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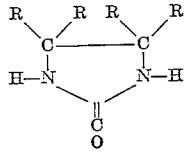
# 3,058,849
# CREASE-RESISTANT RESIN COMPOSITION FOR TEXTILES

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14 Claims. (Cl. 117—139.4)

This invention relates to, and has as among its principal objectives, the provision of certain thermosetting resinous compositions and formulations adapted to provide such compositions that have particular utility and are especially suited for being employed and applied as finishes for improving the crease and wrinkle resistance and the dimensional stability of textile materials. It also has reference to, and it is among the objects of the invention to provide, a method for utilizing such resinous compositions and the formulations that yield them for improving the crease resistance and dimensional stability of cloth and fabric constructed from various textile fiber materials and to furnish crease resistant and dimensionally stable textile materials by practice of the method and utilization of the resinous compositions of the invention.

Resinous compositions, in accordance with the present invention, are comprised of a thermoset adduct of from 1.0 to 4.0 moles of formaldehyde with each mole of a ring-substituted 2-imidazolidinone represented by the structure:



wherein R may be hydrogen, phenyl, or an alkyl radical containing from 1 to 4 carbon atoms with the limitation that at least one R is phenyl, or a 1 to 4 carbon atom alkyl radical. It is usually more beneficial for the resinous composition to be comprised of an adduct containing in the neighborhood of 2.0 to 2.5 moles of formaldehyde with each mole of ring-substituted 2-imidazolidinone.

Advantageously, the thermoset resinous formaldehyde adduct compositions of the invention are provided from an aqueous formulation that is adapted to be applied to a textile material and cured in situ to form the resinous composition on the textile material as a crease resistant finish. Such a formulation may be comprised of an aqueous solution containing the ring-substituted 2-imidazolidinone and the formaldehyde, which may be partially adducted in the formulation, along with a suitable curing catalyst, which may be an acid catalyst or other type of curing accelerator for assisting in the formation of the thermoset formaldehyde adduct. Usually, the aqueous, catalyst-containing formulations for providing the resinous compositions may be prepared as water white solutions having a pH between about 2.0 and 7.0 that may contain as much as 70 percent by weight of active solids, based on the weight of the formulation.

When heated and evaporated to dryness, the formulations produce a hard, brittle, clear, water-insoluble, thermoset, resinous composition. The formulations, as indicated, are adapted to be prepared as concentrated, dilutable stocks that do not require additional activating additaments or ingredients for subsequent use in the finishing of textile materials. The formulations of the invention may be stored for considerable periods without occasioning deleterious consequences and without



diminishing their efficacy for the desired purpose. In addition, the formulations are ordinarily compatible with a varety of other materials such as various latexes, silicone emulsions and the like that are often used for textile finishing purposes.

The crease resistance and dimensional stability of cloth, fabric and other textile fiber materials may be significantly improved by a method which comprises subjectng the textile material to an applicating, thermoset resin-providing formulation in accordance with the invention, impregnating the material with between about 5 and 30 percent by weight and, for many purposes, with between about 5 and 15 percent by weight of active solids from said formulation, based on the dry weight of said textile material, and subsequently exposing the impregnated textile material to a resin curing temperature between about 150 and 200° C. for a period of time between about one and five minutes. Lower curing temperatures generally require longer curing times. Advantageously, the aqueous applicating formulation may contain between about 2 and 25 percent by weight of solids although in many instances it may be convenient for the formulation to be employed with an active solids content of between 5 and 15 percent by weight. It is usually beneficial for the formulation to have a minor proportion, generally between about 0.02 and 1.0 percent by weight, of a wetting agent incorporated therein to assist in the wet pick up of the active solids from the applicating formulation by the textile material. Improved curing results may often by obtained by drying the impregnated textile material at any water evaporating temperature beneath the curing temperature as, for example, in the neighborhood of 100° C. so that it may be substantially free from water before it is subjected to a curing temperature. It frequently may be desirable to employ a temperature of about 175° C. for about one to two minutes in order to cure the applied resinous material.

