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3,014,042

CERTAIN 2-(3-METHYLOL IMIDAZOLIDONE-2-YL-1)-ETHYL ACYLATES AND PROCESS

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The present invention relates to improvements in the treatment of cellulosic fibrous material. More particularly, the invention is especially concerned with the treatment of cellulosic textile materials such as fibers, yarn or fabrics prepared principally from cotton, viscose, cellulose acetate or other cellulosic compounds, said treatment involving the impregnation of the textile material with novel finishing compositions comprised in important part of thermosetting resin-forming compounds together with certain improved softening agents, the latter compounds independently constituting new compositions of matter. As herein employed, the term "impregnation" is meant to include procedures by which the material being treated is coated with the finishing compositions. The invention is concerned further with novel processes for the production of the softening agents of this invention; as well as with cellulosic textile materials possessing improved physical properties, said cellulosic textile materials having been treated with the finishing compositions of this invention as herein described.

The modification of cellulosic textile material by chemical treatment has, over recent years, resulted in an increased interest in such materials for uses which were heretofore restricted to synthetic textiles. By way of illustration, a prominent application lies in the production of so-called "wash and wear" fabrics wherein the cellulosic textile material is treated by initially impregnating the material with a finishing composition consisting of an aqueous solution containing a thermosetting resin-forming compound, and ordinarily, an acid-producing catalyst. The impregnated cellulosic textile material is subsequently dried and cured, thereby at least partially polymerizing the resin-forming compound in situ and effecting a chemical bonding of the resin or resin-forming compound with the cellulosic textile material. The polymerization and bonding processes are accelerated by the presence of the acid-producing catalyst.

When treated in this manner, the impregnated cellulosic textile material has been found to evidence substantially improved crease-resistance and dimensional stability. Unfortunately however, certain disadvantages ordinarily accompany the treatment of cellulosic textile material by impregnation with a thermosetting resin-forming compound as herein above described. Included among these disadvantages are a loss of tear-strength in the impregnated material, and the impartation thereto of a harsh hand or feeling. In order to overcome these disadvantages, softening agents have frequently been incorporated in, or utilized in conjunction with the finishing composition with which the cellulosic textile material is treated.

Most softening agents are long-chain organic compounds which have the ability to lubricate the fibers of the material being treated, thus permitting the fibers more freedom of movement and improving the tear-strength of yarn or fabric containing the treated fibers. In addition, an enhanced hand is produced by the amount of softening agent located on the surface of the treated material.

To qualify as an efficient softening agent for use in conjunction with the treatment of cellulosic fibrous material, and particularly cellulosic textile material, by impregnation with a thermosetting resin-forming compound as hereinbefore described, the softening agent should evidence or possess the following desirable characteristics. Both

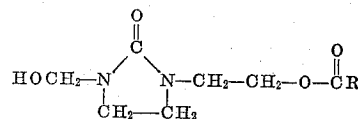
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the chlorine retention of the softening agent and its tendency to yellow or cause yellowing in the treated material should be at a minimum. The softening agent should also improve the tear-strength and hand of the treated material without adversely affecting the crease-resistance or other important physical properties of the treated material to any substantial extent. In addition, it is desirable that the softening agent be compatible with and stable in the finishing composition so as to permit incorporation therein, thus facilitating a simultaneous impregnation of the material to be treated with both the resin-forming compound and the softening agent.

The first softening agents employed for the treatment of cellulosic textile material were of the anionic type, such as sulfated tallows or tall oils. These compounds proved initially effective, but their effectiveness was dissipated rapidly during subsequent laundering. Cationic softening agents such as quaternarized fatty amines have also been considered attractive because of their substantive pick-up by the cellulosic textile material. Their use, however, frequently suffered the attendant disadvantage of an increased tendency toward yellowing in the treated material, while their effectiveness was often lost after relatively few launderings. The disadvantages of the prior art can now be overcome and still further improvements realized, through the practice of the present invention.

This invention is based upon the discovery that certain novel heterocyclic organic compounds, viz. methylolated imidazolidonyl ethyl acylates wherein the acylate radicals possess at least about 8 carbon atoms, serve as improved softening agents for cellulosic fibrous materials, and are particularly suited for use in conjunction with the treatment of cellulosic fibrous material with a thermosetting resin-forming compound. Illustratively, by treating, i.e. impregnating or coating a cellulosic textile material with a finishing composition comprised of an aqueous solution containing a thermosetting resin-forming compound, at least one of the novel softening agents of this invention and, preferably, an acid-producing catalyst, and by subsequently drying and curing the treated material, impregnated cellulosic textile material can be obtained possessing a high degree of tear-strength and evidencing a smooth hand. Moreover, the improved cellulosic textile products of this invention advantageously retain these desirable physical properties, as well as other properties accrueable as a consequence of impregnation with a thermosetting resin-forming compound, to a substantial extent even after repeated launderings, with little or no concomitant yellowing. Further, in addition to the treatment of cellulosic textile material, the finishing compositions of this invention can be employed in similar manner, and with like advantageous effect, to treat other cellulosic fibrous material, such as paper products prepared principally from cellulosic fibers.

