EFFECT OF VARIOUS TREATMENTS AND ADDITIVES ON WOOD-PORTLAND CEMENT-WATER SYSTEMS

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ABSTRACT

In this study, a series of treatments were considered to determine the impact of such treatments on wood-cement compatibility. These treatments included hot water extraction of water-soluble components in wood, and/or chemical additives. The species involved included lodgepole pine and western larch. The latter species, in a prior study, proved to be highly inhibitory in a series of hydration tests. The data presented in this paper show that substantial improvements in cement setting can be achieved by the removal of water-soluble extractives and sugars in western larch. Such improvements, however, did not take place in lodgepole pine, which data show is far less inhibitory than larch in the untreated state. The addition of chemical additives, especially calcium chloride, appears to enhance compatibility in cement-wood-water mixes.

Keywords: Larch, lodgepole pine, wood-cement particleboard, particleboard, hydration.

INTRODUCTION

In a previous study, we reported the results of monitoring hydration temperatures for wood-portland cement-water systems for a number of wood species in Idaho and adjacent areas (Hofstrand et al. 1981). We pointed out that a wood-portland cement-water system is highly species-sensitive as determined by hydration temperature data. In the study, we included nine species: inland Douglas-fir, ponderosa pine, western hemlock, grand fir, western white pine, Engelmann spruce, lodgepole pine, western redcedar, and western larch.

The basic hydration parameters considered in this series of experiments are shown in Fig. 1. These include six parameters as shown at the bottom of the figure. To simplify terminology, we will use the symbols noted in Fig. 1 in describing our results.

In the prior study referred to above, we found that lodgepole pine interfered

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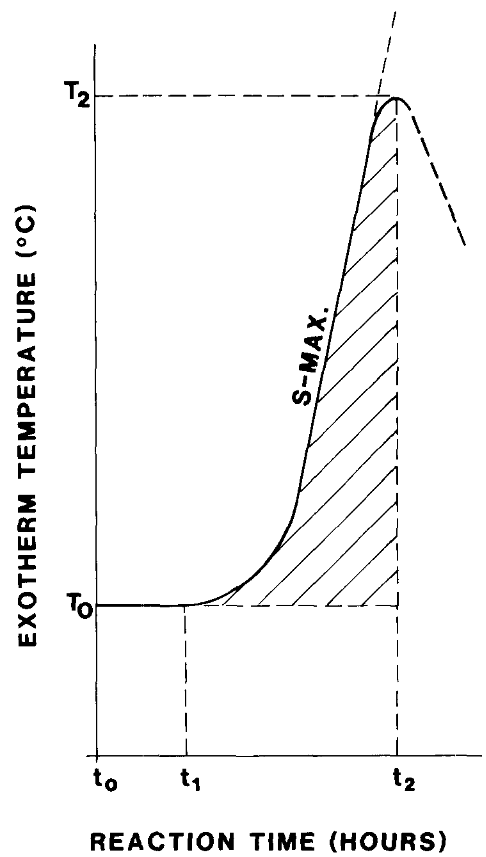

FIG. 1. A schematic representation of the typical behavior of wood-cement reactions. t_0 = initial time, start of hydration experiment; t_1 = time at which exothermic reaction begins; t_2 = time at which maximum temperature is reached; T_0 = temperature at start of experimentation; T_1 = maximum hydration temperature; S_{max} = maximum slope of hydration curve.

the least with cement hydration, while larch proved to be the most inhibitory. The high level of species influence on cement setting has been experienced by a number of researchers (Batyrbaev and Akchabaev 1967; Bugrina et al. 1968; Buzhevich et al. 1968; Davis 1966; Fischer et al. 1974; Sanderman 1966, 1969; Sanderman et al. 1960; Weatherwax 1964). Variations on the setting of wood-cement have also been noted based on geographic location of the species, felling season, and tree components (Sanderman et al. 1960; Stephen et al. 1974).

The considerable influence of species on hardening of cement can constitute a significant problem for a cement-bonded panel industry where residue from a variety of species may have to be accepted as raw material. Thus, special treatments and/or procedures may have to be devised that would ideally enable any species to be accommodated. The results reported in this paper have as their objective the exploration of various wood treatments and chemical additives in the attempt to reduce the inhibitory effects of wood species in wood-cement-water mixes.

