

UNITED STATES PATENT OFFICE

1,996,102

PRODUCTION OF ARTIFICIAL PRODUCTS BY
DRY SPINNING

Henry Dreyfus, London, England

No Drawing. Application May 5, 1931, Serial No.
535,284. In Great Britain July 4, 1930

16 Claims. (Cl. 18—54)

This invention is a continuation in part of the inventions of my prior U. S. applications S. Nos. 328,305 filed 24th December, 1928 and 444,621 filed 15th April, 1930 and relates to the manufacture of artificial filaments, threads, ribbons, films or other products from solutions containing cellulose acetate or other cellulose esters or ethers by the dry spinning method.

My said U. S. application S. No. 328,305 describes the production of improved artificial filaments, threads, or like products by the dry spinning of solutions containing cellulose esters or ethers in a solvent medium containing a volatile substance or substances, a non-solvent liquid or liquids of higher boiling point than the volatile substance or substances and a solvent or solvents of higher boiling point than the non-solvent liquid or liquids, the higher boiling solvent or solvents being preferably employed in a total quantity not exceeding 30% of the weight of the cellulose ester or ether. By this means it was found possible to obtain products of substantially increased extensibility or elasticity, and in particular low filament deniers are obtainable.

My said U. S. application S. No. 444,621 describes the production of products by dry spinning solutions of cellulose esters or ethers in a mixture comprising a volatile non-solvent medium (comprising a volatile solvent and a higher boiling non-solvent) and a higher boiling solvent or solvents in sufficient quantity to confer solvent properties upon the whole medium.

I have now found that valuable results may be obtained by replacing or partly replacing the non-solvent liquid in the spinning solution by vapours of such non-solvent liquid in the evaporative medium, and accordingly the present invention comprises a process for the production of artificial filaments, threads, ribbons, films or other products from solutions of cellulose acetate or other cellulose esters or ethers in a solvent mixture containing a volatile substance or substances and a higher boiling solvent or solvents with or without non-solvent liquids into an evaporative medium, for example air, containing vapours of a non-solvent liquid, for example water, benzene, xylene, methyl, ethyl or propyl alcohol or mixtures thereof.

The specifications referred to above contain a number of examples of suitable volatile substances for making up the spinning solution and of higher boiling solvents therefor. Diethyl phthalate, ethyl lactate, lactic acid, dioxane and diethyl tartrate may be instanced as further examples of suitable higher boiling solvents.

As already indicated, in addition to the vapours of a non-solvent liquid being present in the evaporative medium such a non-solvent medium may also be present in the spinning solution itself. Furthermore the evaporative medium may include vapours of a solvent for the cellulose derivative, for example vapours of acetone. The presence of these solvent vapours, in addition to those produced from the filaments themselves, is also of value in producing very fine filament deniers.

It is preferred that the proportion of higher boiling solvent be not greater than about 50 to 100% of the weight of the cellulose acetate or other ester or ether and preferably not greater than about 30% thereof. Again if a non-solvent liquid be included in the spinning solution its proportion should not be sufficient to approach the stage of precipitation or incipient precipitation before spinning and is preferably not sufficient to produce a precipitated product during the actual spinning.

The temperature and rate of flow of the air or other evaporative medium may be adjusted to obtain any desired type or form of filament (solid, hollow etc.) or cross-section of filament. The size of the jet holes may be adjusted in accordance with the spinning requirements. For example, when running at relatively high spinning speeds, smaller jet holes, e. g. of .08 to .05 mm. or even down to .01 mm. diameter, may be used than with moderate or low spinning speeds. Again, with the higher speeds, higher concentrations of solvent vapour in the evaporative medium are permissible than with lower speeds of spinning to obtain products having similar physical characteristics, thus with the higher spinning speeds one may employ a smaller flow of evaporative medium. Furthermore these factors or other conditions may be varied in accordance with the denier being produced.

