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FLAMEPROOFING PROCESS

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1 Claim. (Cl. 117—102)

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This invention relates to flameproofing and relates more particularly to the flameproofing of cellulose acetate and other organic acid ester of cellulose materials.

It is frequently desirable to flameproof textile fabrics and the like and other materials made of or containing cellulose acetate and other organic acid esters of cellulose. The flameproofing, to be satisfactory, should impart to the treated materials a high resistance to burning and/or retard the propagation of a flame when lit, should be inexpensive and easily applied, and should be relatively permanent to laundering, dry cleaning and the like to which the treated materials are normally subjected. In addition, the flameproofing should not alter the hand or other physical properties of the treated materials in an objectionable manner, and should not appreciably affect the dyeing of the said materials.

It is an important object of this invention to flameproof cellulose acetate and other organic acid esters of cellulose materials with brominated monomeric triallyl phosphate.

Other objects of this invention will be apparent from the following detailed description and claim.

According to the present invention, cellulose acetate and other organic acid ester of cellulose materials, such as textile fabrics and the like, are treated with brominated monomeric triallyl phosphate for the purpose of making the same flameproof. The monomeric triallyl phosphate not only imparts to the treated materials a high resistance to burning, but is also inexpensive and easily applied, and, when applied in the manner and proportions specified hereinafter, is relatively permanent to laundering, dry cleaning and the like to which the materials are normally subjected. The brominated monomeric triallyl phosphate will not, when properly applied, alter the hand or other physical properties of the flameproofed materials in an objectionable manner, nor will it appreciably affect the dyeing of the said materials.

Care should be taken not to employ polymerized triallyl phosphate since several disadvantages attend the use of the brominated polymer as a fire retardant. Thus, a higher add-on of the brominated polymer is necessary to get the equivalent fire retardence as against the use of the brominated monomer. Moreover, the brominated polymerized phosphate ester shows a low resistance to dry cleaning, making for a less durable fire retardant. This may be attributed to the fact that the brominated polymer has more difficulty in penetrating the filaments or fibers of the

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material being treated, particularly cellulose acetate filaments or fibers, and simply stays on the surface thereof. Accordingly, the polymer is more easily removed. It is apparently not due to any increase in solubility since the actual solubility of the brominated polymer is low in dry cleaning solvents. Another disadvantage is that the hand of the coated fabric at the same coating level is stiffened to a greater extent by the use of the brominated polymer than by the use of the brominated monomer.

In flameproofing cellulose acetate and other organic acid ester of cellulose materials, there may be employed from about 2 to 50% by weight of the brominated monomeric triallyl phosphate, based on the weight of the material being treated. For applications where the flameproofed materials will not be laundered or dry cleaned and need not be relatively permanent to these treatments, as little as from about 2 to 5% by weight of the brominated monomeric triallyl phosphate will impart to cellulose acetate and other organic acid esters of cellulose a satisfactory resistance to burning. For applications where the flameproofed materials must be relatively permanent to laundering and dry cleaning, it is preferable to employ from about 15 to 50% by weight of the brominated monomeric triallyl phosphate. The degree of bromination of the monomeric triallyl phosphate may range from about 50 to 100% of theoretical. The best results with respect to relative permanence to laundering and dry cleaning is obtained, however, with a fully brominated material, namely tri-(2,3-dibromopropyl) phosphate.

The brominated monomeric triallyl phosphate may be applied to the cellulose acetate and other organic acid ester of cellulose materials in several different ways. For example, it may be applied to the said materials from solution in a solvent or solvent mixture which will not dissolve the cellulose acetate or other organic acid ester of cellulose. Solutions containing from about 2 to 50% by weight of the brominated monomeric triallyl phosphate give satisfactory results. The brominated monomeric triallyl phosphate may also be applied to the materials to be flameproofed from a dispersion in water or other liquid which will not dissolve the cellulose acetate or other organic acid ester of cellulose. The said dispersions may contain from about 1 to 25% by weight of the brominated monomeric triallyl phosphate. It has been found that the best results are obtained when the application of the brominated monomeric triallyl phosphate is carried out in

the presence of a swelling agent for the cellulose acetate or other organic acid ester of cellulose. The presence of the swelling agent effects a considerable improvement in the relative permanence of the treated materials to laundering and dry cleaning. Following the application of the brominated monomeric triallyl phosphate, the treated materials may be dried in conventional manner as on a tenter.

Solvents that may be employed in preparing a solution of brominated monomeric triallyl phosphate suitable for application to the cellulose acetate or other organic acid ester of cellulose include, for example, benzene, toluene, ethylene dibromide, trichlorethylene, butylacetate and isobutyl methyl ketone. Swelling agents for the cellulose acetate or other organic ester of cellulose materials that may be present with the above solvents during the application of the brominated monomeric triallyl phosphate thereto include, for example, ethylene dichloride, ethanol, chloroform and isopropanol. The solvent or solvent mixture may itself have a sufficient swelling action on the cellulose acetate or other organic acid ester of cellulose in which case no additional swelling agent need be employed.