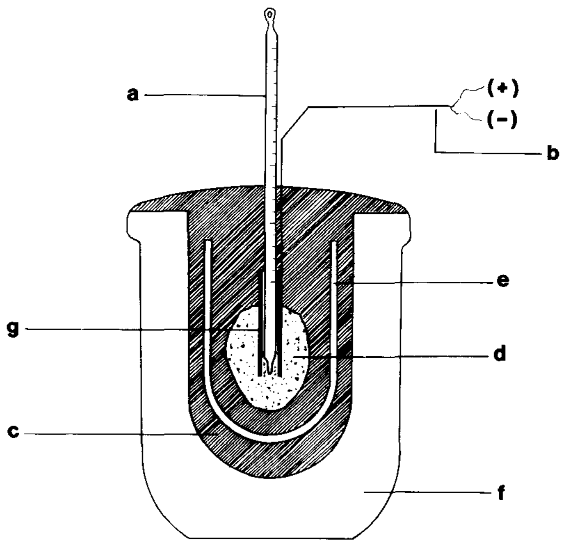

FIG. 2. Design used to determine the heat of hydration of the wood-cement-water mixtures. (a) thermometer, (b) thermocouple wire. (c) fiberglass, (d) wood-cement mixture, (e) thermo-replacement filler, (f) Dewar flask, (g) copper pipe sleeve.

REMOVAL OF SUGARS AND EXTRACTIVES

In prior research, a variety of investigators have concluded that differences in behavior of species when mixed with cement are due primarily to water-soluble substances present in the cell walls of the wood (Biblis and Lo 1968; Bugrina et al. 1968; Fischer et al. 1974; Sanderman 1969; Takahashi et al. 1974; Voitovich and Yavorskii 1972; Weatherwax and Tarkov 1967). On the basis of this prior research information, we initiated a series of tests designed to remove at least a portion of the wood extractives and sugars by the use of hot water and dilute NaOH solution. The objective was to treat the most inhibitory (larch) and the least inhibitory (lodgepole pine) species as identified in our prior research (Hofstrand et al. 1981) to provide useful comparisons.

In this portion of our experiment, 500 grams of hammermilled wood (−20 +40 mesh) for each of the two selected species was heated to boiling in a 2-liter beaker for 6 h. Every 2 h, the wood was washed with boiling distilled water and reheated

TABLE 1. *Results of average (n = 4) values of time and temperature of hydration for treated and untreated lodgepole pine and larch-cement mixtures.*

Species	Treatment	t_2 (h)	Duncan's[1]	T_2 (C)	Duncan's[1]
Lodgepole pine	Untreated	12	a	74.50	b
	Hot water treated	11.63	a	58.50	a
	1% NaOH treated	11.88	a	68.50	a
Larch	Untreated	24.00	c	25.75	b
	Hot water treated	10.38	a	66.50	a
	1% NaOH treated	11.25	b	63.25	a

[1] Means with the same letter are not significantly different ($P < 0.05$).

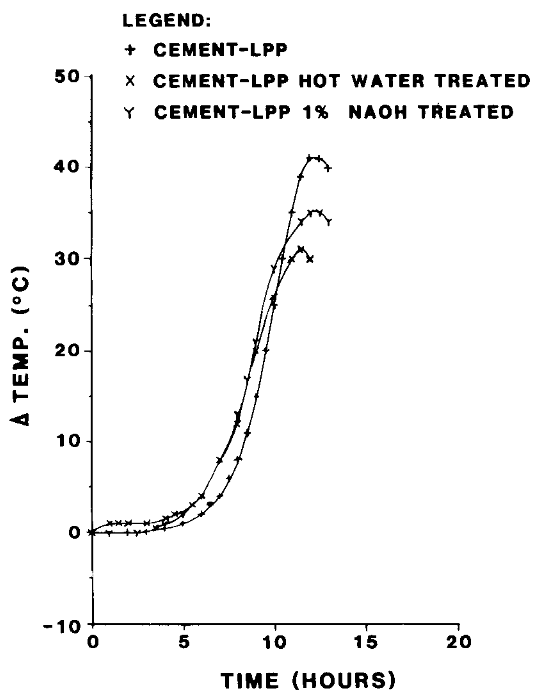

FIG. 3. Hydration data for treated lodgepole pine-cement-water system. The treatment included removal of water-soluble extractives by hot water and dilute solution of sodium hydroxide. A comparison is made with untreated data.

to boiling. After the 6-h heating period, the boiled wood particles were collected by pouring such particles in a 200-mesh screen. The collected wood particles were then washed twice with hot distilled water and dried for 24 h in an oven at 103 C. Two hundred grams of the oven-dry wood for each of the two species were then additionally treated with 1% of NaOH solution for 24 h to determine if further extraction of sugars and extractives could be achieved by this treatment.

