

## UNITED STATES PATENT OFFICE

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## PROCESS FOR TREATING CELLULOSE

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This invention relates to the manufacture of artificial products derived from cellulose. More particularly, it relates to a method of improving the physical properties of synthetic textiles derived from cellulosic materials.

Co-pending application Serial No. 649,777, filed February 23, 1946, for Cellulose derivatives and process of preparing same, discloses a process yielding new cellulose derivatives by reaction of cellulose materials containing free hydroxyl groups, in solution or in dispersion, with polymethylolphenols, followed by a thermal treatment, possibly in the presence of acid catalysts, with the object of forming resiniform bridge linkages of polymethylolphenols between the cellulose chains.

Co-pending application Serial No. 649,778, filed February 23, 1946, for Shaped synthetic products and method of making same, discloses a process in which the polymethylolphenols are slightly condensed and added to solutions of cellulose material for spinning. After spinning the usual operations are performed, followed by drying, whereupon the said thermal treatment is applied to the resulting filaments.

Co-pending application Serial No. 649,779, filed February 23, 1946, for Method of treating pre-formed shaped structures, discloses a kindred process wherein cellulose artificial filaments are impregnated with polymethylolphenols in a finished state, with subsequent subjection to thermal treatment.

In the process disclosed in the aforementioned applications, the bridge linkages are converted into resiniform linkages by heating, preferably in the presence of suitable catalysts. Products containing the resiniform linkages are characterized inter alia by swelling to a degree considerably lower than that of the untreated cellulosic material.

The present invention relates to the treatment of cellulose textiles with a polymethylolphenol (phenol alcohol) under such conditions that the product obtained is nearly white in color and may be bleached by means of active chlorine agents without showing appreciable discoloration.

The polymethylolphenol in question is obtained by treatment of approximately one mole of p-tert-amylphenol with two moles of formaldehyde in an alkaline medium. The phenol is a commercial product sold under the name "Pentaphen."

In accordance with an example for this preparation, 164 g. of p-tert-amyl-phenol are mixed with 200 cc. of 30% Formol and 80 cc. of soda at

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400 g./liter. The phenol dissolves rather rapidly, the temperature rising to about 40° C. This is allowed to stand for about 96 hours, obtaining a yellowish viscous liquid which is diluted with 40% ethanol to reduce the concentration of phenol alcohol to about 6.5%, whereupon the liquid is exactly neutralized with normal sulfuric acid. This product is particularly suitable for the production of textile filaments pursuant to the invention.

The neutral liquid, preferably after slight condensation in the presence of lactic or acetic acid, is added to the cellulose spinning solutions, which are then processed in the usual manner. The liquid may be added to the spinning solution or used for treatment of the spun filaments with subsequent thermal treatment for reaction with the regenerated cellulose and condensation on the fiber in the manner set forth in said co-pending applications.

It is also possible to add traces of lactic or acetic acid, e. g. 0.2%, then impregnate the viscose rayon or fiber with the liquid, air, dry and apply the thermal treatment, i. e. heat to approximately 110–120° C. for 30 minutes.

In these two cases the filaments obtained have a greatly reduced secondary swelling, e. g. 50% to 55% as against 100 to 110% for normal filaments. The product is nearly white, and yellows only slightly, if at all, when bleached with active chlorine agents.

The following example is obviously not intended in any restrictive sense, serving merely to facilitate understanding of the invention:

A thick bundle of 100,000 regenerated cellulose fibers with unit gauge of 1.25 d. obtained from viscose in the usual manner, is thoroughly washed, de-sulfured and bleached in apparatus composed of several vessels. Upon leaving the last vessel, the bundle enters a bath containing 2 grams of laurylpyridinium sulfate, about 1.5% of phenol alcohol obtained as above set forth, and 1 gram of lactic acid per liter of water. The bundle is aired by "foulardage" and passed over a series of rollers heated to 90° C. and then after drying, over more rollers heated to 130° C. in such a manner that the exposure time is about 10 minutes at the higher temperature.

The bundle is then cut into pieces 60 mm. in length, and the fiber is opened by grinding.

The fiber obtained is woolly, has a secondary swelling of 50, and is particularly suitable for mixing with wool. It resists bleaching without appreciable coloration, particularly bleaching with active chlorine.

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As is shown by said co-pending applications the resiniferous compound may be incorporated into the solution from which the yarn is formed, or it may be applied to a preformed yarn or fiber. When applied to a preformed structure, the resiniferous compound is preferably applied in an aqueous solution, though it can be applied from a non-aqueous solution as disclosed in the aforementioned co-pending application, Serial No. 649,779.

The reaction is carried out in the absence of a catalyst. However, an acid catalyst can be used if desired, and, when used, the temperature and times of the thermal treatment are correlated to give the best results. When an acid catalyst is to be used in procedures wherein the resiniferous compound is incorporated in situ during the spinning of the filament, the catalyst can be incorporated as disclosed in co-pending application, Serial No. 649,778. When the catalyst is to be incorporated in a preformed structure, it can be incorporated as disclosed in co-pending application, Serial No. 649,779.

Since it is obvious that various changes and modifications may be made in the above description without departing from the nature or spirit thereof, this invention is not restricted thereto except as set forth in the appended claims.

What is claimed is:

1. The process which comprises condensing in alkaline medium p-tert-amyl-phenol and formaldehyde in molecular ratio of 1:2 and reacting the condensation product with cellulosic material containing free hydroxyl groups and subjecting the reaction products to thermal treatment to form resiniform bridge linkages between the cellulose chains.

2. The process which comprises incorporating dimethanol - p - tert - amyl - phenol obtained as the condensation product of p-tert-amyl-phenol and formaldehyde in a dispersion of a cellulosic material containing free hydroxyl groups, coagulating said dispersion, drying the coagulated material, and subjecting the dried coagulated material to a thermal treatment in the presence of an acid catalyst to produce resiniform bridge linkages between the cellulose chains.

3. A method of producing shaped structures which comprises dissolving in a viscose spinning solution dimethanol - p - tert - amyl - phenol obtained as the condensation product of p-tert-amyl-phenol and formaldehyde, extruding said solution in the shape of the desired structure into a coagulating and regenerating bath, and subjecting the structure to a thermal treatment to form resiniform bridge linkages between the cellulose chains.

4. A method which comprises impregnating a preformed shaped cellulosic structure containing free hydroxyl groups with a solution comprising dimethanol - p - tert - amyl - phenol obtained as the condensation product of p-tert-amyl-phenol and formaldehyde, and subjecting the impregnated shaped structure to a thermal treatment to form resiniform bridge linkages between the cellulose chains.

5. Artificial textiles of regenerated cellulose obtained by the process of claim 1 which will not turn yellow upon bleaching with baths containing active chlorine and which comprises cellulose chains with resinous bridge linkages therebetween of dimethanol-p-tert-amyl-phenol.

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