

DIMENSIONAL STABILIZATION OF WOOD BY VAPOR PHASE CHEMICAL TREATMENTS¹

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ABSTRACT

White pine and Engelmann spruce cross sections (approx. $1\frac{3}{4} \times 1\frac{3}{4} \times \frac{1}{8}$ inches) were treated under various conditions of time, temperature, and moisture content by a vapor phase technique incorporating a number of different reactants. Two of the reactants considered, acetic anhydride and butyl isocyanate, reacted readily with the cell wall material of the wood samples and improved the dimensional stability of the wood by as much as 75%. Sticks $5 \times \frac{3}{8} \times \frac{3}{8}$ inches, with improved dimensional stabilities after reaction of 67%, gave losses in toughness and abrasion resistance of less than 25%. Generally less successful than the butyl isocyanate treatments were those with allyl, t-butyl, ethyl, and phenyl isocyanate.

INTRODUCTION

The hygroscopicity of cellulosic material results in dimensional changes with changes in the surrounding relative humidity. Greater commercial application would undoubtedly be realized and less difficulty would be experienced in many present applications of wood if methods providing adequate control over its swelling and shrinking were to be developed which were not prohibitively costly or detrimental to its strength.

A rather extensive discussion and literature review on the subject of dimensional stabilization of wood and on various techniques capable of imparting dimensional stability are given by Stamm (1964). The method of interest in this research is "bulking," which simply involves depositing non-volatile materials in the cell walls of fibers when they are in a swollen or partially swollen state so that the cellulose chains in the fiber walls are kept separated as the swelling agent is removed. Thus dimensional stabilization effected by bulking

results from restricting the degree of shrinkage rather than the degree of swelling.

The bulking agent may react with the cell wall material as in the reaction of acetic anhydride with the available hydroxyl groups of amorphous cellulose, or it may merely involve deposition of water-soluble salts, sugars, or low-molecular-weight polymers within the cell walls of the fibers (Stamm 1959, 1964). In this research, wood samples were exposed to the vapors of a number of reactants, including acetic anhydride and various isocyanates. Subsequent determinations of the effects of these treatments on the dimensional stability of the samples were made. A vapor phase technique was used in order to reduce problems involving excess chemical take-up during treatment and removal afterward, factors of primary importance in industrial applications.

MATERIALS AND EXPERIMENTAL METHODS

Treating Cell

All treatments reported in this study were conducted in a closed disk-shaped cell with inside diameter of $6\frac{1}{4}$ inches and depth of 1 inch. The cell was constructed of an aluminum alloy having good resistance to acetic anhydride and acetic acid. An aluminum platform which supported the samples over the treating solution inside the cell contained 33 holes of $\frac{1}{8}$ inch radius which permitted the vapors of the solution to move readily through the platform to

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the samples. The cover of the cell was closed tightly by means of wing nuts which screwed on to ten $\frac{3}{16}$ -inch diameter stove bolts extending through the wall of the cell. The mating surfaces of the cell body, cover, and Teflon gasket were grooved to aid in obtaining an effective seal. A handle which could be attached to the cell before treatment and removed afterward provided a means of easily submerging the cell in, and withdrawing it from, an oil bath, where it rested in a horizontal position during the treatments. The oil bath was equipped with a stirrer, two 500-watt heating coils, and a temperature controller.

Dimensional Stability Tests

White pine (*Pinus strobus* L.) and Engelmann spruce (*Picea engelmann* (Parry) Engelm.) cross sections, $\frac{1}{8}$ inch thick by approximately $1\frac{1}{4}$ by $1\frac{1}{4}$ inches in the radial and tangential directions, were used in the study of the effects of the reactions on dimensional stability. They were cut from carefully selected, defect-free specimens of sapwood 1 to 2 ft long with annual rings parallel to two opposite faces. The order of the cross sections from each specimen was maintained during cutting so that optimum matching could be attained in each series of tests. The bulk specific gravity (dry volume basis) of the original white pine specimens varied from 0.34 to 0.42 and the spruce from 0.33 to 0.34. All samples were identified by sets which came from the same original specimen so that variations in dimensions and densities between sets would not confuse the comparison of treatment effects.

