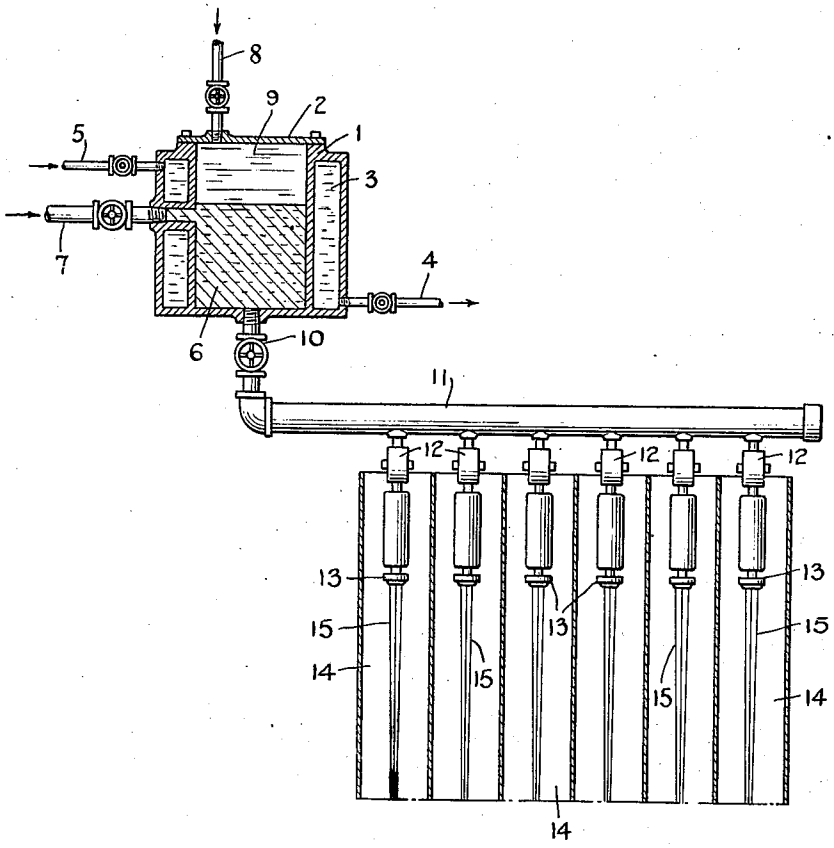
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PREPARATION OF ARTIFICIAL FILAMENTS OR THREADS

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PREPARATION OF ARTIFICIAL FILAMENTS OR THREADS

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This invention relates to the production of filaments or threads from organic esters of cellulose, and relates more particularly to the preparation of such materials by the extrusion of organic esters of cellulose while in molten state.

An object of our invention is to provide a method of preparing filaments or threads from organic esters of cellulose which is simpler and more expeditious than those formerly used.

A further object of our invention is to prepare filaments or threads that are of greater tensile strength and of improved textile properties as compared with filaments or threads produced from organic esters of cellulose by prior processes.

Other objects of our invention will appear from the following detailed description.

Heretofore, in the production of filaments or threads from organic csters of cellulose, either by the dry or evaporative method or by the wet method of spinning, solutions of the organic esters of cellulose in volatile organic solvents were extruded through the orifices of a spinneret into an evaporative coagulating medium. The use of such volatile solvents, however, presents the disadvantages that it is costly to recover the same and even with the greatest care a considerable amount is lost, they are generally inflammable and therefore present a fire risk, and some of the solvent is retained by the yarns and filaments with the resultant effect on the tenacity and other properties of the same.

We have found that the disadvantages arising out of the use of volatile solvents may be avoided by shaping or extruding the organic ester of cellulose through the orifices of a spinneret while the same is in molten condition. In this manner there are produced filaments or threads that are free of residual volatile solvent and therefore of superior tenacity and other textile properties.

Further, we have found that if the solidified filaments or threads are stretched the resulting orientation of the material produces increased tenacity.

