

UNITED STATES PATENT OFFICE

2,339,203

TREATMENT OF CELLULOSIC TEXTILE MATERIAL

Harold W. Stiegler, Stamford, and Linton A. Fluck, Jr., South Norwalk, Conn., assignors to American Cyanamid Company, New York, N. Y., a corporation of Maine

No Drawing. Application August 30, 1941,
Serial No. 409,147

5 Claims. (Cl. 8—136)

This invention relates to the treatment of cellulosic textile materials with methylated methylol-melamine. The invention includes the method of treatment and the treated textile materials themselves.

We have found that cellulosic textile materials such as thread, yarn, and knit and woven cloth of cotton, linen, hemp, jute, regenerated cellulose, cellulose esters, cellulose ethers and other fibrous materials of cellulosic nature may be greatly improved in several important characteristics by the application thereto of methylated methylol-melamine. Methylated methylol-melamine when applied to cellulosic textile fabrics in the amounts and in the manner hereinafter described serves as a general finishing agent filling and weighting the fabrics and giving them a soft, springy hand. The fabric is also given a fair degree of water repellency.

Another and more important result of our treatment of cellulosic fabrics with methylated methylol-melamine is a resistance to shrinking which is imparted to the fabric. We have found that cellulosic materials of the type described above may be made substantially resistant to shrinking by the application of 2 to 8 per cent by weight of methylated methylol-melamine. The shrink resistance obtained as a result of our treatment may be illustrated as follows. Bleached cotton percale samples were impregnated with varying amounts of an aqueous dispersion of methylated methylol-melamine and the fabric tacked to its original size, dried and cured for 7 minutes at 284° F. The fabric samples, together with an untreated control sample, were then washed for 20 minutes at 160° F. in a solution containing ¼ per cent soap and ¼ per cent sodium carbonate. Upon drying it was found that the untreated cotton percale control sample had shrunk 4.4 per cent. The sample of the washed cloth containing 4 per cent of methylated methylol-melamine by weight based on the dry weight of the fabric had shrunk only 1.1 per cent as a result of the washing operation. Another cloth containing 3 per cent of methylated methylol-melamine was found to have shrunk only 1.7 per cent and a cloth sample containing as little as 2 per cent of the methylated methylol-melamine had shrunk 2.5 per cent. Shrinkage measurements were made along the warp of the cloth.

We have also discovered that when the amounts of methylated methylol-melamine added to the cloth is increased to values of the order of 8 to 14 per cent by weight the cellulosic fabric in addi-

tion to having greater resistance to shrinking also possesses remarkable resistance to creasing. This is very surprising as it has heretofore been considered necessary to use as much as 30 per cent by weight of other resinous materials to obtain the same degree of crease resistance.

In order to determine the permanency of the crease and shrink resisting properties of our treated cellulosic fabrics samples of 80 x 80 cotton percale were impregnated with approximately 8 per cent by weight of the methylated methylol-melamine. The fabric was then dried 3 minutes at 250° F. and cured 4 minutes at 284° F. 3½ per cent of diammonium hydrogen phosphate by weight based on the dry weight of the resin was added to the resin dispersion as accelerator. The treated fabrics were washed in a standard washing machine for 4 hours at 160° F. in a ¼ per cent soap and ¼ per cent sodium carbonate detergent solution. Despite this drastic washing the fabrics showed a resin retention of 87.3 per cent. Similar tests made with other accelerators and different curing conditions showed comparable results.

One of the most important features of our new process is the unexpectedly low chlorine retention of the treated materials when subjected to laundering operations involving bleaching steps. Many resin finishes heretofore applied to cellulosic textile fabrics have had the serious disadvantage of picking up chlorine from the bleach. Residual chlorine in the cloth causes considerable damage to the fabric when it is pressed or ironed. During the bleaching and while pressing the cloth, particularly when the pressing conditions are severe, part of the cellulose is converted to oxycellulose with the result that the fabric is weakened. The cloth also has a much greater tendency to turn yellow or scorch during pressing. Cotton percales treated with methylated methylol-melamine in accordance with our invention were tested for chlorine retention after the treated fabrics had been subjected to severe washing and bleaching by commercial laundries. Results show a sharply reduced tendency of the methylated methylol-melamine treated fabric to retain chlorine from bleaching baths as contrasted to the behavior of other well known resinous textile finishing agents. Spot tests for chlorine with starch-potassium iodide solution, pressing the fabric for 20 seconds on a flat press set at its highest temperature (425–450° F.) and chemical analysis for oxycellulose as well as titrations for chlorine show conclusively that the chlorine retention of our treated fabric was extremely small.

