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Study of hydration behaviors of wood–cement mixtures: compatibility of cement mixed with wood fiber strand obtained by the water-vapor explosion process

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Abstract To provide information on the feasibility of using exploded wood fiber strand (WFS) obtained by the water-vapor explosion process in wood–cement mixtures, the compatibility between cement and exploded WFS and its improvement with various additive chemicals were investigated by observation and analysis on hydration behaviors in terms of hydration characteristics: maximum hydration temperature (T_{\max}) and required time (t_{\max}). The three types of cement, six additive chemicals, and exploded WFS (sugi, air-dried and water-soaked) were employed as raw materials in this study. The hydration behaviors of mixtures demonstrated that exploded WFS had strong retarding effects on cement hydration and completely prevented mixtures from setting. The analysis of sugar revealed that the sugar contents of exploded WFS were much higher than those in unexploded wood and increased to about 20-fold (air-dried) and 10-fold (water-soaked), respectively. The degraded polysaccharides became a major factor and played an important role in inhibiting the setting of cement. Moreover, high-performance liquid chromatography analysis proved that the main peaks representing the molecular weight of polysaccharides in extractives of exploded WFS shifted markedly to a lower range of polymerization. $MgCl_2$ was determined to be an effective additive chemical for restraining the inhibitory influences. Addition contents of 2%–3% and 4%–5% were available and acceptable for quick-curing cement and ordinary Portland cement, respec-

tively. As for the furnace-slag cement, the composite additive chemicals of $MgCl_2$ (4%) and CaO (2%) were found to have an obvious accelerating effect.

Key words Wood–cement mixture · Compatibility · Cement hydration reaction · Water-vapor explosion of wood · Additive chemical

Introduction

The rapid decrease in forest resources along with economic development and excessive aggravation of the natural environment is becoming an urgent issue that requires more attention from the technical and economical viewpoints. Recycling wood and the value-added use of wood wastes is therefore important. A project scheme aimed at exploring further utilization of recycling wood wastes – a new wood separation method of water-vapor explosion process (WVEP) for defiberating wood into fibers and fiber strands – was studied and developed at the Forestry and Forest Products Research Institute (FFPRI).¹

The treatment of lignocellulosic resources with high-temperatures and high-pressure steam for short periods followed by sudden decompression (i.e., usually known as “steam explosion”) represents a simple treatment for destructuring wood and plant resources by a combination of chemical and mechanical actions. This process has been reported and widely utilized as lignocellulosic resources fiber separation and conversion technology.^{2–5} However, compared to the steam explosion process, the significant difference for the WVEP is that it does not depend on external steam resources but, rather, makes use of the water in wood to generate water vapor (or steam). With the aid of a blasting force originating from instantaneous expansion of high-temperature and high-pressure water vapor (or steam), the wood or wood wastes located in a hot press were separated into wood fibers and fiber strands from the hemicellulose-rich and pectin-rich middle lamella. Depending on the adjustment of water content and explosion

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parameters (e.g., pressure, temperature, time), the maximum yield and optimum shape of wood fiber and fiber strand could be achieved.

On the other hand, it has been reported that wood–cement mixtures (WCMs) consisting of wood fibers and fiber strands would effectively increase the fracture energy of cement matrixes and enhance the tensile and flexural strength, toughness, and impact resistance of mixtures.^{6,7} In this research, the exploded WFS obtained by WVEP was experimentally mixed with various types of cement with the expectation that it could be an alternative wood material for diversified wood–cement products.

As cement is mixed with wood, the problem initially encountered in achieving high-strength values has been the question of compatibility between cement and wood due to the presence of wood extractives, especially sugars, which appear to inhibit significantly the setting (or curing) of cement.^{8–13} Wood mainly consists of three components: cellulose, hemicellulose, and lignin. During treatment with a high-temperature, high-pressure destructuring process, the degradation of wood components such as hemicelluloses is unavoidable. The severe treatment conditions cause more polysaccharide degradation, releasing the degraded carbohydrates with lower polymerization into water or alkali solution. The retarding effect of exploded WFS on cement hydration is believed to be much stronger than that in unexploded wood.

Based on a consideration of exploring the possibility of using mixtures of cement and exploded WFS to produce low-cost building elements, the present study had three major objectives: (1) observe hydration behaviors and evaluate the influences of WVEP on the compatibility of cement and exploded WFS; (2) examine and analyze the sugar content and molecular weight patterns of polysaccharides in extractives of exploded WFS; and (3) enhance the compatibility of cement and exploded WFS with available additive chemicals at acceptable additive contents.

Materials and methods

Apparatus

The apparatus and measurement methods used in this work are the ones reported by Sandermann et al.⁸ and Weatherwax and Tarkow.¹⁴ The hydration tests were performed in six Dewar flasks with double walls filled with thermal insulation materials (cotton and polystyrene foam), which are similar to those described in our previous papers.^{15,16} Thermocouples (T-CC) connected to a multi-point computing data logger were used to monitor simultaneously the temperature–time changes of mixtures inside the Dewar flasks.

The influence of cement types, wood properties, and additive chemicals on compatibility was determined and evaluated in terms of hydration characteristics: maximum hydration temperature (T_{\max}) and time (t_{\max}) required to reach the T_{\max} . The hydration behavior of mixtures was observed and recorded within a 24-h period.

