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DRY SPINNING PROCESS

Leo Ubbelohde, Berlin-Charlottenburg, Germany

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The present invention relates to an improved method of forming products from aqueous solutions of cellulosic material. While the invention comprehends the formation of films or sheet products of aqueous solutions of cellulosic materials, it is particularly adapted for the formation of filaments of cellulosic material such as the xanthogenate of cellulose, or viscose, or to the formation of films of cuprammonium cellulose.

Heretofore the dry spinning process has been applicable only with respect to solutions of derivatives of cellulose, but according to the present invention the dry spinning process is rendered applicable for the formation of products from aqueous solutions of cellulose such as solutions of viscose or of cuprammonium cellulose.

According to the present invention the aqueous solutions of cellulose prior to the spinning operation are heated to a temperature below that at which the cellulosic material contained in the solution will decompose and the solution while so heated is extruded into an atmosphere of a drying gas.

In carrying out the process according to the present invention an aqueous solution of viscose or other cellulosic material is prepared. The cellulose content of the solution may be normal, that is, about 8%, but in order that the viscosity of the solution will be greater than normal viscose or cuprammonium spinning solutions the solution preferably contains from 15 to 18% of cellulosic material.

After the aqueous solution of the cellulosic material is formed in the usual manner it is permitted to ripen for a period of from 4 to 5 days at a temperature of about 20° C., but the ripening may be carried out according to the known manners at higher temperatures, especially according to the continual ripening processes, which are now well known. For example, by using a somewhat obliquely mounted rotating drum which is maintained at a temperature of about 30° C. and through which the aqueous solution of alkali cellulose is passed at a desired rate, according to the length and the obliquity of the drum and its rotating speed. In such cases the drum may be provided with a double wall for receiving a heat exchange medium and suitably subdivided so that the desired temperature may be maintained at the different parts of the drum.

After the cellulosic solution has ripened for the desired length of time it is heated to a temperature preferably between about 40° C. to 50° C., although in some instances the temperature may drop as low as 30° C. or be raised to about

80° C., although the temperature of the solution should not be permitted to rise above the latter temperature because at about that temperature decomposition of cellulose xanthate or cuprammonium cellulose is liable to take place.

While the cellulosic solution is at the above stated temperature, it is extruded in a well known manner through orifices to form products of the desired shape. If filaments are to be formed the solution is extruded through the usual spinning nozzles. The material leaving the extrusion orifices passes into a stream of heated air or other gas known to be suitable for drying the extruded products. The air or gas in the cell or chamber into which the products are extruded is preferably at a temperature of about 200° C. at the place where the products enter the chamber. The temperature of the air or gas decreases as the extruded products pass through the chamber and the gases in the middle part of the chamber will be at a temperature of between about 150° C. to 180° C., while in the lower part of the chamber the temperature of the gases will be between about 110° C. to 140° C., or possibly as low as 100° C. The use of such elevated temperatures in the cell or chamber is possible due to the fact that the material leaving the extrusion orifices passes through the cell or chamber at a speed such that it does not attain a temperature, before passing out of the cell, at which decomposition of the material will take place.

The carrying out of the method of forming the extruded products as described above has been found to give products of materially better quality in that they have increased tensile strength and are of more desirable cross section. In the case of filaments the cross sections thereof are of a round or lobulated shape instead of a biscuit-like or almond-shaped form such as results from present methods of extruding viscose or cuprammonium spinning solutions. Furthermore, products produced by the above described process have improved dyeing and tensile properties. The nature, and especially the feel of fabrics made from filaments in accordance with the present invention are improved.

The air or gas may be introduced into the spinning cell or chamber into which the products are extruded by introducing it either into the upper or into the lower part of the spinning cell or chamber, so that the air or gas stream travels either in the same direction of the filament after its passage through the extrusion orifices, or in the opposite direction. Suitable

means are provided for maintaining the desired temperatures of the air or gas in the different parts of the spinning cell or chamber.