To obtain the hydration data for the treated wood-portland cement-water system, 200 grams of cement and 15 grams of hammermilled wood particles based on oven-dry weight were dry-mixed in a plastic bag. The mixture was kneaded with 90.5 ml of distilled water (2.7 ml of water per gram of oven-dry wood and an additional 0.25 ml of water per gram of cement) based on prior research experience (Weatherwax and Tarkov 1967). The plastic bag containing the wood-cement-water mixture was wrapped with aluminum foil and placed into a 1-liter Dewar flask. A thermometer was inserted into a 10-cm copper pipe sleeve around which the junction of an iron-constantan thermocouple wire was wrapped with aluminum foil. The thermometer and the thermocouple junction, together with the copper pipe, were inserted into the approximate center of the mixture. The flask was then promptly insulated by placing several layers of fiberglass on the top and sides, and it was then wrapped with masking tape (Fig. 2). All experiments were carried out at an ambient room temperature between 21–23 C.

The temperature was measured at 30-min intervals, except for the first 5 h in

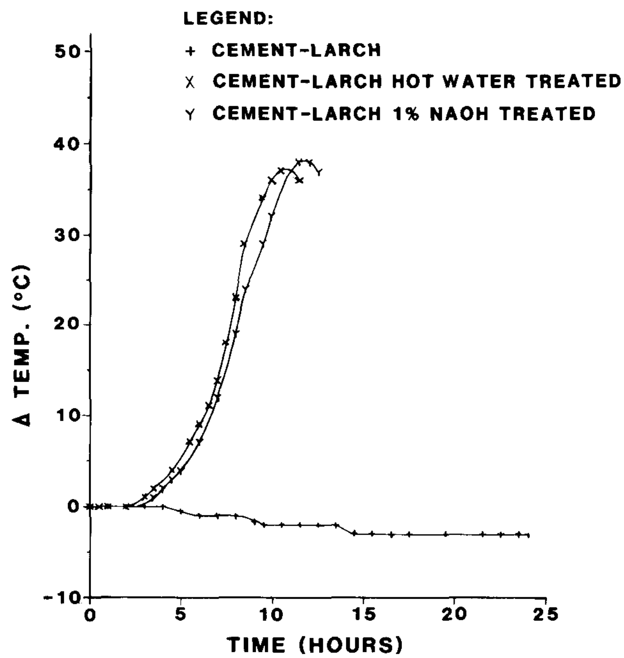

FIG. 4. Hydration data for treated larch-cement-water system. The treatment included removal of water-soluble extractives by hot water and dilute solution of sodium hydroxide. A comparison is made with untreated data.

which no measurable change in temperature took place. Four replications for each species-cement mixture were made.

The results of this portion of the study are recorded in Table 1. Statistical analysis of the results shows no significant differences among the t_2 values of the treated and the untreated logdepole pine-cement mixtures. However, significant differences were found for the maximum temperatures reached for each treatment given to lodgepole pine. The maximum hydration temperature appears to be reduced by the treatments (Fig. 3). The reason for this behavior is not well known. This could perhaps involve possible alteration of the physical characteristics of the wood itself, resulting in decreased thermal conductivity of the wood component in the wood-cement-water mixture.

The picture for larch is dramatically different from that just presented for lodgepole pine: highly significant differences exist between treated and untreated mixtures as Fig. 4 indicates. The results presented in Table 1 and Fig. 4 indicate that the untreated cement-larch-water system completely failed to set over the 24-h test period. However, when larch was treated, the hardening of the mixture took place, producing strong exothermic reaction over a relatively short period of time. Statistical analysis showed no significant differences between the two treatments used (hot water and 1% NaOH). On the contrary, significant differences were encountered between either of these treatments with the untreated larch. Furthermore, hot water performed better in reducing t_2 and in elevating T_2.

