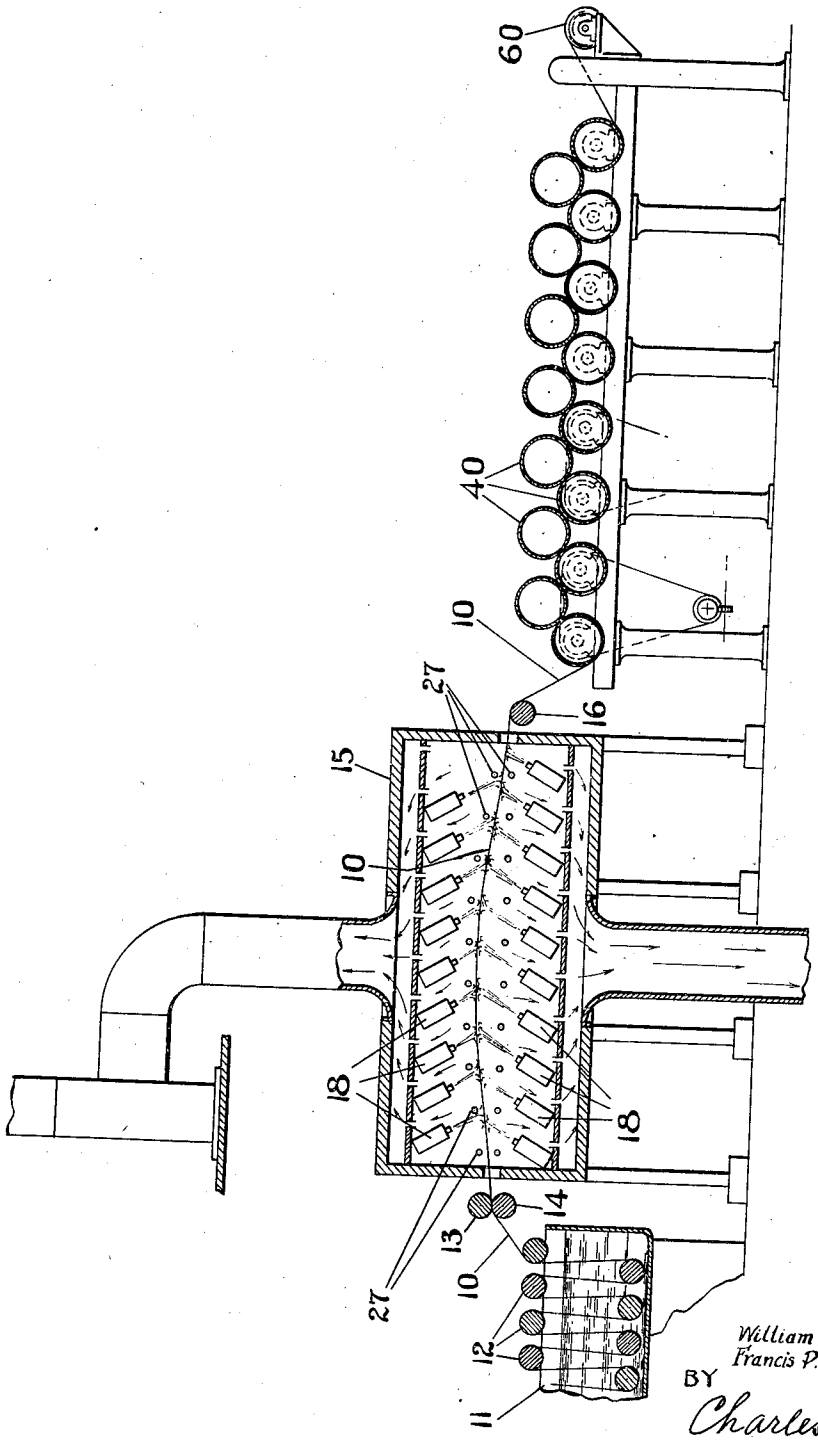
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CELLULOSIC STRUCTURE AND METHOD FOR PREPARING SAME

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CELLULOSIC STRUCTURE AND METHOD FOR PREPARING SAME

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This invention relates to the manufacture of non-fibrous sheets and films which are subject to shrinkage during drying. The invention will be described in terms of the manufacture of regenerated cellulose sheeting cast from viscose. However, it is to be understood that the invention is applicable to the production of cellulosic sheets and films from aqueous alkaline cellulosic solutions generally.

Heretofore, in the manufacture of such sheets and films from viscose, films have been cast in several different thicknesses. The finished sheet produced for ordinary purposes is about 0.001 of an inch thick although thinner or thicker sheets have been produced for other and special purposes.