The effects described above are intensified by the preheating of the cellulosic solution as previously described. The preheating of the cellulosic solution renders it especially well fitted for transformation and shaping, besides assists the process of coagulation and the drying of the extruded products.

In the forming of filaments for textile purposes it is desirable that they be stretched before they are regenerated to cellulose. The passage of the filaments downwardly through the spinning cell or chamber will cause a certain amount of stretch to be imparted to the filaments due to their own weight, but in most instances it is desired to increase the stretch imparted to the filaments, and that may be done by a device which removes the filament from the spinning shaft. The stretch may be further augmented by filament brakes in the spinning cell or chamber or by passing the filaments between several rollers rotating at different speeds. If the filament leaves the spinning nozzle at a speed of 80 to 90 meters per minute it will leave the spinning cell or chamber at a speed of about 200 meters per minute. The filament as it leaves the spinning cell may pass through a thread guide and thereby undergo a certain friction which will give the thread a further stretch so that it will be wound up at a speed of about 200 to 220 meters per minute. The filaments upon leaving the spinning cell will be sufficiently solid and strong to permit their being wound up or being regenerated to cellulose. The extent to which the filaments are stretched may vary widely. For example, the filaments may be stretched only from about 5% to 20% of their length, or from about 50% to 1000% of their length at the time when they leave the spinning nozzle.

As described in my application Serial No. 106,141, it is often desirable to crimp or curl the formed filaments and the crimping or curling of the filaments formed in accordance with the present invention may be accomplished as set forth in my said application and may be performed either during the regeneration of the fibers to cellulose or before regeneration to cellulose, or even after the regeneration to cellulose has been performed.

It is also desirable in many instances to cut the filaments into short staples of desired length. The cutting of the filaments into staples may take place, when viscose filaments are being formed, before the regeneration to cellulose, that is, before the xanthogenate dried and formed to a fiber is transformed into hydrated cellulose.

If the crimping or curling process is performed before the filaments are regenerated to cellulose, the cutting of the fiber into staples may be performed before or after the regeneration to cellulose. If the regeneration to cellulose takes place before the cutting of the filament into staples it is advisable to leave the crimped or curled and cut staples for some time in contact with the regeneration liquid so that tensions and the like within the single staples may be compensated. For example, the filaments crimped or curled and cut into small staples of desired length fall into the regeneration liquid, such as a mineral acid bath, from which the cut fibers may be taken for further after treatment.

Further, the filament formed within the spin-

ning cell and still consisting of cellulose xanthogenate, if viscose solutions are being extruded, may pass to a coagulating bath of well known composition for use in coagulating viscose or xanthogenate, for example, the bath may consist of methyl or alkyl alcohol or of diluted organic acids, such as diluted acetic acid, or of solutions of ammonium salts or of salts of soluble sulphites, such as sodium sulphite or sodium bisulphite or the like. The last mentioned solution is advantageous because the filament consisting of cellulose xanthogenate will in the meantime be purified, that is, desulphurized. Desulphurizing substances such as sodium sulphite, sodium bisulphite, ammonium sulphide or mixtures of those substances or the corresponding sulphates, singly or mixed with each other or with the aforementioned substances may be used in aqueous solution or in addition to the before mentioned alcoholic bath. The alcoholic bath may consist of anhydrous alcohols or of alcohols mixed with water.

The coagulating bath may be inserted in the process after the crimping or curling of the filament has taken place, that is, before the filament gets to the apparatus for cutting it into short staples, or, which is preferred, the fiber may be crimped and cut into staples and then fall into the coagulating and desulphurizing bath and thereafter be regenerated to cellulose.

Substances or materials which are well known in producing common staple fibers or artificial silk may be added to the aqueous solution of the cellulosic material, the addition serving, for example, for matting the product and for giving to it a special character or for imparting to the inner structure of the fiber a special form; for example, for producing small hollow spaces in the fiber or for giving it a faveolate structure. For influencing the inner structure of the filament in this way an addition of starch may be made either in the form of a powder or of a solution of undisintegrated starch. When starch is added to the aqueous solution of the cellulosic material it may be added in the amount of from about 0.5% to 15% calculated on the cellulosic content of the solution.