The calculation of the Inhibitory Index was carried out on the basis of the

TABLE 2. *Inhibitory index for treated and untreated lodgepole pine and larch.*

Species	Treatment	Inhibitory index (%)
Lodgepole pine	Untreated	2.57
	Hot water treated	6.05
	1% NaOH treated	5.57
Larch	Untreated	118.26
	Hot water treated	2.10
	1% NaOH treated	6.21

equation presented in our earlier paper (Hofstrand et al. 1981) and it is presented here for convenience:

$$I = 100[(t_2 - t'_2/t'_2)\ (T'_2 - T_2')(S'_2 - S_2/S'_2)]$$

t_2 = t_2 of wood-cement-water
t'_2 = t_2 of cement-water
T_2 = T_2 of wood-cement-water
T'_2 = T_2 of cement-water
S_2 = S_{max} of wood-cement-water
S'_2 = S_{max} of cement-water.

The results of these calculations, which take into account the various parameters of the hydration tests, are shown in Table 2. As noted in this table, substantial improvement is encountered in larch with hot water or with a dilute solution of caustic soda. A possible explanation for this behavior can be attributed to the removal of sugars and other water-soluble extractives from larch, which appear to be highly inhibitory to setting of portland cement in their natural state. These results appear to be consistent with research carried out in other parts of the world (Jai and Chen 1977; Klar and Van'kov 1971; Krekel 1972). For example, Klar and Van'kov (1971) noted that water-soluble constituents arabinogalactan and sucrose present in European larch produced detrimental results in the hardening of cement.

CHEMICAL ADDITIVES

In the attempt to improve the setting of cement, a number of researchers have explored the inclusions of chemical additives to the wood-cement-water systems.

TABLE 3. *Effect of chemical additives (5%) on the maximum hydration time (t_2) and maximum temperature (T_2) of lodgepole pine and larch-cement mixtures.*

Species	Chemical additives	t_2 (h)	T_2 (C)
Lodgepole pine	$CaCl_2$	5.0	67.0
	NaOH	7.0	71.5
	$MgCl_2$	7.5	63.3
	$CaOH_2$	9.0	47.2
Larch	$CaCl_2$	12.0[1]	26.0
	NaOH	12.0[1]	30.7
	$MgCl_2$	12.0[1]	25.0
	$CaOH_2$	12.0[1]	26.0

[1] Not the setting time.

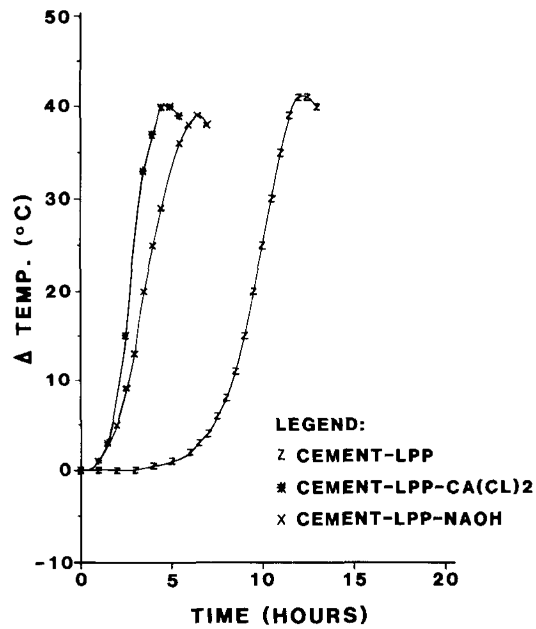

FIG. 5. The effect of calcium chloride and sodium hydroxide on the hydration data for lodgepole pine-cement-water system.

Such additives as monoethanolamine and diathanolamine (Voitovich and Yavorskii 1972; Weatherwax and Tarkov 1967), calcium chloride (Biblis and Lo 1968; Davis 1966; Flaws and Chittenden 1967; Klar 1975; Krekel 1972; Takano et al. 1977) have been used to enhance cement setting. Other additives such as ferric chloride ($FeCl_3$), ferric sulfate ($Fe_2(SO_4)_3$), magnesium chloride ($MgCl_3$) and calcium hydroxide ($Ca(OH)_2$) have been reported as lowering the inhibitory effects of wood on setting of portland cement (Krekel 1972; Mesheheryakova et al. 1969; Takahashi et al. 1974). Prior research on additives is generally inconclusive.

