

UNITED STATES PATENT OFFICE

2,036,854

FIREPROOFING TEXTILE MATERIAL

William Alexander Dickie and Frank Brentnall
Hill, Spondon, near Derby, England, assignors
to Celanese Corporation of America, a corpo-
ration of Delaware

No Drawing. Application October 16, 1931, Serial
No. 569,350. In Great Britain December 20,
1930

5 Claims. (Cl. 91—68)

This invention relates to a treatment of textile materials in order to improve their resistance to fire.

One of the most important points to be considered in dealing with a textile material is to remove or lessen the danger of fire either from the inflammability of the material itself or from hot or burning pieces of the material igniting other objects. A number of substances have been used in order to render textile materials "fireproof", and a considerable degree of success has been obtained. The difficulty of obtaining efficient "fireproofing" is however increased in dealing with fabrics made of or containing organic derivatives of cellulose. These derivatives of cellulose are not usually considered highly inflammable, but at the same time there is added a danger that drops of the molten burning material may ignite other inflammable materials.

It has now been found that efficient "fireproofing" can be obtained for textile materials in general, and especially for cellulose derivative materials, by applying to the materials a substance or composition which, under the action of heat, will yield products which will extinguish a flame, and will also yield products which will hold the hot or charred material together.

Suitable compounds or compositions for carrying this invention into effect are the borates of volatile bases or mixtures of boric acid with salts having a volatile base, or of borates or phosphates with salts having a volatile base. The salts of ammonia and especially the ammonium halides, such for example as ammonium bromide, are excellent examples of salts having a volatile base which may be employed in these mixtures.

As examples of compounds or compositions which may be used for "fireproofing" textile materials mixtures of ammonium phosphate and boric acid, or of ammonium phosphate and ammonium borate and ammonium borate alone may be mentioned. The best composition for use in fireproofing a cellulose derivative material has however been found to be a mixture of ammonium borate or phosphate with an ammonium halide such as ammonium bromide, the added ammonium halide appearing greatly to increase the flame extinguishing properties of the ammonium borate or phosphate. In any of the above compounds or mixtures the ammonium base may be replaced by another volatile base, for example an organic base.

This invention may be applied to the "fireproofing" of textile materials of all kinds. Thus for example cotton, jute, regenerated cellulose or

other cellulosic materials, wool or silk materials may be treated but the process is of greatest value for fireproofing materials made wholly or in part of cellulose derivatives, for example cellulose acetate, cellulose formate, cellulose propionate, cellulose butyrate, or other cellulose ester, or ethyl cellulose, methyl cellulose, benzyl cellulose or other cellulose ether.

In the case of treating materials made of or containing organic derivatives of cellulose, such as those mentioned above, improved results may be obtained by combining the fireproofing treatment of the present invention with a treatment designed to weight the material with a suitable insoluble metallic compound, as for example a phosphate, silicate or other compound of tin. Such weighting treatments are in general carried out by introducing the metallic radicle necessary for the formation of the weighting compound under such conditions that swelling of the cellulose derivative occurs. Such swelling may take place in the weighting metal bath itself or in a pretreatment applied for the specific purpose. Thus suitable swelling agents may be applied to the materials before or together with the weighting metal solution, as for example stannic chloride. Thiocyanates are very suitable for this purpose. An alternative method consists in employing the weighting metal compound, as for example stannic chloride, in a sufficient concentration or at a sufficiently high temperature to swell the cellulose derivative or in employing a compound of the weighting metal which is itself a swelling agent, as for example stannic thiocyanate or stannic chlorothiocyanate. The weighting treatment raises the ironing point of the material, and in conjunction with the fireproofing treatment characteristic of the invention produces a greatly improved product. Such weighting treatments may be applied at any suitable time in relation to the application of the fireproofing treatment. Preferably, however, the weighting treatment is applied before the fireproofing treatment.

