This invention relates to improvements in the treatment of fabrics or other textile materials of the kind known to be susceptible to treatment for the production of crease-resistant products by the aid of resinous condensation products, hereafter termed resins for brevity.

The objects of the invention are to provide improved processes of preparing the impregnating solution and improving direct contact effecting the impregnation, and final condensation. Other objects will appear later. The scope of the invention is defined in the appended claims.

According to one feature of the present invention the textile material is impregnated with a solution of a lightly condensed resinoous product containing a potentially acid substance.

As examples of compounds which liberate acid or increase the acidity of the solution when heated in contact with the textile material, the following may be mentioned:—ammonium acetate, formate, citrate, maleate, tartrate, oxalate, sulphate, chlorate, phosphate (monobasic, dibasic, or tribasic), ammonium ethyl sulphate, ammonium oleyl sulphate or sulphonate, and compounds of similar bases such as monomethylamine, dimethylamine, the ethanolamines, the hydrazines, the cyclohexylamines, etc.

The amino type of compounds appear to liberate acid by reaction between the salt compound and the methyloc compounds of the urea-like substances and/or free formaldehyde which may be present or produced.

Other types of compounds may be used which liberate acid on heating by hydrolysis and/or dissociation, e.g. by hydrolysis such as ammonium acetate or tartrate, but the compound chosen must not be one which prevents penetration of the fibres of the textile material by the impregnating liquor. The amount of the potentially acid substance is variable but for ammonium salts it is convenient to add at least 1 to 2 percent and preferably from 5 to 10 or even 15 percent calculated on the amount of urea, for example.

The invention also includes broadly a process of producing crease-resisting textile materials in which final heating to insolvulize the resinoous nates, is effected by direct contact with steam.

In using urea-formaldehyde condensation products it is preferred to prepare a solution showing a ratio of 1:1.6 of urea to formaldehyde; it is preferred, moreover, that such solution should have a viscosity of about 5 to 7 centipoises (measured at 20° C., water value=1) at a density of 32° to 35° Tw., which can readily be secured by using 40% formaldehyde solution.

With the density practically constant, small changes only in the viscosity of the solution are obtained by the addition of formaldehyde. The density will be controlled to 30° Tw. by adding ammonium salts to the solution. The viscosity of the solution is increased by the addition of formaldehyde. The density is increased by the addition of ammonium salts to the solution.
may be diluted to 60%, 70%, 80%, 90%, concentration, and is stable for 5 hours at 15 to 16° C. After condensing in the cold for two to five hours the solutions are further diluted so as to be 50% of the original concentration (reckoned on the original full strength 100 gm. urea, 200 cc. of 40% formaldehyde solution) and two to three gm. NH₄H₂PO₄ per 100 cc. concentrated mixture is added. This salt may conveniently be added as a solution in the water which is used for the diluting operation.

Fabrics are impregnated with these solutions, squeezed, dried at a low temperature, preferably below 110° C., heated for 2 minutes at 120° C., washed and finished in the usual way.

Alternatively the solutions at 90 to 90% concentration mentioned above instead of being allowed to stand in the cold may be raised to say 70-80° C. and then cooled immediately.

**Example 2**

100 gms. urea are dissolved in 200 ccs. of neutralized 40% formaldehyde solution. This neutral solution is allowed to condense for from 3 to 20 hours at ordinary temperature, and is then diluted to 40% concentration and 2 to 3% ammonium dihydrogen phosphate added (estimated on the concentrated liquor). Fabrics are impregnated, squeezed and dried, preferably below 110° C. During this drying process, the ammonium salt is converted to the free acid, and it is important to check the efficiency of this conversion before proceeding to the final heating. We prefer that the acidity of the impregnated and dried material should be pH=4.5. The goods are then heated for 2 minutes at 120° C., or for 1 minute at 140 to 150° C. Alternatively, the ammonium dihydrogen phosphate may be replaced by two to three per cent ammonium tartrate in which case the impregnated and dried cloth is heated for two minutes at 170° C.

