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Hydration behavior of wood cement-based composite I: evaluation of wood species effects on compatibility and strength with ordinary portland cement

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Abstract As an essential preliminary evaluation for understanding the hydration behavior of wood-cement-water mixtures, an isothermal calorimetry and experimental method were used to measure the hydration heat of wood-cement-water mixtures. The compatibility of 38 wood species with ordinary portland cement was studied using this procedure. Based on the results, all the wood species tested were classified into two groups. The 24 species included in the first group showed a moderating influence on the hydration reaction of cement, and a maximum temperature (T_{max}) peak during the exothermic reaction while the cement set appeared within 24h for each species. The other 14 species inhibited cement hydration completely. According to the maximum hydration temperature (T_{max}) and the time (t_{max}) required to reach the maximum temperature of the mixture, the suitability of each species in the first group was estimated when used as a raw material during production of cement-bonded particleboard. By testing mechanical properties [modulus of rupture (MOR) and internal bonding strength (IB)] during the board-making experiment using the same composition of wood-cement-water, a positive correlation was found between T_{max} and t_{max} and MOR and IB. The results imply that the method can be used as a predictor of the general inhibitory properties and feasibility of using wood species as raw materials prior to manufacture of cement-bonded particleboard.

Key words Cement-bonded particleboard · Hydration reaction of cement · Compatibility · Wood species · Inhibitory property of wood species

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Introduction

Wood-cement panels (WCPs) have been widely used as structural panels because of their good dimensional stability, nailing, insulation, durability, fire resistance, and decay resistance properties compared with wood-based particleboard.[1–7] The compatibility of wood particles with cement used as an inorganic binder during manufacture directly influences the physical and mechanical properties of WCP products.[8–11] Cement hydration is a complex reaction process requiring water, heat, and an alkaline environment. However, wood particles are deleterious to the cement hydration process owing to the soluble sugars and extractives in wood.[12–18] Thus, it is imperative to fully understand the compatibility of wood with cement for better utilization of wood materials and manufacture of WCPs.

Sandermann et al. and others[4,9,19–21] have indicated that the effects of wood on cement might be attributed to various factors, including geographic location, felling season, tree species, wood storage period, tree components, and bark content. The sugars in wood significantly inhibit cement hydration (setting and hardening). Lignin appears to have minimal effect on cement setting.[22] Hardwoods have a lower compatibility with cement than softwood, partly due to the inhibitory properties of hydrolyzable hemicellulose and other extractives present in hardwoods. Water and alkali-soluble extractives and sugars contribute to reducing the release of heat and prolong the setting time of portland cement as measured by the heat of hydration given off during cement setting. Organic acids such as acetic tannic acid and other phenolics may not only inhibit cement hydration, they may also slowly attack and destroy the cement bond, resulting in a reduction in panel strength values and affecting the other panel properties.[23]

Because the process of portland cement hydration, which leads to crystallization and crystal growth, is an exothermic reaction, and higher reaction temperatures engender a better crystalline formation of cement, it is possible to determine the extent of cement hydration (setting and hardening) and the strength values of WCP products by measuring the reaction temperature of wood-cement-water mixtures during the exothermic reaction. Some researchers[24–27] used this theory to research the hydration behavior of wood-cement-water mixtures to estimate the inhibitory characteristics of given wood species.

During the manufacture of WCPs it is usually impossible to use a single wood species as raw material; instead, combinations of wood species are used. Because of the different extractives content among the wood species, with their compatibility being influenced by the species, the properties and strength values of WCPs are sensitive to the wood species used. Adding specific chemicals such as $CaCl_2$, $MgCl_2$, and $Al_2(SO_4)_3$, sometimes called mineralizing agents, improves the compatibility of the wood and the cement. Therefore, understanding and confirming the inhibitory qualities of wood species prior to manufacture is significant for maintaining the stability of WCPs, improving the utilization of wood, and reducing production waste and cost.