Other substances known as additional substances in spinning artificial silk also may be added to the cellulosic solution. Such substances may be removed altogether or partly, so that the inner structure of the filament will show numerous hollow spaces. When adding only a small quantity of the substances mentioned before, the faveolate structure will decrease to microscopic fineness.

Sharp edged materials of a size to impart a roughened or uneven surface to the filaments may be added to the aqueous cellulosic solution. Such substances may consist of pulverized fibers of any kind or infusorial earth or other substances of a similar character. The addition of such substances is of special advantage in improving the spinning properties of the filaments produced and when added should be in an amount of from about 0.1% to 10% calculated on the cellulose content of the cellulosic solution.

The curly or crimp state of the filaments produced as described above may be rendered still more durable, which is of great importance, especially for the utility of staple fiber by adding natural or artificial resins or substances which are able to form artificial resins to the viscose and then condensing or pulverizing or hardening those substances during the forming and curling

of the filament, or after the forming and curling of the filament has been finished. The hardening (condensing or polymerizing) of the added resin-like material, may be performed either by chemical or by physical methods, or by both.

The filament, which may still consist of viscose or of cuprammonium, or may even already be transformed into regenerated cellulose, is passed through a pair of fluted rollers which render it curly, as described in my aforesaid application. The fluted rollers may be maintained at room temperature or may be heated to a temperature of about 30° C. or 60° C. or even higher, for example, to temperatures between 70° C. and 150° C. The rollers may be mounted within a so-called spinning bath to promote the coagulation of the viscose or its regeneration to cellulose, the temperature of the rollers then of course not being above the boiling point of the bath used and normally being below that temperature, or the rollers may only be sprinkled with the liquid which normally would form the spinning bath. In performing the process in this manner, the filament, while entering between the fluted or the like rollers, will undergo a stretching during the curling and coagulating, or during the process of regenerating the cellulose. Of course, the regenerating of the cellulose may be performed by gases such as carbon dioxide, sulphur dioxide or the like which also may be heated, as well as the rollers themselves.

The curling or crimping of the filament and the regeneration of the cellulose may take place successively, although in order to obtain a particular effect, it is preferred that the curling or crimping of the filaments and the regenerating of the cellulose take place simultaneously.

It is to be understood that although reference is frequently herein made to solutions of viscose, cuprammonium may equally well be used; both substances having normally a high concentration of cellulose which may rise to 30% by weight or even more, although normally lower concentrations between about 10% and 25% of cellulose will be used.

According to one example of carrying out the present process a viscose solution containing 16% of cellulose was prepared from a so-called artificial silk pulp. The alkali cellulose made in the usual manner was ripened for a period of three days at a temperature of about 18° C. The alkali cellulose was treated in the usual manner with carbon bisulphide and the xanthogenate formed was dissolved in such a quantity of pure water that 16% of cellulose was found in the resulting solution. The viscosity of the solution thus formed was rather high and a steel ball of a diameter of $\frac{1}{8}$ of an inch took about 60 seconds to sink below the surface so that it was no longer visible. The viscose was then ripened for five days at a temperature of about 20° C. and was then filtered and de-aerated, and after being raised to a temperature of about 40° C. was passed through a spinning nozzle under a pressure of 8 atmospheres. From the spinning nozzle the extruded filaments passed into a spinning cell in which the temperature in the upper part was about 200° C. and in the middle part between 150° C. and 180° C. and in the lower part between 110° C. and 140° C. The filaments entered the spinning nozzle at a speed of 80 to 90 meters per minute and left the spinning cell at a speed of between 200 and 220 meters per minute. The titer of a single filament was between 4 and 5

deniers, while the spun rope had a denier of about 200. The temperature in the upper part of the spinning cell was maintained at a temperature of about 200° C. by a stream of air blown into the upper part thereof at a speed of from about 0.8 to 1 meter per second so that the speed of the air stream was slower than the spinning speed of the filament. The filaments were stretched, curled and crimped and regenerated as described above.