Materials

The weathered wood waste was from sugi wood (*Cryptomeria japonica* D. Don) collected at construction sites based on the consideration of exploring its recycling use as a raw material for WCM. (A significant portion of wood waste generation is seen in the building and construction industry.) Normal sugi wood (air-dried) was also used to make a comparison with weathered wood waste.

The weathered wood wastes with different moisture contents (MC) in air-dried (MC 15%–20%) and water-soaked (MC 150%–200%) conditions, with dimensions of 900 mm (L), 150 mm (W), and 25 mm (T), were used. They were treated by WVEP under explosion conditions of temperature 300°C, pressure 2.5 MPa, and time 4.0 min. With the action of high temperature and high pressure, the weathered wood wastes were first collapsed and softened; then, after sudden decompression followed by rapid expansion of water vapor (known as “explosion”), the weathered wood wastes were split into wood fibers and wood fiber strands (WFSs) with irregular lengths and diameters.

The three commercial cements employed for the hydration reaction test were quick-curing cement (QC), ordinary Portland cement (OC), and B-type furnace-slag cement (FC) (Taiheiyo Cement Co.). Six additive chemicals – aluminum chloride (AlCl_3), calcium chloride (CaCl_2), magnesium chloride (MgCl_2), aluminum sulfate [$\text{Al}_2(\text{SO}_4)_3$], sodium silicate (Na_2SiO_3), calcium oxide (CaO) – were separately selected as single or composite accelerators to modify and improve the compatibility of WCM in this study.

Sample preparation and the hydration reaction test

The exploded WFS, about ϕ 1.0–3.5 mm and 10–30 cm in length as shown in Fig. 1, was either reduced to powder (20–40 mesh) or cut into short fiber strands (2 cm in length). The unexploded wood was similarly reduced to powder,

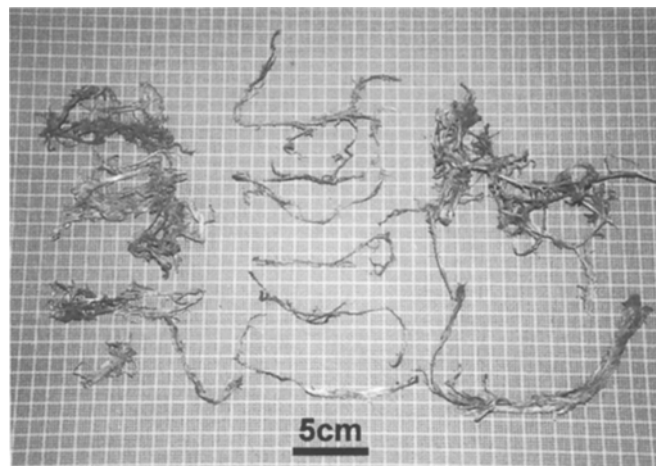


Fig. 1. Wood fiber strand (WFS) obtained in a water-vapor explosion process (WVEP)

dried, and stored in polyethylene bags. All samples for the hydration reaction test were mixed with a mass ratio of cement (200g)/wood (oven-dried, 15g)/distilled water (100ml). To determine any improvement in compatibility, six single additive chemicals were accordingly added to mixtures of wood and cement at an additive content of 4% based on the cement weight. In the case of a selected additive chemical, the contents were examined at additive levels ranging from 1% to 6% of cement weight. The setup for the sample used in the hydration reaction test was described in previous papers.^{15,16}

The hydration reaction test of neat cement was performed as a control corresponding to its mixture. All the tests were conducted at ambient temperatures that ranged from 20° to 23°C. Three replications were run for each specimen for statistical analysis.

Analysis of polysaccharides in wood extractives

The cold-water extractions of the four wood powders (unexploded, exploded, air-dried, water-soaked) were conducted with the addition of 100g wood powder into 1000ml distilled water for 24h at an ambient temperature of 20°C. After the filtered cold-water extractives were added with two volumes of ethanol (99.5%) and stood for 24h at 4°C, the water-soluble degraded polysaccharides in extractives were precipitated. The precipitates collected by centrifugation at 12000g for 30min were subjected to sugar contents measurement and high-performance liquid chromatography (HPLC) analysis. The sugar contents in the four cold-water extractives were quantitatively measured by the anthrone-sulfuric acid method using glucose as the standard.

The molecular weight of the polysaccharides in the four wood extractives was determined by gel filtration chromatography. Chromatography was performed with HPLC [Waters 2690 equipped with a packed column, Waters Ultrahydrogel Linear (7.8mm i.d., 300mm × 2) and RI detector (Waters 2410)]. The solvent was 0.1M NaNO₃, and the flow rate was 0.9ml/min. Pullulan (Shodex Standard P-82, Showa Denko K.K.) was used as a standard for the molecular weight.