The 38 wood species included in this study were selected in China based on the belief that because of the factory sites and wood resources distribution they are potential raw materials for cement-bonded board production. The objectives of the study were to (1) determine the variability of the inhibitory influences of these species on cement hydration for wood-cement-water mixtures; (2) classify these species in terms of their inhibitory characteristics; and (3) simultaneously examine the effect of the hydration reaction on the strength values of the WCPs.

Materials and methods

Apparatus

The experiment (Fig. 1) designed and used in this study consisted mainly of three parts: (1) 12-point temperature-time recorder and plotter; (2) insulating container (thermos flask filled with insulating material such as cotton and mineral wool); (3) platinum (Pt) thermocouple. Accessories included a pipe sleeve, copper pipe, and plastic container.

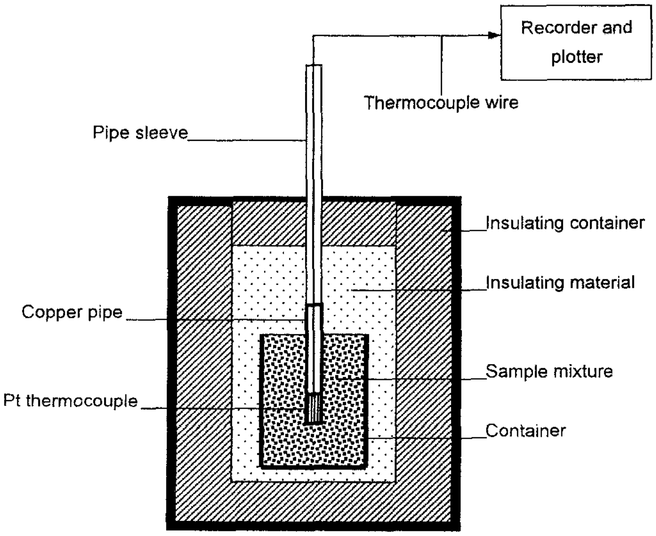

Fig. 1. Apparatus used to measure the hydration characteristics of the wood-cement-water mixture

Sandermann et al.[4] suggested a method for determining the initial hydration of wood-cement composites by measuring the rate of temperature rise; Weatherwax and Tarkow[3,19] also used this technique with slight modification. Moslemi et al.[8,25] suggested using an inhibitory index (I) expressed by an equation for evaluating and classifying the compatibility of wood-cement-water mixtures. For more immediately and uncomplicated explanations of the hydration reaction, the maximum temperature (T_{max}) of hydration and the time (t_{max}) required to reach the temperature were used directly to evaluate the index of compatibility between wood species and cement.

Sample preparation and analysis of the hydration reaction

In total, 38 commercially important wood species were selected for this study (Tables 1, 2) and then air-dried. Because of the difference of hydration temperatures in sapwood and heartwood due to differences in their extractives content,[21,27] wood from a mixture of sapwood and heartwood for each species was stored for more than 3 months in a natural condition prior to use (EMC 20%). After debarking the wood to increase its specific surface and to promote sufficiently separation of extractives that seep from the wood, each species of wood was reduced to powder with a high-speed grinder. The wood powder was screened through 20 mesh and then stored in polyethylene bags until use. The cement used in this study was commercial ordinary portland cement that was stored in sealed plastic bags to avoid quality changes due to moisture problems. The weights of wood powder, cement, and water in the mixture were 15 g (oven-dried), 200 g, and 90 g (including the water in wood species), respectively.

The setup of the sample for the hydration reaction is as follows: First, cement (200 g) and wood powder (15 g oven-dried basis) were dry-mixed in a container. After fully mixing by hand for about 5 min, the mixture was placed in a thermos flask with a thermocouple and covered with insulating materials such as cotton and fiberglass. Within 24 h the exothermic phenomenon of the wood-cement-water mixture was observed and plotted against time. The hydration experiment in this study was conducted at ambient temperatures ranging from 20° to 23°C. The experimented steps and composition in the mixture were based on the research reported by Moslemi et al.[24,25] and Weatherwax and Tarkow.[3,19] The hydration reaction of the mixture indicated that within a 24-h observation period there was an exothermic peak that represented cement setting with a maximum temperature in some species mixed with cement; the time to attain the maximum temperature of hydration was considered the required setting time of the mixture. A total of 39 groups of samples (38 for wood species and 1 for cement alone used as a control sample) were prepared to measure the hydration characteristics. Three replications were conducted for each species for statistical analysis.

