Effect of delay between treatment and drying on toughness of CCA-treated southern pine

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Abstract

Small clear specimens of southern pine were treated with chromated copper arsenate (CCA) type C preservative, maintained in a saturated condition for various periods of time (time delay), and dried using a technique intended to simulate kiln-drying conditions in full-size members. At CCA retentions of 0.6 pound per cubic foot (pcf), toughness was reduced an average of 20 percent but was not further affected by increasing periods of time delay. At CCA retentions of 2.5 pcf, toughness was reduced 36 percent when dried immediately after treatment and by an additional 11 percent when exposed to time-delay periods of up to 28 days. Small clear test specimens may be more sensitive to changes resulting from treatment and processing variables than are specimens of structural sizes. Therefore, results of these experiments should not be assumed to be directly applicable to full-size dimension material.

Introduction

Waterborne preservatives (WBP) command an ever-increasing share of the treated wood market. The chemistry that gives WBP the desired properties of cleanliness, paintability, minimal leachability, and decay resistance may also affect the strength of the treated material. Chromated copper arsenate (CCA) preservatives are one type of WBP that on contact with wood undergo a series of chemical reactions that change the chemicals from a water-soluble mixture to a highly water-insoluble complex, a process known as fixation. The objective of this study was to determine the effects of various time-delay periods on the mechanism and magnitude of strength losses sometimes found in CCA-treated material. Time-delay is defined as the time elapsed between preservative treatment and subsequent kiln-drying.

Fixation

Upon contact with a carbohydrate substrate, CCA fixation is initiated by a sudden and pronounced decrease in pH (9). This decrease contributes to ion-exchange reactions between the copper and the wood substrate and absorption reactions of the hexavalent chromium (9). Dahlgren (8) found that the pH, after reaching a minimum (maximum hydrogen ion concentration), proceeds to a maximum and then slowly fluctuates, finally stabilizing after about 30 days (Fig. 1). In the range of temperatures evaluated (up to 30°C, 86°F), the rate of fixation increased directly with temperature (23). An increase in chemical concentration also led to faster fixation of the arsenic but slower fixation of copper and chromium (13, 16, 23). An increase in any of the input variables imparts more available energy to the CCA-wood system and thereby increases the magnitudes of the pH fluctuations.

Effect on strength

Because of general lack of sensitivity in experimental design, the literature shows inconsistencies regarding the effect of treatment with commercial WBP formulations on the mechanical properties of wood. Virtually all proprietary preservative formulations in common use contain one or more chemical salts capable of degrading wood by partial hydrolysis of cellulose or by other means (19). Among the factors that influence the degree of degradation of WBP-treated wood are the retention, distribution, and composition of the preservative. Several researchers have shown that acidic solutions can hydrolyze the linkage of the strength-inducing cellulose molecule (12, 17, 20, 22). Thus, CCA as

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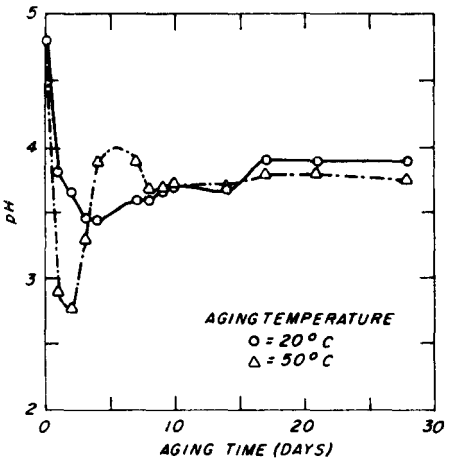

Figure 1. – The pH course during aging of precipitate from Boliden K_{33} (CCA type B). (All measurements carried out at 20°C.) From (8).

an acidic solution has the potential to reduce the strength of wood.