Calculation and data analysis

Analysis of variance was performed on three replications for sugar contents and all hydration characteristics (T_{\max} and t_{\max}). Duncan's new multiple comparison procedure was used to compare the effects of wood properties, cement types, additive chemicals, and additive contents by conducting all pairwise statistical comparison among the sample means at the 5% significance level.¹⁷

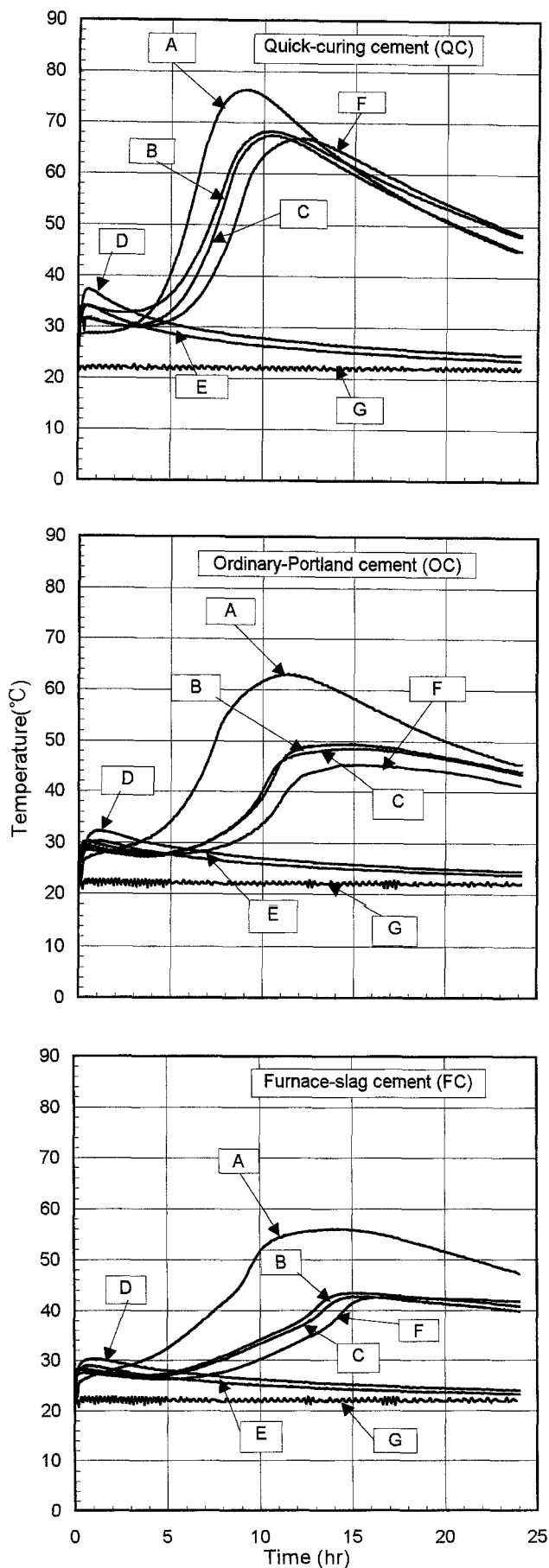
Results and discussion

Preliminary investigation on the inhibitory influences of wood with different properties on hydration behaviors of wood–cement mixtures

In terms of hydration temperature–time curves and hydration characteristics, a comparison of the effects of the cement types and wood properties on the exothermic hydration reaction is presented in Fig. 2. The hydration behaviors of neat cement (controls) and mixtures were found to be different and strongly influenced by cement types and wood properties. The changes observed in hydration characteristics of T_{\max} and t_{\max} monitored during a 24-h period were significant and can be considered to be good, immediate indicators for cement hydration and wood–cement compatibility. It was observed that three types of neat cement have similar patterns of hydration behavior but differ in their T_{\max} and t_{\max} . The neat QC reached the highest T_{\max} of 76.5°C in t_{\max} 9.0h, whereas the neat OC reached an intermediate T_{\max} of 63.0°C in t_{\max} 11.3h. However, the neat FC gave the lowest T_{\max} of 56.0°C in t_{\max} 13.7h. The changes in T_{\max} and t_{\max} of the three types of neat cement are likely mainly due to the differences in the composition of the cement such as C₃A (3CaO·Al₂O₃, tricalcium aluminate), C₂S (2CaO·SiO₂, dicalcium silicate), and C₃S (3CaO·SiO₂, tricalcium silicate) in QC and OC, as well as the contents of ordinary Portland cement clinker in FC.¹⁸

When three types of cement (QC, OC, FC) were added to normal sugi wood, a retarding effect was found due to the reduction in T_{\max} and extension of t_{\max} . The T_{\max} values of mixtures were reduced to 67.0°, 45.3°, and 42.6°C and their t_{\max} values were prolonged to 12.1, 14.4, and 16.7h, respectively. With reference to the classification method proposed in earlier investigations, for example, in the case of OC normal sugi wood could be approximately classified as a wood species with an intermediate inhibitory effect on cement setting. This result supported the earlier conclusion that incompatibility of normal sugi wood was mainly due to the presence of extractives in heartwood.¹⁰ By modifying the compatibility between wood and cement with the addition of selected chemicals and their content, normal sugi wood could be expected to be an acceptable wood species for WCM. Some prior studies reported similar results for the evaluation of the compatibility of cement and sugi wood.^{10,19,20}

The addition of weathered sugi wood wastes (air-dried and water-soaked) to cement showed two typical exothermic peaks, which separately represented the hydration of C₃A and C₃S, observed on the hydration temperature–time curves. The T_{\max} of mixtures, respectively, reached 67.1°–68.2°C at a t_{\max} of 10.2h (QC), 48.5°–49.4°C at a t_{\max} of 13.7–14.6h (OC), and 42.7°–43.3°C at a t_{\max} of 14.8–15.0h (FC). Compared with normal sugi wood, weathered sugi wood wastes showed somewhat better compatibility in terms of higher T_{\max} and shorter t_{\max} , but there were no significant differences according to the Duncan grouping analysis. It should be noted that after long-term outdoor exposure



extractable inhibitory components from weathered sugi wood were relatively decreased owing to multiple effects of natural factors such as rainfall, sunshine, oxidation, and so on.^{14,21} Despite the fact that weathered sugi wood (water-soaked) was immersed in water for more than 3 months, there was no significant difference in hydration behavior compared with air-dried wood.

