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MANUFACTURE OF ARTIFICIAL PRODUCTS

Richard R. Sitzler and Joseph E. Bludworth, Cumberland, Md., assignors to Celanese Corporation of America, a corporation of Delaware

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This invention relates to the manufacture of artificial products such as filaments, yarns, threads, films, foils, ribbons, straw, fibers, bristles and the like having a basis of an organic derivative of cellulose.

An object of our invention is to prepare artificial products such as filaments, yarns, threads, films, foils, ribbons, straws, bristles and the like which deluster readily and retain their delustered or opaque appearance permanently.

Another object of our invention is the preparation of crepe yarns which when woven into fabrics produce improved crepe effects.

Still another object of our invention is to improve the dyeing characteristics of yarns having a basis of an organic derivative of cellulose.

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

Artificial filaments having a basis of an organic derivative of cellulose have been prepared by extrusion of solutions of the cellulose derivatives in a volatile solvent through orifices of a spinneret into an evaporative atmosphere. Yarns made from filaments prepared in this way have found extensive use. While such yarns become delustered in dyeing or scouring baths at elevated temperatures, they have a tendency to become relustered upon the application of heat and moisture as in ironing, or by treating the yarns with certain chemical agents. Yarns and films of a permanent dull finish have been obtained by incorporating certain pigments in the solutions from which the yarns are spun but such pigmented yarns or films are lacking in strength and for this reason are limited in their uses. Due to this lack of strength pigmented yarns are of little value in the preparation of crepe yarns which are to be employed in the preparation of crepe fabrics.

We have now discovered that if polymers of ethylene oxide are added to the solutions employed for spinning yarns of an organic derivative of cellulose, there may be obtained yarns having excellent strength and improved creping properties. When yarns prepared in this manner are subjected to a delustering treatment in aqueous media, the resulting yarns are permanently delustered and on subsequent treatment with agents which normally reluster the usual delustered yarns, the yarns prepared in accordance with our invention retain their original dull or delustered appearance. In addition, we have found that organic derivative of cellulose yarns containing polymers of ethylene oxide have a considerably increased affinity for dyestuffs normally employed for dyeing organic derivative of cellulose materials and may be dyed in deeper shades by such dyestuffs with less processing than yarns having a basis of an organic derivative of cellulose as heretofore prepared. This increased dyeing affinity is not only achieved in yarns which have been delustered but, as stated, yarns in which the ethylene oxide polymer is still present likewise exhibit this desirable property. Moreover, films or foils may be formed from solutions containing polymers of ethylene oxide, which films or foils, upon being given a delustering treatment, are permanently delustered.

The polymers of ethylene oxide employed in accordance with our invention may be prepared in any suitable manner as by contacting liquid ethylene oxide at suitable temperatures with fuller's earth or other similar material containing aluminum oxide. The products of polymerization are mixed polymers and may range from a syrupy liquid through waxy solids to rubbery compounds. By suitable treatment the polymers may be separated into several fractions. Each of the several fractions contains polymers of different degrees of polymerization but of substantially similar or related characteristics. However, each fraction contains polymers having degrees of polymerization different from those of the other fraction. While the heterogeneous mixture of polymers may be employed in our process prior to being separated into individual components, we preferably separate the mixture into its fractions and employ those fractions which are entirely water-soluble and fully compatible with the organic derivatives of cellulose employed.

The ethylene oxide polymers may be added in varying amounts to the solutions from which the yarns, filaments, films, foils or other plastic materials, and the like, may be prepared. We have found that excellent results may be achieved when from 1 to 50%, and preferably from 5 to 10%, of ethylene oxide polymers, based on the weight of the organic derivative of cellulose contained therein, is added to the solutions from which the filaments, films, etc. are prepared. The solutions containing the ethylene oxide polymers, after thorough mixing to obtain uniformity, may be filtered and then extruded through the orifices of a spinneret either into an evaporative atmosphere as in dry spinning or into a coagulative medium as in wet spinning. Such solutions of the organic derivatives of cellulose

may also be cast into films on wheels, bands and the like, and the solvent evaporated therefrom.

