Patented Mar. 15, 1949 2,464,342

UNITED STATES PATENT OFFICE

2,464,342

MANUFACTURE OF WATERPROOF AND WATER-REPELLENT NONINFLAMMABLE FIBROUS MATERIALS

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No Drawing. Application April 16, 1943, Serial No. 483,340

5 Claims. (Cl. 117—137)

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The invention relates to the manufacture of waterproof and water-repellent non-inflammable textiles and fabrics, and to substances adapted to be used in connection with this manufacture.

In patent application Ser. No. 475,575 now Patent No. 2,421,218, May 27, 1947, a method is described for rendering textiles and fabrics not inflammable and water-resistant. These materials are impregnated with a solution of pyrophosphoric acid, thereafter passed through a solution of a condensation product of melamine and formaldehyde, hardened and dried. An insoluble melamine pyrophosphate is formed which is firmly embedded in the synthetic resin coating.

The method is satisfactory and efficient. However, two impregnating steps are needed for its performance. The method also requires a rather accurate control of the working temperature in order to avoid weakening of the fibre during its exposure to the acid solution.

Furthermore, it has been observed that the precipitate sometimes does not sufficiently penetrate into the fibre which causes a somewhat cloudy surface appearance.

It is the object of the invention to improve the former method and to simplify and conduct the same in one bath.

It is an object of the invention to preserve the non-inflammability of the impregnated textiles and fabrics in spite of intensive washing or long time exposure to water and to the atmosphere.

It is a further object of the invention to avoid during the entire manufacturing procedure the contact of the fibres with acid solutions.

It is also an object of the invention to fully preserve the original mechanical strength of the fibre and its original pliability.

Furthermore, it is an object of the invention to process colored goods without impairing in the least their particular color, shade or tint, and to avoid cloudy surface appearance.

It is also an object of the invention to anchor the impregnating substances so firmly in the cell structure of the textiles and fabrics that a removal therefrom or dusting is prevented.

It is another object of the invention to preserve the original soft touch of the treated materials.

With these and other objects in view which will become apparent as this specification proceeds, the invention comprises in its broad aspect the impregnation of fibrous materials, such as particularly textiles and fabrics, with a solution of a melamine-aldehyde condensation product and a phosphoric acid salt of a volatile base, and

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in the incorporation of an insoluble melamine-phosphoric acid compound into the fibre.

The method may be advantageously carried out by the impregnation of the textiles and fabrics with a neutral pyrophosphoric acid salt of a volatile base, such as ammonia or a suitable amide or hydrazine, and by passing the dried or still moist materials through an aqueous condensation solution of melamine and an aldehyde, such as formaldehyde.

The pyrophosphoric acid salt may be directly added to the condensation solution which commences to be slightly cloudy after a long period of standing only.

If, however, this solution is heated to about 100° C., a white insoluble precipitate of melamine and of the pyrophosphoric acid is abruptly formed.

Ammonia has been found to be highly useful as a volatile base; but other suitable bases, such as ethylene diamine, hydrazine or guanidine may be employed.

The present method may be advantageously carried out by using a single impregnating bath containing all the substances forming the insoluble melamine precipitate, through which the fabrics or textiles are passed.

It is advisable to remove sizing substances previously to this treatment.

The processed fabrics or textiles are dried at a temperature of between 100 and 125° C. and preferably at about 120° C.

The textiles and fabrics are hereby provided with an impregnation and coating which renders the same non-inflammable, fire- and water-proof and mildew-proof, and which cannot be removed by washing.

The neutral ammonia salt of the pyrophosphoric acid has been found to be particularly useful in the performance of this invention.

The ammonia which is liberated at the elevated temperature reacts with the formaldehyde of the melamine condensation product, whereby hexamethylenetetramine is formed and the insoluble melaminepyrophosphate is precipitated; the formaldehyde is hereby torn from the melamine compound; hence, it follows that a preferably greater quantity of the condensation product should be employed as it would correspond to the stoicheiometrical relation.

In view of the fact that 2 mols melamine and 1 mol pyrophosphoric acid are required to form the compound and hexamethylenetetramine is produced from 3 mols formaldehyde and 2 mols ammonia, it is desirable to have an excess of

formaldehyde in the condensation bath as otherwise free melamine would be produced.

Example I

126 parts by weight of melamine, 200 parts by weight of formaldehyde slightly alkalized by the addition of sodium-hydrate, are condensed with 500 parts of water by heating the mixture for about 20 minutes with a reflux cooler. The resulting clear solution is cooled.

A concentrated solution containing 36 parts by weight of pyrophosphoric acid is neutralized by slowly adding to the same ammonia and cooling. It is advisable to employ a small excess of ammonia so that the resulting solution has a pH equal to about pH 7.5 which will prevent a premature formation of the precipitate.

A fabric is passed through this impregnating solution in the customary manner. The excess liquid is removed, for instance, by transporting the impregnated article through wringer rolls. Hereafter the fabric is dried at a temperature of between 100 and 125° C. and preferably at about 120° C. A water-insoluble melamine pyrophosphate is formed hereby which is firmly embedded into the fibre. The fabric is preferably rinsed again with water and dried. It is now provided with the desired water resistance and water repellent impregnation which cannot be removed by washing with water. At the same time it has been rendered non-inflammable and mildewproof.