However, the patterns of hydration behaviors of cement mixed with exploded WFS were completely changed and significantly different with the unexploded wood and normal wood. The strong inhibitory effect that resulted in the least compatibility was found when cement was mixed with exploded WFS. The first temperature peaks observed were normally found to be a flash set associated with the hydration of C_3A and appeared within 30min after water addition. Strong inhibition was demonstrated to be due to the extractives of exploded WFS, and thus the hydration temperature gradually decreased without another increase within the 24-h period. The peak of C_3S , which was normally noted to represent the final setting and strength development of cement, entirely disappeared. In this case, it was believed that some undesirable changes occurred in wood components due to treatment of wood with WVEP.

Comparison and analysis of polysaccharides in extractives of unexploded wood and exploded WFS

Wood materials treated with high-temperature and high-pressure processing induced various changes, including extractive removal, hemicellulose degradation and alteration of the strength properties of wood or fibers.²⁻⁵ It should be noted that during WVEP the hemicelluloses could be fractionated simultaneously and converted to water-soluble hemicellulosic sugars, releasing the sugar oligomers or sugars into solution.

The HPLC analysis of the distribution of polysaccharide molecular weight in the extractives of both unexploded wood and exploded WFS (air-dried, water-soaked) are shown in Fig. 3. The evolution values for the polymerization degree of polysaccharides in the extractives of unexploded wood (air-dried, water-soaked) consisted of main peaks ranging from about 81400 to 1900. However, the main peaks represented in exploded WFS (air-dried, water-soaked) shifted markedly to the lower range of polymerization degree (about 54200 to 150). It could be assumed that more water-soluble degraded polysaccharides, oligosaccharides, and even monosaccharides existed in the extractives of exploded WFS in the forms of low-molecular-weight fractions due to hemicellulose degradation.

The total sugar contents in the extractives were measured and are shown in Table 1. The sugar contents determined in extractives of exploded WFS were much higher

Fig. 2. Effects of wood properties on hydration behaviors of wood-cement mixtures (WCM). A, neat cement (control); B, cement + sugi (water-soaked, unexploded); C, cement + sugi (air-dried, exploded); D, cement + sugi (air-dried, exploded); E, cement + sugi (water-soaked, exploded); F, cement + sugi (air-dried, normal); G, ambient temperature

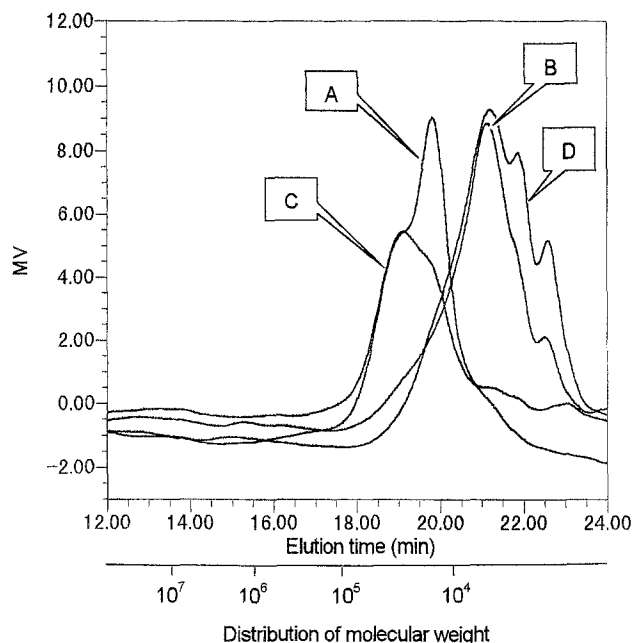


Fig. 3. High-performance liquid chromatography (HPLC) analysis of the influence of a water-vapor explosion process (WVEP) on the distribution of polysaccharide molecular weight in wood extractives. A, sugi, water-soaked, unexploded; B, sugi, water-soaked, exploded; C, sugi, air-dried, unexploded; D, sugi, air-dried, exploded

Table 1. Analysis of sugar contents and pH values in extractives of unexploded wood and exploded wood fiber strand (WFS)

Sample	Grouping ^a	AV \pm SD ^b (mg/ml)	pH
Water-soaked, unexploded	A	0.16 \pm 0.01	6.44 \pm 0.24
Water-soaked, exploded	B	1.57 \pm 0.03	4.67 \pm 0.19
Air-dried, unexploded	A	0.11 \pm 0.00	5.88 \pm 0.10
Air-dried, exploded	C	2.22 \pm 0.04	3.93 \pm 0.36

^aMeans \pm SD with the same capital letter are not significantly different at the 5% significance level (based on the Duncan grouping analysis)

^bEach value is the average of three replications \pm standard deviation

and increased about 20.2-fold (air-dried) and 9.8-fold (water-soaked) compared with unexploded wood. The data for sugar content and molecular weight analysis indicated that wood treated by WVEP was unavoidably not only mechanically defibrated but also chemically modified. Moreover, the hemicelluloses could be degraded to lower molecular weights and easily solubilized during the water extraction. Furthermore, the strength of separated wood fiber and fiber strand was damaged under the severe treatment conditions.