The textile material may be impregnated with the formulation in any desired manner. While it is usually convenient to immerse the textile material in an applicating bath of the formulation and subsequently squeeze it free of excess liquid, suitable impregnating results may also be achieved by spraying, brushing or otherwise coating or applying the formulation on the textile material.

Tetxile materials, particularly cloth and fabric, that are provided in accordance with the invention with a finish application of the thermoset resinous material have significantly enhanced crease and wrinkle resistance and improved dimensional stability. In addition, they have acceptably low chlorine retention characteristics and maintain their tensile strength in a suitable manner, even after exposure to chlorine. They are possessed of a good hand and retain their other desirable physical characteristics so that they may advantageously be employed for various clothing and other cloth and fabric uses. Finished materials in accordance with the invention are generally at least equivalent and may frequently be superior to materials that are provided with conventional melamine and ethylene urea resin finishes employed for obtaining crease resistant properties. The textile materials that may be benefited by the practice of the invention may be of any desired origin, natural, artificial and synthetic, including cotton, wool, silk, viscose and acetate rayon, and acrylic, polyamide, polyester and the like synthetic materials. Frequently, however, the greatest benefit may be secured when the invention is practiced with textile materials that consist of, comprise or contain natural or artificial cellulosic fibers, or both.

The ring-substituted 2-imidazolidinones that are utilized in the practice of the instant invention and their manner of preparation may advantageously be in general ac-

cordance with those which have been disclosed by William F. Tousignant and William W. Bakke in their copending applications, both entitled "2-Imidazolidinones," and having Serial Nos. 836,874 and 836,875 which were filed on August 31, 1959. Such 2-imidazolidinones may be prepared by contacting, at a temperature of from about 175° to 300° C., essentially anhydrous ammonia with a ring-substituted 2-oxazolidinone having the ring-substituting substituents that are desirable in the resulting 2-imidazolidinone. The reaction product may be distilled and further purified by being recrystallized from a suitable solvent, for instance, 4-methyl-2-imidazolidinone may be recrystallized from acetone.

While the 4-methyl-2-imidazolidinone may be converted to a resinous formaldehyde adduct composition from the formulations of the invention with particular advantage, other ring-substituted 2-imidazolidinones may also be beneficially employed. Thus, for example, 5-methyl-2-imidazolidinone, 4-ethyl-2-imidazolidinone, 4,5-dimethyl-2-imidazolidinone, 4-isopropyl-5-methyl-2-imidazolidinone, 4-butyl-5-ethyl-2-imidazolidinone, 4-phenyl-2-imidazolidinone, 4,4,5,5-tetramethyl-2-imidazolidinone, etc. may be utilized.

The formaldehyde that is incorporated in the resinous composition providing aqueous formulation may usually be added conveniently in the form of an aqueous solution, such as one that contains in the neighborhood of 37 percent of dissolved formaldehyde. If desired, compounds that yield formaldehyde such as paraformaldehyde, trioxane and the like may be employed in place of formaldehyde in the preparation of the formulations. As mentioned, some of the formaldehyde in the formulation may adduct with the ring-substituted 2-imidazolidinone dissolved therein before the formulation is cured to a resinous composition. Despite this, the active solids of the formulation remain soluble until they are purposely cured by exposure to a suitable curing temperature.

Various acid curing catalysts and accelerator materials may be employed in the resinous composition-providing formulations of the invention. Advantageously an acid catalyst may be used which is selected from the group consisting of tartaric acid, adipic acid, citric acid and phosphoric acid. Phosphoric acid may be particularly desirable for employment in the practice of the invention. If desired, however, accelerator materials such as the alkanolamine hydrochloride type of catalyst and the like including 2-amino isopropanol hydrochloride, similar to that which is available from the Monsanto Chemical Company under the designation "Catalyst AC" may be employed. Usually, an amount of the catalyst between about 2 and 10 percent by weight, and preferably about 5 percent by weight, based on the weight of active solids in the formulation, may be found to be satisfactory.

