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IMPROVING THE WASH-FASTNESS OF SIZES WITH UREA DERIVATIVES

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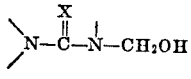
8 Claims. (Cl. 117—161)

This invention relates to the insolubilizing of polyhydroxy sizing materials. In particular, it deals with the fixation on textile materials of sizing materials such as starch, cellulose ether, and polyvinyl alcohol by means of certain urea derivatives.

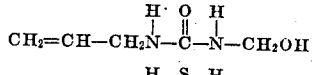
Sizing materials are widely used to impart bulk, stiffness, or strength to yarns and fabrics. But these properties are readily lost when fabric is washed. For many purposes it would be desirable to stiffen fabric permanently or to size textile materials permanently.

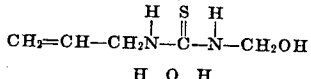
It is an object of this invention to provide a means of stiffening textile materials permanently at the same time retaining the usual appearance of the sized materials. It is an object of this invention to improve the wash-fastness of sizes of hydroxyl containing materials of high molecular weight. It is also an object to provide sizing compositions which are wash-resistant.

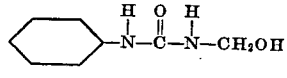
It has been found that certain compounds possessing the structure—

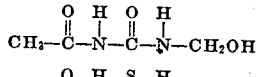


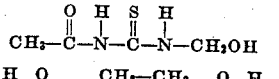
react with polyhydroxy sizes to render such sizes relatively insoluble and, therefore, resistant to removal by washing. In the above structure N, C, H, and O have their usual chemical significance and X represents oxygen, sulfur, or nitrogen in an imino grouping. We have found that a wide variety of compounds possessing the essential grouping shown react with starch, polyvinyl alcohol, and water-soluble cellulose ethers at moderately elevated temperatures and give desirable effects if they contain more than three carbon atoms per molecule. As typical of these compounds, the following are cited—

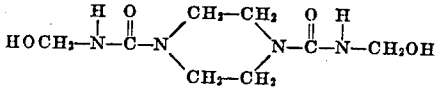


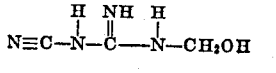


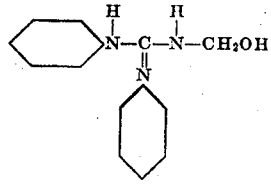




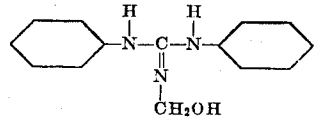




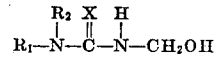




which may also exist in the tautomeric form



These compounds may be represented by the following general formula:



These compounds are monomethylol derivatives or formaldehyde addition products of urea, thiourea, or guanidine, X in the formula thus representing oxygen, sulfur, or nitrogen in an imino grouping. One of the nitrogens of these derivatives is substituted by a group selected from acyl, unsaturated hydrocarbon, aryl, and cyano groups, represented by R_1 in the formula, in which case R_2 is hydrogen, and heterocyclic groups formed with the said nitrogen, wherein R_1 and R_2 are together members of a divalent hydrocarbon group joined to the nitrogen atom.

These compounds react at mildly elevated temperatures with polyhydroxy-containing compounds to produce relatively insoluble reaction products or complexes.

In the preferred application of sizing materials to textile fabrics, yarns, and the like, an aqueous sizing bath is prepared in the usual way and to this bath is added a compound of the type shown above. The preferred compositions contain about 2 parts of such a compound to about 1 to 10 parts of sizing material. Although these urea derivatives are frequently insoluble in water, they are easily dispersed in the sizing bath. There may also be added to the bath an acidic or alkaline catalyst such as acetic acid, ammonium chloride, sodium carbonate, or bicarbonate, borax, etc.

The sizing material is then applied to yarn or fabric by any of the usual sizing methods, such as dipping, or spreading, followed by squeezing with rolls or scraping with a doctor knife. The treated yarn or fabric may then be dried at 150–210° F., if desired, or it may be passed directly into apparatus heated sufficiently hot to dry and react the fixing agent and size. In either case a temperature above 212° F. is necessary to cause reaction. The temperature needed depends upon the particular compound used and the time avail-

able for heating the treated fabric. At 250° F. fifteen minutes or more usually suffices to set the size. At higher temperatures shorter times are effective. The heating may be accomplished in loop driers or closed tenters, or on heated cans.

Variations in the above procedure may be made. The textile material may be impregnated with size and with the fixation agent in separate steps. Also, a ready-to-use composition may be made which contains both size and methylol urea derivative in proper proportions ready for use in the same manner as any prepared size.

After the size has been set by heat, it is often desirable to wash the treated textile material to remove salts, unreacted material, or soluble products resulting from the fixation reaction. Washing, however, is not essential. Fabric may finally be finished in the usual way for starched goods.

The process of insolubilizing sizes or improving the fastness of water-dispersible sizes herein disclosed may be applied to yarn, fabric, thread, or fibers of any material which may be heated over 100° C. without damaging the treated material. The process is thus applicable to such materials as cotton, linen, paper, straw, jute, hemp, the various synthetic fibers, etc.