In accordance with our invention melted organic ester of cellulose of good stability is extruded through orifices of suitable size into a cooling medium to form filaments or threads which are wound on suitable take-up means. As a further carrying of our invention forward, the filaments or threads are stretched, the stretching being preferably effected after they are cooled.

While this invention will be described specifically in connection with cellulose acetate, it is equally applicable to other organic esters of cellulose such as cellulose propionate and cellulose butyrate, or mixed esters such as cellulose aceto-propionate and cellulose aceto-butyrate.

In the drawing, the figure shows one form of apparatus suitable for carrying out the invention, this drawing being merely illustrative.

The apparatus comprises a closed vessel 1 provided with a removable cover 2 and a jacket 3 for receiving a fluid heating medium, such as steam or hot water which may be introduced through the pipe 5 and taken off through pipe 4. The cellulose acetate, 6, can be introduced into the vessel by removing the cover 2 or in the form of a slurry or suspension through valved pipe 7 and may be melted by the heating medium in the jacket. Pressure may be built up in the vessel 1 by means of steam or hot water 9 or hot inert liquid or gas under pressure introduced through pipe 8, this hot liquid or gas aiding in the melting of the cellulose acetate. When the cellulose acetate is melted it is forced, by means of the high pressure in the vessel 1, through the valved outlet 10 into the header 11 and from the latter it is delivered through pump 12 to spinnerets 13. The filaments 15 are extruded through orifices in the spinnerets into spinning cells or chambers 14 through which cool or cold air or other gas passes in order to set the formed filaments. The formed filaments may be collected on any suitable take-up means.

By this invention filaments of low denier, say from less than 2 to 10 or more, which are associated together in required number to form yarn may be produced. However, filaments of large denier, say from 150 to 500 or more, which may be used as horsehair, bristles and the like, may be made, as may be ribbons or bands to be used as artificial straw. The molten cellulose acetate may be extruded through metallic jets or spinnerets of sufficient strength and/or reinforced to withstand the extrusion pressure, and which have orifices of required size, say circular orifices of from 0.05 mm. in diameter or upward in the case of making yarns or bristles, or the orifices may be in the form of slits, when artificial straw is made.

Any suitable temperatures and pressures may be employed for melting and extruding the cellulose acetate. We have found that although it is difficult to melt cellulose acetate at ordinary pressures without decomposition, the cellulose acetate can be readily melted or at least rendered sufficiently plastic to be extruded when it is heated at elevated pressures, especially in the absence of air or other reactive gas. Pressures of 100 to 500

lbs. per square inch are generally suitable and temperatures of 150° C. upwards are employed. For the same effect, the higher the pressure the lower is the required temperature.

We have found that the temperatures and pressures required for melting and extruding the cellulose acetate may be reduced by heating the same under pressure in the presence of water or other aqueous liquid, in which case the pressures employed to melt the cellulose acetate will be from 50 to 250 or more pounds per square inch above atmospheric, and the temperature will be that of boiling water at such pressures. It is preferable to employ the higher pressures, i. e. 150 to 250 or more pounds per square inch and the temperatures corresponding thereto, i. e., about 185 to 210° C.

As an alternative and in some respects preferred method of lowering the temperature required for melting, there is associated with the cellulose acetate relatively non-volatile solvents or plasticizers, in amounts of from 10 to 50% or more of the cellulose acetate present. Examples of such plasticizers are dimethyl phthalate, diethyl phthalate, dimethoxy ethyl phthalate, dibutyl phthalate, triacetin, ethyl orthotoluene sulphonamide, ethyl paratoluene sulphonamide. A mixture of two or more of these plasticizers may be used. These plasticizers may be associated with the cellulose acetate in intimate admixture prior to melting in any suitable manner, such as by finely grinding the ingredients together; dissolving them in a volatile solvent to form a plastic mass, kneading the plastic mass under heat, and working the mass of heated malaxating rolls until practically all the volatile solvent is evaporated, etc.