Some earlier investigations noted that the mechanism of retardation of cement setting involves adsorption of sugar onto the surface of cement particles or early hydration products. This adsorption delays the hydration by making it diffusion-controlled. It is also suggested that the retardation efficiency is related to the molecular weight and stability of polysaccharides in the alkaline aqueous phase of hydrating cement. In the presence of alkali, sugars in wood extractives would react with alkali to form sugar acids that act severely as strong retardants to cement setting.^{11,21–23} Therefore, the

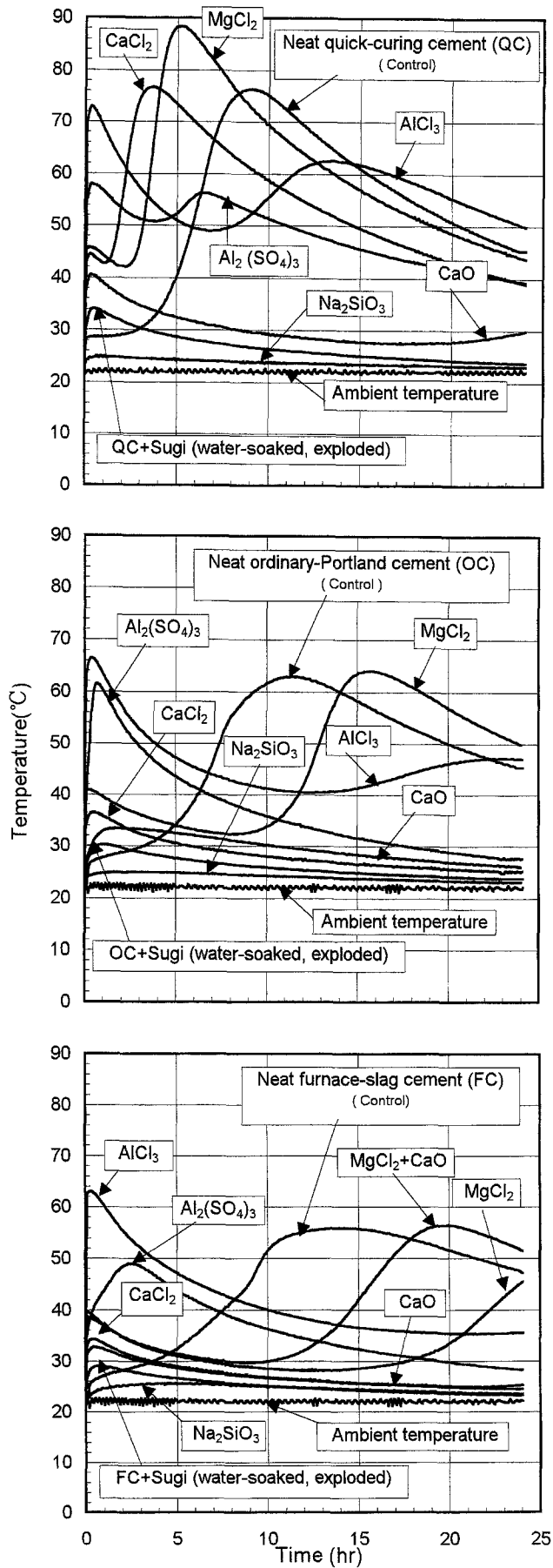
existence of a large amount of degraded polysaccharides due to WVEP would result in a strong retarding influence and would most likely be a major inhibiting factor to cement setting when cement is mixed with exploded WFS. Based on the pH data in Table 1, the water-soluble extractives of exploded WFS presented lower pH values than unexploded ones. The acidic wood extractives decreased the pH of the cement–water mixtures, resulting in an unfavorable cement setting condition.²³

Comparison and selection of available additive chemicals for improving compatibility between cement and exploded WFS

In this study, six additive chemicals were selected to minimize the inhibitory effects, thereby enhancing the compatibility of cement and exploded WFS. In accordance with the three types of cement, the performances of the selected additive chemicals on accelerating cement setting showed different hydration behaviors (Fig. 4).

CaCl₂ and MgCl₂ were found to improve significantly the hydration of QC with a high T_{max} and short t_{max} that are even better than those of neat QC (T_{max} 76.5°C, t_{max} 9.0h). Notably, MgCl₂ showed the highest T_{max} (87.2°C) at a t_{max} of 5.2h (much higher than that of CaCl₂, with 76.8°C); moreover, and an ideal hydration pattern accompanied by a main C₃S exothermic peak was observed. With the addition of MgCl₂, the retarding influence of sugar in the extractive of exploded WFS was effectively restrained; and for this reason, MgCl₂ was considered an acceptable, better additive chemical for QC mixed with exploded WFS. Although with the addition of Al₂(SO₄)₃ and AlCl₃ there were rapid increases in initial hydration temperatures within 1h, the enhancement of hydration of C₃S was relatively weak and slow owing to the lower T_{max} and longer t_{max} than those seen with MgCl₂. Na₂SiO₃ and CaO exerted a negative effect on the hydration reaction of mixtures.