The influence of chemical additives on wood-cement-water systems for the species of interest to us (lodgepole pine, larch) involved two phases. The first phase was designed to determine the influence of chemical additives on the treated (with hot water and 1% NaOH) and untreated wood of the species indicated.

TABLE 4. *Average values (n = 4) of time and temperature of hydration of lodgepole pine and western larch when chemicals are added*

Species	Chemical	t_1 (h)	t_2 (h)	$(t_2 - t_1)$ (h)	T_0 (C)	T_2 (C)	$(T_2 - T_0)$ (C)
Lodgepole pine	$CaCl_2$	1	4.50	3.50	27	66.75	39.75
	NaOH	1	6.63	5.63	34	72.75	38.75
	None	4	12.00	8.00	32	74.50	42.50
Larch	$CaCl_2$	(—)[1]	24.00	(—)	27	26.50	−0.50
	NaOH	(—)	24.00	(—)	31	30.00	−1.00
	None	(—)	24.00	(—)	29	26.00	−3.00

[1] (—) indicates no exothermic reaction taking place within the 24-hour test period.

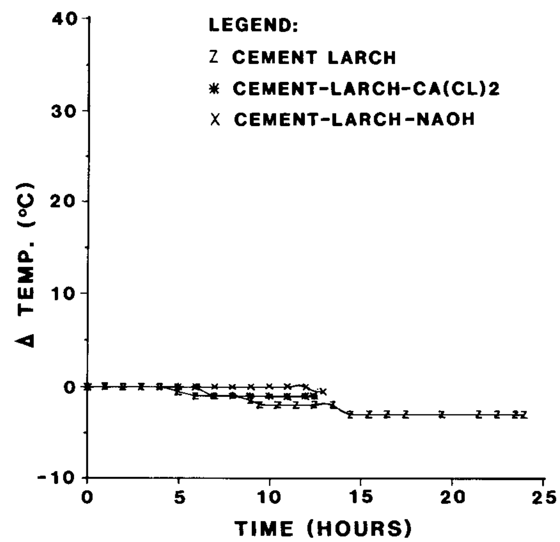

FIG. 6. The effect of calcium chloride and sodium hydroxide on the hydration data for larch-cement-water system.

UNTREATED WOOD

In a series of preliminary tests, 5% solution concentrations of $CaCl_2$, $MgCl_2$, $Ca(OH)_2$, and NaOH were used in combination with lodgepole pine- and larch-cement-water mixes. The proportion of wood, cement and water was that indicated earlier in this paper. The results of these preliminary tests are shown in Table 3. The results show that, among the additives included, $CaCl_2$ and NaOH appear to bring about a better response. These chemicals, therefore, were selected for further experimentation.

The average values for the variables involved in this exothermic reaction of lodgepole pine- and larch-cement mixtures under the influence of the chemical additives are presented in Table 4. The analysis of these results shows that the addition of 5% $CaCl_2$ reduces the reaction time of lodgepole pine from 12 to 4.5 h. It is also interesting to note that the average maximum hydration temperature reached was reduced from 74.5 to 66.8 C. The addition of 5% NaOH reduced the t_2 of lodgepole pine-cement mixture from 12 to 6.6 h. However, the average T_2 values did not change significantly. These results indicate that both chemical accelerators reduced the average reaction time of the lodgepole pine-cement mixtures. The overall difference in the amount of heat evolved was not significantly different when compared to the control specimens. Figure 5 presents a comparison of the effect of chemical additives on the rate of hydration for lodgepole pine. In contrast, the addition of $CaCl_2$ and NaOH to the larch-cement mixture did not produce any noticeable effect in the exothermic reaction of the mixture (Fig. 6). Instead, an endothermic reaction was observed for larch, as Fig. 6 indicates.