Table 1. Hydration characteristics of wood-cement-water mixtures (type I)

Species	Temperature $T_{\max}$ (°C)			Time $t_{\max}$ (h)		
	Average[a]	SD	Grouping*	Average[a]	SD	Grouping*
Cement (control)	59.4	0.29	A	9.2	0.47	FG
Least inhibitory species						
Pterocarya stenoptera C.DC.	54.9	1.47	B	6.7	0.24	BC
Betula platyphylla Suk.	54.8	1.02	B	7.7	0.24	CD
Eucalyptus mcintyrensis	53.8	0.95	BC	5.8	0.24	A
Pinus koraiensis Sieb.	52.7	0.54	C	8.3	0.24	EF
Pinus tabulaeformis Carn.	52.5	2.04	C	8.8	0.47	EF
Eucalyptus cf. rubida	52.3	1.94	C	6.2	0.62	AB
Eucalyptus saligna	51.9	2.92	CD	6.7	0.24	BC
Intermediate inhibitory species						
Populus tomentosa Carr.	50.3	2.92	D	11.2	2.06	IJ
Tilica mandshurica Rupr.	49.9	1.52	E	7.8	0.24	CD
Salix babylonica Linn.	46.5	4.53	EF	13.1	3.00	KL
Toona sinesis Roem.	46.5	1.98	EF	7.4	1.23	BC
Quercus variabilis Bl.	45.8	2.10	FG	14.7	0.47	M
Castanea mollissima Bl.	44.6	1.02	G	6.7	0.24	BC
Fraxinus mandshurica Rupr.	44.2	1.25	GH	12.0	2.84	JK
Platycladus orientalis L.	44.2	3.08	GH	11.8	1.84	J
Quercus mongolica Fisch.	42.5	1.77	HI	11.5	1.08	J
Betula albo-sinensis Burkill	41.7	0.90	I	10.0	0.71	GH
Quercus liaotungensis Koidz	41.4	1.07	I	10.2	0.41	GH
Acea truncatum Bunge	41.1	1.02	J	13.5	0.00	LM
High inhibitory species						
Quercus aliena Bl.	41.7	1.27	I	20.5	0.00	O
Acea mono Maxim	38.2	1.08	J	14.4	1.03	M
Larix olgensis Henry	38.0	1.67	J	20.0	0.00	O
Calophyllum inophyllum L.	36.8	1.06	JK	14.4	0.42	M
Betula costata Trautr.	35.9	0.80	K	16.5	0.71	N

SD, standard deviation

[a] Each value is the average of three replications

*Means with the same capital letter are not significantly different at the 5% significance level

Table 2. Hydration characteristics of wood-cement-water mixtures (type II)

Species	Temperature $T_{\max}$ (°C)	
	Average[a]	SD
Koelreuteria paniculata Laxm.	39.4	0.83
Zizyphus jujuba Mill.	38.3	0.21
Ailanthus altissima Swingle	38.3	0.73
Paulownia tomentosa Steud.	38.0	0.86
Diospyros lotus Linn.	37.6	0.39
Platanus orientalis Linn.	37.3	0.47
Ginkgo biloba Linn.	36.9	1.33
Fraxinus chinesis Roxb.	36.9	0.29
Populus davidiana Dode	35.7	0.16
Phellodendron amurense Rupr.	35.5	0.51
Acer tegmentosum Maxim	35.3	0.40
Betula dahurica Pall.	34.8	0.52
Celtis sinensis Pers.	34.3	0.74
Hevea brasiliensis Muell.-Arg.	32.4	0.43

[a] Each value is the average of three replications

Board preparation and strength measurement

During the board-making experiment the maximum temperature ($T_{\max}$) and the time required to reach it ($t_{\max}$) represented the compatibility of wood species; based on the results 10 wood species from types I and II, respectively, were selected for making the boards. The sample logs were debarked, reduced, and particles screened to chips averaging 2–3 × 1 × 0.2 cm. The chips were air-dried to an average of 12% moisture content (MC), and all wood-cement composite materials were made with a 3:1 weight ratio for cement to wood (c/w) and a 1.00:0.52 weight ratio for cement to water (c/w_a).