The invention includes processes in which the volatile constituents of the solvent mixture are substantially eliminated from the filaments or other products in one operation, the spinning process being carried out, if desired, with a suitable tension applied direct from the nozzles. The products which still contain higher boiling solvents may be further drawn out, as for example between two rollers or other devices rotating at different peripheral speeds. The invention further includes processes in which such additional tension or stretch is imparted to the filaments or other products before the complete elimination of the volatile constituents of the solvent mixture. For instance the filaments

or other products may be subjected to a dry spinning operation to eliminate a substantial proportion of the volatile constituents of the solvent mixture, the said operation being conducted under tension or not as desired, and the filaments or other products, still in a plastic state due to their content of higher boiling solvents and lower boiling constituents, may be stretched or further stretched as previously described. The stretch may be restricted to the desired portion of the filaments or other products by means of a roller, guide, or tension device which offers resistance to their passage as described for example in U. S. application S. No. 476,334 filed 19th August, 1930. The following examples illustrate the invention but are not to be considered as limiting it in any way:—

Example 1

A spinning solution is made up containing 27 parts of cellulose acetate dissolved in a mixture of 65 parts of acetone and 8 parts of ethyl lactate or diacetone alcohol. The solution is spun into an evaporative medium maintained at a temperature of 70 to 80° C. and having a moisture content corresponding to saturation at a temperature 45 to 55° C. The filaments may either be subjected to tension direct from the nozzle, or as described in U. S. application S. No. 476,334 such tension may be wholly or in part confined to the later stages of the spinning. After substantial elimination of the volatile solvent the filaments may be further stretched, as for example between rollers rotating at different peripheral speeds. The filaments obtained have a high extensibility and a medium lustre. Any ethyl lactate or diacetone alcohol remaining in the filaments may, if desired, be removed by any suitable means, as for example by washing.

Example 2

The spinning solution employed in Example 1 is spun into a spinning cell through which passes a counter-current of air regulated to approximately 22 cubic feet per minute. Into the cell at a distance of 6 to 8 inches from the spinning nozzle is injected alcohol vapour, the rate of introduction being about 100 to 120 grams per minute.

Example 3

A spinning solution is made up containing 25 parts of cellulose acetate, 7 parts of water 8 parts of ethyl lactate and 60 parts of acetone, and is spun as described in Example 1 or 2.

Example 4

A spinning solution is made up containing 25 parts of cellulose acetate, 50 parts of acetone, 18 parts of alcohol and 7 parts of ethyl lactate or diacetone alcohol, and is spun as described in Example 1 or 2.

The invention is particularly valuable in the case of obtaining products from solutions of cellulose acetate and especially acetone-soluble cellulose acetate but may also be applied to the manufacture of products from solutions of other cellulose esters, for example cellulose formate, propionate or butyrate or of cellulose ethers, for instance methyl, ethyl or benzyl cellulose or other cellulose ethers.

What I claim and desire to secure by Letters Patent is:—

1. Process for the production of artificial ma-

terials comprising dry spinning a solution of a cellulose derivative in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose derivative.

2. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose acetate.

3. Process for the production of artificial materials comprising dry spinning a solution of a cellulose derivative in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose derivative and stretching the product formed.

4. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose acetate and stretching the product formed.

5. Process according to claim 2, wherein the higher boiling solvent is present in a proportion not exceeding 30% of the weight of the cellulose acetate.

6. Process for the production of artificial materials comprising dry spinning a solution of a cellulose derivative in a mixture comprising a volatile solvent, a higher boiling solvent and a non-solvent for the cellulose derivative into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose derivative.

7. Process for the production of artificial materials comprising dry spinning a solution of a cellulose derivative in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose derivative and is miscible with said mixture.

8. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose acetate and is miscible with said mixture.

9. Process for the production of artificial materials comprising dry spinning a solution of a cellulose derivative in a mixture comprising a volatile solvent, a higher boiling solvent and a non-solvent for the cellulose derivative into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose derivative and is miscible with said mixture.

10. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising acetone and a higher boiling solvent into an evaporative medium charged with the vapors of a substance which is a non-solvent for the cellulose derivative and is miscible with said mixture.

11. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with water vapor.

12. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising a volatile solvent and a higher boiling solvent into an evaporative medium charged with alcohol vapor.

13. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising acetone and ethyl lactate into an evaporative medium charged with water vapor.

14. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising acetone

and diacetone alcohol into an evaporative medium charged with water vapor.

15. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising acetone and ethyl lactate into an evaporative medium charged with alcohol vapor.

16. Process for the production of artificial materials comprising dry spinning a solution of cellulose acetate in a mixture comprising acetone and diacetone alcohol into an evaporative medium charged with alcohol vapor.

HENRY DREYFUS.