Although such films have satisfactory tensile strength, for certain purposes they do not have the required degree of toughness to satisfactorily resist the shock and handling to which they are ordinarily subjected in commercial applications. The tensile strength of a film is measured by the magnitude of the force necessary to rupture it, and depends upon the cohesive forces existing within the structure of the film. The toughness of a film is measured by the length of time it will resist rupture under actual handling conditions, and depends primarily upon its elongation or the degree to which it will yield to a force without breaking. These two factors are clearly distinguishable. For example, a film of as high tensile strength as it has hitherto been possible to produce may lack toughness in that it will fail to yield sufficiently to the shocks encountered in commercial use to avoid premature breaking, cracking or splitting.

In the films of the prior art it has been customary to impart a certain amount of toughness or durability, together with flexibility, by incorporating therein a softener, such as glycerin or ethylene glycol. Besides the question of expense, the use of these materials is attended with certain difficulties. The film tends to become sticky, and various precautions are therefore necessary in order to avoid "caking" of film in rolls or stacks or sheets. For example, film containing above 14% glycerin must usually be sized or provided with a tenuous coating of a suitable substance to avoid this sticking. At still higher glycerin contents the sizing must be so thick that it become a true coating. Additionally, the film becomes so soft that it is extremely difficult if not impossible to handle by means of automatic machinery. Furthermore, the high hygroscopicity of these softeners tends to cause the film to absorb more water than it would in their absence and therefore to vary more widely in dimensions with the relative humidity of the atmosphere. Finally, the use of liquid softeners detracts somewhat from the strength or tenacity of the film. As these objectionable characteristics vary directly with the amount of glycerin or similar softener used, a point will be reached where it is no longer practically possible to increase the softener content. It has been found to be of advantage for some uses to increase the toughness without increasing the softener content and for other uses to secure the advantages of a lower softener content while still retaining the desired degree of flexibility and toughness or durability.

It is therefore an object of this invention to produce non-fibrous sheets and films, having good transparency and flexibility, and of greatly increased durability or toughness compared to films of similar softener content hitherto known.

It is a further object of this invention to produce non-fibrous sheets and films having the desired degree of flexibility, durability and toughness, with lower amounts of softener.

It is a still further object to produce such sheets and films from regenerated cellulose.

Other objects of the invention will appear hereinafter.

The objects of this invention are in general accomplished by conducting the preliminary drying step with a substantially complete absence of tension.

In one method of manufacture of regenerated cellulose sheets and films a viscose solution is forced through an elongated orifice into a coagulating bath. The freshly coagulated film is then led through successive treating baths, such as regenerating, washing, desulfuring, bleaching and glycerinating baths to a drying apparatus. The drying apparatus customarily consists of a series of heated rollers over which the film passes and which subject the film to a considerable amount of tension, both in the longitudinal and transverse directions in order to prevent the formation of puckers and wrinkles and impairment of the transparency of the film. It has now been found in accordance with the present invention that non-fibrous cellulosic sheets and films much tougher per unit of thickness than known heretofore and at the same time retaining good transparency and being free of wrinkles and puckers can be produced by substantially eliminating tension during the first portion of the drying operation and drying with normal, increased or re-

duced tension thereafter. Furthermore, sheets and films produced in this manner will exhibit the desired degree of flexibility and toughness with lower amounts of softener. It has been found in accordance with this invention that, contrary to the process used in making the ordinary film of regenerated cellulose, the special type of film can be made in such a way that free shrinkage in all directions is allowed during the early stage of the drying step. This may be achieved, for example, by drying the film with a substantially complete absence of tension until it contains about 100 to 200% water, based on the weight of the cellulose. This step should not, however, be carried below 100% moisture, since in the lower moisture range complete absence of tension will produce puckers and wrinkles and will impair the transparency of the film. Tension, less than normally used in accordance with the prior art, can, of course, be employed in drying below this moisture content, if desired, but tension cannot be completely eliminated.

The preferred form of apparatus for carrying out the present invention is substantially the same as that shown in U. S. Patent No. 2,099,162 (Eberlin). This apparatus (described in the said Eberlin patent as applicable to the production of all types of film) should be modified so as to achieve this shrinkage and should dry the film without tension down to the required moisture content. It should be emphasized that tension should be substantially eliminated in all directions. For example, in operating the Eberlin drier to produce the ordinary film, shrinkage is substantially completely prevented in the longitudinal direction or direction of travel of the film, due to the fact that the film, after traversing the Eberlin drier, is traveling at substantially as high a rate of speed as before entering said drier. When operating in accordance with the present invention, however, the Eberlin drier must be operated in such a manner that the film within the drier is entirely free to shrink in all directions.