The melting and extrusion of the cellulose acetate whether alone or in the presence of water or plasticizer may be carried out in any suitable apparatus such as a closed vessel in which pressure may be exerted by mechanical means such as pistons, or by hydraulic or pneumatic means such as hot water, hot mineral oil or other inert liquid, or heated nitrogen, steam, carbon dioxide or other inert gas under pressure. When the cellulose acetate is melted it is forced, by means of the high pressure obtaining in the vessel, through a suitable opening in said vessel into a header, and from the header the melted cellulose acetate is pumped into a spinneret from which the filaments are extruded into suitable orifices therein.

In order to harden the filaments extruded from the spinnerets, they may be extruded into a chamber through which cool or cold air or other gas passes or into a cool or cold bath of water or other inert liquid, or if special effects such as dyeing or saponification are desired into a bath containing the ingredients required to cause such effects, such as aqueous solutions of caustic soda or other alkali for saponification, or aqueous or other baths containing dyestuffs or their components for dyeing.

The filaments are then passed around godet rollers or other take-up devices which preferably rotate at a peripheral speed greater than that at which the cellulose acetate is extruded so that the molten cellulose acetate filaments are drawn out as they leave the spinnerets, after which they are wound up, preferably with twisting, by a suitable machine, such as a ring-spinning or cap-twisting device when yarns are made. However, in some instances, especially when the yarns or filaments are subsequently subjected to a stretching operation, we prefer not to draw the filaments out, in which case the godet rollers or other take-up devices are run at a peripheral speed equal to that at which the cellulose acetate is extruded.

We have found that if the filaments or yarns are drawn, and while they are hard and at a temperature not substantially above room temperatures and preferably below the same, say from 0° to 30° C., the tenacity of the same is greatly increased. The amount of stretching is on the order of 20 to 300% or more. This stretching may be done by passing the yarn or filaments around rollers rotating at progressively increasing peripheral speeds or between the positive nip of pairs of rollers rotating at progressively increasing peripheral speeds, and this stretching may be accomplished in only one stage or in a multiplicity of stages. The cooling of the yarns or filaments for stretching may be accomplished through the temperature of the surrounding atmosphere, but particularly good results are obtained by rapidly cooling the yarns or filaments by immersion in a relatively cold non-solvent such as water, which if desired may contain dyes, alkali for saponification, or other desired substance. This stretching operation may be carried out concomitantly with the spinning operation, i. e., after the filaments are extruded and before they are wound onto packages; or as a separate operation, namely after the yarn has been formed and wound onto packages. If desired the stretching may be done on the yarns or filaments while they contain plasticizer. Alternatively the stretching may be done while the yarns or filaments are subjected to the action of steam (preferably wet) or hot water at or near the boiling point.

In those cases where plasticizers are employed, they may be removed from the yarns or filaments after they are formed but prior to their use and either before or after they have been stretched if a stretching operation is performed. This can be accomplished readily by extracting the yarns or filaments with a liquid such as ethyl ether or benzene, that is, a solvent for the plasticizer and not for the cellulose acetate. In some instances where increased pliability is desired, as in the case of artificial straw or bristles, the plasticizer may be advantageously retained.

Any suitable effect materials may be incorporated with the cellulose acetate prior to melting and extrusion to obtain any desired effect. Thus, if yarns or filaments of low luster are to be produced, finely divided titanium dioxide, antimony trioxide, etc., may be incorporated therein. To produce colored yarn, dyes, or finely divided pigments of organic or inorganic character as carbon black, graphite, ultramarine, may be incorporated.

It is to be understood that the foregoing detailed description is merely given by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention, what we desire to secure by Letters Patent is:

1. Process for the production of filaments of organic esters of cellulose, which comprises melting an organic ester of cellulose under a pressure applied directly thereto by a hot aqueous liquid extruding the molten organic ester of cellulose under such pressure through orifices to form filaments, and setting said formed filaments.