More particularly, the softening agents of this invention are the 2-(3-methylol imidazolidon-2-yl-1) ethyl acylates represented by the general formula



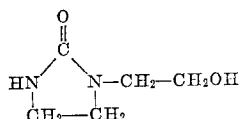
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wherein R designates a monovalent saturated or unsaturated aliphatic radical possessing at least about 8 carbon atoms and preferably from about 12 to about 22 carbon atoms, as for instance, an alkyl radical such as an octyl, decyl, dodecyl, tetradecyl, octadecyl or docosyl radical; an alkenyl radical such as an octenyl, decenyl, tetradecenyl, octadecenyl, or docosenyl radical; and the like. Of the above aliphatic radicals, the designation R preferably represents an alkyl radical. In addition, mixtures of 2-(3-

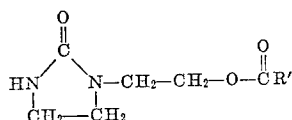
methylol imidazolidon-2-yl-1) ethyl acylates, wherein more than one monovalent aliphatic radical of the type hereinabove described is present can also be employed as softening agents in accordance with this invention.

It has been found that the effectiveness of the softening agents of the invention increases in direct relationship with respect to the number of carbon atoms in the aliphatic radical designated above by R. Thus, by way of illustration, greater tear strength and a smoother hand is evidenced by cellulosic textile material treated with the finishing composition of the invention as herein described, wherein the softening agent is such that R designates an alkyl radical possessing 17 carbon atoms, as compared with identical textile material similarly treated, wherein however, the softening agent is such that R designates an alkyl radical containing only 11 carbon atoms. In this connection, the methylolated imidazolidon-2-yl-1) ethyl acylates represented by Formula I above, wherein R designates a monovalent aliphatic radical possessing less than about 8 carbon atoms have not been found suitable for use as softening agents in accordance with the invention.

The methylolated imidazolidon-2-yl-1) ethyl acylates can be prepared by a two-step process involving the initial esterification of a fatty acid with 2-(imidazolidon-2-yl-1) ethanol, the latter compound being represented by the formula



so as to form the corresponding 2-(imidazolidon-2-yl-1) ethyl acylate intermediate represented by the general formula



wherein R' designates a monovalent aliphatic radical containing from 1 to about 22 carbon atoms. The intermediate product is then methylolated by reaction with either formaldehyde or paraformaldehyde so as to produce the desired 2-(3-methylol imidazolidon-2-yl-1) ethyl acylate. It is to be observed that the softening agents of this invention are produced by selecting the fatty acid reactant such that the acyl radical contained thereby possesses a monovalent aliphatic radical designated by R, wherein R is as defined above, i.e., contains at least about 8 carbon atoms.

The 2-(imidazolidon-2-yl-1) ethanol reactant can itself be obtained from any convenient source. The compound can be, for example, produced by processes such as those described in U.S. Patent 2,517,750, by the reaction of hydroxyethyl ethylene diamine with urea at a temperature of between about 100° C. and about 280° C. The resulting product need not be purified, but can, if desired, be employed in crude for subsequent reaction with a fatty acid as herein described.

The esterification of the 2-(imidazolidon-2-yl-1) ethanol with a fatty acid to produce the 2-(imidazolidon-2-yl-1) ethyl acylate intermediate is carried out by heating a mixture of the reactants at a temperature of between about 30° C. and about 200° C. or slightly higher, and preferably at a temperature of between about 100° C. and about 150° C., while removing the water formed during the course of the reaction. The concentration in which the reactants are employed is not critical to the process, although the ethanol derivative is preferably utilized in at least a stoichiometric proportion. Generally a suitable solvent such as xylene or toluene is also incorporated in the reaction mixture. Such a solvent further serves as an entraining agent, thereby facilitating the removal of water as an azeotropic mixture with the solvent during the course of the reaction. Under such conditions, the esterification is

performed by azeotropically refluxing the reaction mixture. However, the esterification can also be carried out in the absence of an azeotroping agent, as for example, by utilizing an elevated reaction temperature, and ordinarily a reduced pressure, sufficient to permit the removal of water formed during the reaction. In addition, it may also be desirable to incorporate in the reaction mixture a catalytic amount of a conventional direct esterification catalyst such as sulfuric acid or toluenesulfonic acid, etc. such catalyst preferably being employed in a concentration of from about 0.5 to about 5.0 percent by weight based upon the weight of the reactants and more preferably from about 1 to about 2 percent by weight based upon the weight of the reactants, although higher or lower concentrations can also be employed.