A similar comparison could be conducted for OC with six additive chemicals. It was observed that only MgCl₂ generated a completed, enhanced exothermic peak of C₃S that was close to the main peak of neat cement during the 24h observation period. MgCl₂ still proved to have better action in overcoming the retarding effects resulting from exploded WFS, with a T_{max} of 64.2°C (higher than neat cement), even though its t_{max} (15.7h) was longer than that of neat cement (9.0h). Al₂(SO₄)₃ and AlCl₃ showed an initial temperature increment due to hydration of C₃A. However, the hydration of C₃S, which represents the real strength development of WCM, was greatly inhibited or prolonged, even though AlCl₃ demonstrated a low, slow exothermic peak with a T_{max} of 47.6°C at a t_{max} of 22.6h. In the case of QC, CaCl₂ had a completely different hydration behavior, and there was no heat release generated by hydration of C₃S. In the case of Na₂SiO₃ and CaO, no positive influence was observed. Comparing all additives, MgCl₂ was still believed to be the most effective additive chemical for the mixtures. Faced with the existence of high sugar content, MgCl₂ significantly demonstrated better ability to



reduce the inhibitory influence of sugars, thereby improving the setting of mixtures, as the water-soluble degraded polysaccharide is thought to be a major retarding component in the extractives of exploded WFS. A former report expressed the opinion that MgCl₂ had proved to be an effective accelerator for overcoming the inhibitory effects of sugar, such as sucrose.¹¹

When six chemicals were added to the mixtures of FC and exploded WFS, the hydration behaviors were significantly different from those of QC and OC. There was no desirable or acceptable hydration reaction with these additions. Although the addition of MgCl₂ was found to increase the hydration temperature starting at about 12.5h, compared with the t_{max} of neat FC the setting time of the mixture was greatly delayed. AlCl₃ and Al₂(SO₄)₃ reached a higher initial hydration temperature at an earlier hydration stage, and after this maximum value the hydration temperature decreased continuously. CaCl₂, Na₂SiO₃, and CaO had no accelerating effects on the hydration of mixtures within the 24-h observation period. However, when mixtures added with a composite additive chemical of MgCl₂ (4%) + CaO (1%), the hydration reaction was notably improved and enhanced, and the hydration temperature increased to 54.8°C. It is therefore apparent that with the addition of CaO the setting time was apparently shortened to 20.5h. Because FC is a latent hydration material that contains relatively higher silicon dioxide and lower calcium oxide, its reactivity should be lower and slower than that of OC.^{7,18} Addition of CaO would increase its reactivity, resulting in an adjustment of the setting time of cement.

The results of the six additive chemicals added into mixtures of OC and unexploded wood are shown in Fig. 5. The hydration performances and patterns of accelerating cement setting were significantly different from those mixed with exploded WFS (Fig. 4). In the case of unexploded wood, three chloride salts of CaCl₂, MgCl₂, and AlCl₃ as well as CaO were found to have a positive influence in terms of a higher T_{max} or shorter t_{max} than those of neat cement and mixtures of cement and unexploded wood. Corresponding to the appearance of much the degraded polysaccharides leached from exploded WFS and considering the degraded polysaccharides as a main retarding factor to cement setting, the ability of CaCl₂ and AlCl₃ to overcome the retarding effects were greatly weakened. Taking CaCl₂ as an example, an exothermic peak (T_{max} 68°C, t_{max} 4.2h) of C₃S observed in a mixture of cement and unexploded wood completely disappeared in the mixture of cement and exploded WFS. This result showed that the selection and determination of an acceptable additive chemical is strongly related to the change in the components in wood extractives that varies with wood species, wood properties, and treatment of wood.

Fig. 4. Effects of additive chemicals on hydration behaviors of cement mixed with exploded WFS. AlCl₃, CaCl₂, MgCl₂, Al₂(SO₄)₃, Na₂SiO₃, and CaO were added separately at 4% of the cement weight as single additive chemicals. MgCl₂ was added at 4% and CaO at 1% of the cement weight as composite additive chemicals

Determination of additive contents of chemicals for exploded WFS mixed with various cements

To determine the additive contents available for various types of cement, the relation between the hydration characteristics (T_{max} and t_{max}) and additive contents was examined (Table 2).

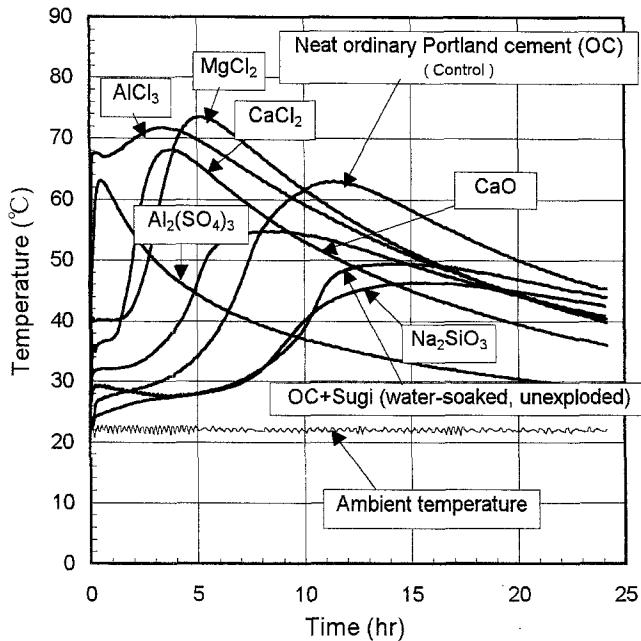


Fig. 5. Effects of additive chemicals on hydration behaviors of cement mixed with unexploded wood (water-soaked). AlCl_3 , CaCl_2 , MgCl_2 , $\text{Al}_2(\text{SO}_4)_3$, Na_2SiO_3 , and CaO were added separately at 4% of the cement weight as single additive chemicals