Filaments spun from solutions containing the ethylene oxide polymers may be the fine filaments having a denier of, say, 1 to 10, such as are used for making yarns, or they may be much heavier, i. e. having a denier of, say, 50 to 2,000, which may be used for artificial bristles or, when flattened, may be used for artificial straw. Our invention may also be advantageously employed for making films, foils and other products where strength and delusterability are important. The solution of organic derivative of cellulose containing the polymers may be cast or extruded to form sheet material ranging in thickness from that of thin foil, to that of film and of sheets of substantial thickness.

The delustering treatment is preferably carried out in hot aqueous media. We have found that water at elevated temperature makes an excellent delustering agent. Wet steam may also be employed. In some instances, the aqueous delustering bath may also contain other substances such as soap, salts, acid salts, pine oil, tetralin, etc. which accelerate or enhance the delustering action of the bath. While we preferably employ these solutions at or near the boiling point, i. e. about 100° C., temperatures as low as 60° C. may be advantageously employed.

Since at least a substantial proportion of the ethylene oxide polymers employed are water-soluble, the action of the delustering bath, i. e. the aqueous medium, causes the polymers contained in the materials being treated to dissolve out of the same to a substantial degree. The time necessary for the removal of the water-soluble ethylene oxide polymers from the materials being treated varies greatly and depends in the main upon the extent to which it is desired to remove them and the relative thickness of the filaments, yarn, foil, film or other material being delustered. When employing water at a temperature of about 100° C. for the delustering bath, a satisfactory degree of delustering may be obtained by maintaining the materials being treated in the bath for from 30 to 120 minutes. By treating the materials containing ethylene oxide polymers in this way, a completely delustered and substantially opaque product may be obtained.

While our invention is particularly applicable for the preparation of permanently delustered filaments, yarns, foils, films and other products having a basis of cellulose acetate, other organic derivatives of cellulose, such as other organic esters and ethers of cellulose, may be employed. Examples of such other organic esters of cellulose are cellulose propionate and cellulose butyrate and mixed esters such as cellulose acetate-propionate and cellulose acetate-butyrate, while examples of cellulose ethers are ethyl cellulose and benzyl cellulose.

In preparing the solution of the organic derivatives of cellulose containing the ethylene oxide polymers any suitable solvent in which both the organic derivatives of cellulose and the ethylene oxide polymers are soluble may be employed. Examples of such solvents are acetone, acetone and ethyl or methyl alcohol, chloroform, ethylene dichloride, ethylene dichloride and ethyl or methyl alcohol, methylene chloride and ethyl or methyl alcohol, and ethylene formal.

In order further to illustrate our invention, but without being limited thereto, the following examples are given:

Example I

To a solution of 1 part by weight of cellulose acetate in 3 parts by weight of acetone there is added 5% on the weight of the cellulose acetate of an ethylene oxide polymer melting at about 50° C. and having a molecular weight of about 4,000. The resulting solution is thoroughly mixed and filtered and is then processed, employing a dry-spinning method, into a yarn of 124 denier having 60 filaments. The yarn thus prepared is backwound on to a stainless steel bobbin and the bobbin holding the yarn is then immersed in boiling water for 15 minutes and is then dried. The yarn produced is completely and permanently delustered and has a tenacity of 1.32 grams per denier. Pigmented yarn of the same denier and having the same number of filaments has a tenacity of only 1.05.

Example II

A film .0026 inch in thickness is poured or cast from the solution prepared as indicated in Example I. This film is found to have a breaking strength of 5,700 pounds per square inch, an elongation of 6% and is transparent. After being treated in boiling water for 15 minutes the ethylene oxide polymer contained therein is removed, the film becoming completely opaque. The strength of the resulting opaque film, i. e. the breaking strength, is found to have risen to 6,700 lbs. per square inch and the elongation to 14.2%.