Further analysis of the literature reveals no consensus on the quantitative effects of WBP treatment on mechanical properties of wood. However, several general conclusions appear warranted. Energy absorption characteristics and impact resistance are most affected by WBP treatments with reductions ranging to 50 percent (5, 6, 7, 10, 14, 21). Tensile strength and modulus of rupture also seem to be affected, but these reductions may be insignificant depending upon level of treatment (5, 7, 10, 11, 14, 21). Compression strength and modulus of elasticity seem to be unchanged and in some cases compression strength may be increased by WBP treatment (4, 5, 10, 11, 14, 15).

Time delay

Fixation reactions proceed during the time-delay period and are ultimately finalized during the kiln-drying process. The length of the time-delay period may influence the effects of CCA treatment on strength as a result of the fluctuating pH conditions imposed during the fixation process. Thus, time delay may have important implications on the mechanism and magnitude of strength losses (5, 6, 10, 19) sometimes found in CCA-treated material.

Scope

Toughness tests were used to monitor strength because it was assumed that this property would be most sensitive to fixation effects. These tests evaluated the effects of time-delay periods of 1, 24, 168, 336, and 672 hours on small clear specimens of southern pine sapwood treated to nominal retention levels of 0.6 and 2.5 pounds per cubic foot (pcf, or 9.6 and 40.0 kg/m^3, respectively). These time-delay periods were chosen to represent critical periods based upon the Dahlgren curve (Fig. 1). Results will be useful in guiding future research on effects of treatment and drying on the strength of WBP-treated material and may be useful in identifying processing practices that eliminate or minimize strength loss.

Methods and materials

Longleaf (*Pinus palustris* Mill.) and slash (*Pinus elliottii* Engelm.) pine logs were cut from trees growing on the Harrison Experimental Forest (DeSoto National Forest) near Saucier, Miss. Trees were at least 12 inches in diameter at breast height; logs were 6 feet long with growth rates of 6 to 10 rings per inch in the sapwood portion of the tree. These guidelines minimized material variability in the mechanical properties and assured more uniform preservative treatment between specimens.

All test logs were end-coated to retard drying and checking and were shipped to the Forest Products Laboratory (FPL) for processing. They were sawn into 1.25- by 1.25-inch (3.2 by 3.2 cm) sticks with the rings oriented parallel to a face. Sticks containing 100 percent sapwood were selected for further processing and were air-dried to approximately 20 percent moisture content (MC). During this air-drying process, molds developed on the surface of some stock. Three hundred and eighty-four specimens with dimensions of 0.8 by 0.8 by 11 inches (2 by 2 by 28 cm) were machined from sticks showing no macroscopic evidence of mold or stain. Microscopic analysis was not performed because it was assumed that any nonvisible infestations, if present, were removed during machining from the 1.25- to 0.8-inch (3.2 to 2.0 cm) cross-sectional dimensions.

These 384 specimens were randomly sorted into 11 groups: 10 treatment groups of 32 specimens each and a control group of 64 specimens. All pressure treatments were performed by FPL personnel using standard full-cell procedures as outlined in AWPA Standard C2-1980 (2). The control group was treated with water and solution concentrations of subsequent CCA treatments were determined from the amount of water uptake by the controls. Five groups were pressure treated with CCA-type C preservative to a target retention of 2.5 pcf (40 kg/m^3); the other five groups were treated to a targeted 0.6 pcf (9.6 kg/m^3). Each specimen was weighed before and after treatment.

CCA retentions were calculated by the weight gain method:

$$R = \frac{3.81\ C\ (WT - WI)}{L \cdot W \cdot D}$$

where

R = retention (pcf)
WT = weight treated (g)
WI = weight initial (g)
C = solution concentration expressed as a decimal (weight of salt/weight of water plus salt)
L, W, D = length, width, and depth of specimen (in.) before treatment
3.81 = unit conversion constant

The six specimens exhibiting greatest deviation from the mean retention level were culled from each group of 32 specimens. Twelve specimens deviating most from the mean water pickup were culled from each control group. Each group of specimens then was stickered and sealed in a moisture-proof, heat-resistant polyethylene bag and stored at 80°F (26.6°C) during the time delay between treatment and drying. At intervals

of 1, 24, 168, 336, and 672 hours, measured from the time of release of the treatment pressure, a randomly chosen group of bagged specimens from each of the two retention levels (2.5 or 0.6 pcf) were placed in an oven maintained at 190°F (87.8°C).

