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MANUFACTURE OF ARTIFICIAL FILAMENTS, THREADS, AND FILMS.

No Drawing. Application filed December 20, 1921. Serial No. 523,758.

To all whom it may concern:

Be it known that we, CHARLES WILFRED PALMER and WILLIAM ALEXANDER DICKIE, subjects of the King of Great Britain, both of Spondon, near Derby, England, have invented certain new and useful Improvements Relating to the Manufacture of Artificial Filaments, Threads, and Films, of which the following is a specification.

It has been proposed to manufacture artificial filaments and films from solutions of cellulose acetate in organic solvents that are soluble in or miscible with water, by squirting or extruding the cellulose acetate solutions into aqueous coagulating baths.

Hitherto it has not been possible to obtain strong and elastic filaments or films by such methods, owing to the fact that the cellulose acetate is not coagulated by water in the form of a structurally continuous gel, but with a discontinuous granular or reticulated internal structure, so that the resulting threads or films are lacking in strength and elasticity.

We have found that ammonium thiocyanate and other thiocyanates, such for example as those of sodium, potassium and calcium, are capable, owing to a latent solvent power which they have for cellulose acetate, of exerting a swelling and coagulation controlling action on the coagulating filaments or films and thereby tending to preserve continuity of structure and to prevent the too complete shrinkage of the cellulose acetate which ordinarily occurs as the result of the removal of the organic solvent by the aqueous coagulating bath and causes precipitation of the cellulose acetate with a discontinuous structure.

According to the present invention, in order to obviate the difficulty before referred to, we add ammonium thiocyanate or other thiocyanates to the coagulating baths, or we may incorporate the same with the cellulose acetate solutions to be spun or formed into filament or films.

By means of the invention we have found that it is possible to obtain strong, elastic, translucent and lustrous filaments or films.

The invention applies in particular to the manufacture of artificial filaments or films from acetone-soluble cellulose acetates, but may also be employed with other acetates of cellulose.

In practising the invention the thiocyanates should not be employed in too great quantity or concentration, as otherwise they could exert too great a solvent power and prevent coagulation of the cellulose acetate. On the other hand they should be in sufficient quantity or concentration to exert the required modifying influence on the shrinking action of the water.

In carrying out the invention, solutions of cellulose acetate in any organic solvents miscible with water (both hereinafter in the claiming clauses included in the term miscible with water) or soluble in water may be employed, for example acetone, or acetone-alcohol mixtures, with or without suitable plastifying agents, as for instance benzyl alcohol.

The manner in which the invention may be carried into effect is illustrated by the following example, it being understood that this is given only by way of illustration and is in no way limitative.

A solution of 16 parts of cellulose acetate in 84 parts of acetone is forced in the known way through the capillary orifices of squirting nozzles into a solution of ammonium thiocyanate in water, said solution containing about 115 to 120 grams of ammonium thiocyanate per litre of water. The coagulated filaments are carried through the bath under a guide and are wound up continuously in any known way. The thiocyanate carried forward in the thread is removed by washing, and the purified translucent threads of cellulose acetate are dried. The filaments issuing from the squirting nozzle are endowed with considerable strength, and the combined action of the water and the thiocyanate swelling agent are such that the cellulose acetate solution may be drawn out to filaments of great tenuity from orifices of relatively large diameter. In this way it is easily possible to obtain artificial silk filaments of very low denier, for example as fine as 1 or 2 or less denier, that is to say as fine as natural silk.

It is understood that the limits of concentration of the thiocyanate salt may be varied to suit desired conditions.

Mixtures of the thiocyanates may likewise be employed as before mentioned, in the coagulating bath. For instance, mixtures of any two or more of the thiocyanates of ammonium, sodium, potassium and calcium may be employed in about the same total

proportions as above mentioned, say about 115 to 120 grams total thiocyanate per litre of water, but the limits of concentration may be varied as above mentioned. If a plasticising agent is employed, the solution might contain 16 parts of cellulose acetate dissolved in 80 parts of acetone containing about 4 parts of benzyl alcohol as plasticising agent.

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

1. In a process for the manufacture of filaments, threads or films, extruding cellulose acetate in solution into an aqueous coagulating bath containing thiocyanate in solution.

2. In a process for the manufacture of filaments, threads or films, extruding a solution comprising cellulose acetate and an organic solvent thereof miscible with water, into an aqueous coagulating bath containing thiocyanate in solution.

3. In a process for the manufacture of filaments, threads or films, extruding a solution comprising cellulose acetate and an organic solvent thereof miscible with water, into an aqueous coagulating bath containing ammonium thiocyanate in solution.

4. In a process for the manufacture of filaments, threads or films, extruding a solution comprising cellulose acetate and acetone into an aqueous coagulating bath containing ammonium thiocyanate in solution.

In testimony whereof we have hereunto subscribed our names.

CHARLES WILFRED PALMER.
WILLIAM ALEXANDER DICKIE.

Witnesses:
THOS. H. COOK,
A. A. GUNNING.