Acetylation of Jute: Effects on Strength, Rot Resistance, and Hydrophobicity

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Synopsis

Jute cloth was acetylated with a limited amount of liquid acetic anhydride at 120°C. Weight gains of up to 20% were obtained. At a weight gain of 11%, achieved after a reaction time of 2 h, resistance to fungal attack was greatly improved. The cloth was also even in color, and the reduction in strength was minor. Tests on equilibrium moisture content and water uptake showed that the acetylated jute was much more hydrophobic than the unmodified material. It also shrank less when leached in water. Hence, dimensional stabilization was obtained.

INTRODUCTION

Jute is a material much used for transport and storage of food, especially in the developing countries. It is also a material susceptible to decay, and large quantities of food are destroyed due to deteriorated storage sacks. However, conventional preservatives cannot be used since they would contaminate the food.

Jute is also used in interior decoration products, e.g., as a carrier for linoleum and plastic floor coverings and instead of wallpapers. In these applications its swelling and shrinkage upon moisture sorption and desorption is a disadvantageous property.

Acetylation of wood has been shown to reduce both its dimensional instability and susceptibility to decay.[1] There are similarities in the chemical composition of wood and jute, i.e., both are lignocellulosic materials, although the cellulose content of jute (approximately 73%) is considerably higher than that of wood, and the lignin content is lower (approximately 13%). Jute also contains 13% hemicellulose and minor amounts of other components such as pectin and extractives.[2]

The purpose of this study was to investigate the effects of acetylation on rot resistance, hydrophobicity, and strength properties of jute. A simplified acetylation procedure, employing neither a catalyst nor an organic cosolvent, but only liquid acetic anhydride in a limited amount was used.[1,3]

Acetylation of jute has previously been studied, for example by Callow,[4] who primarily investigated discoloration of jute by light, and also the effect of acetylation on strength and resistance to abrasion.

Journal of Applied Polymer Science, Vol. 37, 3437–3447 (1989)
 CCC 0021-8995/89/123437-11$04.00

EXPERIMENTAL

Acetylation

Samples of jute cloth, 1 × 1 m, were oven-dried and weighed. The cloth was folded and put into a stainless steel mesh basket, which was dipped into acetic anhydride for 1 min. The basket was allowed to drain for 3 min before being placed in a preheated (120°C) reactor of stainless steel (volume 7 L). The reaction time was varied from 5 min to 24 h. After the reaction was completed, a vacuum was applied for 2 h to remove excess acetic anhydride and byproduct acetic acid. The cloth was again oven-dried and weighed and the weight gain due to acetylation was calculated as a percentage based on the weight of the oven-dried, unmodified material.

The amount of acetic anhydride taken up by the material after dipping and draining was determined by weighing. A small series of acetylations was carried out at a reaction temperature of 90°C. Acetylation with mixtures of acetic anhydride and acetic acid was also performed at 120°C.

Tests of Properties of Acetylated Jute

Samples of jute cloth with varying weight gains were placed in a fungus cellar in moist unsterile soil at approximately 25°C. White, brown and soft rot fungi and tunneling bacteria were present in the soil. The samples were inspected after 2, $5\frac{1}{2}$, and 8 months.