When MgCl_2 was added to the QC with contents that ranged from 1% to 3%, hydration characteristics of mixtures were significantly improved, resulting in an increase in T_{max} from 55.2°C to 80.2°C, and the t_{max} was shortened from 24.1 h to 6.0 h. Compared to the mixtures without additives, the addition of 1% MgCl_2 caused an increment of about 21.1°C for the T_{max} , and the t_{max} decreased, leading to a perfect exothermic peak of C_3S that occurred within about 24 h. With further addition of the chemical, the changes in both T_{max} and t_{max} became relatively smaller. For example, at an addition level of 4%, there was only about 5.7°C of increase in T_{max} and 2 h decrease in t_{max} , compared to the figures after addition of 3% MgCl_2 . Based on the Duncan grouping analysis, the results indicated that the hydration characteristics (T_{max} , t_{max}) of mixtures with 1%–3% MgCl_2 (addition level) were significantly different at the 5% significance level. The T_{max} and t_{max} of mixtures when 2%–3% MgCl_2 was added could be respectively classified with those of neat QC (control). Thus, 2%–3% of MgCl_2 was considered an acceptable additive content for QC. In addition, owing to the fact that QC has a higher content of C_3S than OC, the additive chemical should be controlled at a lower addition level. A higher additive chemical content would cause a violent reaction and produce rapid rigidity and fluidity loss, resulting in operation difficulty such as forming and molding.

For the OC, additive contents of MgCl_2 varied from 1% to 6%. When the additive content was 4%, a perfect peak of C_3S located in 15.7 h was observed. Based on the Duncan grouping analysis, with the addition of 4%–5% MgCl_2 the T_{max} and t_{max} of mixtures could be respectively classified in the B group, which was the same with those

Table 2. Effects of additive chemicals and their contents on hydration characteristics of cement mixed with exploded WFS (sugi, water-soaked)

Composite	T_{max}^a (°C)	Grouping ^b	t_{max}^a (h)	Grouping ^b
Neat quick-curing cement (control)	76.5 ± 1.7	B	9.0 ± 0.2	A
Cement + exploded WFS	34.1 ± 0.3	D	0.5 ± 0.1	— ^c
+ MgCl_2 1%	55.2 ± 1.6	C	24.1 ± 1.8	C
+ MgCl_2 2%	71.3 ± 1.0	B	12.5 ± 1.3	B
+ MgCl_2 3%	80.2 ± 2.1	A	8.0 ± 0.5	A
+ MgCl_2 4%	85.9 ± 1.6	A	6.0 ± 0.3	A
Neat ordinary Portland cement (control)	63.0 ± 1.4	B	11.3 ± 0.2	B
Cement + exploded WFS	30.3 ± 0.3	C	0.9 ± 0.1	—
+ MgCl_2 1%	31.8 ± 0.7	C	0.3 ± 0.2	—
+ MgCl_2 2%	34.1 ± 0.3	C	0.3 ± 0.1	—
+ MgCl_2 3%	37.3 ± 0.5	C	0.2 ± 0.1	—
+ MgCl_2 4%	64.2 ± 0.8	B	15.7 ± 0.2	B
+ MgCl_2 5%	67.0 ± 0.8	B	13.0 ± 0.5	B
+ MgCl_2 6%	73.5 ± 0.3	A	7.4 ± 0.5	A
Neat furnace-slag cement (control)	56.0 ± 1.8	A	13.7 ± 0.7	A
Cement + exploded WFS	29.0 ± 0.1	C	0.7 ± 0.2	—
+ MgCl_2 1%	31.5 ± 0.3	C	0.5 ± 0.2	—
+ MgCl_2 2%	36.9 ± 0.2	B	0.9 ± 0.3	—
+ MgCl_2 3%	39.5 ± 0.5	B	0.7 ± 0.2	—
+ MgCl_2 4%	45.6 ± 0.1	B	24.0 ± 0.3	C
+ MgCl_2 4% + CaO 1%	54.8 ± 0.7	A	20.5 ± 2.0	B
+ MgCl_2 4% + CaO 2%	60.6 ± 1.2	A	13.1 ± 1.5	A

^aEach value is the average of three replications ± standard deviation

^bMeans with the same capital letter are not significantly different at the 5% significance level (based on a Duncan grouping analysis)

^cOnly one peak appeared within 1 h during the 24-h observation period

Table 3. Effects of additive contents on hydration characteristics of cement mixed with exploded WFS (sugi, air-dried)

Composite	T_{\max}^a (°C)	Grouping ^b	t_{\max}^a (h)	Grouping ^b
Neat ordinary Portland cement (control)	63.0 ± 1.4	A	11.3 ± 0.2	B
Cement + exploded WFS	32.3 ± 0.2	B	0.6 ± 0.2	– ^c
+MgCl ₂ 1%	32.6 ± 0.3	B	0.4 ± 0.3	–
+MgCl ₂ 2%	33.5 ± 2.4	B	0.4 ± 0.1	–
+MgCl ₂ 3%	36.2 ± 2.1	B	0.2 ± 0.1	–
+MgCl ₂ 4%	61.2 ± 0.2	A	17.4 ± 0.2	C
+MgCl ₂ 5%	64.5 ± 2.3	A	13.9 ± 0.5	B
+MgCl ₂ 6%	67.8 ± 1.6	A	8.6 ± 0.5	A