The details of the invention will be more clearly apparent by reference to the following description taken in connection with the accompanying drawing illustrating one embodiment of apparatus suitable for carrying out the present invention and in which the figure is a fragmentary diagrammatic elevational view of a drying apparatus.

Referring now to the drawing, the film 10 is led through the final treating tank 11, usually containing a softener which impregnates the film, over rotating rollers 12 and squeeze rollers 13 and 14, into the preliminary drying apparatus indicated generally at 15. This consists of an enclosure containing a series of jets 18 directing streams of hot air or open gas flames against the sides of the film. Positioned adjacent and below the path of travel of the film may be placed nickel rods 27 in order to support the film and keep it away from the immediate vicinity of the burners. If desired, the jets 18 positioned above the film may be omitted, in which case it may be desired to omit the nickel rods 27.

After emerging from the preliminary drying apparatus 15, the film is led over a roller 16, which travels at a lower peripheral rate of speed than rollers 12, 13 and 14. The rate should be so adjusted that the film, during its passage through the drying chamber 15, is under substantially no tension and is therefore allowed to shrink freely in all directions. At the same time, of course, the roller 16 should not be so excessively slow that the film is allowed to pile up in the chamber 15. The film should be so constantly withdrawn from the chamber 15 that substantially the same amount of film remains therein at all times.

The rate of travel through the chamber 15 and the amount and temperature of the heat supplied should be such that the moisture content of the film is reduced to the required amount (preferably between 100% and 200%) in the substantially complete absence of tension. It is obvious that these factors can be appropriately controlled by one skilled in the art.

After being led over roller 16 the film travels over a series of the well known drier rollers 40, the speed of which, together with that of roller 16, is so adjusted that tension, whether normal, reduced or elevated, is applied to the film from this point down to the point at which it is completely dry. This is necessary in order to secure film of good transparency and to avoid the formation of puckers and wrinkles.

In order to test the toughness of various types of film under actual handling conditions, the following test has been devised. A sample of the film to be tested is formed into a bag, and 200 grams of dry white beans inserted therein. The bag is closed so as to leave the film tightly wrapping the beans, without loose flaps, and sealed by means of a piece of pressure sensitive regenerated cellulose adhesive tape. The bag is then repeatedly dropped from a height of two feet onto a glass plate, in an atmosphere of 35% relative humidity. The number of times that it can be dropped without breaking is a direct measure of its toughness, and may be considered, for the purpose of this specification, as the toughness factor.

The following table indicates the results of such a test, an average of 24 samples being taken in each case, on samples of ordinary regenerated cellulose film dried under tension in accordance with the prior known art, and regenerated cellulose film toughened according to this invention, each being approximately 0.001 inch thick.

Per cent glycerin	Drops before breaking	
	Untoughened film	Toughened film
0	67	125
14.3	170	248

It will be seen that film processed according to this invention is much tougher than film produced in accordance with the prior art with the same amount of softener, the improvement in all cases being at least 25%, and in most cases far in excess of this.

Films or pellicles prepared in accordance with the present invention and containing normal amounts of a softener such as glycerin exhibit greatly improved durability or toughness over similar films containing the same amounts of softener, and dried according to the prior art, as shown by the above table. Despite the fact that the glycerin content is the same, they also exhibit increased freedom from stickiness. For example, regenerated cellulose film prepared in accordance with the present invention and containing 14% glycerin will not exhibit objectionable sticking for approximately seventeen days at 80% relative humidity and one pound per

square inch pressure, whereas regenerated cellulose film prepared in the manner known heretofore, and containing the same amount of glycerin, under the same conditions will exhibit objectionable sticking in approximately two to three days.

In the practice of this invention it has also been found possible to use much lower amounts of softener than are used in the ordinary type of film, and still retain the required degree of toughness. The advantages of such a procedure are, as pointed out above, increased freedom from stickiness, less susceptibility to changes in atmospheric moisture, and greater tenacity. The cost factor in using lower quantities of glycerin is obvious.

In all cases, in addition to the above advantages, film prepared according to this invention exhibits freedom from wrinkles and puckers and no impairment of transparency.