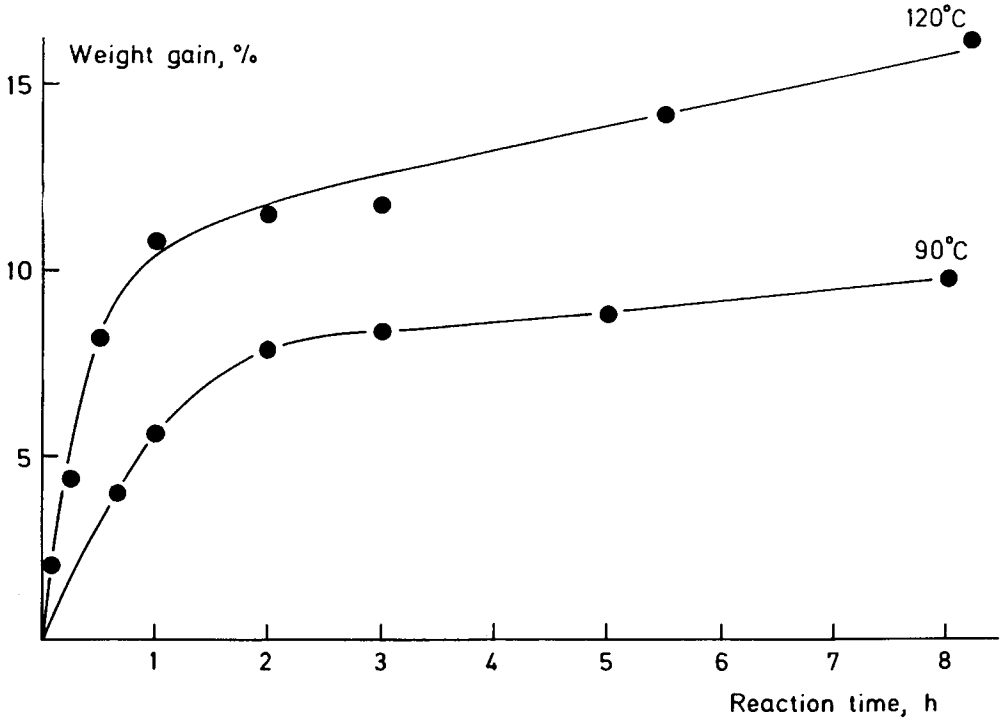

Fig. 1. Weight gain vs. reaction time for acetylation of jute at 90 and 120°C.

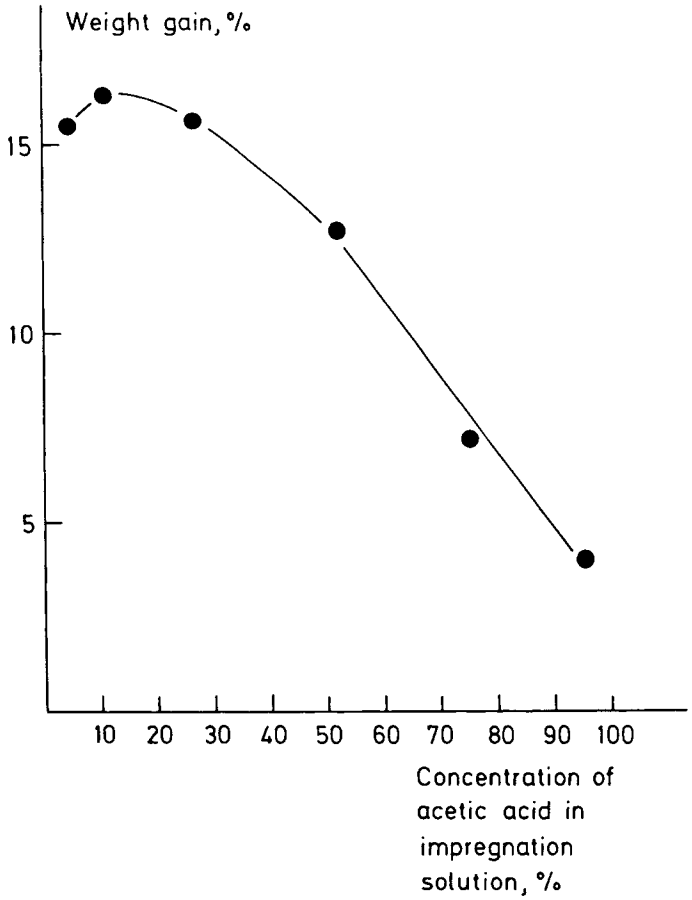

Fig. 2. Weight gain vs. concentration of acetic acid in impregnation solution for acetylation of jute at 120°C for 5 h.

TABLE I
Results from Fungus Cellar Test of Acetylated Jute with Varying Weight Gains

Reaction temperature (°C)	Weight gain (%)	Number of Specimen	Average rating[a] after (months)		
			2	$5\frac{1}{2}$	8
	Control	10	3	—[b]	—
90	4.0	5	2	3[c]	—
90	5.6	5	1.8	3	—
120	8.3	5	0.4	2.6	—
120	11.5	5	0	1.8	3
120	14.2	5	0	0.2	0.6
120	16.2	5	0	0	0

[a]Rating: 0 = no attack, 1 = slight attack, 2 = heavy attack, 3 = destroyed.
[b]Samples disintegrated.
[c]Three of the samples disintegrated.