^aEach value is the average of three replications ± standard deviation

^bMeans with the same capital letter are not significantly different at the 5% significance level (based on a Duncan grouping analysis)

^cOnly one peak appeared within 1 h during the 24-h observation period

Table 4. Effects of additive contents on hydration characteristics of cement mixed with exploded WFS (sugi, water-soaked, 2 cm in length)

Composite	T_{\max}^a (°C)	Grouping ^b	t_{\max}^a (h)	Grouping ^b
Neat ordinary Portland cement (control)	63.0 ± 1.4	A	11.3 ± 0.2	B
Cement + exploded WFS	29.9 ± 0.1	C	1.0 ± 0.2	– ^c
+MgCl ₂ 1%	31.3 ± 0.2	C	0.6 ± 0.1	–
+MgCl ₂ 2%	33.4 ± 0.4	C	0.3 ± 0.1	–
+MgCl ₂ 3%	37.3 ± 0.3	C	0.2 ± 0.1	–
+MgCl ₂ 4%	50.0 ± 0.2	B	18.3 ± 1.8	C
+MgCl ₂ 5%	60.4 ± 0.2	A	13.1 ± 0.1	B
+MgCl ₂ 6%	65.2 ± 0.3	A	9.4 ± 0.1	A

^aEach value is the average of three replications ± standard deviation

^bMeans with the same capital letter are not significantly different at the 5% significance level (based on a Duncan grouping analysis)

^cOnly one peak appeared within 1 h during the 24-h observation period

of neat OC (control). Thus, an additive MgCl₂ content of 4%–5% was considered appropriate and acceptable for the mixtures.

Similar to the FC, the single additive chemical of MgCl₂ was found to be useful for enhancing the hydration reaction of mixtures. However, the accelerating effect of MgCl₂ + CaO was significant. When added with 2% CaO, the T_{\max} and t_{\max} of mixtures were adjusted to 60.6°C and 13.1 h; and in terms of the Duncan grouping analysis, the figures could be respectively classified into a group that was consistent with those of neat FC. Thus, 4% MgCl₂ + 2% CaO was also determined to be an acceptable additive content for the mixtures.

Table 3 shows the hydration characteristics of cement mixed with exploded WFS (air-dried). Although compared with the figures in Tables 1 and 2 the sugar contents were found to be increased about 20.2-fold and were much higher than that of exploded WFS (water-soaked), it was found that not less than 5% MgCl₂ is required to obtain hydration characteristics close to those of neat OC based on the Duncan grouping analysis. There were no significant differences in additive content between the water-soaked and air-dried exploded WFS when mixed with OC.

When OC was directly mixed with short exploded WFS in 2-cm lengths, as shown in Table 4, strong retarding effects were similarly observed, and the higher additive content of the chemical was considered necessary for enhancing the compatibility of mixtures. According to the

Duncan grouping analysis, for enhancing the hydration reaction of mixtures at least 5% of MgCl₂ was needed for the mixture as well. Based on these results, it should be pointed out that in this case hydration characteristics of mixtures had little relation to the surface area of the exploded WFS. This finding is attributed to the fact that during WVEP the degraded components are mostly deposited on the surface of exploded WFS because wood degradation resulting from the penetration of high-temperature and high-pressure steam flow mainly took place on primary cell walls and middle lamellae, which are rich in hemicelluloses and pectins.

Conclusions

The exploded WFS showed a strong retarding influence on the hydration reaction of WCM in comparison with normal and unexploded wood. The results revealed that WVEP would result in significant changes in wood components.

The sugar contents of exploded WFS were much higher, increasing about 20-fold (air-dried) and 10-fold (water-soaked) in comparison with unexploded wood. The large amount of degraded polysaccharides due to water–heat degradation of wood components (e.g., hemicelluloses and pectins) was the major factor that inhibited the setting of WCM.

There were significant differences in the distribution of polysaccharide molecular weight in extractives between unexploded wood and exploded WFS. In contrast to unexploded wood, HPLC analysis showed that the main peaks representing polysaccharide molecular weight in extractives of exploded WFS shifted markedly to a lower degree of polymerization.

Among six chemicals, $MgCl_2$ was determined to be an effective additive for restraining the inhibitory influence of degraded polysaccharides. Addition levels of 2%–3% and 4%–5% of $MgCl_2$ were considered sufficient for QC and OC, respectively. As for the FC, the composite additives $MgCl_2$ (4%) plus CaO (2%) were found to have an obvious accelerating effect.

In the case of exploded WFS (air-dried), not less than 5% of $MgCl_2$ is required to enhance setting of the mixture, even though a higher sugar content was measured than in exploded WFS (water-soaked). There were no significant differences between the water-soaked and air-dried exploded WFS regarding the contents of $MgCl_2$ when mixed with OC.

It was also shown that there were similar hydration behaviors of WCM whether mixed with wood powder or short exploded WFS (2 cm in length), and the same levels of $MgCl_2$ were needed to overcome retarding effects. This implies that the hydration characteristics of mixtures had little relation to the surface area of exploded WFS.

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