The heated fabric is washed in 0.5% warm soap solution for 5 minutes, well rinsed with water and dried in the usual manner.

The viscosity of the concentrated mixture above was 6.5 centipoises (water value=1) in one experiment after twenty hours. If the solution is allowed to stand it does not gelatinize but deposits crystals of melting point 126° C. These may be collected, and used in aqueous solution in concentration of 20-25% by weight in a familiar manner to the dilute solution referred to above, the amount of catalyst being 2 to 4% on the solid content. Free acid is not advisable on grounds of stability.

The crystalline product from the neutral reaction mixture gives a dimethyl urea of high solubility which is important since we can prepare powders suitable for use in making solutions for impregnation.

After the final heating of the goods for the production of crease-resisting and recovering effects, the material may be washed or finished according to desired known methods, either mechanical or chemical, e.g. the product may be treated with swelling agents or with substances which react with either or both the cellulose and condensation product.

**Example 3**

Goods composed wholly or partly of rayon are impregnated in a suitably diluted solution of a low viscosity condensation product prepared with an alkaline catalyst and then catalyzed with a potentially acid substance, e.g. NH₄H₂PO₄. As a specific instance, we may mention the following:—15 kgs. of urea are dissolved in 33 kgs. of neutralized 40% formaldehyde solution and 1.3 litres of ammonium hydroxide (0.88 density) added. The mixture is either raised to boiling and cooled rapidly or allowed to stand at room temperatures for from 3 to 6 hours. In these circumstances, the viscosity is usually found to lie between 5 and 7 centipoises, (water value=1).

The mixture is then diluted to 50 to 60% and a suitable catalyst added, e.g. 1.5% tartaric acid or from 2 to 3% ammonium dihydrogen phosphate or ammonium tartrate, calculated on the concentrated mixture. The cloth is impregnated, squeezed and thoroughly dried at a temperature preferably not exceeding 110° C., until, in the case of potentially acid substances, the necessary acidity which we prefer to be pH 4.5 has developed.

The goods are then heated for 2 minutes at 170° C. in the case of tartrate and either 1 min. at 140-150° C. or 2 mins. at 120° C. in the case of phosphate. Alternatively, the heating may be accomplished by passing the material through a chamber containing steam under similar conditions of time and temperature. After heating, the goods are washed, dried and finished according to known means.

Particularly in the case of steaming at the above-mentioned lower temperatures it was expected that the durability, flexibility and handle of the goods were superior to those of similar samples where condensations are effected by means of cylinders, hot air chambers, etc. Cotton goods may also be treated in the above manner but with less advantage than in the case of rayon, both as filament and as staple fibre. This treatment is applicable broadly e.g. when using substances such as tartaric acid instead of potentially acid substances.

**Example 4**

1 molecular proportion of urea is stirred at low temperature with a 10% excess of 1 molecular proportion of 40% formaldehyde solution after the addition of sufficient alkali (sodium hydroxide) to ensure an alkalinity of N/100. 360 gms. of urea are dissolved in 500 ccs. of neutral 40% formaldehyde solution and 5 ccs. of normal sodium hydroxide solution. The mixture was cooled to 20° C. and stirred for 6 hours and allowed to stand with cooling for a further period of 18 hours, when crystalline monomethyl urea commenced to separate. Further cooling of the liquor produced a total yield of 75% of monomethyl urea of melting point 110° C.

In view of the great instability of this substance in acid solution, we prefer to use potentially acid catalysts whose aqueous solutions are non-acidic, e.g. triammonium phosphate (heating for 20 minutes at 110 to 120° C. or 1 minute at 140 to 150° C.) or ammonium tartrate (2 minutes at 170° C.).

28 grams of this monomethyl urea was dissolved in 75 ccs. of water and 1 gram of tertiary ammonium phosphate was added. The textile material was impregnated, the excess of liquid removed and the material dried at a low temperature. It was then heated for 2 minutes at 120° C.