For each wood-cement composite, aluminum sulfate [$Al_2(SO_4)_3$], accounting for 4% of the weight of cement, was added as a mineralizing agent to accelerate the setting of the wood-cement-water mixture and to increase the early-stage strength of the board to avoid breaking the board when it is separated from cauls and mold. The aluminum sulfate was first dissolved in water (based on the weight of cement), and the wood was then mixed with cement prior to addition of the solution. After being fully mixed, the solution was added to the mixture. The material was hand-blended for approximately 5 min until it was uniformly mixed and wetted. The wet material was hand-moved, molded in a forming box into a 40 × 40 cm^2 size mat, and then placed on a caul plate that had been coated with mineral oil (used as a release agent). The target density of the board was 1.2 g/cm^3. The mat was cold-pressed to 12-mm stops between caul plates and then was locked in a mold to maintain pressure. After 24 h the hardened mat was removed from the molds and caul plates; it was then stored for postcuring at an ambient temperature of 20°C. After 28 days the mat was cut into samples of 20 × 10 cm^2 and 5 × 5 cm^2, respectively, and tested to determine the modulus of rupture (MOR) and internal bonding (IB) strength. Three boards were made from each species, and six replications were conducted.

Calculation and data analysis

For calculation and statistical analysis of the data, Duncan's new multiple comparison procedure[28] was applied. We obtained all pairwise statistical comparisons for the sample means of hydration characteristics of the wood-cement-water mixture.

Results and discussion

Measurement of hydration reaction of wood-cement-water mixture

Figure 2 shows the hydration temperature of ordinary portland cement as a function of time. There are at least two normal peaks observed in the temperature–time curve of cement paste alone within 24h.[29] It has been generally agreed that the first peak indicates the formation of the calcium-aluminum phase of C_3A ($3CaO{\cdot}Al_2O_3$, tricalcium aluminate) and heat release of the initial cement hydration; the second peak indicates the hydration of C_3S ($3CaO{\cdot}SiO_2$, tricalcium silicate) and shows the maximum exothermic reaction of cement.[15,21,29] The first peak appears less than 1h after water application, whereas the second peak, which represents the final cement setting, takes more than 10h.

The hydration temperature of portland cement mainly depends on its components. The maximum hydration temperature T_{max} and time t_{max} to reach it should be comparable for all portland cements.[3,4,24] Any additives, such as wood species, that may change the setting rate of the cement would lead to a change in the exothermic rate of hydration and thus change both T_{max} and t_{max}.

We studied the hydration reactions of 38 wood-cement-water mixtures and their hydration exothermic curves appeared as different but regular types within 24h. Four of them are shown in Fig. 3.

Determinations for two species

The hydration temperatures for 39 group of species samples are listed in Tables 1 and 2. Thirty-nine temperature curves were obtained based on the means of the data for each group. Based on the presence of one or two exothermic peaks for the hydration reaction within a 24-h period, the temperature-time curves of 38 species were divided into two types (I and II), as illustrated in Fig. 4.

For type I species, although the t_{max} of each wood-cement-water mixture is before or after that for cement alone, the hydration temperature curves of the mixtures were similar to that for cement alone, with two exothermic peaks occurring in each curve within 24h. The first temperature peak appeared within about 30min of the water application. The temperature then decreased but increased again within 24h, with the appearance of the second main exothermic peak (e.g., *Pterocarya stenoptera* C.DC., *Betula platyphylla* Suk., *Pinus tabulaeformis* Carn., and *Salix babylonica* Linn.).