Strength tests of acetylated jute were performed according to Swedish Standard SS 25 12 67 (ISO 5081-1977). The apparatus used was an Instron 1122 CRT (constant rate of traverse). Samples measuring 70 × 320 mm, including a 10 mm fringe in each side, were cut out of jute cloth with varying weight gains. Testing length in the machine was 200 mm. Tests were also done on the yarn originating from the web. Dry strength as well as wet strength was examined and the strength was measured in both the warp and the weft direction.

The hydrophobicity of the jute was tested as equilibrium moisture content and water uptake. The latter was performed by laying samples of acetylated jute cloth in deionized water, allowing them to sink and then draining them shortly, after which they were weighed. The equilibrium moisture content was determined at different relative humidities (RH), placing samples of acetylated jute cloth in a climate cabinet and weighing after equilibrium was obtained.

Jute cloth was soaked in deionized water, to study shrinkage. Two cycles of 2 days in water followed by air drying and oven drying were executed. Shrinkage was measured in both the directions of the warp and the weft.

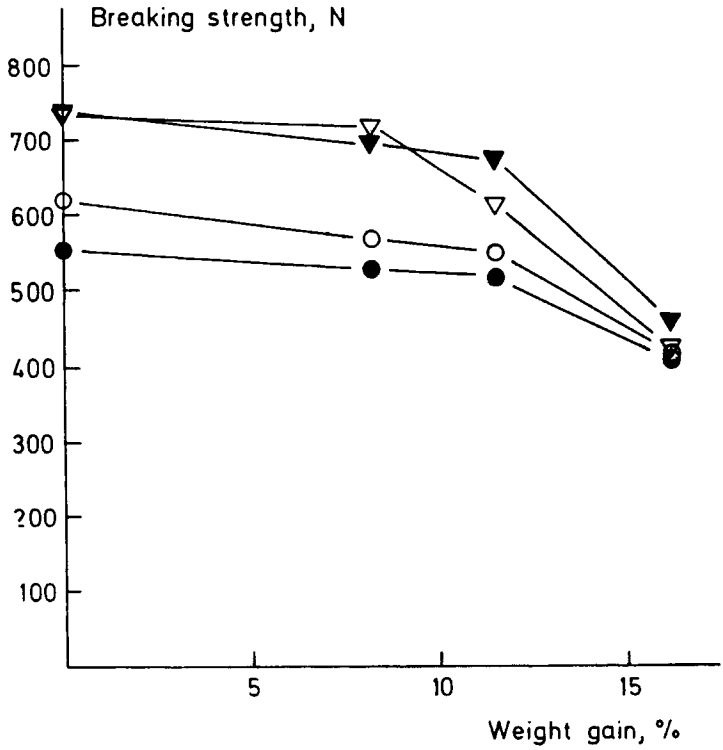

Fig. 3. Dry and wet breaking strength of acetylated jute cloth vs. weight gain, weft and warp direction: (○) dry strength, warp; (▽) dry strength, weft; (●) wet strength, warp; (▼) wet strength, weft.

RESULTS AND DISCUSSION

The uptake of acetic anhydride after dipping and draining was determined as 120%. This is a large excess of acetic anhydride, since it is sufficient for a maximum weight gain of 50%, assuming that no acetic anhydride is lost by hydrolysis due to water present in the jute, and taking into account the byproduct acetic acid formed in the acetylation.

The result of the acetylation can be seen in Figure 1. At 120°C a weight gain of about 11% was obtained after a reaction time of 1 h, while at 90°C the weight gain was about 10% after an 8-h reaction time. After 24 h the weight gain was 20%, using a reaction temperature of 120°C. The cloth became patchy when acetylated at 120°C for 5 h or more, probably due to the long residence time at an elevated temperature.