The completion of the esterification of the 2-(imidazolidon-2-yl-1) ethanol with the fatty acid as hereinabove described can be determined conveniently by a cessation in the formation of water that is produced during the course of the reaction. Upon completion of the esterification, the resulting 2-(imidazolidon-2-yl-1) ethyl acylate intermediate can be recovered from the crude reaction product in any suitable manner. A satisfactory procedure, for instance, comprises distilling off any remaining solvent, generally under reduced pressure, and thereafter cooling the residue to about room temperature, or somewhat lower, whereby the desired intermediate is recoverable in solid form. The product can subsequently be purified by recrystallization from a suitable solvent such as methanol. Other suitable recovery procedures can also be employed. For example, the crude product obtained upon completion of the esterification can be cooled as indicated above, without the prior distillation of any remaining solvent. The desired intermediate can then be separated in solid form; any solvent that is present remaining in the liquid phase. Moreover, the crude reaction product itself can be employed for the subsequent methylolation, thus completely obviating the necessity for utilizing a recovery procedure.

The methylolation of the 2-(imidazolidon-2-yl-1) ethyl acylate intermediate is carried out by heating an aqueous mixture or solution containing the intermediate and either formaldehyde or paraformaldehyde at a temperature of between about 20° C. and about 100° C. or slightly higher, and preferably at a temperature of between about 30° C. and about 60° C. The proportion in which the reactants are employed is not critical to the process although stoichiometric proportions are preferably used. Thus, for example, one mole of formaldehyde is required to methylolate the secondary amino-nitrogen atom contained in each mole of the 2-(imidazolidon-2-yl-1) ethyl acylate intermediate. It has been found particularly desirable to carry out the methylolation in the presence of a catalytic amount of an alkaline catalyst such as sodium hydroxide, potassium hydroxide, triethanolamine, sodium carbonate, or the like, such alkaline catalyst preferably being employed in a concentration sufficient to engender a pH value of from about 8 to about 10 in the reaction mixture. Higher or lower concentrations of catalyst can also be utilized, although increasing saponification of the resulting product may render the use of higher catalyst concentrations less desirable. In addition, a suitable solvent such as water, methanol, ethanol, etc. is generally incorporated in the reaction mixture.

Upon completion of the methylolation, the resulting 2-(3-methylol imidazolidon-2-yl-1) ethyl acylate can be recovered in any convenient manner. A suitable recovery procedure, for instance, involves neutralizing any remaining alkaline catalyst by the addition of dilute acid such as dilute sulfuric acid or dilute acetic acid, and thereafter distilling off the solvent, when present, preferably under reduced pressure. The 2-(3-methylol imidazolidon-2-yl-1) ethyl acylate product can subsequently be recovered in solid or semisolid form, i.e. as a solid, paste, gel, etc., by cooling the residue to about room temperature or somewhat lower. The crude reaction product can then

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be purified, if desired, by recrystallization from a suitable solvent such as methanol or employed directly for use as a softening agent as herein described. Generally the 2-(3-methylol imidazolidon-2-yl-1) ethyl acrylate is dispersed in water and employed as an aqueous dispersion. Such dispersions are readily dissolved or emulsified in the finishing compositions of this invention.

In further accord with this invention, the 2-(3-methylol imidazolidon-2-yl-1) ethyl acrylates prepared by the above reactions are employed as softening agents for cellulosic fibrous material by incorporating the product in a finishing composition consisting of an aqueous solution containing a thermosetting resin-forming compound, at least one of the softening agents of this invention, and preferably, an acid-producing catalyst such as those commonly utilized in conjunction with the production of thermosetting resin-forming compounds. Suitable catalysts, for example, include 2-amino-2-methylpropanol-1-hydrochloride, magnesium chloride, zinc chloride, zinc fluoroborate, monoethanolamine hydrochloride, and the like. The use of such a catalyst, however, is not essential to the invention. Of the thermosetting resin-forming compounds suitable for use in the invention, particular good results can be realized using an aminoplastic resin-forming compound such as the 1,3-dimethylol-5-alkyl-perhydrotriazones, urea-formaldehyde, melamine-formaldehyde, tetramethylol acetylene diureine or cyclic ethylene urea-formaldehyde (1,3-dimethylol imidazolidone-2), etc.