For type II of species, the first temperature peak appeared within about 30min of the water addition; strong inhibition due to the extractives of species was demonstrated, so the temperature gradually decreased without another increase within the 24-h period. As shown in Table 2, 14 species were considered to belong to this type (e.g., *Koelreuteria paniculata* Laxm., *Platanus orientalis* Linn., *Phellodendron amurense* Rupr.).

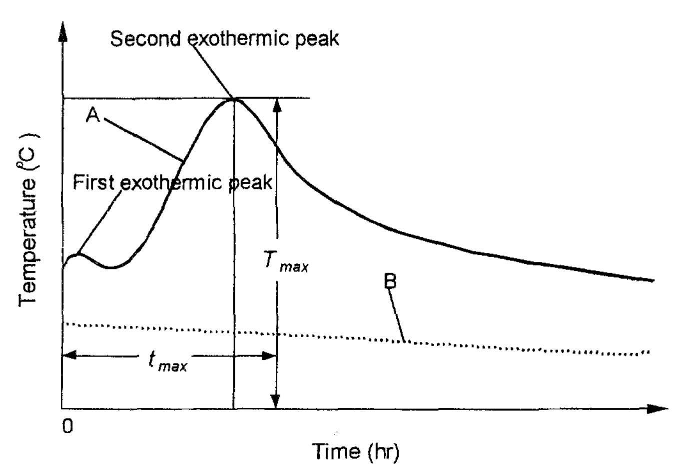

Fig. 2. Index of hydration temperature-time curve of cement. *A*, temperature-time curve of cement; *B*, ambient temperature; T_{max}, maximum hydration temperature; t_{max}, time required to reach maximum temperature

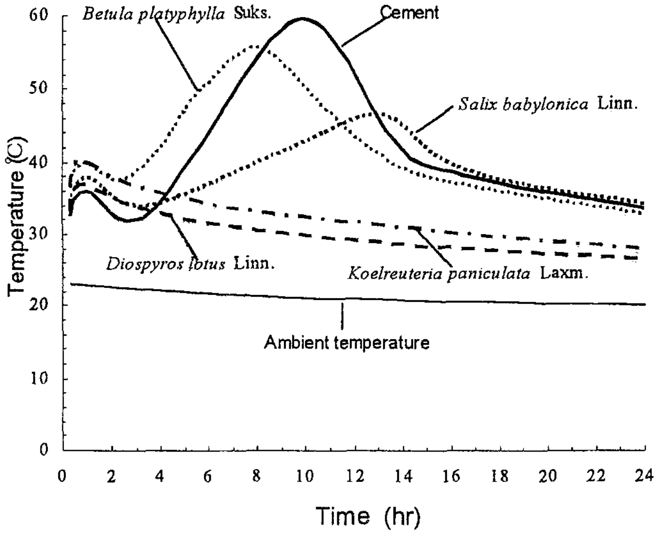

Fig. 3. Effects of wood species on hydration characteristics of wood-cement-water mxitures