Based upon a preliminary kiln-drying run, 46 hours were needed to dry WBP-treated 2-inch nominal dimension lumber at 190°F, but only 6 hours to dry the small toughness specimens. To simulate the drying of full-size material, test specimens were bagged in polyethylene during the first 40 hours of heating. Then the bags were opened and the specimens dried from approximately 70 percent down to 20 percent MC during the last 6 hours. Bagging the specimens thus had served to retard drying as was intended.

Unfortunately, a seam failed in the bag containing specimens treated to 0.6 pcf and scheduled for a 14-day time delay; the MC of these specimens was approximately 2 percent after 40 hours. The effect of this mishap is evident in the results discussed later.

The authors acknowledge that this drying technique might be more severe than conventional drying of CCA-treated lumber at the same temperature; elevated temperatures do have a greater effect on strength at high moisture levels. However, our purpose was to identify the optimum time delay for minimizing strength loss, not to quantify the drying or treatment effect. Thus, we believe that it is of no consequence if the drying of the test specimens did not accurately simulate conventional kiln-drying.

After drying, the specimens were allowed to equilibrate under conditions controlled at 80°F (26.6°C) and 65 percent relative humidity (approximately 12% equilibrium moisture content (EMC)).

The pH of a randomly selected specimen in each group was measured before treatment, after treatment, at the end of the time-delay sequence, and at the time of testing. The pH of the wood was measured as follows:

1) A small quantity of wood was ground into sawdust.
2) Weight was determined for the freshly ground sawdust.
3) The sawdust was soaked in a constant volume of freshly boiled and cooled, deionized water with a known pH.
4) This slurry was placed in a sonic bath for 10 minutes.
5) The slurry was filtered and the pH of the filtrate was measured.

Toughness tests were conducted on an FPL toughness test machine as described in ASTM D 143(1). The widths, depths, and weights of the specimens were measured immediately before each toughness test. Immediately after testing, a 1-inch section was cut from near the failure point to determine MC and specific gravity. A correction for the weight of the preservative in the specimens was included in these measurements. All specimens and controls were tested for toughness in a random order. This eliminated any possible time bias during the test period due to operator experience or machine adjustment.

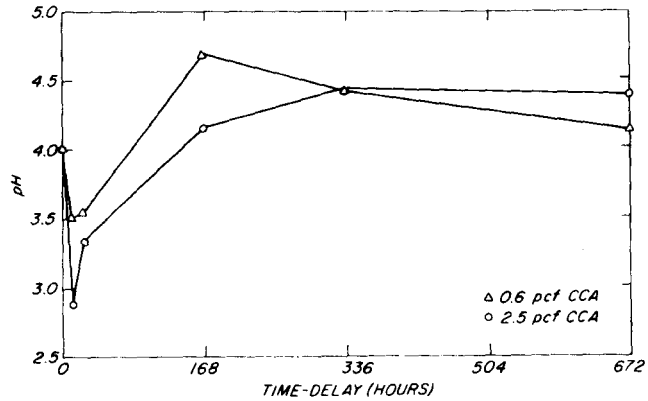

Figure 2. – Variation of wood pH due to increasing periods of time delay for small clear specimens of southern pine treated to two CCA retention levels.

Results and discussion

Treatment

The pH of the treated wood obtained over various time delays (Fig. 2) approximated the Dahlgren pH fixation curve (Fig. 1) rather well. It is known that pH is a reasonable indicator of the rate of CCA fixation (8, 9) and that the rate of fixation is directly related to preservative concentration and temperature (13, 16). It is evident that the pH of the two higher-energy systems (i.e., the Dahlgren 50°C system and our 2.5 pcf system) responded in similar manners. These systems quickly dropped to a pH of 2.7 to 2.9 and eventually stabilized at a pH higher than their lower-energy counterparts at approximately 4.3. The pH of the two lower-energy systems (i.e., the Dahlgren 20°C system and our 0.6 pcf system) also responded with this pattern; the systems reached a pH of about 3.5, then slowly stabilized at a pH of about 4.1.