Figure 2 shows how the reaction rate was influenced by acetic acid present in the impregnation solution. Up to 30 w % of acetic acid did not affect the

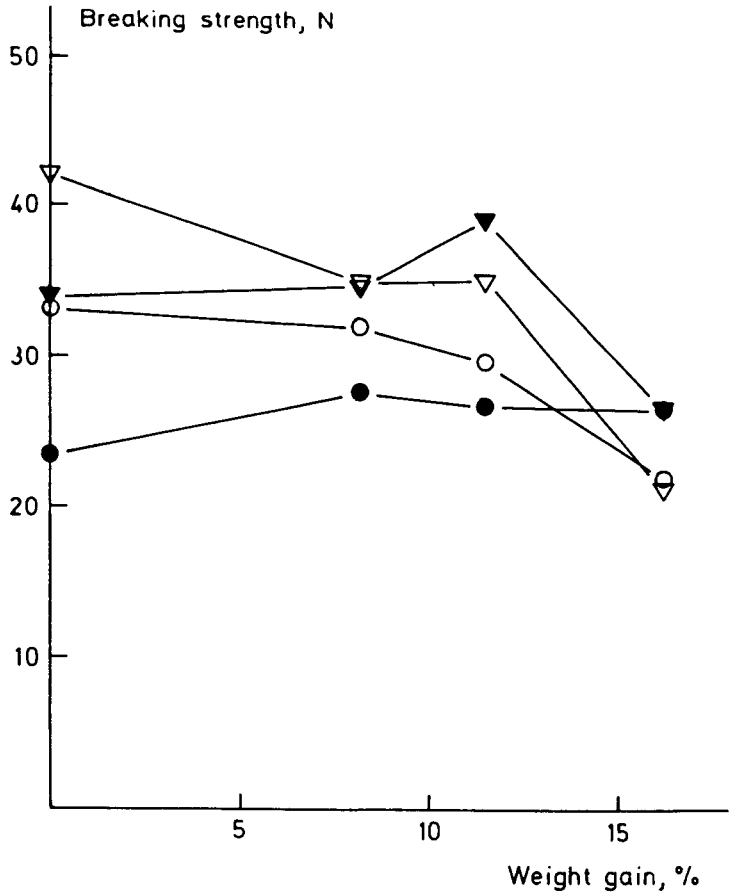

Fig. 4. Dry and wet breaking strength of yarn from acetylated jute cloth of varying weight gain, weft and warp direction: (○) dry strength, warp; (▽) dry strength, weft; (●) wet strength, warp; (▼) wet strength, weft.

weight gain considerably. A maximum in weight gain at 10–20% acetic acid was observed. This was probably due to the swelling effect of acetic acid, allowing for easier penetration of acetic anhydride into the fiber. At higher concentrations of acetic acid the dilution of acetic anhydride led to a decreased reaction rate.

Acetylated jute cloth is resistant to fungal attack, as can be seen in Table I. A weight gain of 11% resulted in no attack after 2 months and very little attack after 5 months. However, after 8 months the samples were heavily attacked. A 16% weight gain was required for the jute to totally withstand 8 months in the fungus cellar, but also at a 12–14% weight gain the biological resistance increased markedly.

The mechanism for protection of lignocellulosic materials against microbiological degradation through chemical modification, e.g., acetylation, is not completely understood. It is believed to be due to two factors: Greatly decreased moisture sorption in the cell walls and blocking of the hydroxyl groups of the cell wall components in such a way that the enzymes of the

