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PRODUCTION OF ARTIFICIAL MATERIALS

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This invention relates to the production of artificial filaments, yarns, threads, ribbons, films or other materials from solutions of cellulose acetate, formate, propionate, butyrate or other carboxylic acid esters or other esters of cellulose.

In the past considerable research has been made with a view to discovering processes which are capable of yielding uniformly saponified cellulose ester materials. We have now found that very uniformly saponified materials may be obtained by carrying out the saponification continuously with the production of the filaments, ribbons, threads or the like, for example continuously with their production by wet or dry spinning processes.

Accordingly, therefore, the present invention comprises a combined process for the production of filaments, yarns, threads, ribbons, films or other materials from solutions of cellulose esters by spinning the solutions, for example by wet or dry spinning processes, or otherwise forming the materials therefrom and applying a saponifying treatment continuously with the production of the materials.

Preferably the spinning is carried out in such a manner that the filaments, threads or other materials are still in a relatively swollen condition or contain substantial quantities of solvent when subjected to the saponifying treatment. By this means a very high degree indeed of uniformity in the saponification may be obtained. In the case of wet spinning the desired swollen condition of the filaments or other materials or the desired content of solvent may be achieved by spinning the solutions of cellulose esters into coagulating baths containing solvents or swelling agents for the cellulose ester and/or by carrying the coagulated filaments or other products into a subsequent treatment bath containing such solvents or swelling agents. Alternatively or in addition solvents or swelling agents may be incorporated in the spinning solution of the cellulose ester. In all cases, whether the solvent or swelling agent be incorporated in the spinning solution, in the coagulating bath, or in a subsequent treatment bath, high boiling solvents or swelling agents are preferable. Processes for carrying out such wet spinning operations are described in our prior U. S. Patent Nos. 1,465,994 and 1,467,493, U. S. application S. No. 402,785 dated 26th October, 1929, and British application No. 1814/29 dated 18th January, 1929. In the case of dry spinning, to obtain the very high degree of uniformity of saponification, the filaments may be subjected to the saponification treatment while still retaining substantial quantities of solvent. The retention of solvent may be facilitated by incorporating a relatively high boiling solvent or swelling agent in the spinning solution.

The filaments, threads and the like so treated, while retaining their strength, have a subdued lustre and are capable of yielding cross-dyed effects, for example with substantive dyestuffs, when used in combination with the normal unsaponified filaments, threads etc. As stated above, owing to the fact that the filaments, threads and the like undergo the saponification treatment in a swollen condition, they are very uniformly saponified. Furthermore, the process obviously results in increased efficiency and cheapness as compared with processes in which the saponification is carried out upon the finished threads or filaments.

The saponifying treatment may be effected by means of any suitable saponifying agents, for example caustic alkalies or ammonia in aqueous or alcoholic solution. In the case of nitrocellulose any suitable so-called de-nitrating agent may be employed. In the case of organic esters of cellulose the conditions of saponification are preferably such as to produce only a partial or superficial saponification.

The following examples illustrate the invention but are not to be considered as in any way limiting it.

Example 1.

A 25% solution of cellulose acetate in acetone is extruded through suitable spinning nozzles into a coagulating bath containing 60 parts by volume of diacetone alcohol to 40 parts of water. The swollen filaments are carried through a guide to a roller driven at a suitable peripheral speed to draw out the filaments to the required denier, e. g. to a denier as low as 0.75, and the associated filaments are passed directly into a trough containing a 2½% solution of caustic alkali in water or alcohol, and the yarn is thereafter reeled and washed or otherwise treated as desired.

Similarly the above spinning solution may be used in conjunction with a coagulating bath containing 50 parts of diacetone alcohol to 50 parts of rectified spirit or containing a 45% solution of ethyl lactate or containing an aqueous solution of 30% of diacetone alcohol and 15% of acetone or again a 25% solution of cellulose acetate in a solvent mixture consisting of 25 parts of diacetone alcohol to 75 parts of acetone may

be extruded into a bath containing a 40% aqueous solution of diacetone alcohol, the filaments being in all cases passed directly into aqueous or alcoholic caustic soda.

Example 2

Cellulose acetate yarn spun by any known method of dry or evaporative spinning, e. g. using the various forms of apparatus described in U. S. Patent No. 1,601,125, U. S. application S. No. 615,682 dated 29th January, 1923, U. S. Patent No. 1,541,104, and U. S. application S. No. 236,448 dated 29th November, 1927, or U. S. applications S. Nos. 311,213–4 dated 8th October, 1928, is passed immediately after leaving the hot spinning cell or like apparatus, and while containing solvent, over a wick, rotating roller or other device supplied with a 5% solution of caustic soda in water or methyl or ethyl alcohol, the yarn being then collected on bobbins or in any other desired manner. An intermediate or subsequent washing may if desired be applied, but it is not essential. If no washing is effected any lubricant applied to the yarn for facilitating subsequent operations should be inert towards the saponifying agent and should preferably be applied subsequent to the saponifying treatment.

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

1. Process for the manufacture of artificial materials comprising organic esters of cellulose, comprising as a continuous operation forming the products by a spinning process from a solution of the organic ester of cellulose in a solvent, and subjecting them while still containing solvent to a saponifying treatment.

2. Process for the manufacture of artificial materials comprising cellulose acetate, comprising as a continuous operation forming the products by a spinning process from a solution of the cellulose acetate in a solvent, and subjecting them while still containing solvent to a saponifying treatment.

3. Process for the manufacture of artificial materials comprising organic esters of cellulose, comprising as a continuous operation forming the products by a spinning process from a solution of the organic ester of cellulose in a solvent and subjecting them while still containing solvent to a treatment resulting in partial saponification.

4. Process for the manufacture of artificial materials comprising organic esters of cellulose, comprising as a continuous operation forming the products by a spinning process from a solution of the organic ester of cellulose in a solvent and subjecting them while still containing solvent to a treatment resulting in superficial saponification.

5. Process for the manufacture of artificial materials comprising organic esters of cellulose, comprising as a continuous operation forming the products from a solution of the organic ester of cellulose in a solvent by a wet spinning process and subjecting them while still containing solvent to a saponifying treatment.

6. Process for the manufacture of artificial materials comprising cellulose acetate, comprising as a continuous operation forming the products from a solution of the cellulose acetate in a solvent by a wet spinning process and subjecting them while still containing solvent to a saponifying treatment.

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