The sizing materials which are useful in this process are those high molecular weight compounds containing a multiplicity of hydroxyl groups. Without doubt, the water-soluble or water-dispersible properties of these sizes are due to the hydroxyl groups, which can react with such compounds as the hydroxymethyl urea derivatives herein shown useful for fixing sizes. The useful sizing agents include cellulose ethers, such as water-soluble methyl or ethyl cellulose, hydroxyethyl cellulose and the like, polyvinyl alcohol, and starch, such as corn, rice, tapioca, sago, wheat, or potato starch, flours which contain a high percentage of starch, and the modified forms of starch. The size may be modified with fillers, pigments, softeners, resins, etc.

While the compounds herein disclosed are applicable for the processes outlined, they give somewhat different effects depending upon their molecular weight and molecular arrangement. Relatively small molecules give stiffer and harsher finishes than those of larger molecular size. The compounds containing long chain substituents give soft, smooth finishes and a certain degree of water-repellency may be secured. Stiffness of finish will, of course, also depend upon the kind, type and amount of sizing agent used.

Typical uses of these hydroxymethyl urea derivatives in the process of sizing fabrics are illustrated by the following examples.

Example 1.—Cotton sheeting was coated with a composition consisting of 20 parts of 7.5% aqueous hydroxyethyl cellulose solution (high viscosity), 1 part methylol allyl thiourea, and 0.25% of soda ash. It was heated at 265° F. for ½ hour and washed. A very stiff finish resulted which was not appreciably softened on subsequent washings.

Example 2.—Cotton lawn was impregnated with a coating paste composed of 10 parts of 7.5% aqueous hydroxyethyl cellulose, 1 part of methylol phenyl thiourea in 25% alcoholic solution, and 0.2% soda ash. It was dried, ironed for 1¼ minutes with a medium-hot iron and washed. The finish was very stiff and fast to washing.

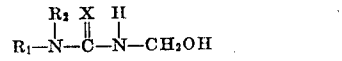
Example 3.—Rayon fabric was coated with a composition containing 7.6% potato starch, 4.75% methylol acetyl thiourea, and 0.2% soda ash. It was dried, heated ½ hour at 265° F., and washed. A good, permanent stiffness resulted.

Example 4.—Cotton sheeting was coated with a suspension of 5 parts monomethylol cyanguanidine in 100 parts of 8% potato starch containing 0.25% soda ash. It was air-dried, heated at 265° F. for ½ hour, and given a thorough laundering. The stiff finish was retained.

Example 5.—Cotton fabric was impregnated with an aqueous paste comprising 7.25% tapioca starch, 2.27% of the methylol compound resulting from the reaction of N,N'-dicarbamido-piperazine with 2 mols formaldehyde, and 0.3% soda ash. It was heated ½ hour at 265° F. and washed. A very stiff finish resulted, fast to further washing.

We claim:

1. A process of treating textile material to impart thereto a substantially wash-resistant size finish which comprises coating the material with an aqueous sizing preparation containing a formaldehyde addition product of the general formula:



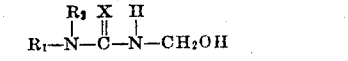
and a hydroxyl-containing size of the group consisting of starch, water-soluble cellulose ethers, and polyvinyl alcohol, and subsequently heating the coated material to a temperature in excess of 212° F., thereby to effect reaction between the formaldehyde addition product and the size, said addition product being the monomethylol derivative of a member of the group consisting of urea, thicurea, and guanidine, X in the formula thus representing a member of the group consisting of oxygen, sulfur, and nitrogen in an imino grouping, said derivative being substituted on a nitrogen atom with a group selected from acyl, unsaturated hydrocarbon, aryl, and cyano groups, represented by R_1 in the formula, in which case R_2 is hydrogen, and heterocyclic groups formed with the said nitrogen, wherein R_1 and R_2 are together members of a divalent hydrocarbon group joined to the nitrogen atom.

2. The process of claim 1 in which the size is starch.

3. The process of claim 1 in which the size is polyvinyl alcohol.

4. The process of claim 1 in which the size is a water-soluble cellulose ether.

5. Textile material carrying as a substantially water-insoluble size a complex reaction product formed by heating above 212° F. a hydroxyl-containing size of the group consisting of starch, water-soluble cellulose ethers, and polyvinyl alcohol with a formaldehyde addition product of the general formula:



said addition product being the monomethylol derivative of a member of the group consisting of urea, thiourea, and guanidine, X in the formula thus representing one of the group consisting of oxygen, sulfur, and nitrogen in an imino grouping, said derivative being substituted on a nitrogen atom with a group selected from acyl, unsaturated hydrocarbon, aryl, and cyano groups, represented by R_1 in the formula, in which case R_2 is hydrogen, and heterocyclic groups formed with the said nitrogen, wherein R_1 and R_2 are together members of a divalent

hydrocarbon group joined to the nitrogen atom.

6. The textile material of claim 5 on which the complex reaction product is formed from starch.

7. The textile material of claim 5 on which the complex reaction product is formed from polyvinyl alcohol.

8. The textile material of claim 5 on which the complex reaction product is formed from water-soluble cellulose ether.

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