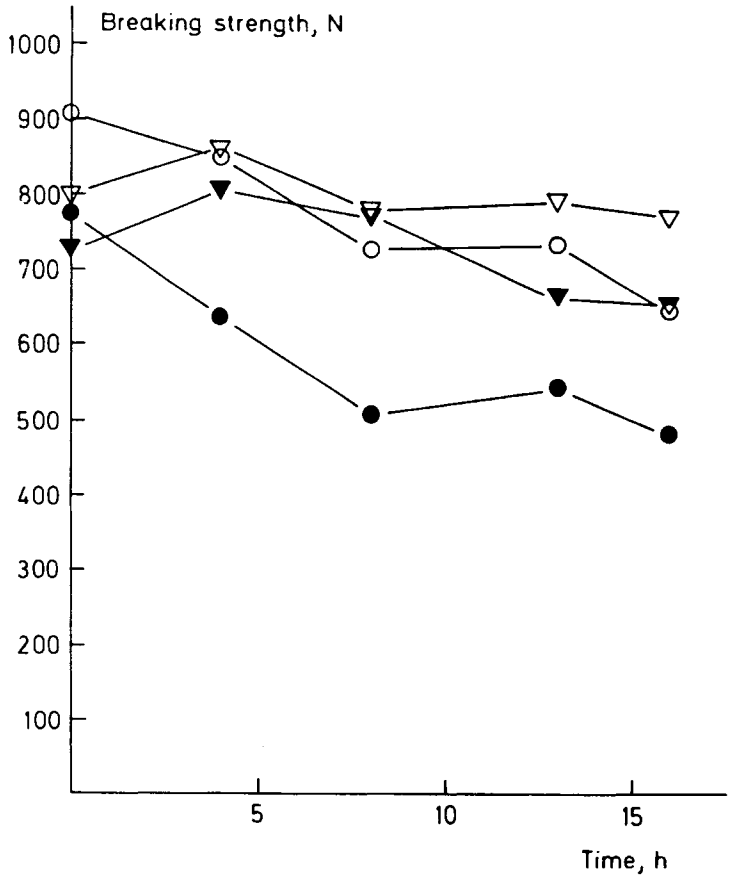

Fig. 5. Dry and wet breaking strength of oven-dried, unmodified jute cloth vs. time of oven-drying at 120°C: (○) dry strength, warp; (▽) dry strength, weft; (●) wet strength, warp; (▼) wet strength, weft.

wood degrading microorganisms cannot recognize them as attackable substrates.

Figure 3 shows that the strength of the web was reduced due to acetylation. At a weight gain of 11% the reduction in dry strength was only 11% in the warp direction and 16% in the weft direction, while at a weight gain of 16% the strength reductions were 33 and 42%, respectively. For yarn (Fig. 4) the dry strength was reduced in a way similar to that of the web, while the wet strength was essentially independent of the degree of acetylation. The results differ from those obtained by Callow,[4] which showed an increase in wet strength due to acetylation. The strength was the same for samples acetylated at 90 and 120°C, the comparison being made at 6 and 8% weight gain, respectively.