Two cross-sections selected from adjacent positions in the original specimen were used in each treatment. A cross-section to serve as a control was taken from the position between every two pairs of cross-sections designated for treatment. After selection, the samples were dried in a forced-convection oven at 110 C for 4 hr. The oven-dry weight of the samples was then determined to one-thousandth of a gram, and the radial and tangential dimensions to one-thousandth of an inch with a

dial gauge. The dry samples were stored in desiccators containing calcium chloride for later treatment. A number of samples to be treated at moisture contents other than oven-dry were stored, over saturated salt solutions or in a conditioned laboratory maintained at 50% relative humidity. Conditioned samples were weighed again before treatment to determine their actual moisture content.

Treatment of the cross sections was carried out with the vapors from 20 ml of reactant solution in the bottom of the cell. The amount was increased to 40 ml for the stick form specimens, as discussed later. (All solution compositions given in this paper are on a per cent by volume basis.) After the assigned treating time, the cell was withdrawn from the bath, cooled in running water for 5 min, emptied and cleaned. After removal from the cell, the cross-sections treated with isocyanates and their controls were washed by soaking in dimethyl formamide for 4 hr, acetone for 1 hr, and water for 12 hr. Acetylated samples were soaked only in water for 12 hr. They were then placed between flat aluminum screens and allowed to air-dry for at least 24 hr before being dried in an oven for 4 hr at 110 C. The dry wood samples were weighed to determine the extent of reaction, and their tangential and radial dimensions were measured. The controls generally lost 1 to 2% in weight because of washing; therefore, the weight loss in per cent of the controls was added to the net weight gain of the corresponding treated samples to give the gross weight gain, which was taken as a measure of the extent of reaction.

The swollen dimensions of the cross sections were measured after the sections had been soaked in distilled water for 24 hr. The per cent swelling of the samples and the anti-shrink efficiency (ASE) obtained by treatment were calculated according to the following equations:

$$\% \text{ directional swelling} = \frac{\left(\begin{array}{c} \text{swollen dimension} \\ - \text{dry dimension} \end{array} \right)}{\text{dry dimension}} \times 100\%$$

TABLE 1. *Effect of temperature on the vapor phase acetylation of oven-dry white pine cross sections¹*

Temperature (C)	Weight increase (%)	Antishrink efficiency (%)
110	14.1	43.2
120	17.8	53.3
130	20.5	56.2

¹ Treated with acetic anhydride vapor only for 60 min.TABLE 2. *Effect of moisture content on the vapor phase acetylation of white pine cross sections¹*

Moisture content ² (%)	Weight increase (%)	Antishrink efficiency (%)
0.0	19.8	61.5
4.8	19.0	59.5
11.6	18.1	58.8

¹ Treated with acetic anhydride vapor only for 60 min at 130 C.² Based on the oven-dry weight of the wood.

$$\begin{aligned} & \% \text{ volumetric} \\ & \text{swelling (S)} = R + T + RT/100 \\ & \% \text{ ASE} = \frac{\left(\begin{array}{l} \text{S of the control} \\ - \text{S of the treated sample} \end{array} \right)}{\text{S of the control}} \\ & \quad \times 100\%, \end{aligned}$$

where $R = \% \text{ radial swelling}$
 $T = \% \text{ tangential swelling}$

Toughness and Abrasion Tests

Five-inch long sticks of white pine and spruce having rings parallel to two opposite faces and radial and tangential dimensions of $\frac{3}{8}$ inch were used for toughness and abrasion tests. The specific gravity of the pine sticks varied from 0.40 to 0.45 and that of the spruce from 0.33 to 0.34.

The 5-inch samples for treatment and subsequent strength tests and their controls were selected randomly. These samples were dried for 6 hr at 110 C. After their dry weight and dimensions had been recorded, they were stored in desiccators over calcium chloride before treatment. All strength specimens were treated at this dry condition.

The pine sticks were treated at 130 C with vapors of a solution containing 85% acetic anhydride and 15% dimethyl formamide. Spruce sticks were treated at 130 C with vapors of a solution containing 65% butyl isocyanate and 35% dimethyl formamide. A number of these treated samples were cut into five 1-inch sections which were used to determine the variation of specific gravity and dimensional stability from end to end.