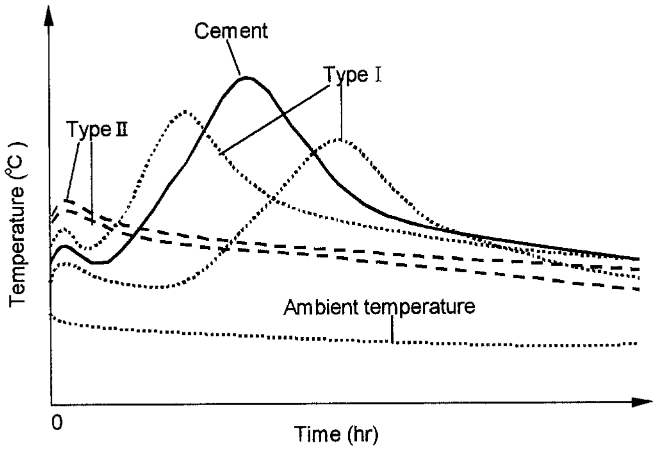

Fig. 4. Hydration curve types of wood-cement-water mixtures

The first peak for most of the 38 species (except *Celtis sinensis* Pers. and *Hevea brasiliensis* Muell.-Arg.) reached a high temperature, even higher than that for cement alone (34.6°C). The high temperature for the wood-cement-water mixture is due to the possibility that some of the extractives in wood are thought to accelerate slightly the hydration of tricalcium aluminate (C_3A) during the cement phase, leading to a flash setting, which releases high heat at the beginning of the hydration process.[29] For this reason, the first peak cannot be used to distinguish the compatibility of species. It can be concluded that the high extractives content in type II wood species completely retard the hydration of tricalcium silicate (C_3S) and inhibit cement setting, resulting in disappearance of the second peak, which represents the final cement setting within 24h or a greatly delayed setting time of cement beyond 24h. Therefore, the type II species were thought to severely retard cement hydration and so are not suitable materials. If it were necessary to use these species for production, additional treatment would be required prior to manufacture of WCPs.[15,23,25,30,31]

Comparison of T_{max} in type I species

All type I wood species more or less inhibited hydration, with decreased T_{max} of cement. The T_{max} of these species was apparently lower than that of cement alone. *Pterocarya stenoptera* C.DC. had the highest T_{max} (54.9°C), and *Betula costata* Trautr. had the lowest (35.9°C) with 23.5°C temperature difference compared to that of cement alone. Statistical analysis using Duncan's new multiple comparison for T_{max} indicated that there were significant differences among the type I species, and 24 species were grouped from B to K. Both hardwood species (e.g., *Pterocarya stenoptera* C.DC. and *Betula platyphylla* Suk.) and softwood species (e.g., *Pinus koraiensis* Sieb. and *Pinus tabulaeformis* Carn.) were represented in type I species. The maximum temperature for cement alone (group A, controls) was significantly different from that of the wood-cement-water mixtures.

Comparison of t_{max} in type I species

The advanced and delayed exothermic cement hydration reaction, as estimated by the time (t_{max}) to reach maximum hydration temperature, may be used to determine how soon a wood-cement panel can be removed from closed caul plates and molds during the cold-press process. Statistic analysis of Duncan's new multiple comparison for t_{max} in type I species showed that there were significant differences between the means of samples. The t_{max} of 24 type I species were grouped. Eight species in which t_{max} ranged from 5.8 to 7.8h, were classified as groups A–E. The t_{max} of the cement alone, regarded as the normal hydration reaction time, was classified as group FG (FG overlapped F and G groups) and ranged from 8.3 to 10.2h; there were four such species. There were 12 species grouped from F to O, in which t_{max} was >10.2h. As shown in Fig. 2, the t_{max} of eight species, with T_{max} >50°C, was 6.2–8.8h, with the exception of 11.2h for *Populus tomentosa* Carr.; and they were earlier than the t_{max} (9.2h) of cement alone. These species were thought to have comparatively high compatibility with cement due to their high T_{max} and short t_{max}, and the same general trend was observed with strength values of boards in the late group.

Comprehensive evaluation for compatibility

Based on the experimental results for wood-cement-water mixtures, Sandermann and Kholer[9] classified the wood used for the manufacture of WCPs as follows: highly suitable, suitable, and less suitable. These authors suggested that using highly suitable wood species and portland cement, even without additive chemicals, can produce WCPs of high quality.

Because the maximum temperature (T_{max}) of the cement used in this study was 59.4°C lower than that used by Sandermann and Kholer,[9] in accordance with Duncan's comparison for T_{max} and t_{max} and reference to the classification method used by Sandermann and Kholer,[9] we classified the wood species included in type I as follows.

1. *Least inhibitory species*: The maximum temperature (T_{max}) was >50°C, and the time (t_{max}) was <10h: 7 wood species.
2. *Intermediate inhibitory species*: The maximum temperature (T_{max}) was >40°C, and the time (t_{max}) was <15h: 12 wood species. If these wood species were used as raw materials for WCPs, specified additive chemicals and related technology should be prepared.
3. *Highly inhibitory species*: The maximum temperature (T_{max}) was <40°C or time (t_{max}) >15h: 5 wood species. These woods are considered unsuitable for the manufacture of WCPs. If it is necessary to use these species for production, extractives must be removed prior to manufacture.