By way of further illustration, an applicating formulation for providing a crease-resistant finish was prepared by entering 31 grams of 4-methyl-2-imidazolidinone, 63 grams of 37 percent by weight aqueous solution of formaldehyde, 4.4 grams of 2-amino isopropanol hydrochloride catalyst, 37 percent solids, and 272 grams of water in a suitable cnotainer and stirring to give a uniform solution. The pH of the resulting solution was about 2. A sample of 80 x 80 pure finished cotton sheeting was dried and weighed and then dipped in the formulation bath with the excess being squeezed out on wringer rolls. The dipping and wringing operations were then repeated. The sample was dried at 100° C. for five minutes, and cured at 150° C. for 4½ minutes. The sample was then washed in a mild soap solution, rinsed, dried at 100° C. for 5 minutes, and weighed to determine the pick up of resin (based on the dry weight of the fabric). A portion of the sample was then placed in a 0.25 percent available chlorine solution for 15 minutes, and given 6 separate 1 minute rinses in clear water. The sample was then dried and conditioned at 70° F. and 65 percent relative humidity for 24 hours. AATCC standard tests 69–52 and 66–53 were made to determine the chlorine damage and wrinkle resistance of the samples, respectively. Tensile tests were made and the color of the sample after chlorination and scorching at 365° F. for 30 seconds was noted. The results of these tests were compared to the test results from a blank (a sample of the same cotton sheeting that received no resin treatment) and to the test results from a sample of the same cotton sheeting that had been treated with a commercial ethylene urea resin using identical conditions throughout. The results of the tests are presented in Table I.

*Table I*

| Sample | Percent pickup | Wrinkle angle,[1] deg. | Percent loss in tensile strength [2] | Color after scorch [3] |
|---|---|---|---|---|
| 4-methyl-2-imidazolidinone resin. | 7.4 | 154 | 36.4–29.0 | Excellent. |
| Commercial ethylene urea resin. | 8.9 | 155 | 36.8–34.5 | Do. |
| Blank | None | 79 | 7.2– 0.0 | Do. |

[1] The wrinkle angle was measured in the warp direction. The wrinkle resistance was measured by AATCC method 66–53 in which the sample is given a standard crease and the angle of recovery is noted after the creasing pressure is released. An angle of 0° indicates no recovery or no wrinkle resistance while an angle of 180° indicates full recovery or perfect wrinkle resistance.

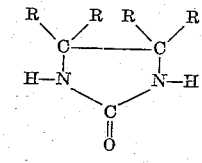
[2] The first figure is the percent loss in tensile strength after treatment, chlorination, and scorching, and the second is the percent loss after just the resin treatment measured according to AATCC method 69–52.

[3] There was no apparent color developed after scorching.

Results generally similar to those obtained in the above example may be obtained with other thermosetting formaldehyde ring-substituted 2-imidazolidinone adduct resinous finish compositions and formulations for providing them that are employed in the foregoing manner on cotton, rayon and other textile cloth and fabrics in order to improve their crease and wrinkle resisting characteristics.

What is claimed is:

1. Applicating formulation that is adapted to provide crease-resisting thermoset resinous textile finishes upon being subjected to resin curing temperatures between about 150° and 200° C. for a period of time between about one and five minutes, said formulation comprising, in aqueous solution, a ring-substituted 2-imidazolidinone having the general formula:



wherein R is selected from the group consisting of hydrogen, phenyl, and alkyl radicals containing from 1 to 4 carbon atoms with the limitation that at least one R is selected from the group consisting of phenyl and a 1 to 4 carbon atom alkyl radical; between about 1.0 to 4.0 moles of formaldehyde for each mole of ring-substituted 2-imidazolidinone; and a catalyst for curing the ring-substituted 2-imidazolidinone and the formaldehyde to a resinous formaldehyde adduct composition.