The acetylated samples and the corresponding controls were washed by soaking

in water for 12 hr; the samples treated with the isocyanate solution and controls were washed by soaking for 4 hr in dimethyl formamide, 1 hr in acetone, and 12 hr in water. These samples were conditioned for 7 days to a moisture content in equilibrium with 50% relative humidity. The conditioned samples were tested for toughness on a Forest Products Laboratory Intermediate Toughness Tester using the weight in the low position, 4-inch span and 60° angle. The pull was in the tangential direction. One-inch sections were cut from the ends of the broken toughness samples and were abraded with the abrasion tester devised by Loos (1966). This tester consisted of a disk wood sander mounted on a lever arm designed to apply a 500 g load on the test specimen. The sander was geared down to 85 rpm. The radial surface of the specimen was abraded using no. 80 grit disks. The abrasion resistance of the samples was taken as the reciprocal of the change in tangential dimension after 200 revolutions.

RESULTS AND DISCUSSION

Treatment with Acetic Anhydride

Most of the work reported in the literature on the acetylation of wood has been liquid phase, or partially liquid phase, as in the impregnation of wood with a swelling agent or catalyst and then exposure to vapors of acetic anhydride. Any impregnation step which involves extreme liquid absorption generally requires a considerable time and results in a waste of chemical. For this reason, acetylation was included in this study to determine its applicability to a completely vapor phase, relatively high

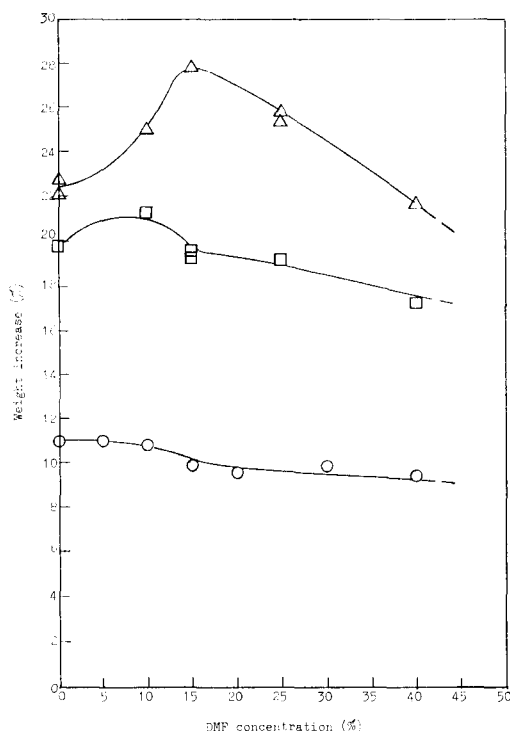


FIG. 1. Relationship between the per cent weight increase of oven-dry white pine cross sections and the concentration of DMF in the liquid treating solution for vapor phase acetylation at 130 C. Circles: 20-min treating time, Set A samples. Squares: 60-min treating time, Set B samples. Triangles: 120-min treating time, Set B samples.

temperature system. It was also included as a means of judging the performance of the other reactions investigated.

Results of acetylation reactions conducted between 110 and 130 C are given in Table 1. The normal boiling points of acetic anhydride and dimethyl formamide (DMF) are 140 and 153 C, respectively. The relationship between treating rate and temperature would be expected to vary appreciably with treating time, *i.e.*, at lower levels of treatment, the effect of increasing temperature on the rate should be more pronounced.

The effect of sample moisture content on the extent of acetylation is shown in Table 2 for several 60-min treatments. Increasing the moisture content of the cross sections before treatment caused a decrease in the

concentration of the acetic anhydride in the wood and in the vapor surrounding the wood during treatment and resulted in a slight decrease in the reaction rate. The rate-depressing effect of the moisture is not serious. It is evident that air-dried wood which generally has a moisture content of less than 15% can be acetylated readily. However, because of the reaction of the anhydride with water to form acetic acid, it is desirable to have the moisture content as low as possible.

