Acid Resistance of Aldehyde Finishes on Cotton

INTRODUCTION

We have reported previously on the ability among various aldehydes to crosslink cellulose in cotton.[1] In continuing this work, it was found that the few aldehydes with appreciable reactivity to cotton gave crosslinks with noticeable differences in their resistance to acid hydrolysis. Data on this acid resistance may be helpful in defining the chemical structures formed by aldehydes and cellulose.

EXPERIMENTAL

Aldehydes and other agents were applied to cotton printcloth by padding an 8–12% solution containing 1.8–2.0% $MgCl_2 \cdot 6H_2O$, except that 1.0% $Zn(BF_4)_2$ was used with 10% dimethoxytetrahydrofuran. Fabric was then dried for 7 min at 70°C, cured for 3 min at 160°C, and washed. Wrinkle recovery angles of the fabrics were determined by the AATCC Recovery Angle Method. Fabric samples were hydrolyzed in 0.1*N* or 0.5*N* hydrochloride acid for 30 min at 40°C and then rinsed and dried. Retention of the wrinkle recovery angle of the washed fabric through hydrolysis was taken as indicating retention of the finish or crosslinks on the cotton. No other measure of crosslinking was found suitable to detect changes at the levels of crosslinking involved. Durable press ratings, as used in the earlier work, supported conclusions drawn from wrinkle recovery angles but were less precise.

RESULTS AND DISCUSSION

In Table I are wrinkle recovery angles of fabric after treatment with crosslinking agents and after treatment and acid hydrolysis. The agents include three aldehydes that are reactive to cotton—formaldehyde, glyoxal, and glutaraldehyde (pentanedial). Also included are 2,5-dimethoxytetrahydrofuran, a reactive acetal, and, for comparison, dimethyloldihydroxyethyleneurea [4,5-dihydroxy-1,3-bis(hydroxymethyl)-2-imidazolidinone], a common amide-formaldehyde agent that gives a relatively acid-resistant finish. The agents are arranged in the table in the order of decreasing acid resistance in the finish.

Formaldehyde is believed to make a methylene crosslink. Glutaraldehyde has been proposed to give a substituted methylene crosslink.[3] Glyoxal is likely to give a substituted methylene crosslink also. In such structures the crosslink from glutaraldehyde is a methylene group with a relatively nonpolar substituent containing two hydrogens on the α-carbon. The crosslink from glyoxal is a methylene group with a polar substituent containing a single hydrogen on the α-carbon. From data on the hydrolysis of ethyl acetals,[4] these structures would be predicted to give acid resistance in the observed order, that is, a little less resistance with glyoxal than with formaldehyde and decidedly less resistance with glutaraldehyde.

TABLE I
Wrinkle Recovery of Treated Cotton Printcloth

Finishing agent	Concentration applied (% ows)	Wrinkle recovery angles (w + f) (°)		
			After hydrolysis	
		Original	0.1*N* acid	0.5*N* acid
Formaldehyde	8	275	273	275
Glyoxal	10	258	254	251
Dimethyloldihydroxyethyleneurea	12	283	267	236
Glutaraldehyde	12	284	256	216
Dimethoxytetrahydrofuran	10	261	225	206

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2,5-Dimethoxytetrahydrofuran reacted appreciably with cotton only when applied from a pad bath with low pH. This suggested that the active agent is succinaldehyde from hydrolysis of the hemiacetal anhydride. However, succinaldehyde would be expected to give a finish with acid resistance as high or higher than that from glutaraldehyde. The low acid resistance suggests that the dimethoxytetrahydrofuran formed a crosslink with a different structure than that formed by aldehydes. With dimethoxytetrahydrofuran, two functional groups may react with cellulose to form crosslinks with an ethylene or tetrahydrofuran group between cellulose chains.

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