This similarity in pH fluctuation allows us to relate the fluctuations in wood pH due to the CCA fixation process over the various levels of time-delay periods to the toughness of the treated material. That is, we can assume that any effects on toughness are a result of the fluctuations in the pH of the treated material.

Average salt retentions at the targeted 0.6 pcf level for the five time-delay periods ranged from 0.60 to 0.62 pcf and coefficients of variation from 3.9 to 6.9 percent (Table 1). At the 2.5 pcf level, the average retentions ranged from 2.74 to 2.94 and coefficients of variation from 3.7 to 6.8 percent. Though actual retentions are higher than targeted, the small differences are assumed to be of no consequence to the experiment.

Equilibrium moisture content

Average EMCs of specimens at the nominal 0.6 pcf level are consistent (≃13.6%) except for the 336-hour time-delay period (11.5%)(Table 2). The specimens of this group are those that were inadvertently dried to very low MCs because of the bag failure. The lower average EMC of this group occurred at least partially because these specimens approached final equilibrium under adsorbing conditions while others were under

TABLE 1. —*Actual retention, coefficient of variation, and range of CCA-treated small clear specimens of southern pine.*

Nominal retention (pcf)	Property	Time delay (hours) 1	24	168	336	672	Avg. of all specimens
0.6	Retention (pcf)	0.615	0.604	0.606	0.619	0.612	0.611
	Coefficient of variation (pct)	(4.9)	(6.3)	(6.9)	(3.9)	(3.9)	(5.2)
	Range (pcf)	0.571-0.668	0.523-0.662	0.559-0.662	0.551-0.659	0.564-0.654	0.523-0.663
2.5	Retention (pcf)	2.745	2.848	2.939	2.840	2.928	2.860
	Coefficient of variation (pct)	(6.8)	(5.5)	(3.7)	(5.2)	(3.7)	(5.0)
	Range (pcf)	2.337-3.034	2.406-3.091	2.730-3.122	2.634-3.072	2.718-3.141	2.337-3.141

TABLE 2 — *Moisture content after equilibration in 12 percent EMC Conditions and specific gravity[a] of CCA -treated small clear specimens of southern pine*

Nominal retention (pcf)	Property	Time delay (hours) 1	24	168	336	672
2.5	Moisture content	13.87	14.12	14.46	14.36	15.14
	Specific gravity	.64	.62	.59	.62	.60
0.6	Moisture content	13.51	13.59	13.65	11.51	13.61
	Specific gravity	.58	.60	.60	.59	.59
0.0	Moisture content	13.34	—	—	—	—
	Specific gravity	.57				

[a]Based on ovendry weight and volume at 126.

TABLE 3 — *Average toughness and standard deviation (in. -lb) of CCA-treated small clear specimens of southern pine*

Nominal retention (pcf)	Property	Time delay (hours) 1	24	168	336	672
25	Toughness	1265	1235	1067	1182	1043
	Standard deviation	356	284	243	345	226
06	Toughness	1532	1647	1565	2025	1516
	Standard deviation	413	533	301	420	411
00	Toughness	196 7				
	Standard deviation	41 4				

desorbing conditions. The average EMCs of the nominal 2.5 pcf treatments were higher than those for the 0.6 pcf level. They also tended to increase slightly with the increasing time delay: EMC at 1 hour was 13.9 percent; at 672 hours, 15.1 percent.