From the results of this section, it is possible to surmise that the effects of the chemical additives (accelerators) are to speed up the exothermic reaction of cement when combined with low inhibitory species. However, the chemical additives do not appear to neutralize the detrimental effect of high inhibitory species on the

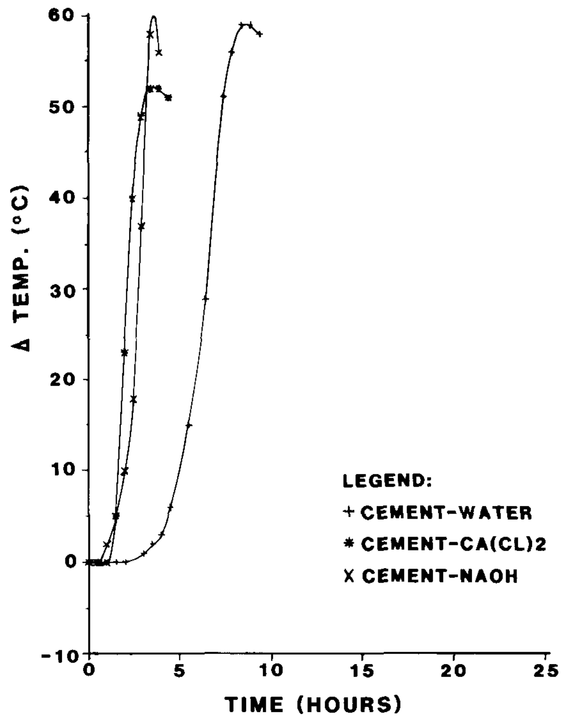

FIG. 7. The effect of calcium chloride and sodium hydroxide on the hydration data for cement-water system (no wood in the mix).

exothermic reaction of cement. This statement is in contrast with earlier conclusions made by Biblis and Lo (1968). They reported a significant reduction in the setting time of southern pine-cement mixtures with the addition of $CaCl_2$ solutions. These authors believed such time reductions were due to the neutralizing effects of $CaCl_2$ on the large amounts of sugars contained in the wood.

Another possible explanation is that the influence of the chemical additives is to speed up the rate of hydration of plain cement without reacting with the wood substances. In this respect, Pazner (1978) noted that the use of activated magnesite cement overcomes the problem of species having high percentages of sugars and tannins. He notes that hardening of cement is so fast (5–15 min) that no opportunity is given to detrimental wood substances to impair the exothermic reaction of the cement.

The influence of both chemical additives on a cement-water mixture (without wood) is illustrated in Fig. 7. A comparison of this figure with Fig. 5 indicates that a proportion of reduction in the average t_2 values occurs with the addition of either chemical additive regardless of the presence of lodgepole pine particles. However, the average maximum hydration temperature reached by the wood cement mixtures under the influence of the chemical additives was reduced when compared to the values reached by the control specimen (cement-water with chemical additives). This behavior suggests that the addition of wood particles tends to interfere with the heat conductivity in the system.

TABLE 5. *Average value for time and temperature of hydration for treated lodgepole pine and larch-cement mixtures with and without the addition of chemical additives.*

Species	Treatment	n	Additive	t_1 (h)	t_2 (h)	$t_2 - t_1$ (h)	T_0 (C)	T_2 (C)	$T_2 - T_1$ (C)
Lodgepole pine	Hot water	4	$CaCl_2$	1.5	4.13	2.63	32	73.00	41.00
	1% NaOH	4	$CaCl_2$	0.5	4.63	4.13	29	70.25	41.25
	Hot water	4	None	4.0	11.63	7.63	27	58.50	31.25
	Hot water	4	NaOH	2.0	7.13	5.13	33	65.75	32.75
	1% NaOH	4	NaOH	1.0	7.63	6.63	34	68.75	34.75
	1% NaOH	4	None	3.5	11.81	8.31	27	62.50	35.50
Larch	Hot water	4	$CaCl_2$	0.0	4.50	4.50	29	70.00	41.00
	1% NaOH	4	$CaCl_2$	0.5	5.25	4.75	29	71.00	42.00
	Hot water	4	None	2.0	10.38	8.38	29	66.50	37.50
	Hot water	4	NaOH	1.0	6.68	5.68	32	69.00	37.00
	1% NaOH	4	NaOH	0.5	7.25	6.75	33	69.00	36.00
	1% NaOH	4	None	1.0	11.25	10.25	25	63.25	38.25

TREATED WOOD

In this test series, we intended to determine the effect of $CaCl_2$ and NaOH on the exothermic behavior of cement when combined with wood previously treated with hot water or 1% solution of NaOH, as described earlier. Again, two species—lodgepole pine and larch—were included.