The time and temperature of final condensation are obviously capable of variation without sacrificing the efficiency of the invention. We are aware that it is possible to execute many variations on the above examples without sacrificing
the novelty of our invention, e. g. other bases than ammonia may be employed, most ammonium salts and potentially acid substances can be used with condensation products and mixtures of condensation products of urea or its isomer ammonium cyanate, thioureia, biuret, phenol, adipamide, dicyandiamide, ammonium thio-cyanate, etc. Methyol ureas are particularly suitable alone or in suitable mixture with or without urea and formaldehyde. In the case of mixtures prepared at room temperatures it is possible to add the ammonium salt before or during condensation provided that such catalyzed mixtures are neutral or alkaline.

We declare that what we claim is:—
1. Process of improving the crease-resistance of textile fabrics which comprises impregnating the fabric with a solution of a reaction product of urea and formaldehyde consisting mainly of
2. Process of improving the crease-resistance of textile materials which consists in impregnating the material with a solution of partially condensed resin components which is unstable in acid solution, and which solution contains a potentially acid ammonium salt, and heating the impregnated material to insolubilize the resin and produce a crease-resistant product.
3. Process of improving the crease-resistance of textile material which consists in impregnating the material with a partially condensed solution of urea and formaldehyde which is unstable in acid solution, which solution contains a potentially acid ammonium salt, and heating the impregnated material to insolubilize the resin and produce a crease-resistant product.

DISCLAIMER


Hereby enters disclaimer to claims 1, 2, 3, 4, 5, and 7 in said specification. [Official Gazette December 24, 1946.]
the novelty of our invention, e.g. other bases than ammonia may be employed, most ammonium salts and potentially acid substances can be used with condensation products and mixtures of condensation products of urea or its isomer ammonium cyanate, thiourea, biuret, phenol, adipamide, dicyandiamide, ammonium thio-cyanate, etc. Methyliol ureas are particularly suitable alone or in suitable mixture with or without urea and formaldehyde. In the case of mixtures prepared at room temperatures it is possible to add the ammonium salt before or during condensation provided that such catalyzed mixtures are neutral or alkaline.

We declare that what we claim is:

1. Process of improving the crease-resistance of textile fabrics which comprises impregnating the fabric with a solution of a reaction product of urea and formaldehyde consisting mainly of a methyliol urea and containing a potentially acid ammonium salt and then heating the impregnated fabric to insolubilize the resin.

2. Process of improving the crease-resistance of textile materials which consists in impregnating the material with a solution of partially condensed resin components which is unstable in acid solution, and which solution contains a potentially acid ammonium salt, and heating the impregnated material to insolubilize the resin and produce a crease-resisting product.

3. Process of improving the crease-resistance of textile material which consists in impregnating the material with a partially condensed solution of urea and formaldehyde which is unstable in acid solution, which solution contains a potentially acid ammonium salt, and heating the impregnated material to insolubilize the resin and produce a crease-resisting product.

4. Process of improving the crease-resistance of textile materials which consists in impregnating the material with a solution of a partial condensate of urea and formaldehyde containing an ammonium salt in quantity from 1 to 15 per cent of the amount of urea, and heating the impregnated material to insolubilize the resin and produce a crease-resisting product.

5. Process as in claim 2 in which an ammonium salt of a strong mineral acid is used.

6. The process of improving the crease-resistance of a textile fabric by insolubilizing of resin within the fibre in which the fabric is impregnated with a partially condensed solution of urea and formaldehyde containing a potentially acid substance and in which the insolubilizing is effected by heating the impregnated material in contact with steam.

7. A crease-resisting textile fabric containing a synthetic resin insolubilized by heating a partially condensed resinous product in situ in presence of acid liberated from an ammonium salt of a mineral acid.

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FREDERICK CHARLES WOOD.

DISCLAIMER


Hereby enters disclaimer to claims 1, 2, 3, 4, 5, and 7 in said specification. [Official Gazette December 24, 1946.]