Toughness

Average toughness values and standard deviations are in Table 3. It is evident that the mishap involving the bag broken during drying affected the toughness results because the values for these specimens (0.6 pcf, 336-hr. time delay) are considerably higher than for any other treatment group. The higher values cannot be associated with the lower EMC of the specimens in this group because the moisture differences are small and energy-absorption characteristics are generally not affected by MC. It may be that CCA-treated wood is not degraded if rapidly dried to very low moisture levels. However, further research to explain this disparity in toughness results was inconclusive and no substantiated evidence can be found to document why the specimens in the broken bag performed so well.

Analysis

A two-way analysis of variance (ANOVA) was conducted to test for the effects of retentioh level and time delay on toughness (Table 4). The results for the 336-hour time-delay period at the nominal 0.6 pcf

TABLE 4. — *Analysis of variance for the effects of preservative retention and time-delay period upon the toughness of CCA-treated small clear Specimens of southern pine.*

Source of variation	Degree of freedom	Sum of squares	F	Probability level
TREATMENTS				
Retention	2	280994	103.	<.0001
Control versus others	1	178162	131.	<.0001
0.6 versus 2.5	1	102832	75.7	<.0001
Time	4	9411	1.73	<.1433
Time by retention	3	4526	1.11	N.S.
ERROR	297	403398		
TOTAL	306	698328		

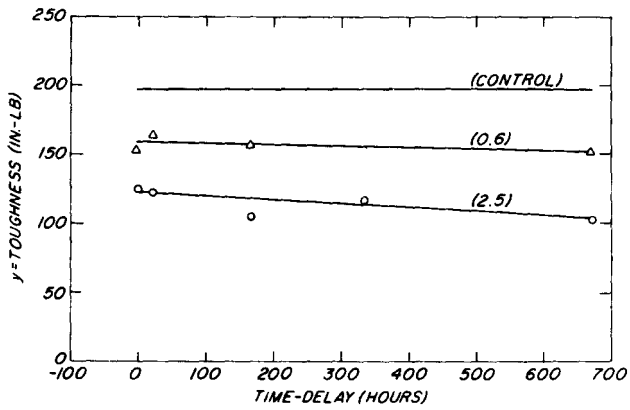

Figure 3. — Effects of time-delay (hours) on the toughness of small clear specimens of southern pine. Estimated regression equation for CCA-treated southern pine at 0.6 pcf is y = 158.152 - 0.0105 (x) (R^2=0.005) and for 2.5 pcf is y=122.389 - 0.0273 (x) (R^2 = 0.049) where y = toughness (in.-lb.) and x = time-delay (hr.).

retention level were not included in the analysis because it was obvious by inspection that these data do not reflect the true treatment response.

A Bartlett's test (18) for homogeneity of variance showed that the variances between treatment cells were significantly different. The variation in toughness of the untreated controls was similar to that at the 0.6 pcf treatment level, but at the 2.5 pcf level the variation was substantially less (Table 3).

While variance heterogeneity does not allow for strict interpretation of the ANOVA, general trends can still be observed and tested, although the precision of the tests is somewhat diminished. The interaction between treatment level and time delay was not significant (Table 4). When retention levels were pooled, time delay also was not significant, but this test did have a significance level of a<.1433. Thus, considering the decreased sensitivity of the ANOVA due to the variance heterogeneity, the effect of time delay should not be dismissed entirely. The toughness of small clear specimens of southern pine was reduced by CCA treatment: when pooled across time delay, the toughness of the treated material was significantly lower than the controls (a<.0001). In addition, the 2.5 pcf CCA retention level was quite different than the 0.6 pcf level (a<.0001).

Because the ANOVA assumed equal spacing of the time-delay periods, the sensitivity of the analysis was further reduced by the unequal (nonlinear) time-delay periods employed (1, 24, 168, 336, and 672 hours). Because these time intervals were chosen to monitor specific time-delay periods of interest as described by Dahlgren (8), a more sensitive analytical procedure was needed. A regression analysis (of the form $y = b_o + b_1 x$) was conducted on the effect of time delay versus toughness. The results for the 336-hour time delay at the nominal 0.6 pcf retention level again were not included in the analysis. The regression equations of time (x = hours) versus toughness (y) were

$$y = 158.8 - 0.010(x)$$

for the 0.6 pcf CCA retention level with an R^2 value of 0.005 and

$$y = 122.4 - 0.027(x)$$

for the 2.5 pcf CCA retention level with an R^2 value of 0.049 (Fig. 3).