Correlativity between characteristics of hydration and properties of board

In accordance with the results of the hydration reactions of 38 species, six and four species, respectively, in types I and

Table 3. Hydration characteristics and strength values of wood-cement board

Species	$T_{\max}$ (°C)	$t_{\max}$ (h)	MOR[a] (MPa)	SD	IB[a] (MPa)	SD
Type I						
Pterocarya stenoptera C.DC.	54.9	6.7	8.79	0.86	0.48	0.24
Pinus koraiensis Sieb.	52.7	8.3	9.25	1.55	0.62	0.17
Salix babylonica Linn.	46.5	13.1	7.25	1.40	0.55	0.20
Quercus mongolica Fisch.	42.5	11.5	8.65	0.97	0.51	0.22
Calophyllum inophyllum L.	36.8	14.4	6.18	1.20	0.41	0.16
Betula costata Trautr.	35.9	16.5	4.92	2.31	0.33	0.09
Type II						
Koelreuteria paniculata Laxm.	39.4		5.24	0.96	0.29	0.13
Platanus orientalis Linn.	38.3		3.45	0.99	0.31	0.13
Phellodendron amurense Rupr.	35.5		4.12	0.62	0.38	0.18
Hevea brasiliensis Muell.-Arg.	32.4		3.31	1.21	0.20	0.10

[a] Each value is the average of six replications

II were chosen to make boards to examine the relation between hydration characteristics and strength values. Significant identity of the hydration characteristics with strengths was observed with maximum hydration temperature ($T_{\max}$), as shown in Table 3. There was a general trend of higher hydration temperature ($T_{\max}$) and shorter reaching time ($t_{\max}$) with higher MOR and IB strength values. The type I wood species with a higher $T_{\max}$ and shorter $t_{\max}$, which represented good compatibility with cement, considerably affected the properties of the board. The MOR and IB strengths of type I species were significantly higher than those of type II. However, even though the type II wood species were shown to have a relatively higher $T_{\max}$ (>40°C at the first exothermic peak), the strength of the panels was lower than that of type I because their hydration reactions with cement were completely inhibited, with no second exothermic peak within 24h. This finding appears to be consistent with those of other researchers who found close relations between the hydration characteristics of wood-cement-water mixtures and strength.[21,32]

Although the correlation between hydration characteristics and strength is not considered to be suitable in all case due to a large variety of diverse woods that are not homogeneous in their effects on cement, it appears that the hydration characteristics of wood-cement-water mixtures can be expected to be a general estimation of panel strength, especially in specific areas and species chosen as raw materials prior to WCP production.

Conclusions

On the basis of the data from hydration of wood-cement composites and strength tests collected in this series of experiments, the following conclusions were drawn.

1. The use of hydration characteristics ($T_{\max}$) and ($t_{\max}$) of wood-cement composites is a simple, recommendable method of directly and quantitatively measuring the inhibitory effect of wood species. $T_{\max}$ and $t_{\max}$ can be considered the most important parameters and indicators for estimating the compatibility of given species.

2. The temperature–time curves of mixtures differed significantly among the total 38 wood species, representing different compatibilities with cement. Altogether 24 and 14 species, respectively, were classified in type I or II based on the presence of a second exothermic reaction peak within 24h.

3. In terms of the hydration characteristics ($T_{\max}$ and $t_{\max}$) of wood-cement-water mixtures, the compatibility of type I species were divided into three levels: least, intermediate, and highly inhibitory species for WCP production. The type II species were not deemed acceptable raw materials for board production because there a complete hydration reaction was not observed.

4. There was a favorable identity between hydration characteristics ($T_{\max}$, $t_{\max}$) and MOR and IB values, which means that $T_{\max}$ and $t_{\max}$ can be used as predictors to determine the sensitivity of board strengths of wood species and to determine the feasibility of species application.

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