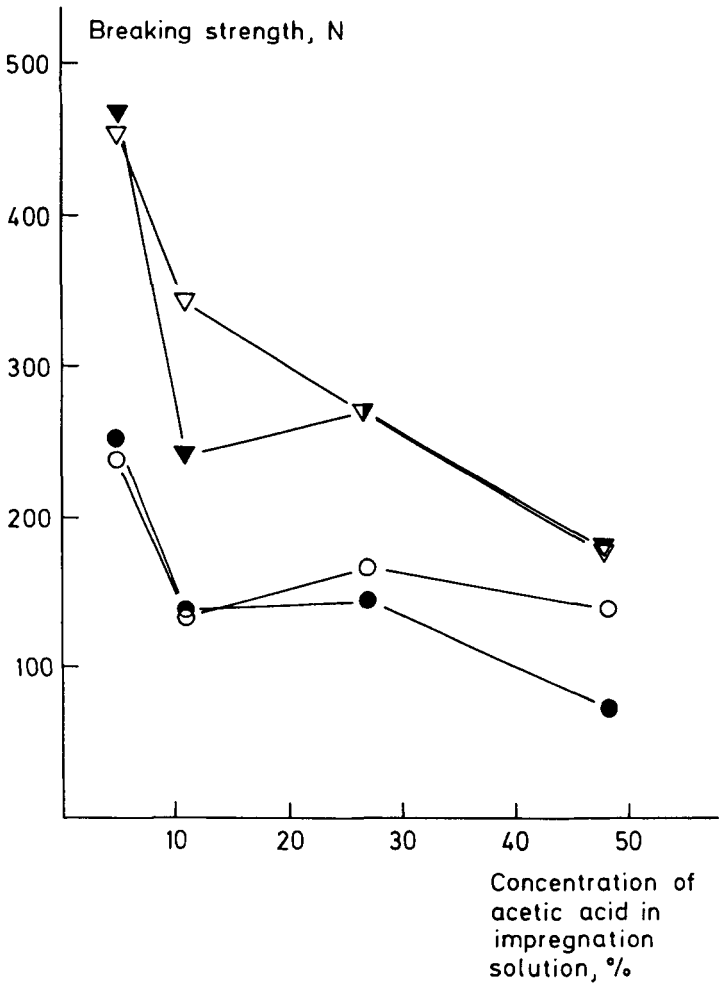

Fig. 6. Dry and wet breaking strength of acetylated jute cloth (warp and weft direction) vs. concentration of acetic acid in impregnation solution, 15% weight gain: (○) dry strength, warp; (▽) dry strength, weft; (●) wet strength, warp; (▼) wet strength, weft.

Part of the strength loss can be explained by the prolonged heat treatment to which the samples were subjected. For example, a sample that was acetylated for 2 h was first oven-dried overnight at 105°C followed by acetylation and evacuation at 120°C for 2 h each. The sample was then again oven-dried at 105°C overnight. The oven drying was done in order to determine the weight gains as accurately as possible but is, of course, not necessary for the acetylation. Figure 5 shows strength losses due to heat treatment (oven drying at 120°C). The reduction in wet strength was larger than the reduction in dry strength, which may explain the above-mentioned deviation from the results obtained by Callow.

Figure 6 clarifies how the strength was affected by acetic acid present in the impregnation solution. The considerable strength reduction suggests that acid degradation of the cellulose chains, mainly responsible for the strength of the material, must have occurred presumably through breakage of glucosidic

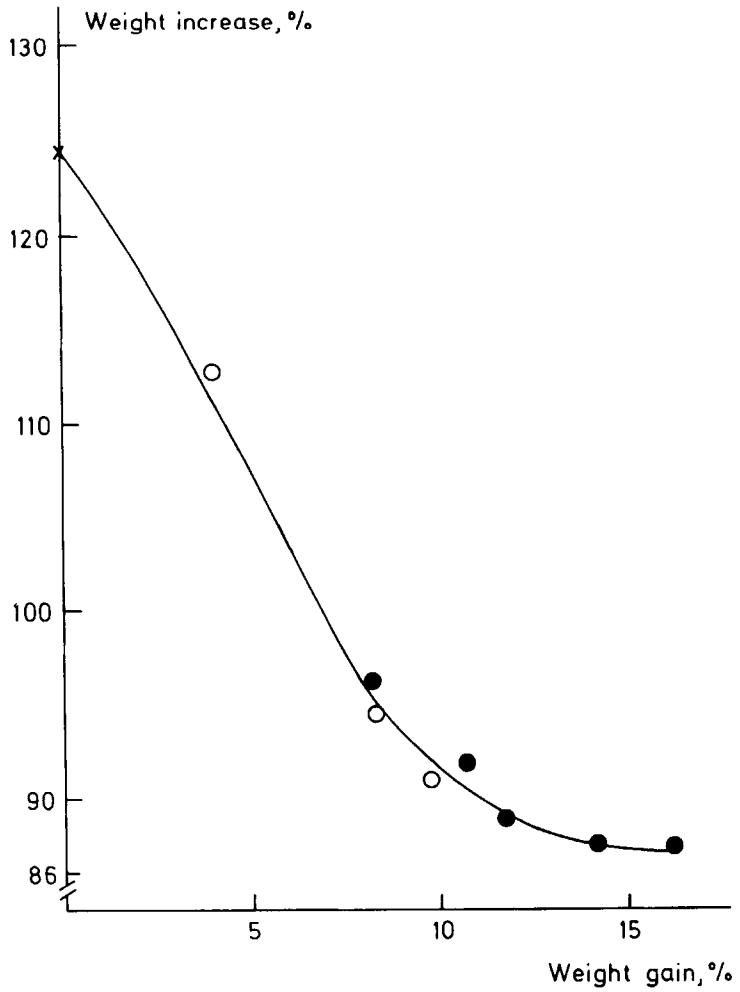

Fig. 7. Weight increase due to water uptake during water soaking of acetylated jute cloth with varying weight gains: (×) control; (○) acetylated at 90°C; (●) acetylated at 120°C.