More particularly, the finishing compositions of this invention are generally comprised of from about 3 percent to about 15 percent by weight and preferably from about 5 percent to about 12 percent by weight of the thermosetting resin-forming compound, from about 0.1 percent to about 5 percent by weight and preferably from about 0.3 percent to about 2 percent by weight of at least one of the softening agents of this invention and, when the use of an acid-producing catalyst is desired, from about 0.5 percent to about 3 percent by weight and preferably from about 1 percent to about 2.5 percent by weight of said acid-producing catalyst, water constituting the principal remaining constituent of the finishing composition. It has also been found desirable to incorporate in the finishing compositions small amounts of both a surface-active wetting agent, for example, from about 0.1 percent to about 0.5 percent by weight of a compound such as an alkylphenyl polyethylene glycol ether or the like, and a bodying agent, as for instance, from about 1 percent to about 5 percent by weight of a compound such as polyvinyl alcohol, a cold water-soluble starch ether, or the like. Still other finishing agents, such as water-repelling agents, can also be incorporated in the finishing compositions in minor amounts. In addition, the proportions of the individual components of the finishing compositions of the invention can, in the light of this disclosure, be varied by those skilled in the art within somewhat broader ranges, depending upon the amounts desired to be picked-up by the material being treated. Moreover, satisfactory results can be obtained by incorporating the softening agents of the invention, preferably in amounts as hereinabove described, in any finishing composition conventionally employed to impregnate a cellulosic textile material with a thermosetting resin-forming compound, and particularly with an aminoplastic resin-forming compound.

In practice, the cellulosic fibrous material to be treated is immersed in or otherwise subjected to contact with the finishing composition of the invention until impregnated or coated therewith to a desired extent, in accordance with conventional padding operations. The treated material is then dried, generally while substantially wrinkle-free, and preferably by air at a temperature of up to about 110° C. Thereafter, the treated material, still desirably maintained substantially wrinkle-free, is cured by heating the material at a temperature of from about 130° C. to about 160° C. and preferably at a temperature of from about 145° C. to about 155° C., for a period of from

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about 1 to about 5 minutes. Other conventional drying and curing operations can also be employed. The cured material is subsequently generally neutralized and washed, especially when an acid-producing catalyst has been incorporated in the finishing composition. The neutralization can be effected, for example, by immersing the cured material in an aqueous solution containing small amounts i.e. up to about 1.0 percent by weight of sodium carbonate. The cured material is then washed with water until all traces of alkali are removed, and again dried. Ordinarily, temperatures of up to about 60° C. have been found satisfactory for use in such operation.

It is to be observed that the amount of finishing composition that impregnates the material being treated can be controlled during the padding operation. Thus, for example, by decreasing the roll pressure of the padding mangle, when such apparatus is employed, the material being treated is impregnated with increasing amounts of the finishing composition. The amount impregnated can be controlled by varying the proportions of the various components of the finishing composition. In accordance with the present invention, suitable techniques are employed to insure the impregnation of the material being treated with preferably from about 3 percent to about 15 percent and more preferably from about 5 percent to about 12 percent by weight of the thermosetting resin-forming compound, preferably from about 0.1 percent to about 5 percent and more preferably from about 0.3 percent to about 2 percent by weight of the softening agent of the invention, and, when the use of an acid-producing catalyst is desired, preferably from about 0.5 percent to about 3 percent and more preferably from about 1 percent to about 2.5 percent by weight of said acid-producing catalyst. Further, good results can be achieved by impregnating any cellulosic fibrous material with conventional amounts of a thermosetting resin-forming compound and a softening agent of this invention in an amount as herein prescribed.

The invention, and the advantages accruable in accordance therewith, can be illustrated further by the following specific examples of its practice.

EXAMPLE I

A reaction mixture consisting of 624 grams (6 moles) of hydroxyethyl ethylene diamine and 360 grams (6 moles) of urea was charged to a 5 liter 3 neck flask, stirred, and slowly heated to a maximum temperature of 200° C. until ammonia evolution essentially ceased. The crude liquid which resulted was vacuum stripped and the residue recrystallized from acetone to obtain 2-(imidazolidon-2-yl-1)ethanol is an essential quantitative yield.

EXAMPLE II

To a 500 milliliter three-neck flask equipped with a reflux condenser and decanting head, 142 grams (0.5 mole) of stearic acid, 64.5 grams (0.5 mole) of 2-(imidazolidon-2-yl-1)ethanol obtained as described in Example I and 300 grams of xylene were charged. The charge was refluxed at a temperature of 150° C. for a period of ten hours, while water slowly formed during the ensuing reaction was collected in the decanter as an azeotropic mixture with xylene. When approximately 175 grams of xylene had been removed as such azeotropic mixture, the temperature of reflux increased to 170° C. and was maintained at that level for an additional eight hours during which time further quantities of a water-xylene azeotropic mixture were collected. Upon completion of the reaction, as indicated by a cessation in the formation of water, heating was discontinued. The remaining xylene was removed by distillation at a maximum temperature of 140° C. and a pressure of about 30 millimeters of mercury. The residue was dissolved in benzene and the free stearic acid present (8 percent by weight of the residue) removed as ammonium stearate by bubbling ammonia through the benzene solution, followed by the filtration of the ammonium stearate pre-

