of 2-4 pentanediol, 2-methyl based on the combined weight of cellulose and 2-4 pentanediol, 2-methyl.

4. In a method for the production of cellulosic pellicles wherein a thin pellicle is formed from an aqueous alkaline cellulosic dispersion by coagulating in an acid coagulating bath, the improvement which comprises treating said pellicle with a solution containing, as a softener for said pellicle, sufficient 2-4 pentanediol, 2-methyl to incorporate 8–25% of 2-4 pentanediol, 2-methyl into said pellicle.

5. In a method for the production of regenerated cellulose pellicles, wherein a thin pellicle is formed from an aqueous alkaline cellulosic dispersion by coagulating in an acid coagulating bath and regenerating the cellulose in said pellicle, the improvement which comprises treating said regenerated cellulose pellice with a solution containing, as a softener for said pellicle, sufficient 2-4 pentanediol, 2-methyl to incorporate 8–25% of 2-4 pentanediol, 2-methyl into said pellicle.

6. In a method for the production of regenerated cellulose pellicles, wherein a thin pellicle is formed from an aqueous alkaline cellulosic dispersion by coagulating in an acid coagulating bath and renegerating the cellulose in said pellicle, the improvement which comprises treating said regenerated cellulose pellicle with a solution containing, as a softener for said pellicle, sufficient, 2-4 pentanediol, 2-methyl to incorporate 12–16% of 2-4 pentanediol, 2-methyl into said pellicle

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