Again in the fireproofing of materials made of or containing a cellulose ester additional safeguards may also be applied, for example by subjecting the cellulose ester material to a partial or superficial saponification, e. g. to an extent corresponding to a loss of 3% of the weight of the fibre.

The treatments may be applied to the materials at any stage of their production or treatment and may be applied together with other treatments. Thus, the treatments may be applied to yarn prior

to its being made up into fabric, it may be applied to the fabric, or to an article made from the fabric.

According to one mode of carrying the invention into effect the material to be treated is impregnated by immersion in a solution of the compound or composition to be applied, and is then wrung out and dried. Drying of the fabrics may take place for example on a stenter. Other methods of impregnation, such for example as padding, printing or spraying may be employed.

The following examples illustrate the invention but are not to be considered as limiting it in any way. In particular considerable variations may be made in the proportions of fireproofing substances applied and in the concentrations in which they are applied.

Example 1

A cellulose acetate fabric is immersed for a short time in an aqueous solution containing 20% of ammonium bromide and 5% of ammonium borate. The fabric is wrung out and dried on a stenter. Instead of applying the fireproofing agents by a bath treatment they may conveniently be applied by a padding treatment.

Example 2

Previously scoured cellulose acetate fabric is entered and steeped for 1 hour with occasional turning in a bath maintained at about 20° C. containing 280 grams per litre of stannic chloride ($\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$) and 243 grams per litre of ammonium thiocyanate. It is then lifted, drained well and washed, first in two baths of cold water and then in two baths of water at 45° C. until free from soluble salts. The goods are then entered into a bath at 40° C. containing 12% of sodium dihydrogen phosphate, worked for $\frac{1}{2}$ hour, lifted, rinsed and soaped for 20 minutes in a bath containing $2\frac{1}{2}$ grams per litre of olive oil soap at 50° C. The goods are then rinsed and are padded or impregnated with the fireproofing composition as described in Example 1. By employing trisodium phosphate instead of sodium dihydrogen phosphate for the fixation of the tin a saponification of the cellulose acetate may be effected.

Example 3

Previously scoured cellulose acetate fabric is entered into a bath at 20° C. containing 280 grams of stannic chloride and 182 grams of ammonium

thiocyanate per litre. After steeping for 1 hour with occasional turning the goods are lifted, drained, washed and further treated as described in Example 2 for the fixation of the tin compound. After rinsing the goods are padded with an aqueous liquor containing 15% of ammonium bromide and 10% of ammonium phosphate. They are then dried as described in Example 1.

What we claim and desire to secure by Letters Patent is:—

1. Process for the treatment of materials comprising organic derivatives of cellulose which comprises applying a weighting treatment to the said materials and then applying to the said materials a halide of a volatile base and a substance selected from the group consisting of a phosphate of a volatile base and a borate of a volatile base.

2. Process for the treatment of materials comprising cellulose acetate which comprises applying a weighting treatment to the said materials and then applying to the said materials a halide of a volatile base and a substance selected from the group consisting of a phosphate of a volatile base and a borate of a volatile base.

3. Process for the treatment of materials comprising cellulose esters which comprises applying a weighting treatment to the said materials and then applying to the said materials a halide of a volatile base and a substance selected from the group consisting of a phosphate of a volatile base and a borate of a volatile base, and which also comprises the step of saponifying the materials.

4. Process for the treatment of materials comprising cellulose acetate which comprises applying a weighting treatment to the said materials and then applying to the said materials a halide of a volatile base and a substance selected from the group consisting of a phosphate of a volatile base and a borate of a volatile base, and which also comprises the step of saponifying the materials.

5. In a process for the manufacture of materials comprising organic derivatives of cellulose, in which the materials are weighted with an insoluble metal compound, the step which consists in applying to said materials a halide of a volatile base and a substance selected from the group consisting of a phosphate of a volatile base and a borate of a volatile base.

WILLIAM ALEXANDER DICKIE.
FRANK BRENTNALL HILL.