Dimethyl formamide (DMF) is miscible with acetic anhydride and lends itself readily to a one-step vapor phase treating process (1957). The effect of varying the concentration of DMF in the liquid acetylating solution on the degree of reaction for three different treating times is shown in Fig. 1. For the lower degrees of reaction, DMF exhibited little influence on the reaction except at higher concentrations where the corresponding reduction in anhydride concentration reduced the reaction rate. DMF concentrations up to 15% exerted a slight positive effect on the 60-min reaction and concentrations up to 35% considerably increased the degree of reaction for treatments for 120 min. The appearance of a maximum in the curves and the shift of this maximum toward 15% DMF with increasing reaction times suggests that the factor controlling the extent of the reaction is the degree to which the cell wall structure is opened up, *i.e.*, the number of reaction sites or hydroxyl groups exposed. It is felt that at shorter reaction times the acetic acid formed as a by-product of the acetylation of wood swells the amorphous cell wall material and exposes a sufficient number of hydroxyl groups to permit a weight increase of approximately 20%. As is shown by Fig. 2, acetylation without DMF proceeds very slowly above 20% weight increase, whereas acetylation incorporating 15% DMF in the liquid reaction solution continues quite rapidly toward 28% weight increase. Measurement of the volumetric swelling of white pine cross sections from oven dry to saturation in acetic acid, acetic anhydride, and DMF showed that the swelling of the

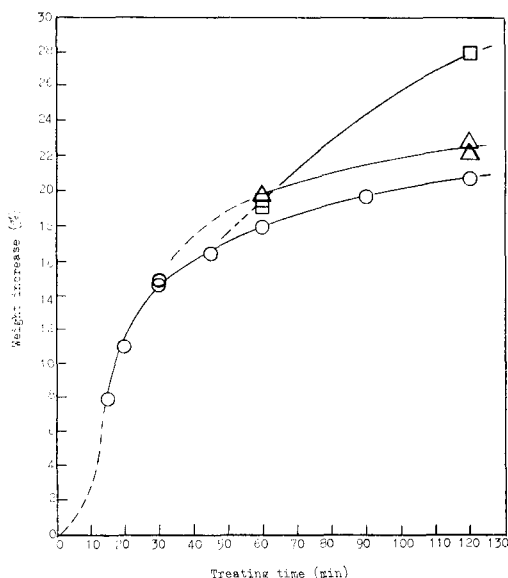


FIG. 2. Relationship between the per cent weight increase of oven-dry white pine cross sections and the treating time for vapor phase acetylation at 130 C. Circles: Set A cross sections, 0.0% DMF. Triangles: Set B cross sections, 0.0% DMF. Squares: Set B cross sections, 15.0% DMF.

cross sections in these chemicals relative to that in water was 87% for acetic acid, 20% for acetic anhydride, and 133% for DMF.

The relationship between the antishrink efficiency and the per cent weight increase of the white pine cross sections upon acetylation is given in Fig. 3 and is in agreement with results reported by Tarkow, Stamm, and Erickson (1955). Approximately 30% weight increase, corresponding to about 75% ASE, appears to be the maximum obtainable by acetylation.

Table 3 shows the variation in the fiber direction of specific gravity and antishrink efficiency of two 5-inch pine sticks having different levels of treatment. The radial and tangential dimensions of these sticks were $\frac{3}{8}$ inch. The degree of treatment as reflected both by the specific gravity and the antishrink efficiency decreased from the ends to the centers of the sticks. The existence of this treatment gradient supports the conclusion of previous investigators (Goldstein et al. 1961) that vapor phase treatment of wood is probably feasible for

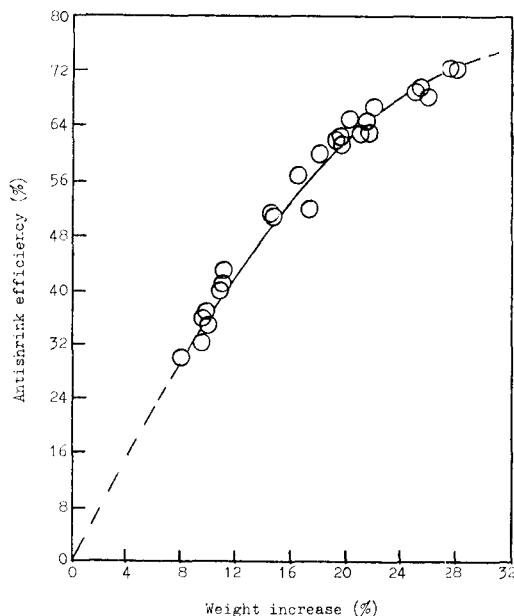


FIG. 3. Relationship between the antishrink efficiency and the oven-dry weight increase of white pine cross sections upon acetylation.

only thin specimens. Because of the slow rate of diffusion of the reactants into the wood, treatment of thicker specimens requires a longer time and yields less uniform results.

Other investigators have shown that the physical strengths of wood are not seriously affected by acetylation and are actually improved in some cases (Stamm 1964). The toughness and abrasion resistance of 5-inch samples acetylated in this study by a vapor phase procedure are shown in Table 4. Samples treated to 43 and 67% ASE retained at least 90% of their original toughness and over 80% of their original abrasion resistance. The fact that the samples treated to 67% ASE exhibited a relative abrasion resistance of 1.09 cannot be considered significant because of the large variance associated with this group of samples.

Treatment with Isocyanates

The vapor phase treatment of spruce cross sections with phenyl isocyanate was confounded by the polymerization of this

TABLE 3. *Longitudinal variation of specific gravity and antishrink efficiency of 5-inch white pine sticks after vapor phase acetylation at 130 C¹*

Sample	Weight increase (%)	Section ²	Oven-dry specific gravity ³	ASE (%)
1	13.3	1	0.42	63
		2	0.41	53
		3	0.41	47
		4	0.41	53
		5	0.42	59
2	23.9	1	0.52	73
		2	0.49	70
		3	0.49	71
		4	0.48	64
		5	0.49	80

¹ The liquid acetylating solution consisted of 15% DMF and 85% acetic anhydride; treating times were 2 hr for sample 1 and 5 hr for sample 2.

² Sections were 1 inch long and were numbered consecutively from end to end of the stick.

³ The oven-dry specific gravity before treatment of sample 1 was 0.40 and that of sample 2 was 0.42.

chemical on the inside of the treating cell and on the surfaces of the wood samples. Because of this polymerization, the results for any given set of treating conditions were unpredictable. The phenyl isocyanate did not polymerize appreciably unless DMF was present in the treating solution; however, without DMF the cross sections would remain unswollen and the isocyanate could not enter and bulk the wood structure. Although this one-step treating process was unsatisfactory, samples which were soaked

in DMF prior to treatment with vapors of phenyl isocyanate exhibited a bulking effect of the order expected from their chemical content, indicating a relatively small amount of isocyanate polymerized outside the cell walls. (A weight increase of 65% resulted in antishrink efficiencies of 77%.) The impregnation of wood samples with liquid DMF followed by exposure to phenyl isocyanate vapor was the treating procedure used successfully by Clermont and Bender (1957).

Butyl isocyanate was found to be much more adaptable to a completely vapor phase treating process than was phenyl isocyanate. Some of the conditions and results of the butyl isocyanate treatments of spruce cross sections are given in Table 5. The data indicate that the most effective concentration of DMF in the treating solution was about 35%. Only a slight change in per cent weight increase of the samples resulted from varying the DMF concentration from 25% to 45% for either the 120 min treatments or the 60-min treatments.

Table 5 shows that the cross sections, dry or containing moisture, cannot be adequately treated with butyl isocyanate unless DMF, or possibly some other swelling agent which is also a solvent for the isocyanate, is present. The moisture in one sample caused it to be partially swollen; but since butyl isocyanate is not soluble in water, the treat-

TABLE 4. *Effect of vapor phase acetylation on the toughness and abrasion resistance of white pine¹*

	Low level treatment		High level treatment	
	Control	Treated	Control	Treated
Number of samples ²	4	5	4	5
Treating time (min)	0	90	0	240
Weight increase (%)	0	12.4	0	23.3
Antishrink efficiency ³ (%)	0	43	0	67
Toughness (inch-lb)	11.51	10.78	10.72	9.99
Standard deviation	1.63	1.69	0.78	1.73
Abrasion resistance	22.4	18.7	20.0	21.8
Standard deviation	2.8	2.7	2.0	4.0
Values relative to controls				
Toughness	1.00	0.94	1.00	0.93
Abrasion resistance	1.00	0.83	1.00	1.09

¹ Oven-dry samples were acetylated at 130 C with vapors of an acetic anhydride solution containing 15% DMF.

² The samples for the two levels of treatment were from different longitudinal positions in the original specimen; however, the nine samples within each level were from laterally adjacent positions and were randomly designated to serve as a control or to be treated.

³ Antishrink efficiencies were predicted from Fig. 3.

TABLE 5. Vapor phase reaction of oven-dry spruce cross sections with butyl isocyanate at 130 C

Treating time (min)	DMF in reaction solution ¹ (%)	Weight increase (%)	ASE (%)
120	15	41.0	74.6
120	25	47.6	76.0
120	35	48.9	78.1
120	45	48.2	76.0
120	55	37.3	71.8
120	0	1.5	3.6
120 ²	0	2.4	3.9
60	25	32.8	65.9
60	35	33.6	67.9
60	45	30.5	64.5
30	35	18.6	51.8
240	35	51.3	76.6
60 ³	35	26.3	58.6
60 ⁴	35	11.1	28.8

¹ The liquid treating solutions were composed of butyl isocyanate and dimethyl formamide with the compositions given on a per cent by volume basis.

² Treated at a moisture content of 7.9% rather than at an oven-dry condition.

³ Treating temperature 120 C.

⁴ Treating temperature 110 C.

ment was not significantly benefited by its presence.

The effect of impregnating the spruce cross sections with DMF prior to treatment with butyl isocyanate is shown in Table 6. Increasing the per cent DMF in the samples from 0 to about 50% improved the effectiveness of the 15-min treatments. When the concentration of the DMF in the wood was increased to 81%, no further improvement in the reaction was evident. This is not surprising since the wood should have been completely swollen at 50% DMF content. Comparison of samples in Table 6 with those in Table 5 reveals that treatment of the impregnated samples was faster than the one-step vapor phase treatment of oven-dry samples.

The effect of temperature on the vapor phase butyl isocyanate treatments can be seen by comparison of samples in Table 5. The reaction rate was appreciably improved by increasing the temperature from 110 to 130 C; however, as was mentioned previously with respect to the anhydride treatment, the relationship between per cent weight increase and temperature will

TABLE 6. Effect of impregnating oven-dry spruce cross sections with dimethyl formamide prior to treatment with butyl isocyanate vapors at 130 C

Treating time (min)	DMF in wood ¹ (%)	Weight increase (%)	ASE (%)
30	37	29.5	66.2
15	55, 81	20.6	60.6
15	31, 46	14.9, 16.6	51.0, 57.5
15	10	9.6	32.3

¹ The oven-dry samples were submerged in dimethyl formamide (DMF) for short periods of time ranging from a few seconds to 90 sec, wrapped in aluminum foil and allowed to equilibrate for 2 hr before treatment.

vary with treating time, *i.e.*, extent of reaction.

The effect of the moisture content of the spruce cross sections before treatment on 30-min vapor phase butyl isocyanate treatments is shown in Fig. 4. The effectiveness of the treatments dropped rapidly with increasing moisture content. Since butyl isocyanate and water are not miscible, it is thought that moisture retarded the take-up of isocyanate and also reduced the concentration of the isocyanate in the vapor surrounding the cross sections, consequently reducing the reaction rate.

The relationship between the per cent weight increase and treating time for vapor

TABLE 7. Longitudinal variation of specific gravity and antishrink efficiency of 5-inch spruce sticks after vapor phase treatment with butyl isocyanate at 130 C¹

Treating time (min)	Weight increase (%)	Section ²	Oven-dry specific gravity ³	ASE (%)
75	13.6	1	0.36	48
		2	0.35	47
		3	0.35	48
		4	0.35	54
		5	0.37	59
180	20.3	1	0.37	67
		2	0.35	62
		3	0.35	66
		4	0.35	64
		5	0.37	72

¹ The liquid treating solution consisted of 35% DMF and 65% butyl isocyanate.

² Sections were 1-inch long and were numbered consecutively from end to end of the stick.

³ The oven-dry specific gravities before treatment for the first stick was 0.34 and for the second stick 0.33.

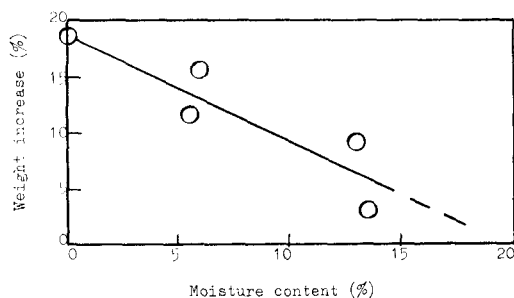


FIG. 4. Relationship between the per cent weight increase and the moisture content of spruce cross sections treated for 30 min at 130 C with vapors of a butyl isocyanate-dimethyl formamide solution (35% DMF).

phase butyl isocyanate treatments at 130 C is given by Fig. 5. Under the conditions used, the treating rate for the isocyanate reaction was considerably faster than the acetylation rate (Fig. 2). Only a general comparison can actually be made since different wood species were treated.

The antishrink efficiency of spruce cross sections as a function of the weight increase upon reaction with butyl isocyanate is shown in Fig. 6. Comparison of this relationship with that for acetylation of white pine reveals that for a given antishrink efficiency a greater weight increase was required in the spruce-isocyanate system than in the pine-anhydride system. The greater weight increase was necessitated primarily by the fact that the volumetric swelling of the spruce was larger than the swelling of the pine and therefore required more bulking. The average volumetric swelling of the untreated spruce cross sections was 13.4% while that of the pine cross sections was 9.9%. A secondary factor which contributed to the higher bulking requirement for the isocyanate treatment was that this treatment caused an increase in the total volume change of the samples from the oven-dry, untreated to the completely water-swollen condition. This increase in total volume change was 0.8% at 50% ASE and 1.7% at 70% ASE.

The variation of specific gravity and antishrink efficiency in the fiber direction from end to end of 5-inch spruce sticks (% by %

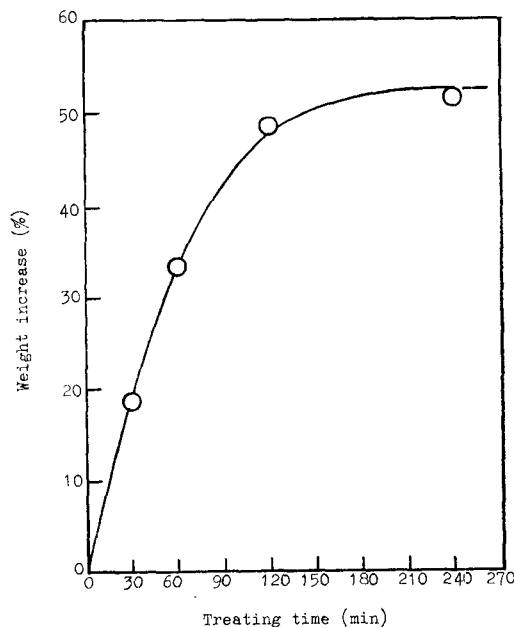


FIG. 5. Relationship between the per cent weight increase of oven-dry spruce cross sections and the treating time for vapor phase treatment with butyl isocyanate at 130 C (Liquid treating solution: 35% dimethyl formamide in butyl isocyanate).

inch) after vapor phase treatment with butyl isocyanate is presented in Table 7. A treating gradient is shown to exist, but it is much less pronounced than the gradient exhibited by the acetylated pine sticks (Table 3). The difference between them can most probably be attributed to the lower specific gravity of the spruce which generally indicates a greater ease of diffusion through the wood.

The effect of vapor phase butyl isocyanate treatments on the toughness and abrasion resistance of 5-inch long spruce sticks is shown in Table 8. Approximately a 25% reduction of the original values of these strengths was incurred upon treatment to 47 and 67% ASE. It is doubtful that a strength reduction of this order would be serious in potential applications of the treated wood.

Treatments of spruce cross sections with ethyl, tertiary-butyl, and allyl isocyanates proceeded much slower than those with butyl isocyanate. For reactions involving

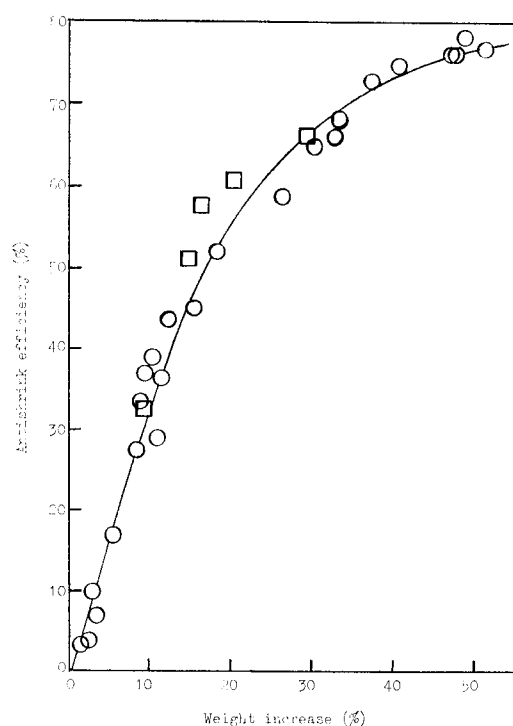


FIG. 6. Relationship between the antishrink efficiency and the oven-dry weight increase of spruce cross sections upon reaction with butyl isocyanate. Circles: Completely vapor phase treatment with solutions containing various amounts of dimethyl formamide. Squares: Treatment after impregnation with dimethyl formamide.