Example III

Yarn prepared in the manner indicated in Example I and still containing the ethylene oxide polymer therein is delustered by treatment for one hour at 98–100° C. in a bath containing 20 grams per liter of sodium oleate. The delustered yarn is then dyed for 30 minutes at 80° C. in a bath containing 1% of 1:4-dimethyl-aminoanthraquinone based on the weight of the yarn, 0.25 gram per liter of sodium oleate soap and 3 ccs. per liter of "Celascour" (a mixture of Turkey red oil, xylene and water). The yarn is then rinsed well in water at 65° and then in cold water. By this treatment the yarn is delustered and dyed a blue shade, the degree of delustering and depth of shade being greater than that produced on cellulose acetate yarn which is free from ethylene oxide polymer.

Where the above blue dyestuff is replaced by Celliton Blue AF or by Setacyl Direct Discharge Blue G equally satisfactory results are obtained.

Example IV

Yarn prepared in the manner indicated in Example I, and while still containing the ethylene oxide polymer in the yarn structure, is treated in a bath comprising 1% of 1:4-dimethyl-aminoanthraquinone based on the weight of the yarn to be dyed, 0.25 gram per liter of sodium oleate soap and 3.0 ccs. per liter of "Celascour." The yarn is immersed in the bath maintained at 98–100° C. for one hour. The yarn is simultaneously delustered and dyed a blue shade by this treatment. The depth of shade and degree of delustering are considerably greater than that achieved when cellulose acetate yarn not containing the ethylene oxide polymer is given a like treatment in the bath.

Example V

To a solution of 1 part by weight of cellulose acetate in 3 parts by weight of acetone is added 10% on the weight of cellulose acetate of ethylene

oxide polymer melting at about 50° C. and having a molecular weight of about 4,000. The solution is mixed and filtered and is then dry spun into a yarn of 180 denier having 40 filaments. The resulting yarn is then given a crepe twist of 70 turns per inch during an up-twisting operation while being subjected to the action of steam during the actual insertion of the twist. This crepe yarn is employed for weaving a crepe fabric having 74 picks per inch on a warp of 120 ends of a 100 denier, 40 filament cellulose acetate yarn of 5 turns per inch. The original width of this fabric is 45 inches. When this fabric is given a boil-off at 98° C. in an aqueous boil-off bath to develop the crepe, the boil-off width decreases to 37 inches after treatment for 60 minutes. The resulting fabric presents a pronounced and pleasing crepe effect.

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. In a process for the production of permanently delustered crepe fabrics, the steps of applying a crepe twist to filamentary materials having a basis of an organic substitution derivative of cellulose and having uniformly incorporated throughout the mass thereof a polymerized ethylene oxide having a consistency from a syrupy liquid to a solid in an amount equal to from 1 to 50% based on the weight of the organic substitution derivative of cellulose present in said filamentary materials, weaving said crape twisted filamentary materials into a fabric, and then subjecting said fabric to the action of a hot aqueous medium so as to cause said fabric to exhibit a crepe effect and a permanent deluster.

2. In a process for the production of permanently delustered crepe fabrics, the steps of applying a crepe twist to filamentary materials having a basis of cellulose acetate and having uniformly incorporated throughout the mass thereof a polymerized ethylene oxide having a consistency from a syrupy liquid to a solid in an amount equal to from 1 to 50% based on the weight of the cellulose acetate present in said filamentary materials, weaving said crepe twisted filamentary materials into a fabric, and then subjecting said fabric to the action of a hot aqueous medium so as to cause said fabric to exhibit a crepe effect and a permanent deluster.

3. In a process for the production of permanently delustered crepe fabrics, the steps of applying a crepe twist to filamentary materials having a basis of cellulose acetate and having uniformly incorporated throughout the mass thereof a polymerized ethylene oxide having a consistency from a syrupy liquid to a solid in an amount equal to from 5 to 10% based on the weight of the cellulose acetate present in said filamentary materials, weaving said crepe twisted filamentary materials into a fabric, and then subjecting said fabric to the action of a hot aqueous medium so as to cause said fabric to exhibit a crepe effect and a permanent deluster.

RICHARD R. SITZLER.
JOSEPH E. BLUDWORTH.