This application is a continuation-in-part of my application for Dry-spinning process, Serial No. 42,053, filed September 25, 1935.

I claim:

1. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between approximately 30° C. and 80° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the shape of the final product and passing the same into a current of a gaseous drying atmosphere, and regenerating the formed product to cellulose.
2. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between approximately 40° C. and 50° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the shape of the final product and passing the same into a current of a gaseous drying atmosphere, and regenerating the formed product to cellulose.
3. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between approximately 30° C. and 80° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the shape of the final product and passing the same into a current of a gaseous drying atmosphere, stretching the product, and regenerating the formed product to cellulose.
4. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between approximately 40° C. and 50° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the shape of the final product and passing the same into a current of a gaseous drying atmosphere, stretching the product, and regenerating the formed product to cellulose.
5. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature of between about 30° C. and 80° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the shape of the final product, passing said product into a current of a gaseous drying atmosphere decreasing in temperature from about 200° C. to about 100° C., removing said product from said gaseous drying atmosphere before it attains a temperature at which the material of which it is formed will be decomposed stretching the product, and regenerating the formed product to cellulose.
6. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature of between about 40° C. and 50° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the

shape of the final product, passing said product into a current of a gaseous drying atmosphere decreasing in temperature from about 200° C. to about 100° C., removing said product from said gaseous drying atmosphere before it attains a temperature at which the material of which it is formed will be decomposed stretching the product, and regenerating the formed product to cellulose.

7. A process for dry spinning artificial filaments of aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between about 30° C. to 80° C., thereafter, and while the solution is at such a temperature pressing the same through fine orifices into a current of a drying atmosphere above 100° C., removing the resulting filaments from said drying atmosphere before they attain a temperature at which the material of which they are formed will decompose, and regenerating the formed filaments to cellulose.

8. A process for dry spinning artificial filaments of aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between about 30° C. to 80° C., thereafter, and while the solution is at such a temperature pressing the same through fine orifices into a current of a drying atmosphere above 100° C., removing the resulting filaments from said drying atmosphere before they attain a temperature at which the material of which they are formed will decompose, stretching the formed filaments, and regenerating the formed filaments to cellulose.

9. A process for dry spinning artificial filaments of aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between about 40° C. to 50° C., thereafter, and while the solution is at such a temperature pressing the same through fine orifices into a current of a drying atmosphere above 100° C., removing the resulting filaments from said drying atmosphere before they attain a temperature at which the material of which they are formed will decompose, and regenerating the formed filaments to cellulose.

10. A process for dry spinning artificial filaments of aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between about 40° C. to 50° C., thereafter, and while the solution is at such a temperature pressing the same through fine orifices into a current of a drying atmosphere above 100° C., removing the resulting filaments from said drying atmosphere before they attain a temperature at which the material of which they are formed will decompose, stretching the formed filaments, and regenerating the formed filaments to cellulose.

11. A process for dry spinning artificial filaments of viscose which comprises heating a solution of viscose to a temperature between about 30° C. and 80° C., thereafter, and while the viscose solution is at such temperature, pressing it through fine orifices into a current of a drying atmosphere gradually decreasing in temperature from about 200° C. to about 110° C., removing the resulting filaments from said drying atmosphere before they attain a temperature at which the material of which they are formed will decompose stretching the filaments, and regenerating the filaments to cellulose.

12. A process for dry spinning artificial filaments of viscose which comprises heating a solution of viscose to a temperature between about

40° C. and 50° C., thereafter, and while the viscose solution is at such temperature, pressing it through fine orifices into a current of a drying atmosphere gradually decreasing in temperature from about 200° C. to about 110° C., removing the resulting filaments from said drying atmosphere before they attain a temperature at which the material of which they are formed will decompose stretching the filaments, and regenerating the filaments to cellulose.