The invention, although particularly useful for the production of regenerated cellulose sheets and films from viscose, is also applicable to the production of sheets and films from aqueous alkaline cellulosic solutions coagulable in an acid coagulating bath including, for example, regenerated cellulose produced from cuprammonium cellulose and lowly etherified or lowly esterified cellulose. The invention finds its greatest use in its application to films and sheets of gel regenerated cellulose, that is, cellulosic films which have been coagulated and/or regenerated, washed, bleached, desulfured and glycerinated but not yet dried. However, improved results can also be obtained in treating sheets and films which have been previously dried, by rewetting the same and redrying them in accordance with the present invention.

Many changes and modifications of the invention as above specifically set forth may obviously be made without departing from the nature and spirit of the invention. It is therefore to be understood that the invention is not to be limited except as set forth in the appended claims.

We claim:

1. In the method of producing non-fibrous, cellulosic sheets and films from aqueous alkaline cellulosic solutions the steps comprising coagulating said sheets and films, purifying the same, preliminarily drying the same to a point at which they contain between 100% and 200% moisture based on the weight of the cellulosic material, with substantially complete elimination of tension, and completing the drying of said films under tension.

2. In the method of toughening non-fibrous, cellulosic sheets and films cast from aqueous alkaline cellulosic solutions, the steps which comprise passing said films through a bath containing a softening agent, preliminarily drying the same to a point at which they contain between 100% and 200% moisture based on the weight of the cellulosic material, with substantially complete elimination of tension, and completing the drying of said films under tension.

3. In the method of toughening regenerated cellulose sheets and films, the steps which comprise passing said films through a bath containing a softening agent, preliminarily drying the same to a point at which they contain between 100% and 200% moisture based on the weight of regenerated cellulose, with substantially complete elimination of tension, and completing the drying of said films under tension.

4. In the method of toughening regenerated cellulose sheets and films the steps which comprise passing said films through a bath containing glycerin, preliminarily drying the same to a point at which they contain between 100% and 200% moisture based on the weight of regenerated cellulose, with substantially complete elimination of tension, and completing the drying of said films under tension.

5. In the method of producing non-fibrous, cellulosic sheets and films from aqueous alkaline cellulosic solutions, the steps comprising coagulating said sheets and films, purifying the same, preliminarily drying the same, with substantially complete elimination of tension, until the moisture content has been reduced to such an extent that further drying under tension will result in an improvement in toughness of the said sheets and films over that which will result in the absence of said preliminary drying step, said sheets and films after said preliminary drying step containing at least 100% moisture based on the weight of the cellulosic material, and completing the drying of said films under tension.

6. In the method of toughening regenerated cellulose sheets and films, the steps which comprise passing said films through a bath containing glycerin, preliminarily drying the same, with substantially complete elimination of tension, until the moisture content has been reduced to such an extent that further drying under tension will result in an improvement in toughness of the said sheets and films over that which will result in the absence of said preliminary drying step, said sheets and films after said preliminary drying step containing at least 100% moisture based on the weight of regenerated cellulose, and completing the drying of said films under tension.

7. In the method of toughening non-fibrous, cellulosic sheets and films cast from aqueous alkaline cellulosic solutions, the steps which comprise passing said films through a bath containing a softening agent, preliminarily drying the same, with substantially complete elimination of tension, until the moisture content has been reduced to such an extent that further drying under tension will result in an improvement in toughness of the said sheets and films over that which will result in the absence of said preliminary drying step, said sheets and films after said preliminary drying step containing at least 100% moisture based on the weight of the cellulosic material, and completing the drying of said films under tension.

8. The process which comprises passing continuous gel regenerated cellulose web through a drier in substantially complete absence of tension in the web, until the water content has been reduced to 200% to 100% based on the weight of the cellulose material whereby the web is permitted to shrink freely, and thereafter drying while preventing shrinkage of the web.

9. In the method of toughening regenerated cellulose sheets and films, the steps which comprise preliminarily drying wet regenerated cellulose sheets and films to a point at which they contain between 100% and 200% moisture, based on the weight of regenerated cellulose, with substantially complete elimination of tension, and completing the drying of said films under tension.

10. In the method of toughening regenerated cellulose sheets and films, the steps which comprise passing said films through a bath containing a softening agent, preliminarily drying the same, with substantially complete elimination of tension, until the moisture content has been re-

duced to such an extent that further drying under tension will result in an improvement in toughness of the said sheets and films over that which will result in the absence of said preliminary drying step, said sheets and films after said preliminary drying step containing at least 100% moisture based on the weight of regenerated cellulose, and completing the drying of said films under tension.

11. The process of claim 1 when the cellulosic sheet is a lowly etherified cellulose.

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