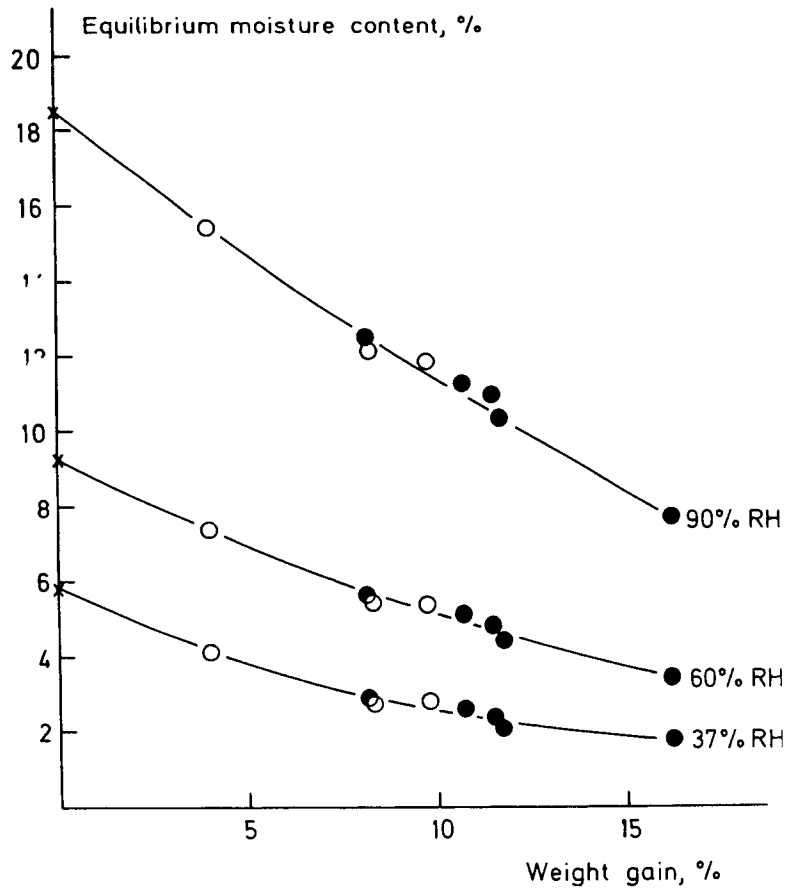

Fig. 8. Equilibrium moisture content (EMC) vs. weight gain for acetylated jute cloth at varying relative humidities and 24°C: (×) control; (○) acetylated at 90°C; (●) acetylated at 120°C.

bonds. The concentration of acetic acid should therefore be kept below 10% in an industrial process. Furthermore, the moisture content of the jute, when acetylated, should be kept as low as possible, since all water in the jute will cause hydrolysis of acetic anhydride to acetic acid. In order to make comparison possible, the strength measurements were done on samples with the same weight gain (15%). A reaction time of 5 h was used in all the experiments, except the one with 50% acetic acid in the impregnation solution, where a reaction time of 8 h was needed to obtain 15% weight gain.

Figures 7 and 8 clearly show that the material becomes more hydrophobic with increasing degree of acetylation. At a weight gain of 16%, for example, the water uptake was reduced by 30% compared with the control samples (cf. Fig. 7). At the same weight gain, the equilibrium moisture content was reduced by 70% at 37% RH, and by 63% at 60% RH (cf. Fig. 8). It should be taken into consideration that a certain amount of hydrophobic substances, e.g., oils added in the manufacturing process and natural extractives, was probably extracted from the jute in the impregnation step. Hence, it is