Although the R^2 values are low, this in part reflects the large natural variability in wood when tested for toughness compared to the relatively small effect due to time delay rather than a complete lack of relationship. However, the means of the 2.5 pcf group show a decreasing trend over time (126.5, 123.5, 106.7, 118.2, and 104.3). Additionally, a discordancy test for upper outliers (3) shows two observations at the 2.5 pcf retention level and 336-hour time-delay period to be outliers (a<.01). When these two values were deleted the new mean and standard deviation were 111.4 and 24.5, making the general negative linear trend of the deleted data even more apparent (126.5, 123.5, 106.7, 111.4, and 104.3). In fact, the pattern of the means of the deleted data might even suggest a cubic model, but this type of model, when tested, did not greatly increase the R^2 value (R^2 = 0.085 at 2.5 pcf). The means of the 0.6 pcf group also appear to exhibit a negative linear trend over time but to a lesser extent than the 2.5 pcf level. Therefore, only straight-line models were considered.

Statistically, the slope of the nominal 0.6 pcf retention level can be considered flat (a<.4765), whereas the slope of the nominal 2.5 pcf retention level has a significantly (a<.01) negative slope. Thus, it appears that at the 0.6 pcf retention level, toughness is not affected by increasing periods of time delay but, at the 2.5 pcf retention level, toughness decreases as time delay increases.

However, a statistical test for equality of slopes does not rule out the possibility that both of these regressed slopes are decreasing at the same rate. The possibility of the two retention levels having the same intercept was tested and found to be implausible. This agrees with the difference due to treatment found to exist in the ANOVA.

Summary and conclusions

The objective of this study was to establish the effects on toughness of various delay periods between treatment with **CCA** and subsequent kiln-drying. The

toughness of small clear specimens of southern pine sapwood was measured after they had been 1) treated with CCA to either 0.6 or 2.5 pcf retention, 2) maintained in a saturated condition at 80°F for various time periods and 3) dried at 190°F using a technique intended to simulate kiln-drying in full-size members.

The simulated kiln-drying was probably more severe than common industry practice. This factor may have caused lower average toughness results, but we do not believe that it influenced the time-delay trends observed.

Toughness tests were chosen to measure the strength response to time delay because this property is usually affected more by treatment, thus increasing the likelihood of detecting a time-delay effect. We do not expect that other properties would show the same quantitative response to time delay, yet the same general trends and conclusions made from toughness results are probably applicable to other properties as well.

The delay period between treatment and drying had no significant effect on the toughness of small clear specimens of southern pine treated to 0.6 pcf (9.6 kg/m^3).

Increasing periods of delay between treatment and drying had significant ($a<.01$) negative effects on the toughness of small clear specimens of southern pine treated to 2.5 pcf (40.0 kg/m^3). Based upon regression analysis at the 2.5 pcf level, the expected loss in toughness is about 9 percent greater (based on untreated controls) for a time delay of 672 hours than for 1 hour.

Toughness was significantly reduced by treatment itself, ranging from about 16 to 23 percent at the 0.6 pcf treatment level and from 36 to 47 percent at the 2.5 pcf level depending on time delay. Although our drying may have been more severe than common practice, these reductions are similar to those observed by Bendtsen and Gjovik (5) for work to maximum load values (rapid bending tests) in specimens treated to comparable levels of CCA and air-dried after treatment.

We conclude that the time delay between treatment with CCA and subsequent kiln-drying has little practical significance as far as strength losses are concerned. The maximum expected loss in toughness due to time delay is 9 percent (2.5 pcf-672 hr.); other important strength properties are most likely affected even less.

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