cipitate. The benzene was then evaporated and the residue recrystallized from acetone whereby 2-(imidazolidon-2-yl-1) ethyl stearate was obtained in a 160 grams (81 percent) yield as a white powder. Analysis of the product indicated the following properties: melting point: 63° C.; elemental analysis for nitrogen; calculated for $C_{23}H_{44}N_2O_3$: 7.08 percent; found 7.04 percent. The production of the 2-(imidazolidon-2-yl-1)-ethyl stearate was also carried out in the absence of a xylene entraining agent by refluxing a mixture consisting of 156 grams (1.2 moles) of 2-(imidazolidon-2-yl-1) ethanol and 284.5 grams (1 mole) of stearic acid at a temperature of 150° C. under a pressure of 30 millimeters of mercury for a period of 15 hours and then at a temperature of 200° C. under similar pressure for an additional 5 hour period, while removing the water formed during the course of the reaction. Analysis indicated all of the stearic acid had been reacted. Upon cooling to room temperature, the 2-(imidazolidon-2-yl-1)ethyl stearate thus formed was recovered as a white solid, in an essentially quantitative yield.

Fifty grams (0.126 mole) of 2-(imidazolidon-2-yl-1)-ethyl stearate obtained as described above, 4 grams (1.33 moles based upon formaldehyde) of paraformaldehyde, 0.5 gram of sodium hydroxide and 150 ml. of methanol were charged to a 500 milliliter three-neck flask equipped with a reflux condenser and decanter head and refluxed at a temperature of 65° C. for a period of two hours. Upon completion of the reaction, the caustic was neutralized by the addition of alcoholic hydrochloric acid and the methanol solvent removed by vacuum distillation. The residue was dissolved in boiling acetone, and the insoluble precipitate which was also formed was removed by filtration. The filtrate was then cooled to a temperature of 20° C., whereupon 8.3 grams of crystalline product believed to be 2-(3-methylol imidazolidon-2-yl-1) ethanol was formed and removed by filtration. Acetone was then stripped from the filtrate by vacuum distillation at a reduced pressure below about 40 millimeters of mercury, whereupon 39 grams of 2-(3-methylol imidazolidon-2-yl) ethyl stearate was recovered as a light-yellow waxy solid, having a melting point of 46° C. When dispersed in an equal weight of water, a white paste was obtained. The product was found to be an effective softening agent in accordance with the invention.

Since the above methylolation resulted in some saponification, a reaction omitting the use of caustic was also carried out by heating a mixture containing 100 grams (0.25 mole) of 2-(imidazolidon-2-yl-1) ethyl stearate obtained as described above, 9.0 grams (0.30 mole) of paraformaldehyde and 110 grams of water with stirring at a temperature of 60° C. for a period of six hours. The reaction mixture was then cooled to room temperature whereupon 2-(3-methylol imidazolidon-2-yl-1) ethyl stearate was recovered as an aqueous paste in an essentially quantitative yield. The product was found to be an effective softening agent in accordance with the invention.

EXAMPLE III

In a manner as described in Example II 260 grams (1 mole) of a commercial grade of palmitic acid (Emersol 621; acid number: 256.3; equivalent weight: 217) and 156 grams (1.2 mole) of 2-(imidazolidon-2-yl-1)-ethanol obtained as described in Example I were heated to a temperature of 200° C. under a reduced pressure of 30 millimeters of mercury until the presence of palmitic acid could no longer be detected in the reaction mixture, while removing the water formed during the course of the reaction. Upon completion of the reaction, the product was cooled to room temperature whereupon 2-(imidazolidon-2-yl-1) ethyl palmitate was obtained in an essentially quantitative yield as a white solid.

A mixture containing 2-(imidazolidon-2-yl-1) ethyl palmitate product obtained as described above 36 grams (1.2 mole based upon formaldehyde), of paraformal-

hyde and 434 grams of water was heated with stirring at a temperature of 60° C. for a period of six hours. The reaction mixture was then cooled to room temperature whereupon 2-(3-methylol imidazolidon-2-yl-1) ethyl palmitate was recovered as an aqueous paste in an essentially quantitative yield. The product was found to be an effective softening agent in accordance with the invention.

EXAMPLE IV

In a manner as described in Example II, 200 grams (1 mole) of a commercial grade of lauric acid (Neofat 12; equivalent weight: 200.5) and 156 grams (1.2 mole) of 2-(imidazolidon-2-yl-1) ethanol obtained as described in Example I were heated to a maximum temperature of 200° C. under a pressure of 30 millimeters of mercury until the presence of lauric acid could no longer be detected, while removing the water formed during the course of the reaction. Upon completion of the reaction the product was cooled to room temperature whereupon 2-(imidazolidon-2-yl-1) ethyl laurate was obtained in an essentially quantitative yield as a white solid.