2. The formulation of claim 1, wherein said ring-substituted 2-imidazolidinone is 4-methyl-2-imidazolidinone.

3. The formulation of claim 1, wherein the catalyst is an acid catalyst selected from the group consisting of tartaric acid, adipic acid, citric acid and phosphoric acid.

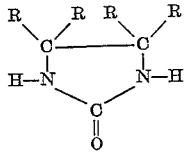
4. The formulation of claim 1, wherein the catalyst is an alkanolamine hydrochloride.

5. The formulation of claim 1, wherein the catalyst is present in an amount between about 2 and 10 percent by weight, based on the weight of the active solids in the formulation.

6. The formulation of claim 1, wherein the pH is between about 2.0 and 7.0.

7. The formulation of claim 1, containing as much as 70 percent by weight of dissolved active solids, based on the weight of the formulation.

8. Method for improving the crease resistance of textile materials which comprises impregnating the textile material with between about 5 and 30 percent by weight of active solids from a resinous formaldehyde adduct composition-providing formulation containing, in aqueous solution, a ring-substituted 2-imidazolidinone having the general formula:



wherein R is selected from the group consisting of hydrogen, phenyl, and alkyl radicals containing from 1 to 4 carbon atoms with the limitation that at least one R is selected from the group consisting of phenyl and a 1 to 4 carbon atom alkyl radical; between about 1.0 to 4.0 moles of formaldehyde for each mole of ring-substituted 2-imidazolidinone; and a catalyst for curing the ring-substituted 2-imidazolidinone and the formaldehyde to a resinous formaldehyde adduct composition; and subsequently exposing said impregnated textile material to a resin curing temperature between about 150° and 200° C. for a period of time between about one and five minutes.

9. In the method of claim 8, wherein the ring-substituted 2-imidazolidinone that is dissolved in said aqueous formulation is 4-methyl-2-imidazolidinone.

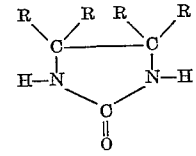
10. The method of claim 8, wherein the formulation for impregnating the textile material contains between about 2 and 25 percent by weight of dissolved active solids.



11. The method of claim 8 and including the step of drying the impregnated textile material substantially free from water before exposing it to a curing temperature.

12. In the method of claim 8, exposing the impregnated textile material to a resin curing temperature of about 175° C. for a period of time of about one to two minutes.

13. A textile material provided with a crease resistant finish that is comprised of a thermoset resinous composition comprising an adduct of from 1.0 to 4.0 moles of formaldehyde with each mole of a ring-substituted 2-imidazolidinone having the general formula:



wherein R is selected from the group consisting of hydrogen, phenyl, and alkyl radicals containing from 1 to 4 carbon atoms with the limitation that at least one R is selected from the group consisting of phenyl and a 1 to 4 carbon atom alkyl radical.

14. The textile material of claim 13 containing between about 5 and 30 percent by weight of the finish, based on the weight of the textile material.

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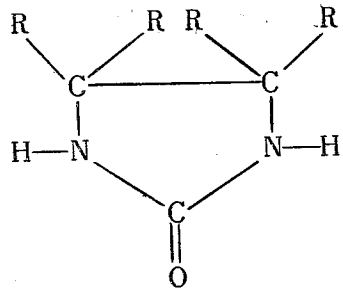
# UNITED STATES PATENT OFFICE
# CERTIFICATE OF CORRECTION

atent No. 3,058,849 October 16, 1962

William W. Bakke et al.

It is hereby certified that error appears in the above numbered patnt requiring correction and that the said Letters Patent should read as orrected below.

Column 1, lines 31 to 37, the formula should appear as hown below instead of as in the patent:




column 3, line 28, after "cent" insert -- by weight --; line 60, for "cnotainer" read -- container --.

Signed and sealed this 7th day of May 1963.

SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents