

Effect of Several Wood Factors on Dimensional Stabilization of Southern Pines

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ONE OF THE MAIN DISADVANTAGES of using wood is that it shrinks and swells with changes in relative humidity. Extensive work has been done in the area of dimensional stability by a number of investigators with many species (7), but little information is available. Therefore, this study was undertaken in order to obtain quantitative information on stabilizing different kinds of wood of the southern pines using several bulking agents.

Bulking involves replacing the water in the cell wall, usually with a water-insoluble chemical. Aqueous solutions of several thermosetting resin-forming systems have been tried with considerable success. Best results have been obtained when the weight of resin taken up in impregnation is equal to about 40 percent of the oven-dry weight of the wood (10).

Polyethylene glycol has been found to be quite successful in stabilizing wood (3). Recent work by Stamm (9) indicates that polyethylene glycol retention, as well as shrinkage reduction, is optimum when the polymer is impregnated into wood that is initially either at zero or saturated moisture conditions. In the dry condition the polymer is taken up by capillary action, whereas in the saturated condition penetration is by solvent exchange and diffusion. Capillary uptake is effective only when the polymer moves in the fiber direction.

Stabilizing wood with vinyl monomers is a new technique. The polymerization of these monomers in wood can be done by gamma irradiation (8) or by heat with a suitable catalyst (6). Methyl methacrylate, styrene, and styrene-acrylonitrile have been reported to reduce the shrinkage of wood (1, 4, 5, 8).

Experimental Procedure

General Study

End-matched wood samples measuring 1 by 1 inch in cross-section and 1/2 inch in thickness along the grain were obtained from a loblolly pine (*Pinus taeda* L.) board. The average specific gravity was 0.488 ± 0.023

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The effectiveness of five dimensional stabilizing agents and three impregnation methods on southern pine was determined. Four southern pine species were studied in order to determine the effect of wood factors. The best dimensional stability was obtained when the wood was preswollen and the chemical was impregnated by a diffusion process. In general, polyethylene glycol gave the highest anti-swell efficiency value, followed in the order by phenolic resin, styrene-acrylonitrile, styrene, and methyl methacrylate, respectively. Better stabilization was obtained with the low density corewood than with the high density maturewood.

(green volume basis). These samples were randomly assigned to various treatment classes. Altogether, five kinds of chemicals and three treating processes were studied. The chemicals were 50 percent aqueous solution of polyethylene glycol-1000 (Dow's Carbowax), 25 percent solid content of phenolic resin (Borden's Compregnite, Monsanto's Resinox 468, and Synco's 352), methyl methacrylate, styrene, and styrene-acrylonitrile (60:40 volume). The three impregnation processes were capillary uptake, solvent exchange, and vacuum-pressure. The variables are shown in Table 1. There were 14 replications for the control and seven replications for each chemical and impregnation combination.

Treatment by solvent exchange was accomplished by first saturating the wood with water for more than 2 weeks; each day a vacuum was applied for about 15 minutes. With polyethylene glycol, the samples were soaked in the polymer for 48 hours. With vinyl monomers, the samples were first soaked in an intermediate solvent for 48 hours, then the solvent was exchanged with the monomer six times at intervals of 24 hours, each time using fresh monomers. Acetone was used as an intermediate solvent for methyl methacrylate, ethanol for styrene-acrylonitrile, and both ethanol and dioxane for styrene. Ten percent carbon tetrachloride was added to the styrene system as an accelerator.

In the capillary uptake treatment, the oven-dry samples were soaked directly in the appropriate treating chemicals. An initial vacuum was applied to the treating solution to remove the air from the wood and then released to permit penetration by diffusion for 24 hours.

In the vacuum-pressure treatment, the samples that were previously conditioned to about 20 percent EMC over a saturated calcium sulfate solution in a desiccator were soaked in the desired chemical in an open container, and placed inside a retort for an initial vacuum of 22 inches of mercury for 30 minutes, followed by a 60 psi air pressure for 3 hours.

The samples treated with polyethylene glycol were stored in non-drying conditions and allowed to equalize

by diffusion for 1 week. This was accomplished by placing them on a stainless steel rack inside a desiccator containing some water. The treated samples were then slowly air-dried for several days before placing them in an oven at 220°F. for at least 24 hours.

The samples treated with phenolic resin were cured at 300°F. in accordance with the manufacturer's recommendations. The time required for the center of the sample to reach the desired curing temperature had to be determined in order to obtain a uniform cure. To do this, a small hole was drilled to the center of several dummy samples and a thermocouple wire inserted in the hole. The change in temperature of the wood was determined with a Leeds & Northrup potentiometer. The time to reach 300°F. was 40 ± 5 minutes, and an additional 10 minutes was used to insure a complete cure.

With the vinyl monomers, the samples were weighed immediately after impregnation and quickly wrapped in aluminum foil to avoid monomer loss. They were then irradiated in an air atmosphere with a Cobalt-60 gamma source. The dose rate was 1.3 x 10⁶ rads/hr. The irradiation times were as follows: styrene, 79 hours; methyl methacrylate, 73 hours; styrene-acrylonitrile, 24 hours. From preliminary investigations these times were found to give a monomer conversion of about 90 percent for methyl methacrylate and styrene-acrylonitrile, and, except for the capillary uptake samples, at least 50 percent for styrene. After polymerization, the treated

samples were allowed to dry in a vacuum oven at 122°F. to constant weight in order to remove the water and the unpolymerized monomer in the wood.

All the treated samples were evaluated by first exposing them to a particular atmospheric condition, and then recording their dimensions and weights at equilibrium. Dimension measurements were made with a dial gage accurate to 0.001 inch and weight readings with a Sartorius top-loading balance accurate to 0.001 gram. The samples were subjected to four moisture conditions: oven-dry (220°F.), 15 percent R.H. (potassium acetate), 75 percent R.H. (sodium chloride), and distilled water (36 hours of soaking). The samples were conditioned by exposing to the atmosphere inside a desiccator containing the appropriate saturated salt solution at a controlled temperature of 90 ± 1°F.

The following physical values were obtained:

$$\text{Swelling (\%)} = \left[\frac{D_t - D_1}{D_1} \right] 100$$

$$\text{Anti-Swell Efficiency or ASE (\%)} = \left[\frac{S_o - S_t}{S_o} \right] 100$$

$$\text{Treatment Level (\%)} = \left[\frac{W_t - W}{W} \right] 100$$

$$\text{Bulking Coefficient (\%)} = \left[\frac{V_t - V}{V} \right] 100$$

Table 1. — DIMENSIONAL STABILITY DATA OF LOBLOLLY PINE TREATED BY VARIOUS METHODS.

Bulking Agent and Impregnation Method	Average Volumetric Swelling		Anti-Swell Efficiency (O.D.-Soaked)	Bulking Coefficient	Treatment Level
	Fraction (ΔS_M) (15-75% RH)	Total (S_T) (0-100% RH)			
Polyethylene Glycol					
Capillary Uptake	0.73	1.65	87.8	12.7	61.8
Solvent Exchange	1.10	1.28	90.5	11.8	63.4
Vacuum-Pressure	1.07	1.66	87.7	11.9	53.2
Phenolic Resin					
Vacuum-Pressure					
Resinox 468	1.48	2.82	79.2	12.7	78.2
Compregnite	2.08	3.11	77.0	13.9	66.8
Synco 352	2.09	3.34	75.3	12.6	77.8
Methyl Methacrylate					
Capillary Uptake	4.47	11.44	15.5	-0.9	102.1
Solvent-Exchange (Acetone) ¹	2.37	7.25	46.5	6.3	93.1
Vacuum-Pressure	3.19	7.98	41.1	4.4	93.8
Styrene					
Capillary Uptake	4.76	14.68	-8.5	-2.0	25.0
Solvent Exchange (Ethanol) ¹	2.00	3.57	73.6	10.7	69.2
Solvent Exchange (Dioxane) ¹	3.24	6.95	48.7	6.4	49.3
Vacuum-Pressure	1.93	4.26	68.5	9.5	56.1
Styrene-Acrylonitrile					
Capillary Uptake	2.97	7.32	45.9	3.5	99.8
Solvent Exchange (Ethanol)	2.56	4.03	70.2	9.2	106.4
Vacuum-Pressure	2.11	3.52	74.0	11.0	98.4
Control (Untreated)	4.92	13.54	-----	-----	-----

¹Intermediate solvent.

in which D_i is the initial dimension, D_f is the final dimension, S_c is the swelling control sample, S_t is the swelling of treated sample, W is the oven-dry weight of wood, W_t is the oven-dry weight of treated wood, V is the oven-dry volume of wood, and V_t is the oven-dry volume of treated wood. The volumetric swelling value was determined by the summation of the radial and tangential swelling values.

Detailed Study

Ten trees from each of four species of southern pine, namely, loblolly pine (*Pinus taeda* L.), shortleaf pine (*P. echinata* Mill.), longleaf pine (*P. palustris* Mill.) and slash pine (*P. elliottii* Engelm.) were randomly selected for sampling from a forest near Bogalusa, Louisiana. Each tree was bored at heights of 1 foot and 10 feet with an increment borer to determine which trees had high (>0.62) and low (<0.62) specific gravity (oven-dry basis) in the corewood (0-15 years) and in the mature-wood (25-40 years), respectively. Selected trees were then felled and taken to a mill where cants were cut from the corewood and maturewood sections and sawed so that the growth rings would be located parallel to the opposite edges as shown in Figure 1. This procedure allowed for accurate dimensional measurements in the radial and tangential planes of wood. After planing the cants to 1 by 1 inch in cross-section, samples measuring 1/2 inch in the fiber direction were cut from them. As with the general study, the small longitudinal direction was chosen to facilitate end-matching, and to eliminate the factor of chemical penetration. These samples were then randomly assigned to various classes as follows:

Variables	Levels
Species	4
Age Class	2
Density Class	2
Treatment Class	4
Replicates	7
Total	448

The four treatment classes were: polyethylene glycol/solvent exchange, methyl methacrylate/solvent exchange, phenolic resin/vacuum-pressure, and styrene-acrylonitrile/vacuum-pressure. The procedure for treatment was the same as that described in the general study, except for the methyl methacrylate. Also, all the samples were directly soaked in distilled water from the oven-dry condition and therefore only total swelling data were obtained. The methyl methacrylate samples were first saturated with methanol from the air-dry condition, then exchanged with the monomer five times at intervals of 24 hours each time. The use of methanol as a swelling agent was found to prevent internal checking in wood and also gave higher stability value than acetone.

Discussion of Results

General Study

Polyethylene glycol-1000 was found to be an effective bulking agent as indicated by the high anti-swell efficiency (ASE) values of about 90 percent shown in Table 1. These values are consistent with published reports (3, 9). Impregnation of the polymer by the capil-

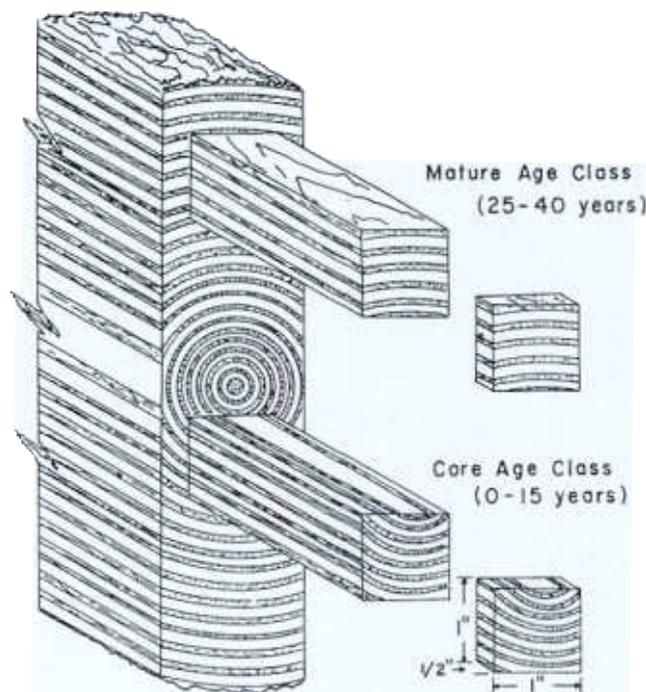


Figure 1. — A diagrammatic representation of the sampling procedure showing how samples were obtained.

lary uptake method proved to be quite effective and was comparable with the vacuum-pressure treatment. Pre-swelling of the wood with water, as in the solvent exchange method, gave maximum bulking; therefore somewhat better results were obtained with this process than with the other treating methods since more chemical could get into the cell wall structure.

Phenolic resins were also found to be an effective bulking agent. ASE values of over 75 percent were obtained with all the three commercial resins. Resinox 468 appears to be superior since it shows the lowest swelling values and therefore the highest ASE values (Table 1). The apparent superiority of this resin may be offset by the physical appearance after curing. A crusty residue of resin which had exuded during curing was found on both the Resinox and Synco samples, but not on the Compregnite samples.

The dimensional stability data for the vinyl monomers are also shown in Table 1. With the exception of styrene-acrylonitrile, more effective stabilization was achieved when wood was pre-swollen prior to treatment, since lower swelling and higher ASE values were obtained with the solvent exchange process than with the other treating methods. The styrene-acrylonitrile samples gave somewhat better values than either the styrene or methyl methacrylate samples. The ASE values for styrene-acrylonitrile were 74 percent with the vacuum-pressure method and 70 percent with the solvent exchange process. The values for styrene were 74 percent by solvent exchange with ethanol and about 50 percent with dioxane; for methyl methacrylate, 46 percent by solvent exchange with acetone and 41 percent by vacuum-pressure. However, with the capillary uptake process using styrene and methyl methacrylate, low ASE values

were obtained, due probably to surface tension effect which results in low treatment level. In addition, a shrinkage phenomenon was observed upon treatment, as indicated by the negative bulking coefficient (Table 1). The reasons for this phenomenon are not clearly understood. It is known that on conversion of monomer into polymer, shrinkage of the system is observed. Whether the decrease in oven-dry volume is due to cross-linking with the cellulose thus causing the entire system to shrink upon polymerization, or whether it is due to some type of internal stress, is a matter for conjecture. A probable cause, namely, collapse, was not observed.

Those samples which were treated with the three vinyl monomers used in this study developed some internal checks when they were treated by the solvent exchange and vacuum-pressure processes and dried after irradiation. Visually, no checks were found in the samples treated by the capillary uptake. Check formation was probably due to water in the cell wall and subsequent shrinkage of the sample during polymerization. When a non-aqueous swelling agent such as methanol was used to pre-saturate the wood before exchanging it with the monomer, no checking developed.

The relationship between apparent fiber saturation point, equilibrium moisture content (based on oven-dry treated weight), and total volumetric swelling of wood can be expressed mathematically as the slope of the moisture content - swelling line and may be approximated:

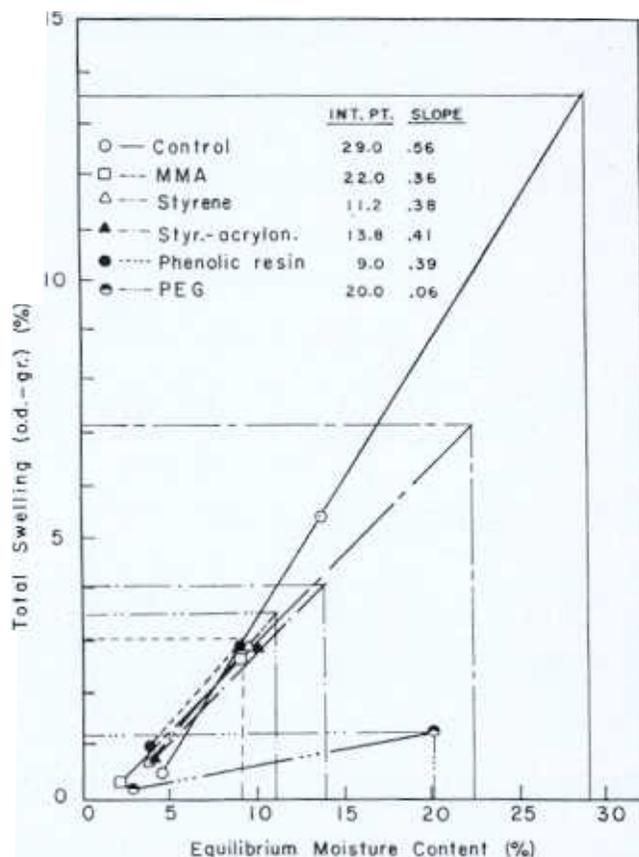


Figure 2. — Relationship of moisture content (based on oven-dry weight of treated wood) and swelling showing the significance of the slope of line as an indication of dimensional stability.

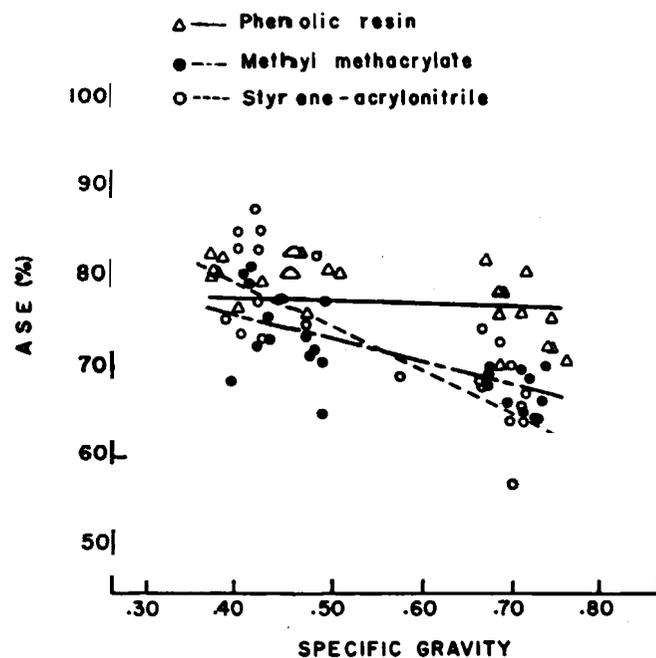


Figure 3. — Relationship of volumetric anti-swelling efficiency and specific gravity for several bulking agents in shortleaf pine.

$$\frac{dS}{dM} \approx \frac{\Delta S_M}{\Delta M} = \frac{S_T}{M_T}$$

where ΔM is the change in equilibrium moisture content at two relative humidities; S_T is the total swelling between oven-dry and soaked (swollen) conditions; ΔS_M is the change in swelling between two relative humidities; and M_T is the apparent fiber saturation point, as determined by the intersection point method (12), provided a linear relationship exists between moisture content and shrinkage or swelling.

Theoretically, an ideally stabilized wood showing the least amount of dimensional change between two humidity conditions should be one that has the lowest slope which would correspond to a high M_T value and a low S_T value for the same ΔM range. Figure 2 was developed from the solvent exchange data in Table 1 for each of the chemicals used. In each case, the plot shows that the intersection point for treated wood is lower than the control or untreated wood. However, all treated samples have higher dimensional stability because of bulking, which is reflected in the lower slopes. In the case of polyethylene glycol, higher stability was also due to large equilibrium moisture changes. Since polyethylene glycol is hygroscopic at high humidities, it is assumed that the additional water was sorbed by the polymer in the cell lumens with no appreciable increase in volume due to swelling.

Detailed Study

The basic dimensional stability data of the four southern pine species for various age class and specific gravity combinations are shown in Table 2 in terms of volumetric swelling. The general trend is for the high-

Table 2. — AVERAGE VOLUMETRIC SWELLING (O.D. TO SOAKED CONDITION) FOR VARIOUS SPECIES OF SOUTHERN PINE AND WOOD FACTOR COMBINATIONS.

Species and Wood Factor ¹	Untreated Wood	Treated Wood			
		Polyethylene Glycol	Phenolic Resin	Methyl Methacrylate	Styrene-Acrylonitrile
(percent)					
Loblolly Pine					
L-C	16.3	3.5 (14.8, 60.5) ²	3.6 (17.7, 76.2)	4.9 (9.7, 100.6)	4.4 (8.8, 106.6)
H-C	24.1	4.1 (19.7, 35.1)	5.4 (20.5, 47.2)	7.2 (14.3, 57.8)	8.9 (10.9, 51.8)
L-M	14.2	1.9 (14.1, 59.7)	3.3 (16.6, 78.3)	4.3 (9.4, 101.8)	3.7 (8.3, 106.3)
H-M	23.6	5.3 (20.0, 30.6)	6.4 (20.2, 45.0)	8.8 (14.9, 49.7)	9.6 (11.6, 42.9)
Shortleaf Pine					
L-C	12.4	5.7 (10.8, 73.0)	2.5 (13.8, 90.2)	3.1 (6.7, 130.2)	2.5 (6.0, 146.5)
H-C	18.9	3.3 (17.8, 36.5)	5.3 (18.9, 47.9)	5.8 (15.0, 62.9)	6.3 (13.8, 62.1)
L-M	15.3	2.3 (14.3, 62.5)	3.2 (16.4, 86.3)	4.2 (9.1, 117.9)	3.1 (8.7, 134.0)
H-M	19.5	4.1 (17.0, 36.1)	4.9 (19.5, 15.3)	6.5 (13.5, 58.5)	6.9 (10.7, 59.5)
Longleaf Pine					
L-C	11.5	3.2 (11.2, 45.7)	1.6 (13.5, 61.6)	3.6 (8.1, 84.6)	4.3 (7.4, 53.8)
H-C	14.1	4.6 (13.1, 31.8)	3.4 (15.3, 49.0)	4.9 (10.2, 58.8)	5.4 (9.6, 42.4)
L-M	15.1	2.5 (14.1, 51.1)	3.9 (14.5, 64.7)	3.9 (8.6, 74.8)	4.4 (8.1, 80.4)
H-M	20.3	3.7 (16.7, 36.5)	5.5 (18.4, 49.9)	6.1 (13.1, 58.1)	8.0 (10.2, 50.1)
Slash Pine					
L-C	10.7	3.7 (8.2, 59.9)	1.2 (9.6, 61.2)	2.3 (5.6, 106.5)	4.2 (4.7, 60.2)
H-C	9.2	3.6 (7.4, 21.0)	5.0 (6.9, 20.6)	3.3 (5.4, 47.5)	4.0 (5.5, 28.7)
L-M	15.0	1.7 (15.1, 56.3)	3.7 (16.9, 81.9)	4.5 (9.6, 97.0)	3.9 (8.6, 100.3)
H-M	20.3	3.9 (17.8, 35.6)	5.1 (19.8, 52.0)	7.5 (12.8, 58.2)	8.2 (10.0, 56.0)

¹C = corewood (0-15 years); M = maturewood (25-40 years); L = low specific gravity (<0.62 O.D. basis); H = high specific gravity (>0.62).

²The first value is bulking coefficient; the second value is treatment level.

density maturewood to give the least dimensional stability, followed in the order by the high-density corewood, the low-density maturewood, and the low-density corewood. The average swelling values were respectively, 20.9, 16.6, 14.9, and 12.7 percent for the untreated samples and 6.28, 5.03, 3.58, and 3.39 percent for the treated samples.

In the analysis of variance using volumetric ASE as the variable (Table 3), the main effects of treatment were highly significant. A comparison of the means (Table 4) and their differences shows that those samples that were treated with polyethylene glycol and phenolic resin gave better ASE results than those treated with the vinyl monomers. Apparently, the vinyl monomers do not penetrate the cell wall of wood as readily as either polyethylene glycol or phenolic resin. As shown in Table 2, the bulking coefficient of the vinyl monomers is low even though the treatment level is high.

The effect of species was significant although Duncan's test on the six possible combinations showed that two were significantly different at the 5 percent level of probability. This finding showed that shortleaf pine had significantly greater ASE value than slash and longleaf pines. Loblolly pine would be significantly different from longleaf and slash pines if the levels of probability were 6 and 7 percent, respectively. This result is interesting since one would not expect any species differences in the southern pines due to the similarity of their anatomical characteristics. The differences may be attributed to the more resinous nature of slash and longleaf pines, thus making them slightly more difficult to treat because of reduced permeability

(2). Apparently the higher resin content in these species did not contribute to better stabilization when the wood was treated with bulking agents, even though these two species had lower untreated swelling values (Table 2) due probably to natural bulking effect of the resin in the cell wall. Generally, when the less resinous species, namely, loblolly and shortleaf pines, were treated with a non-volatile bulking agent such as polyethylene glycol

Table 3. — ANALYSIS OF VARIANCE FOR VOLUMETRIC ANTI-SWELL EFFICIENCY.