Results of these tests are presented in Table 5. An analysis of these data indicates that the addition of chemical accelerators into the treated wood-cement mixtures produced a significant reduction in the time required to reach maximum temperature (t_2) as well as an increase in the maximum temperature of hydration (T_2).

The addition of 5% calcium chloride to the hot water-treated lodgepole pine-cement mixture resulted in a reduction of the average t_2 values from 11.63 to 4.13 h, with an increase in the average reaction temperature of 27% (58.5 C to 73 C). However, the addition of 5% NaOH to the treated wood cement mixture gave more conservative results, reducing the average reaction time from 11.63 to 7.13 h. The reduction in the average t_2 values was accompanied by an increase of the exothermic temperature of approximately 7 C or an increase of 12.4%. Similar results were obtained by the addition of the chemical accelerators to the 1% NaOH-treated lodgepole pine-cement mixtures. Figures 8a, b illustrate a comparison showing that both chemical additives substantially improve the hydration of treated lodgepole pine-cement mixtures. This figure further shows that between the two additives tested, $CaCl_2$ had more influence on the hydration data.

A similar pattern to that of lodgepole pine emerges when chemical accelerators are added to western larch-cement mixtures. Significant reduction in reaction time (10.38 h reduced to 4.5 h) occurred when 5% ($CaCl_2$ was added to the hot water-treated larch-cement mixtures. The increase in the maximum temperature of hydration was modest (from 66.5 C to 69 C). The addition of 5% NaOH to the treated larch-cement mixture resulted in a reduction of the average reaction time (t_2) of approximately 37% (10.38 to 6.68 h). A similar trend was exhibited when the chemical accelerators were added to the 1% NaOH-treated larch-cement mix-