13. A process for forming artificial filaments of aqueous solutions of cellulosic material which comprises forming an aqueous solution containing from 15% to 18% of cellulosic material, ripening such solution for a period from about four days to five days at a temperature from about 20° C. to about 30° C., heating the ripened solution of cellulosic material to a temperature of between about 30° C. and 80° C., thereafter, and while the solution is at such temperature, pressing it through fine orifices into a current of a drying atmosphere, stretching the resulting filaments, and regenerating the formed filaments to cellulose.

14. A process for forming artificial filaments of aqueous solutions of cellulosic material which comprises forming an aqueous solution containing from 15% to 18% of cellulosic material, ripening such solution for a period from about four days to five days at a temperature from about 20° C. to about 30° C., heating the ripened solution of cellulosic material to a temperature of between about 40° C. and about 50° C., thereafter, and while the solution is at such temperature, pressing it through fine orifices into a current of a drying atmosphere, stretching the resulting filaments, and regenerating the formed filaments to cellulose.

15. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between approximately 30° C. and 80° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the shape of the final product and passing the same into a current of a gaseous drying atmosphere, and regenerating the formed product to cellulose.

16. A process for forming products from aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between approximately 40° C. and 50° C., thereafter, and while the solution is at such temperature, forming the solution into substantially the shape of the final product and passing the same into a current of a gaseous drying atmosphere, and regenerating the formed product to cellulose.

17. A process for dry spinning artificial filaments of aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between about 30° C. to 80° C., thereafter, and while the solution is at such a temperature pressing the same through fine orifices into a current of a drying atmosphere, and regenerating the formed filaments to cellulose.

18. A process for dry spinning artificial filaments of aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between about 40° C. to 50° C., thereafter, and while the solution is at such a temperature pressing the same through fine orifices into a current of a drying atmosphere,

and regenerating the formed filaments to cellulose.

19. A process for dry spinning artificial filaments of aqueous solutions of cellulosic material which comprises heating a solution of such material to a temperature between about 40° C. to 50° C., thereafter, and while the solution is at such a temperature pressing the same through fine orifices into a current of a drying atmosphere, stretching the formed filaments, and regenerating the formed filaments to cellulose.

20. A process for forming artificial filaments which comprises heating an aqueous solution of cellulosic material to a temperature between about 40° C. to 50° C., pressing said solution while at said temperature through fine orifices into a current of a drying atmosphere gradually decreasing in temperature from about 200° C. to 110° C., stretching the resulting filaments, cutting said filaments into short staples, and regenerating the short staples to cellulose.

21. A process for forming artificial filaments which comprises forming an aqueous solution of cellulosic material, containing starch in a quantity of from about 0.5% to 15% calculated on the cellulose content of the solution, heating said solution to a temperature between about 40° C. to 50° C., passing said solution while heat-

ed at such temperature through fine orifices into a current of a drying atmosphere, stretching the resulting filaments, and regenerating the filaments to cellulose.

22. A process for forming artificial filaments which comprises forming an aqueous solution of cellulosic material containing a resin dissolved therein, heating said solution to a temperature between about 40° C. to about 50° C., pressing said solution while at said temperature through fine orifices into a current of a drying atmosphere, stretching the resulting filaments, and regenerating the filaments to cellulose and hardening the resin contained therein.

23. A process for forming artificial filaments which comprises forming an aqueous solution of cellulosic material containing therein a dissolved natural resin, heating said solution to a temperature between about 30° C. to 80° C., pressing said solution through fine orifices into a current of a drying atmosphere, stretching the resulting filaments, curling or crimping said filaments, cutting said curled or crimped filaments into staples of desired length, regenerating said staples to cellulose, and hardening the added natural resin.

LEO UBBELOHDE.