A mixture containing 340 grams (1 mole) of the 2-(imidazolidon-2-yl-1) ethyl laurate obtained as described above, 36 grams (1.2 mole based upon formaldehyde) of paraformaldehyde and about 370 grams of water was heated with stirring at a temperature of 60° C. for a period of six hours. The reaction mixture was then cooled to room temperature whereupon 2-(3-methylol imidazolidon-2-yl-1) ethyl laurate was recovered as an aqueous paste in an essentially quantitative yield. The product was found to be an effective softening agent in accordance with the invention.

EXAMPLE V

Finishing compositions were prepared containing the following: 1,3-dimethylol imidazolidone-2, 7.5 percent by weight; 2-amino-2-methyl-propanol-1 hydrochloride, 0.5 percent by weight; various amounts of different softening agents as indicated below in Table A; 0.24 percent by weight of an alkyl phenyl polyethylene glycol ether wetting agent, and 5 percent by weight of a cold water-soluble starch ether bodying agent, the remaining component being water. Samples of cellulosic textile material were then treated with the finishing compositions and tested by the procedures described below. The results obtained are tabulated below in Table A.

Treating procedure

The fabric used was desized percale sheeting having a 96 x 90 (warp-fill) count. Numbered sheets each measuring 10 in. x 24 in. cut in the warp direction were conditioned to constant weight at a temperature of 70° F. and 65 percent relative humidity. After immersion in the finishing composition at 50° C. for 2.5 minutes, the sheets were put through a hand wringer and then reimmersed to the finishing composition for another 2.5 minutes. A second pass was made through the wringer and the specimens were allowed to air-dry, ironed at a low heat to remove wrinkles, oven-dried at 110° C. for 10 minutes and cured at 150° C. for five minutes. After washing with a 0.5 percent sodium carbonate solution, the sheets were given three cold water rinses and a boiling water rinse. The sheets were then brought again to constant weight at a temperature of 70° F. and 65 percent relative humidity.

Test methods

Tear strength.—The tear strength was determined according to Federal Specification CCC-T-191b test method 5132. Three specimens, each measuring 4 inches x 2.5 inches, with the long dimension parallel to the warp direction, were cut from each sheet. By means of an Elmendorf Tear Tester, the force in grams required to tear through 43 mm. of fabric was obtained in triplicate. The averages of the values are recorded below in Table A.

Chlorine retention.—Damage caused by retained chlorine was determined according to the AATCC tentative test method 69—52. Six specimens, each measuring 4 inches x 6 inches were cut from each sheet with the long dimension parallel to warp direction. The test specimens were held in boiling water for three minutes and then three of the specimens were immersed for 15 minutes in a weight of test solution (containing .25 percent available chlorine) equal to 50 times the weight of the specimen. The control specimens and treated specimens were given six fresh water rinses and then dried by ironing at a rayon heat for approximately 10 seconds. After conditioning at 65 percent humidity and 70° F. for 24 hours, the control and treated specimens were placed in a

in Example II above, softening agent No. 2 is a conventional glyoxalidine softening agent, 1-(2-hydroxyethyl)-2-heptadecyl imidazoline. Also included in the table for comparison are data obtained from runs in one of which water was substituted for the finishing composition (untreated) and in another of which no softening agent was employed (control). As further tabulated, the concentration of softening agent in the finishing composition is indicated in percent by weight; the dry pick-up represents the amount of finishing composition retained by the treated material after drying and curing and is indicated in percent by weight of the fabric; the amount based upon the weight of softening agent on the fabric is similarly indicated.

TABLE A

Softening Agent Employed	Concentration of Softening Agent in Finishing Composition	Dry Pick-Up	Amount of Softening Agent on Fabric	Tear Strength	Chlorine Retention (Breaking Strength)		Crease Resistance (Percent Angle of Recovery)	Discoloration (Reflectance Deviation)
					Control	Bleached		
Untreated.....				1,109	30.8	24.8	49.5	—1.0
Control.....				433	19.0	12.3	81.8	—0.5
No. 1.....	0.5	4.85	0.29	544	17.1	11.1	72.7	—1.0
No. 1.....	1.0	5.93	0.61	656	17.5	12.5	76.0	—1.0
No. 1.....	2.0	6.47	1.60	688	17.8	16.5	70.0	—1.0
No. 2.....	0.5	4.45	0.21	603	16.7	7.0	75.5	—3.0
No. 2.....	1.0	7.90	0.69	587	17.7	10.8	80.0	—4.5
No. 2.....	2.0	9.28	1.50	613	17.8	9.8	76.0	—6.0

Scorch Tester (type SO-3, manufactured by the Atlas Electrical Devices Company, Chicago, Illinois) at 365° F. for 30 seconds. The actual damage was determined by recording the breaking strength in kilograms of each specimen with an Amthor Tensile Tester, (manufactured by the Amthor Testing Instrument Co., Brooklyn, New York). The average values of the breaking strengths of both the control and treated specimens are recorded below in Table A.