ethyl isocyanate, which has a normal boiling point of 60 C, temperatures above 90 C were not used in order to limit the pressure within the treating cell. The lower temperatures may have been largely responsible for the slower treating rate. Spruce cross sections treated for 120 min at 90 C with an ethyl isocyanate solution containing 50% DMF showed 6.8% weight increase and 11.5% ASE. A similar treatment at 100 C with a *t*-butyl isocyanate solution containing 40% DMF resulted in 1.7% weight increase and 4.0% ASE. The slower rate for this treatment is probably due to steric influences on the reactivity of the tertiary molecule.

Spruce cross sections treated with allyl isocyanate to weight increases of 2.1 and 8.2% were exposed to 20 megarads of gamma radiation from a Cobalt-60 source in an attempt to initiate radical polymerization and thus form cross-links between the cellulose chains in the cell walls. Although a small amount of polymerization may have occurred, subsequent measurements indicated no restriction on the degree of swelling of the cross sections and therefore no effective cross-linking. Chain transfer reactions were probably important in limiting the extent of polymerization and cross linking.

TABLE 8. Effect of vapor phase butyl isocyanate treatment on the toughness and abrasion resistance of spruce¹

	Low level treatment		High level treatment	
	Control	Treated	Control	Treated
Number of samples ²	4	5	4	5
Treating time (min)	0	75	0	240
Weight increase (%)	0	13.9	0	31.1
Antishrink efficiency ³ (%)	0	47	0	67
Toughness (inch-lb)	11.52	8.86	11.37	8.16
Standard deviation	0.96	0.52	1.55	1.15
Abrasion resistance	24.2	18.8	24.4	18.0
Standard deviation	1.4	1.6	2.8	1.6
Values relative to controls				
Toughness	1.00	0.77	1.00	0.72
Abrasion resistance	1.00	0.78	1.00	0.75

¹ Oven-dry samples were treated at 130 C with vapors of a butyl isocyanate solution containing 35% DMF.

² The samples for the two levels of treatment were from different longitudinal positions in the original specimen; however, the nine samples within each level were from laterally adjacent positions and were randomly designated to serve as a control or to be treated.

³ Antishrink efficiencies were predicted from Fig. 6.

SUMMARY AND CONCLUSIONS

White pine and Engelmann spruce cross sections were treated under various conditions of time, temperature, and moisture content by a vapor phase technique incorporating acetic anhydride and allyl, butyl, t-butyl, ethyl, and phenyl isocyanate. The volumetric swelling of the cross sections from the oven-dry to the water-soaked condition was readily reduced by as much as 75% upon reaction with either acetic anhydride or butyl isocyanate. The other treatments investigated also improved the dimensional stability of the cross sections but were generally less successful.

Because of the favorable results of the acetic anhydride and butyl isocyanate treatments on the dimensional stability of the cross sections, the effect of these chemicals was determined on the toughness and abrasion resistance of wood (generally considered to be the two mechanical properties of wood that are most adversely affected by both chemical and thermal treatments).

Conclusions from this investigation are:

1. Vapor phase treatment provides an effective means of modifying the properties of wood; however, its practical application is probably restricted to relatively thin specimens.

2. Acetic anhydride and butyl isocyanate can be readily adapted to a vapor phase treating process. Vapor phase acetylation of wood can be carried out with or without dimethyl formamide in the treating solution; however, the beneficial effect of the

dimethyl formamide is appreciable only for the greater degrees of reaction. For the treatment of wood with butyl isocyanate, the presence of dimethyl formamide, or possibly some other swelling agent and solvent, in the treating solution is essential.

3. Vapor phase treatment with acetic anhydride or butyl isocyanate can impart high dimensional stability to wood without seriously affecting its strength. Tests of the toughness and abrasion resistance of white pine and spruce sticks treated to 67% ASE showed that the acetic anhydride and butyl isocyanate treatments reduced these strengths by less than 10% and 25%, respectively.

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