When the quantity of brominated monomeric triallyl phosphate applied to the cellulose acetate or other organic acid ester of cellulose material is within the upper portion of the range hereinabove specified, there is some tendency for the hand of the said materials to change. If this change in hand is undesirable, it may be readily eliminated by lightly rinsing the material in an organic solvent in which the brominated monomeric triallyl phosphate is soluble, but in which the cellulose acetate or other organic acid ester of cellulose is not soluble. The organic solvents listed above for preparing solutions of the brominated monomeric triallyl phosphate are satisfactory for this purpose. The change in the hand of the treated materials may also be eliminated by laundering or dry cleaning the said materials.

The following examples are given to illustrate this invention further.

Example I

A cellulose acetate fabric is dipped for 15 seconds in a solution containing 195 grams per liter of tri-(2,3-dibromopropyl) phosphate in a solvent mixture made up of 2 parts of toluene, 1 part of isopropanol and 1 part of isobutyl methyl ketone, the parts being by volume, and maintained at room temperature. The fabric is blotted gently, dried at room temperature for 15 minutes and in an oven at 80° C. for 10 minutes. The add-on of the tri-(2,3-dibromopropyl) phosphate is 25% by weight based on the weight of the fabric.

The treated fabric is given eleven launderings in accordance with Test No. 2 as described on page 102 of the A. A. T. C. C. manual for 1947–1948. After the eleventh laundering, the fabric is tested for flame resistance generally in accordance with A. S. T. M. Test D626–41T to be suitable for a fabric formed of cellulose acetate or other organic acid ester of cellulose yarns or fibers.

However, since the application of the flame to cellulose acetate fabric, flameproofed in accordance with the present invention, will cause a certain amount of fused material to be produced, the procedure outlined in the A. S. T. M. test referred to should be slightly modified. Thus, the fused material should be broken up by hand before applying the weights or tension to start and to continue the tear according to the test. In the case of non-esterified cotton textiles, there will be no fused portion to break up if the flameproofing compounds of the present invention are used.

The laundered fabric has a char length of 3⅜ inches which is identical to the char length of the unlaundered fabric indicating that the flameproofed fabric is resistant to laundering. An untreated cellulose acetate fabric is completely consumed in this test.

Example II

A cellulose acetate fabric is dipped for 120 minutes in a solution containing 111 grams per liter of tri-(2,3-dibromopropyl) phosphate in a solvent mixture made up of equal parts by volume of ethylene dichloride and toluene, and maintained at room temperature. The fabric is squeeze rolled, dried at room temperature for 60 minutes and in an oven at 80° C. for 10 minutes. The add-on of the tri-(2,3-dibromopropyl) phosphate is 33.5% by weight based on the weight of the fabric. The hand of the fabric is slightly stiff, but this stiffness is eliminated by rinsing the fabric for 10 seconds in the above solvent mixture.

The treated fabric is given six dry cleanings in a Laudereometer, with each dry cleaning consisting of a 25 minute tumbling with soaped dry cleaning solvent, such as "Stoddard Solvent," in the ratio of one pound of fabric per gallon of solvent. The soap is prepared in the manner described on page 9 of Commercial Standard CS59–44 of the Department of Commerce and is used in the ratio of 270 ml. of soap per gallon of dry cleaning solvent. After each tumbling period, the fabric is given two five minute rinses with unsoaped dry cleaning solvent, blotted and oven dried at 80° C. for 10 minutes.

The dry cleaned fabric has a char length of 2¾ inches when tested in the manner set forth in Example I, indicating that the flameproofed fabric is resistant to dry cleaning.

Example III

A swatch of cellulose acetate fabric ½'' x 3'' is dipped for 15 seconds in a solution containing 80 grams per liter of partially brominated monomeric triallyl phosphate having a bromine content of 49% of theoretical dissolved in carbon tetrachloride, and maintained at room temperature. The treated fabric is blotted, dried for 60 minutes at room temperature and for 10 minutes at 80° C. The add-on of the partially brominated monomeric triallyl phosphate is 10% by weight based on the weight of the fabric.

The treated fabric is suspended vertically and ignited with a small luminous flame. When the flame is removed from the fabric there is no afterflame.

Example IV

A cellulose acetate fabric has applied thereto 2.3% by weight, based on the weight of the fabric, of tri-(2,3-dibromopropyl) phosphate in the manner set forth in Example III except that the solvent is toluene and the concentration therein of the phosphate ester is 10 grams per liter. The treated fabric has a char length of only 3⅜ inches when tested in the manner set forth in Example I.

It is to be understood that the foregoing detailed description is given merely 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:

Process for flameproofing cellulose acetate textile materials which comprises impregnating the said materials with tri-(2,3-dibromopropyl) phosphate dissolved in a mixture of ethylene dichloride as swelling agent and toluene as solvent, the amount of tri -(2,3-dibromopropyl) phosphate added to said textile materials being about 15 to 50% by weight, based on the weight of said textile materials drying the treated materials and slightly rinsing the said textile materials with a solvent for the tri-(2,3-dibromopropyl) phosphate which is not a solvent for the cellulose acetate, to improve the hand of the flameproofed textile materials while substantially retaining their flameproof properties.

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