Crease-resistance.—Crease-resistance was determined by measuring the angle of recovery according to Federal specification CCC-T-191b test method 5212. Specimens, each measuring 4 cm. x 1.5 cm.; cut parallel to both the warp and fill direction were placed into a specimen holder to produce a fold 1.8 cm. from the free end. The specimens were subjected to a 1.5 pound weight for five minutes and then allowed to recover for five minutes while held on a protractor. The percent angle of recovery in the warp direction is recorded below in Table A.

Several of the advantages accruable in accordance with the invention, viz. improved tear strength accompanied by less chlorine retention and discoloration, can readily be seen from the above table.

EXAMPLE VI

In a manner similar to that described above in Example V, finishing compositions were prepared containing different softening agents of this invention in varying amounts. In Table B below, softening agent No. 1 is 2-(3-methylol imidazolidon-2-yl-1)ethyl stearate produced as described in Example II above, softening agent No. 2 is the corresponding palmitate, produced as described in Example III above, and softening agent No. 3 is the corresponding laurate produced as described in Example IV above. These finishing compositions were employed to treat cellulosic textile material essentially as described above in Example V, and the treated material tested as further described therein.

TABLE B

Softening Agent	Amount of Softening Agent in Finishing Composition	Dry Pick-Up	Amount of Softening Agent on Fabric	Tear Strength	Crease Resistance (Percent Angle of Recovery)	Discoloration (Reflectance Deviation)
Untreated.....				1,032	46.1	—0.5
Control.....		4.25		432	80.6	—1.0
No. 1.....	0.5	4.95	0.25	496	80.6	—0.5
No. 1.....	1.0	6.45	0.61	528	81.6	—0.5
No. 1.....	2.0	6.90	1.20	565	81.1	—1.5
No. 2.....	0.5	5.60	0.28	496	79.4	—0.5
No. 2.....	1.0	6.60	0.63	533	77.7	—0.5
No. 2.....	2.0	9.50	1.65	560	78.3	—1.0
No. 3.....	0.5	5.70	0.29	496	77.7	—1.0
No. 3.....	1.0	6.40	0.62	512	76.7	—1.0
No. 3.....	2.0	8.60	1.49	544	77.2	—1.0

Discoloration.—Specimens, each measuring 4 inches x 6 inches, were subjected to accelerated aging conditions in an oven at a temperature of 100° C. for a period of 96 hours. The amount of yellowing was determined by measuring the reflectances on a Photovolt Reflectometer (Model 610) before and after aging. A deviation of —1.0 indicates a trace of discoloration, a deviation of —2.0 indicates noticeable yellowing, while a deviation of —3.0 or more indicates considerable yellowing.

In the table softening agent No. 1 is 2-(3-methylol imidazolidon-2-yl-1)ethyl stearate, produced as described

EXAMPLE VII

In a manner similar to that described above in Example V, finishing compositions were prepared containing different softening agents in varying amounts. In Table C below, softening agent No. 1 is 2-(3-methylol imidazolidon-2-yl-1)ethyl stearate produced as described above in Example II, softening agent No. 2 is a commercially available cationic softening agent. These finishing compositions were employed to treat cellulosic textile material essentially as described above in Example V and the treated material tested as further described therein.

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TABLE C

Softening Agent	Amount of Softening Agent in Finishing Composition	Dry Pick-Up	Amount of Softening Agent on Fabric	Tear Strength	Crease Resistance (Percent Angle of Recovery)	Discoloration (Reflectance Deviation)
Untreated Control		5.85		1,056	47.7	-1.0
No. 1	0.5	6.05	0.30	416	82.2	-1.0
No. 1	1.0	6.85	0.65	485	83.9	-1.0
No. 1	2.0	8.45	1.47	528	83.9	-1.0
No. 2	0.5	6.14	0.31	544	83.3	-1.5
No. 2	1.0	6.33	0.60	469	82.2	-3.0
No. 2	2.0	7.02	1.24	485	81.7	-3.0
				491	81.7	-4.0