Source	D.F.	Mean Square	F Ratio
Total	447		
A (Treatment)	3	3,992.48	129.04**
B (Species)	3	410.17	13.26**
AB	9	176.08	5.69**
C (Age Class)	1	2.64	0.08N.S.
BC	3	367.36	11.87**
AC	3	687.34	22.22**
ABC	9	210.42	6.80**
D (Density Class)	1	3,436.77	111.08**
CD	1	111.68	3.61N.S.
BD	3	249.49	8.06**
BCD	3	413.63	13.37**
AD	3	126.13	4.08**
ACD	3	135.13	4.36**
ABD	9	41.09	1.33N.S.
ABCD	9	80.07	2.59**
Error	384	30.94	

** Significant at the 0.01 level of probability.

N.S. = Not significant.

Table 4. — AVERAGE VALUES OF PERCENTAGE VOLUMETRIC ANTI-SWELL EFFICIENCY FOR DIFFERENT CLASSES OF VARIABLES.

	Species				Sp. Gravity		Group Average
	Loblolly	Slash	Longleaf	Shortleaf	Low	High	
Treatment							
Polyethylene Glycol	82.2	75.9	76.2	81.9	81.0	77.1	79.1
Phenolic Resin	77.4	80.0	76.5	76.4	79.7	75.4	77.6
Methyl Methacrylate ¹	69.8	68.8	69.3	71.1	72.4	67.1	69.8
Styrene-Acrylonitrile	69.1	62.7	63.6	71.4	71.0	62.4	66.7
Age Class							
Corewood	76.8	70.9	69.4	75.7	75.5	70.9	73.2
Maturewood	72.5	72.8	73.4	74.4	76.6	70.1	73.3
Group Average	74.6	71.9	71.4	75.3	76.0	70.7	

¹Methanol was used as a swelling agent.

or phenolic resin, better dimensional stability was obtained than when treated with the vinyl monomers. The accumulation of resin, however, is generally more pronounced near the pith and therefore the variations among species are mainly in the corewood rather than in the maturewood (Table 4). The effect of resin was not shown as a main effect in the analysis of variance because of confounding interactions with specific gravity and other variables; therefore, the effect of age class was not significant (Table 3).

The effect of specific gravity was found to be significant. The less dense material gave higher ASE values. Table 4 shows that the average ASE value for the low density group was 76 percent, compared with 70 percent for the high density group. This result is expected when the swelling behavior of untreated and treated wood is compared. It is known that the volumetric shrinkage of untreated wood is linearly proportional to specific gravity (11) if the cell lumen remains constant and if there is no bulking of the extraneous material in the cell wall. If stabilization of wood by bulking did not alter this linear relationship, the ASE values for shortleaf pine treated with various chemicals decreased with an increase in specific gravity; therefore, their corresponding swelling values for treated wood would increase curvilinearly upward. The regression lines of ASE versus specific gravity for the other southern pines showed the same trend.

Conclusions

From this work, the following conclusions are drawn:

- 1) Pre-swelling the wood is necessary to obtain the highest ASE values.
- 2) The slope of moisture content - swelling (or shrinkage) line appears to be an effective method of evaluating stabilization treatments. Smaller differences could be detected by measuring the dimensional changes between two humidities of interest than by using the entire hygroscopic range.
- 3) The non-volatile bulking agents generally are somewhat better stabilizing agents than the vinyl monomers, since more quantities of the non-volatile chemicals can enter the cell wall structure of wood. The highest ASE value was obtained with polyethylene glycol, followed

by phenolic resin, styrene-acrylonitrile, styrene, and methyl methacrylate, in that order. The ASE of methyl methacrylate can be increased with a suitable swelling agent.

4) There are some differences in the ASE values among the southern pines, but these differences are mainly in the corewood and due to resin content. The less resinous species, namely, loblolly and shortleaf pines, gave higher ASE values than slash and longleaf pines.

5) The predominant wood factor influencing dimensional stability appears to be specific gravity. The ASE value increases with a decrease in specific gravity.

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