2. Process for the production of filaments of cellulose acetate, which comprises melting cellu-

lose acetate under a pressure applied directly thereto by a hot aqueous liquid, extruding the molten cellulose acetate under such pressure through orifices to form filaments, and setting said formed filaments.

3. Process for the production of filaments of cellulose aceto-propionate, which comprises melting cellulose aceto-propionate under a pressure applied directly thereto by a hot aqueous liquid, extruding the molten cellulose aceto-propionate under such pressure through orifices to form filaments, and setting said formed filaments.

4. Process for the production of filaments of cellulose aceto-butyrate, which comprises melting cellulose aceto-butyrate under a pressure applied directly thereto by a hot aqueous liquid, extruding the molten cellulose aceto-butyrate under such pressure through orifices to form filaments, and setting said formed filaments.

5. Process for the production of filaments of organic esters of cellulose, which comprises melting an organic ester of cellulose under a pressure applied directly thereto by hot water, extruding the molten organic ester of cellulose under such pressure through orifices to form filaments, and setting said formed filaments.

6. Process for the production of filaments of cellulose acetate, which comprises melting cellulose acetate under a pressure applied directly thereto by hot water, extruding the molten cellulose acetate under such pressure through orifices to form filaments, and setting said formed filaments.

7. Process for the production of filaments of organic esters of cellulose, which comprises melting an organic ester of cellulose under a pressure applied directly thereto by a hot aqueous liquid, extruding the molten organic ester of cellulose under such pressure through orifices to form filaments, and cooling the formed filaments at least until they are solidified.

8. Process for the production of filaments of cellulose acetate, which comprises melting cellulose acetate under a pressure applied directly thereto by a hot aqueous liquid, extruding the molten cellulose acetate under such pressure through orifices to form filaments, and cooling the formed filaments at least until they are solidified.

9. Process for the production of filaments of organic esters of cellulose, which comprises melting an organic ester of cellulose under a pressure applied directly thereto by hot water, extruding the molten organic ester of cellulose under such pressure through orifices to form filaments, and cooling the formed filaments at least until they are solidified.

10. Process for the production of filaments of cellulose acetate, which comprises melting cellulose acetate under a pressure applied directly thereto by hot water, extruding the molten cellulose acetate under such pressure through orifices to form filaments, and cooling the formed filaments at least until they are solidified.

11. Process for the production of filaments of organic esters of cellulose, which comprises melting an organic ester of cellulose under a pressure applied directly thereto by hot water, extruding the molten organic ester of cellulose under such pressure through orifices to form filaments, cooling the formed filaments at least until they are solidified, and stretching said filaments.

12. Process for the production of filaments of cellulose acetate, which comprises melting cellulose acetate under a pressure applied directly thereto by hot water, extruding the molten cellulose acetate under such pressure through orifices to form filaments, cooling the formed filaments at least until they are solidified, and stretching said filaments.

13. Process for the production of filaments of cellulose aceto-propionate, which comprises melting cellulose aceto-propionate under a pressure applied directly thereto by hot water, extruding the molten cellulose aceto-propionate under such pressure through orifices to form filaments, cooling the formed filaments at least until they are solidified, and stretching said filaments.

14. Process for the production of filaments of cellulose aceto-butyrate, which comprises melting cellulose aceto-butyrate under a pressure applied directly thereto by hot water, extruding the molten cellulose aceto-butyrate under such pressure through orifices to form filaments, cooling the formed filaments at least until they are solidified, and stretching said filaments.

15. Process for the production of filaments of organic esters of cellulose, which comprises melting an organic ester of cellulose under a pressure applied directly thereto by hot water, extruding the molten organic ester of cellulose under such pressure through orifices to form filaments, cooling the formed filaments at least until they are solidified, and stretching said filaments in the presence of a cooling medium.

16. Process for the production of filaments of cellulose acetate, which comprises melting cellulose acetate under a pressure applied directly thereto by hot water, extruding the molten cellulose acetate under such pressure through orifices to form filaments, cooling the formed filaments at least until they are solidified, and stretching said filaments in the presence of a cooling medium.

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