The instant method may be performed by subjecting the textiles and fabrics to repeated impregnation in order to intensify its efficiency or to obtain special effects.

The relative quantities of the impregnating substances may be adjusted during a first impregnating phase in such a manner that no synthetic resin is produced, the formaldehyde and the melamine being used up in toto for the formation of the hexamethylenetetramine or the melaminepyrophosphate.

Hereafter a second impregnation may be conducted on the lines which are described in Example I, the fabric after the first treatment being still in a liquid absorbent state.

A final product is hereby obtained which contains a considerably larger quantity of fibre-embedded melaminepyrophosphate without increase of the artificial resin.

A specific example of performing the invention in this manner is given in the following:

Example II

126 parts by weight of melamine, 260 parts slightly alkalized formaldehyde and 500 parts of water are condensated to form an artificial resin. A concentrated solution of 36 parts of pyrophosphoric acid is added which has been neutralized and slightly alkalized with ammonia.

The fabric is passed through this solution and exposed to air for several hours. Hereafter it is conducted through the impregnating bath described in Example I. The thus produced article has a superior water repellency.

The ammonia may be substituted by other amides the aqueous solutions of which are alkaline, react with formaldehyde and form the desired precipitate at an elevated temperature.

Instead of the formaldehyde other aldehydes may be employed which by condensation with melamine form a water-insoluble colorless resin and are separable from the melamine condensation product so that the insoluble melamine phosphoric acid compound may be formed.

The fabric processed in accordance with the instant invention excels in permanent non-inflammability which is not reduced if the articles are rinsed with water or located therein even for a longer period. The fibre is not weakened by the impregnation; it is water resistant and water repellent which greatly shortens the drying time. The precipitate is formed within the cellular structure of the fibre which, therefore, remains unchanged at its surface. Consequently, dark colored fabrics, such as uniform cloth, can be successfully treated without necessitating an additional coloring procedure. The touch of the textiles processed according to the invention is appealing and soft and the fabrics retain their pliability.

There is a further important advantage inherent with this invention, insofar as the textile and fabric acquires a certain resistance against the attack of fungi and particularly mildew creating bacteria. This advantage results from the pronounced water repellency of the treated goods and from the fact that the bacteria do not find food for their growth on the sterile coating.

The water resistance and repellency is apparent from the fact that the processed fibres even if colored with a watersensitive dyestuff do not impart any dyestuff to water. If located in water for a long period the water remains clear.

It was found that at the same time the supershine of artificial silk is eliminated which assumes the dull luster of first quality natural silk.

The invention may also be carried out by partly or completely substituting the melamine formaldehyde condensation product by another hardenable synthetic resin.

For instance, a mixture of thiourea and melamine may be treated with formaldehyde and the mixed condensation product may be incorporated into the fibre; either an excess of formaldehyde may be employed, or the melamine condensation product may be substantially used up by the ammonium pyrophosphate or the relative quantities may be varied in another suitable manner.

The examples given above are not considered to be a limitation of the application or of the methods and products of the invention, because variations in percentages of the material used, in the time and temperature of the different heating steps in the sequence thereof, in the mode of application and in the use of the products of reaction can be made by one skilled in the art to suit different requirements.

We claim:

1. A method of rendering textiles and fabrics, non-inflammable, water- and mildew-proof, comprising impregnating the said materials with an aqueous solution of ammonium pyrophosphate and of a melamine-formaldehyde condensation product, drying the impregnated materials at an elevated temperature and incorporating thereby into the same a water insoluble melamine pyrophosphate.

2. A method of rendering textiles and fabrics, non-inflammable, water- and mildew-proof, comprising impregnating the said materials with the aqueous solution of ammoniumpyrophosphate containing a slight excess quantity of ammonia, and of a melamine-formaldehyde condensation product, drying the impregnated materials at an elevated temperature and incorporating thereby into the same a water insoluble melamine pyrophosphate.

3. A method of rendering textiles and fabrics, non-inflammable, water- and mildew-proof, comprising impregnating the said materials with the aqueous solution of ammoniumpyrophosphate and of a melamine-formaldehyde condensation product containing a slight excess quantity of formaldehyde, drying the impregnated materials at an elevated temperature and incorporating thereby into the same a water insoluble melamine pyrophosphate.

4. A method of rendering textiles and fabrics, non-inflammable, water- and mildew-proof, comprising impregnating the said materials with the aqueous solution of ammoniumpyrophosphate and of a melamine-formaldehyde condensation product, the latter being maintained in the solution in a quantity slightly in excess to its stoichiometrical relation to the pyrophosphate.

5. A method of rendering textiles and fabrics, non-inflammable, water- and mildew-proof, comprising impregnating the said materials with a slightly alkaline aqueous solution of ammoniumpyrophosphate and of a melamine-formaldehyde condensation product, drying the impregnated materials at a temperature of about 120° C., and again washing and drying the said materials.

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