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VISCOSE SPINNING PROCESS

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The invention relates to a process for the production of fibres, filaments, bristles, strips, foils and like products from viscose solutions.

It has long been known to extrude viscose solutions into coagulating baths containing salts having an alkaline reaction to form shaped products. A process for the coagulation of viscose solutions in film form on a solid support with the aid of caustic alkali solutions has been proposed but owing to the slowness of the coagulation, this process could not be commercialised. It proved impossible to produce filaments by the same technique, though it was subsequently found practicable to use coagulating baths containing alkali carbonate.

The process according to the present invention makes it possible, using caustic alkali coagulating baths, to produce fibres, filaments and the like, as well as foils, from viscose solutions without the necessity of employing supports for the products during their formation.

According to the present invention a process for the production of fibres, filaments, bristles, strips, foils and like products comprises extruding, through a suitably shaped orifice, a viscose solution having a degree of xanthation of 0.25 to 0.45, and of which the cellulose has a degree of polymerization of at least 400, into an aqueous coagulating bath containing from 50% to 75% of caustic alkali at a temperature of 50–100° C., decomposing the xanthate products by the action of heat, washing them free from caustic alkali, stretching them and drying them. The "degree of xanthation" is the molecular ratio of —CSS— units to glucose $C_6H_{10}O_5$ units.

Preferably, according to the invention, the caustic alkali concentration of the viscose solution should be relatively high and preferably from 8.5% to 13%. The cellulose concentration in the viscose solution is preferably between 6% and 11%.

The products, consisting of partially decomposed cellulose xanthate, leave the coagulating bath in a highly swelled condition. They are immediately subjected to a heat treatment to complete the decomposition of the xanthate. This heat treatment is preferably effected by the application of steam at 100° to 180° C. until the xanthate is decomposed. With a take-up speed of 50 m./min., the length of the path of treatment is, for example, from 6 to 16 m.

Following this heat treatment, the articles are washed to free them from caustic alkali. Since aqueous solutions in which the concentration of caustic alkali solution is from 4% to 8% have the greatest swelling action on cellulose hydrate, this dangerous range of concentration must be passed through as rapidly as possible. It has therefore proved expedient to carry out this treatment in from two to three stages. Thus, for example, in a first stage the caustic alkali content of the products may be reduced by treatment with 3–5% caustic alkali solution (which may contain salts such as sodium sulphate or sodium sulphide in order to lower the swelling action) to such an extent that the products can thereafter be completely freed from caustic alkali by washing them with water. The first treatment may be carried out at a temperature of from 70° to 100° C. and the remaining caustic alkali completely removed by further treatments with hot water. The liquids employed in the treatments may be guided in counter-current to the travel of the products. In order to accelerate the treatments, it has proved desirable to employ devices which, while they feed the products forward yet maintain them in the treatment bath for an appreciable time. Thus filaments may make several turns round a feed roller in the bath, e. g. a cage-like drum or crossed double-roller, before being fed forward from the roller. The coagulating bath and the baths employed for the subsequent treatments may be regenerated if desired.

The overall consumption of caustic alkali solution and sulphuric acid hitherto usual in the viscose industry is considerably reduced by the process of the present invention, because the caustic alkali present in the viscose solution enters the coagulating bath as such and requires no neutralisation.

The stretching of the products is preferably effected at least to the major extent before they reach a condition of maximum swelling. They may, however, be further stretched at a later stage. The first stretching can conveniently be performed between the extrusion orifice and the stage of heat treatment.

After washing to remove the caustic alkali, and before drying, the articles may be cleaned and softened. Any other treatments usual in the rayon industry may be applied at this point.

The following example will serve to illustrate this invention but is not to be regarded as limiting it in any way:

Example

A viscose solution containing 9% of alpha-cellulose and 8.5% of NaOH, having a degree of xanthation of 0.4, a viscosity of 635 poises at 20° C. and formed from cellulose having a degree of polymerisation of 450, was spun through a spinneret having 60 holes each of a diameter of 0.07 mm. into a coagulating bath consisting of a 71% aqueous solution of caustic alkali (NaOH) at 92° C. The filaments formed were led out of the coagulating bath and a tension of 18 g. was imparted thereto by means of a brake-type stretching device. The filaments were then guided in several turns round a crossed double roller, a length of filament of 7.2 m. being carried by the roller, and treated with steam at 100° C. The filaments were then led with a stretch of 40% under a tension of 70 g., to a second double roller system, at which they were sprinkled with 3% caustic alkali (NaOH) solution at a temperature of 92° C. over a distance of 1.6 m. The supply of bath solution was so regulated that the escaping caustic alkali solution had a concentration of 4.2% and the filaments still contained from 150% to 300% of NaOH, calculated on the cellulose. The products were then washed free from caustic alkali by treatment with water at 90° C. in two stages. A stretch of from 4% to 6% was applied to the products in each interval between these three treatments for the removal of caustic alkali. The filaments were then collected in a spinning centrifuge at a take-up rate of 55 m./min. The cakes obtained were softened and dried in the usual manner. The cellulose of these filaments had a degree of polymerisation of 450. Thus no decomposition of the cellulose occurred at any time during the spinning process. The air-dried rayon having a count of 300 deniers had a strength of 1.9 g./den. and an elongation of 14%. The filaments had a substantially non-serrated, circular cross-section and were not stuck together. 1 kg. of the dry filament contained 55 mg. of elementary sulphur.

I claim:

1. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of heat, washing them free from caustic alkali, stretching and drying them.

2. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 8.5 to 13% of caustic alkali, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of heat, washing them free from caustic alkali, stretching and drying them.

3. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 6 to 11% of alpha cellulose, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulation bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of heat, washing them free from caustic alkali, stretching and drying them.

4. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 6 to 11% of alpha cellulose and 8.5 to 13% of caustic alkali, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of heat, washing them free from caustic alkali, stretching and drying them.

5. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 6 to 11% of alpha cellulose and 8.5 to 13% of caustic alklai, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of steam, washing them free from caustic alkali, stretching and drying them.

6. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 6 to 11% of alpha cellulose and 8.5 to 13% of caustic alkali, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of steam, washing them free from caustic alkali, first by treatment with dilute caustic alkali solution, then by treatment with hot water.

7. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscous solution containing 6 to 11% of alpha cellulose and 8.5 to 13% of caustic alkali, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of steam, washing them free from caustic alkali, first by treatment with dilute caustic alkali solution, containing a substance reducing the swelling of the products, then by treatment with hot water.

8. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 6 to 11% of alpha cellulose and 8.5 to 13% of caustic alkali, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., decomposing the xanthate products by the action of steam, washing them free from caustic alkali, first by treatment with dilute caustic alkali solution, then by treatment with hot water, the said caustic alkali or the said water being carried to flow in counter-current to the travel of the products.

9. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 6 to 11% of alpha cellulose and 8.5 to 13% of caustic alkali, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., stretching the products, thereafter decomposing the products by the action of heat, thereafter washing the products free of caustic alkali and drying them.

10. Process for the production of fibres, filaments, bristles, strips and foils which comprises extruding through a suitably shaped orifice, a viscose solution containing 6 to 11% of alpha cellulose and 8.5 to 13% of caustic alkali, having a degree of xanthation of 0.25 to 0.45 and of which the cellulose has a degree of polymerisation of at least 400 into an aqueous coagulating bath containing from 50 to 75% of caustic alkali, at a temperature of 50–100° C., stretching the products, thereafter decomposing the products by heating them, thereafter washing the products free of caustic alkali first by treatment with dilute caustic alkali solution and then by treatment with hot water, stretching the products between such washing stages and drying the products.

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