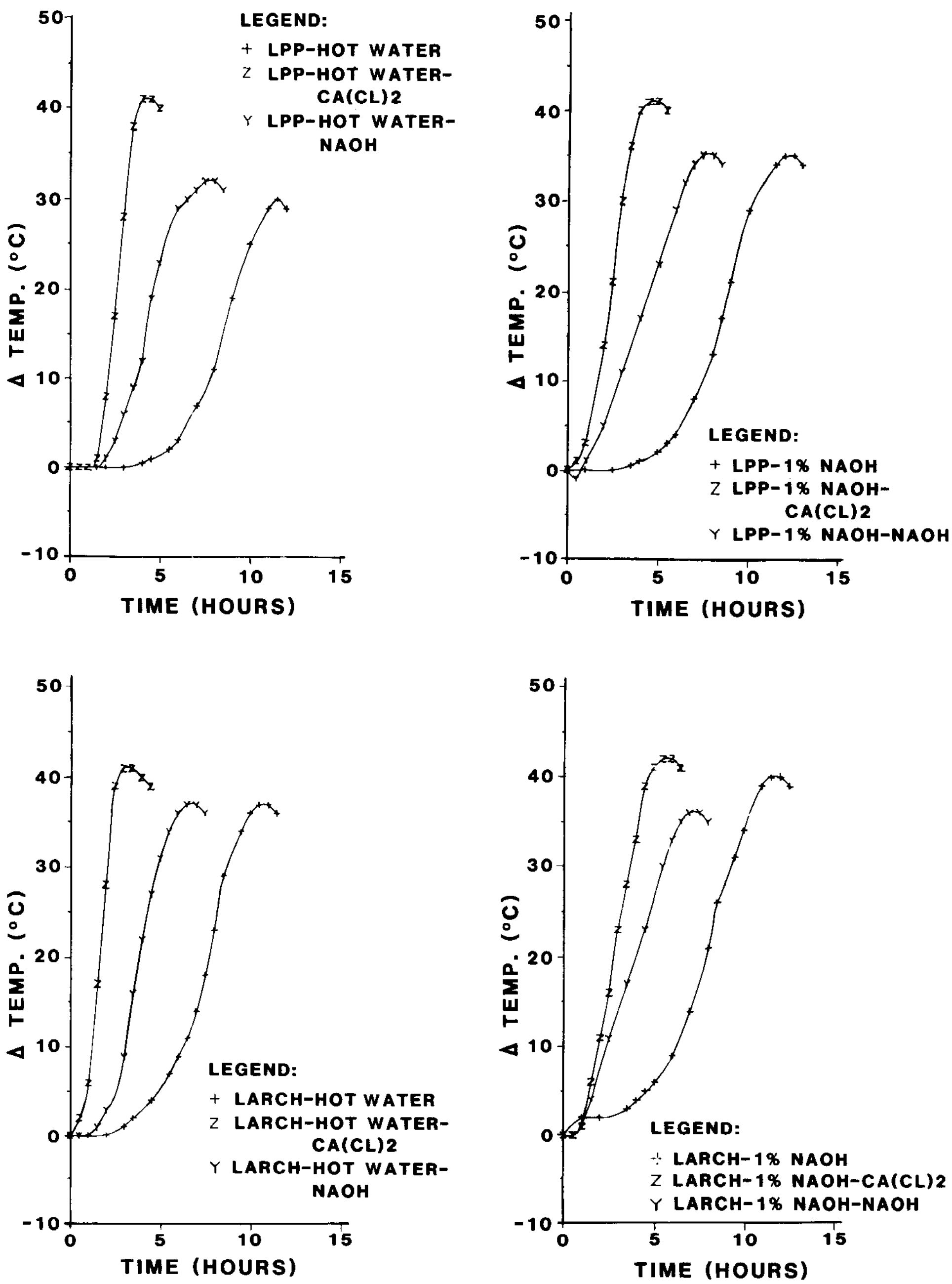

FIG. 8. The effect of chemical additives (calcium chloride and sodium hydroxide) on the previously treated wood (hot water extraction and extraction by dilute solution of sodium hydroxide). (a) effect of chemical additives on hot water treated lodgepole pine, (b) effect of chemical additives on 1% NaOH treated lodgepole pine, (c) effect on hot water treated larch, and (d) effect on 1% NaOH treated larch.

tures (see Table 5). A comparison has been provided in Figs. 8b and c. These figures resemble closely those obtained with lodgepole pine-cement mixtures.

Examining t_2 and T_2 data of the treated wood-cement mixtures as influenced by the addition of chemical accelerators indicates that the action of the additives is to speed up or increase the exothermic reaction of the cement. It is possible, therefore, that the function of the treated wood in the wood-cement mixture is primarily to take part as a filler material. The presence of the treated wood on the cement mixture is believed to reduce the thermal conductivity of the heat generated by the hydration of the cement. This is based on the assumption that the chemical components remaining in the wood after extractives and water-soluble sugars have been removed are primarily cellulose, hemicellulose, and lignin. These substances are insoluble in water and are characterized by a highly stable chemical composition.

The data obtained in this section and statistically analyzed would suggest that among the procedures employed, hot water treatment with the addition of a 5% $CaCl_2$ will produce the best results for larch-cement mixtures. On the other hand, the simple addition of 5% $CaCl_2$ will generate the best results for the untreated lodgepole pine-cement mixtures.

CONCLUSIONS

On the basis of the data collected in this series of experiments, it can be concluded that substantial improvements in cement setting can be achieved by removal of water-soluble extractives and sugars in larch. The addition of chemical additives, particularly $CaCl_2$, can further improve the cement setting in a larch-cement-water system. Such improvements are not, however, applicable to all species. Lodgepole pine, which is normally not an inhibitory species, needs no treatment with either hot water or dilute solutions of caustic soda. However, probably stronger bonds can be achieved between wood (larch or lodgepole pine) and cement by addition of $CaCl_2$. Our prior research indicates that $CaCl_2$ enhances crystalline formation in cement and thereby increases the mechanical interlocking between the cement binder and the wood particles.

Earlier in this paper, we referred to our prior research examining nine species including larch and lodgepole pine (Hofstrand et al. 1981). What could we state with regard to the seven species we did not include in this study (western white pine, grand fir, Englemann spruce, western redcedar, ponderosa pine, Douglas-fir, and western hemlock) as they pertain to the effect of hot water or dilute NaOH extraction? It is likely that either hot water or NaOH extraction would improve cement setting in a wood-cement mix for highly inhibitory species such as Douglas-fir and western hemlock. However, this procedure is probably not necessary for the other species (i.e., western white pine, grand fir, Englemann spruce, western redcedar and ponderosa pine), as they are not so inhibitory as larch to the setting of cement. The addition of additives, particularly calcium chloride, is likely to accelerate setting in all cases.

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