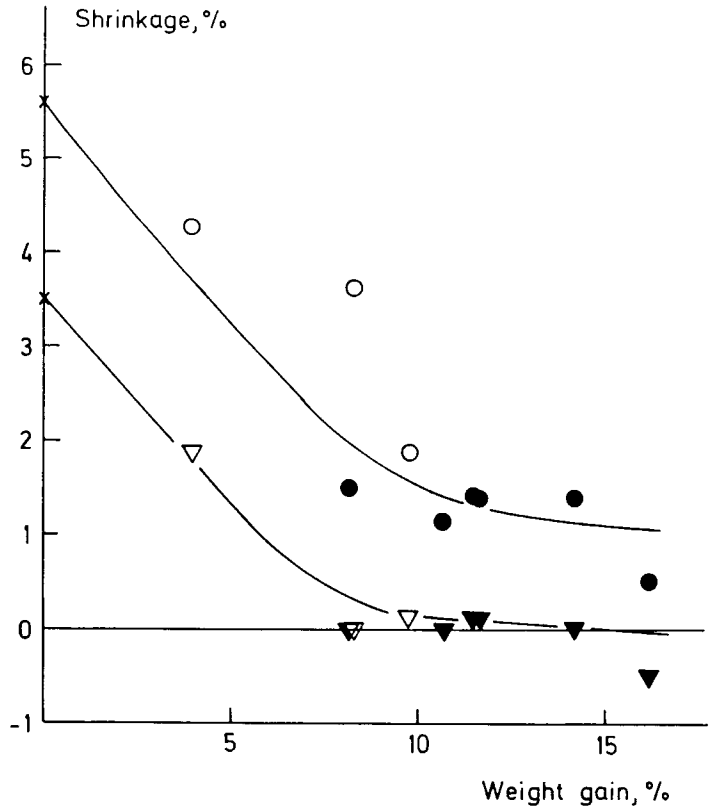

Fig. 9. Shrinkage of acetylated jute cloth with varying weight gains after two water soaking and oven-drying cycles, warp and weft direction: (×) control; (○) warp, acetylated at 90°C; (●) warp, acetylated at 120°C; (▽) weft, acetylated at 90°C; (▼) weft, acetylated at 120°C.

possible that the effect on hydrophobicity is even greater than what is shown in these results.

Acetylated jute shrinks much less than unmodified jute when leached in water; i.e., dimensional stability has been obtained. Figure 9 shows that the total shrinkage after two cycles of water soaking and oven-drying decreased from 6% for a control to 2% for highly acetylated samples in the warp direction and from 3.5% to 0% in the weft direction.

Water soaking of lignocellulosic materials causes swelling of the fibers when water is sorbed into the cell walls and is hydrogen-bonded to the hydroxyl groups of the cell wall components. Swelling of the jute fibers in turn causes shrinkage of the spun and woven jute cloth. Acetylation of the material bulks the fibers and keeps them in a permanently swollen state. Further swelling, upon moisture uptake, is limited by the natural structure of the fiber. This is believed to be the main mechanism for dimensional stabilization. Blocking of hydrophilic hydroxyl groups with less hydrophilic acetyl groups, resulting in lower equilibrium moisture content and lower water uptake, may also contribute to the dimensional stabilization.

CONCLUSIONS

- Acetylation of jute cloth is feasible according to the method described. Weight gains ranging up to 20% can be obtained.
- A weight gain of 11%, achieved after a reaction time of 2 h, gives markedly increased resistance to fungal attack. At this weight gain, the strength reduction is small and the color is still even. Higher weight gains give even better resistance to fungi, but require longer reaction times, and the web becomes patchy and loses much of its strength.
- It is important to keep a low concentration of acetic acid in the impregnation solution; otherwise acid degradation will cause reduction in strength of the web.
- Acetylated jute is considerably more hydrophobic than unmodified jute.
- The acetylated jute cloth shrinks less when exposed to water, i.e., it is dimensionally stabilized.

The authors wish to thank TASSO AB, Halmstad for supplying the jute cloth. Thanks are due to Dr. Thomas Nilsson, the Swedish University of Agriculture, for performing the fungus cellar tests and to Carl-Håkan Andersson, the Institute of Textile Research (TEFO), for the use of the strength testing machine. Mrs. Eva Kristenson is thanked for drawing the figures.

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Received October 15, 1987
Accepted July 1, 1988