The methylated methylol-melamine which we employ in our process is ordinarily, and preferably, applied to the cellulose fabrics in the form of an aqueous dispersion containing from about 2 to 15 per cent or more of the methylated methylol-melamine. The aqueous dispersions may be applied to the fabrics in various ways known to those in the art; thus, for example, the dried fabrics to be treated may be immersed in the resin dispersion and then passed through suitable rolls as in a padder or mangle to secure uniform impregnation and to remove excessive resin. The fabric, however, may be impregnated by other methods such as for example by spraying or with suitable boxes located on the mangle. Our invention is not limited to any particular method of impregnating the cellulosic fabric and other methods will of course occur to those skilled in the art.

As pointed out above the cellulosic fabrics may be rendered substantially resistant to shrinkage by the application of about 2 to 8 per cent by weight of the resin based on the weight of the fabric. Larger amounts as for example 8 to 14 per cent by weight of the resin will not only shrink-proof the fabric but also impart thereto a high degree of crease resistance. Accordingly our invention contemplates the application of 2 to 14 per cent by weight of methylated methylol-melamine to the cellulosic material.

After the material has been impregnated with the methylated methylol-melamine dispersion, or solution, it is dried and the resin cured in the fabric by the application of elevated temperatures. In order to speed up the curing of the resin and decrease the heating time a suitable catalyst may be added to the aqueous resin dispersion. Oxalic acid, diammonium hydrogen phosphate and methyl acid pyrophosphate have given us particularly good results. Other catalysts such as triethanol amine phthalate, zinc chloride, acetic acid, dilute mineral acids, as HCl, and others have been used with satisfactory results. Still other catalysts for the curing of methylated methylol-melamine resins are known to chemists in the resin art and may be employed.

The curing temperatures are in general quite low and may vary considerably from about 200° F. to about 300° F. with a corresponding reduction in time of cure with increase of temperature. The drying and curing operation is flexible and may be varied to suit the equipment available to the processor. High temperatures of the order of 280° F. to 300° F. will cure the resin to a substantially water-insoluble state in 3 or 4 minutes. Where facilities are not available for curing the resin at moderate temperatures within the range of about 240° F. to 280° F., the fabric after impregnating with the desired amount of methylated methylol-melamine may be framed to width as on a pin tenter, dried, batched up on

a shell and allowed to stand hot to obtain total drying and heating time sufficient to dry the cloth and insolubilize the resin in situ. With some fabrics a drying and curing time of two or three hours may be required at 200° F.

The methylated methylol-melamine which we employ is prepared by known methods. Two to six moles of formaldehyde is reacted with one mole of melamine to form a condensation product believed to be mostly methylol-melamine. This product is then reacted with two to six moles of methanol whereby methylated methylol-melamine is formed. Excessive polymerization should be avoided since the resin is preferably employed by us as an aqueous dispersion, or solution, and products polymerized excessively tend to be difficultly soluble in water. We have also found that excessive polymerization of the product tends to give the cellulosic fabric a stiff hand.

What we claim is:

1. The process which comprises impregnating cellulosic textile materials with 2 to 14 per cent by weight of a water-soluble methylated methylol-melamine in the form of an aqueous solution and thereafter drying the textile material and curing the methylated methylol melamine whereby it is rendered substantially water-insoluble.

2. The process which comprises impregnating cotton textile fabrics with 2 to 14 per cent by weight of a water-soluble methylated methylol-melamine in the form of an aqueous solution and thereafter drying the fabric and curing the methylated methylol melamine whereby it is rendered substantially water-insoluble.

3. The process of rendering cellulosic textile fabrics resistant to shrinking and creasing which comprises impregnating said fabrics with 8 to 14 per cent by weight of a water-soluble methylated methylol-melamine in the form of an aqueous solution and thereafter drying the fabric and curing the methylated methylol melamine whereby it is rendered substantially water-insoluble.

4. The process of rendering cellulosic textile fabrics resistant to shrinking which comprises impregnating said fabrics with 2 to 8 per cent by weight of a water-soluble methylated methylol-melamine in the form of an aqueous solution and thereafter drying the fabric and curing the methylated methylol-melamine whereby it is rendered substantially water-insoluble.

5. A cellulosic textile fabric resistant to shrinking containing 2 to 14 percent by weight of a heat-cured and substantially water-insoluble methylated methylol melamine, said fabric having been impregnated with an aqueous solution of a water-soluble methylated methylol melamine and thereafter dried and heated to cure the resin to a substantially water-insoluble condition.

HAROLD W. STIEGLER.
LINTON A. FLUCK, JR.