EXAMPLE VIII

To test the permanence of the softening agents of the invention, finishing compositions were prepared as described above in Example V. Run No. 1 employed a finishing composition containing no softening agent; run No. 2 employed a finishing composition containing 2 percent by weight of 2-(3-methylol imidazolidon-2-yl-1)ethyl stearate produced as described above in Example II. The finishing compositions were employed to treat cellulosic textile material essentially as described above in Example V using ten specimens of cotton percale sheeting with each finishing composition. After calculating the amount of finishing composition retained by the treated material (ad-on) samples were cut from the first specimen of each series and subjected to the Elemendorf Tear Test as described above in Example V and the Monsanto Crease-Recovery Test as described in Monsanto Technical Service Bulletin T 30-45.1, published by the Monsanto Chemical Co., Springfield, Mass. All of the specimens were placed in a General Electric Filter-Flo washer, given four short cycle washings, allowed to air-dry, ironed, again brought to constant weight and weighed. This process was repeated after 8, 16, and 24 washings. The short cycle washing consisted of a three-minute interval for filling with 17 gallons of water at 48° C., a four-minute agitation with about 100 grams of a commercially available detergent (Tide), a three-minute interval during which the wash water was removed, a three-minute interval for filling with 17 gallons of rinse water at 38° C., a two-minute rinse agitation and a three-minute damp-dry spin. The results obtained are indicated below in Table D. In the table the average weight of the fabric specimens and the dry pick-up is indicated in grams.

TABLE D

Run No.	Average Fabric wt.	Average Dry Pick-Up	Tear Strength	Crease Resistance (Percent Angle of Recovery)
(After padding, drying and curing)				
1	19.664	0.947	432	77.7
2	20.743	1.112	645	83.3
(After four washings)				
1	0.027	2.85	437	78.8
2	0.119	10.60	640	83.3
(After eight washings)				
1	0.057	6.02	432	77.7
2	0.155	13.93	640	80.0

(After sixteen washings)

	Average Weight Loss	Percent Loss of Dry Pick-Up	Tear Strength	Crease Resistance (Percent Angle of Recovery)
1	0.085	8.90	448	77.2
2	0.174	17.26	640	80.0

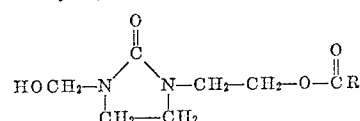
(After twenty-four washings)

	Average Weight Loss	Percent Loss of Dry Pick-Up	Tear Strength	Crease Resistance (Percent Angle of Recovery)
1	0.099	10.45	461	77.2
2	0.192	17.26	667	80.0

In addition, the hand of the cellulosic textile material treated in accordance with the invention as described above was evaluated objectively by manually feeling the fabrics and visually observing their drape. In all cases, an improved hand was found for the material treated in accordance with the invention when compared with the control fabric (no softening agent). This improved hand was still evident after twenty-four washings.

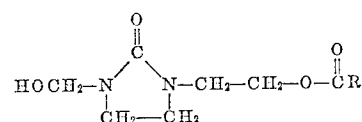
What is claimed is:

1. As new compositions of matter, 2-(3-methylol imidazolidon-2-yl-1)ethyl acylates of the formula



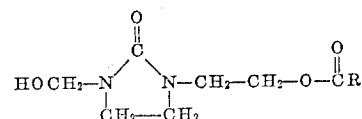
wherein R designates a monovalent aliphatic radical selected from the group consisting of the alkyl and alkenyl radicals of from 8 to 22 carbon atoms.

2. As new compositions of matter, 2-(3-methylol imidazolidon-2-yl-1)ethyl acylates of the formula



wherein R designates an alkyl radical of from 12 to 22 carbon atoms.

3. As new compositions of matter, 2-(3-methylol imidazolidon-2-yl-1)ethyl acylates of the formula



wherein R designates an alkenyl radical of from 12 to 22 carbon atoms.

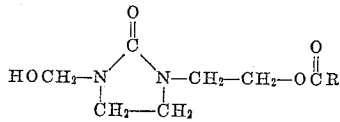
4. As a new composition of matter, 2-(3-methylol imidazolidon-2-yl-1)ethyl stearate.

5. As a new composition of matter, 2-(3-methylol imidazolidon-2-yl-1)ethyl laurate.

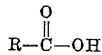
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6. As a new composition of matter, 2-(3-methylol imidazolidon-2-yl-1)ethyl palmitate.

7. A process for the production of a 2-(3-methylol imidazolidon-2-yl-1) acylate of the formula



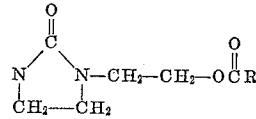
wherein R designates a monovalent aliphatic radical selected from the group consisting of the alkyl and alkenyl radicals of from 8 to 22 carbon atoms, which process comprises the steps of heating a mixture of an aliphatic carboxylic acid of the formula



wherein R is as defined above, with 2-(imidazolidon-2-yl-1) ethanol, at a temperature of from about 30° C. to

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about 200° C., thereby producing a 2-(imidazolidon-2-yl-1)ethyl acylate of the formula



wherein R is as defined above, and subsequently heating a mixture of said 2-(imidazolidonyl-2-yl-1)ethyl acylate with a methylolating agent selected from the group consisting of formaldehyde and paraformaldehyde, at a temperature of from about 20° C. to about 100° C.

8. The process according to claim 7 wherein R designates an alkyl radical of from 12 to 22 carbon atoms.

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