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Hydration of Portland Cement Pastes Containing Untreated and Treated Hemp Powders

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Abstract: In this study, the residual of hemp products as biomass wastes has been ground into powders to be used in cement-based materials. The effect of untreated and alkaline-treated hemp powders on the hydration of portland cement paste was studied by isothermal calorimetry analysis, thermogravimetric analysis, Fourier transform infrared spectroscopy analysis, and scanning electron microscopy analysis. The results show that partially replacing cement with untreated and alkaline-treated hemp powders can delay cement hydration and reduce the degree of hydration. Compared with untreated hemp powders, alkaline-treated (washed or nonwashed) hemp powders show a lower set delay effect on cement hydration; moreover, distilled-water-washed treated hemp powders delay less than nonwashed treated hemp powders. The study also shows that coarse hemp powders exert a lower set delay effect on cement hydration than fine hemp powders. Generally, the distilled-water-washed treated coarse hemp powders exert the minimum delay effect on cement hydration. **DOI: 10.1061/(ASCE)MT.1943-5533.0003209.** © *2020 American Society of Civil Engineers.*

Author keywords: Cement hydration; Hemp powders; Alkaline treatment; Degree of hydration; Set delay effect.

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Introduction

The application of plant-based biomass wastes to concrete provides a sustainable way to develop green construction materials. Many researchers have attempted to use different forms of plant-based biomass wastes, such as plant fibers (Chakraborty et al. 2013b; Dewi and Wijaya 2017; Li et al. 2004), plant chips (Guo et al. 2019), and plant powders (Sakr 2006; Tay 1990). Biomass powders (often called ash), such as rice husk ash (Habeeb and Mahmud 2010) or corncob ash (Adesanya and Raheem 2009), are often the residue after combustion. They can be used as a replacement of pozzolans in concrete. Research on the direct use of plant-based powders without combustion is limited. Recently, Matos et al. (2015) showed that a reasonable partial replacement of cement with cork powders in self-compacting concrete could result in a good strength level and suitable durability.

Although plant-based powders have the potential to be used in construction materials, several researchers reported the application of plant-based powders can reduce the mechanical properties of concrete (Guerra et al. 2012; Karim et al. 2017). There are some challenges toward its good application. For example, Karim et al. (2017) studied palm oil shell powder blended cement and concluded the partial replacement of cement with palm oil shell powders can increase the setting time of concrete. Plant-based powders, similar to fibers and chips, are mainly composed of cellulose, hemicellulose, lignin, and extractives. These organic components can delay cement hydration by forming a protective layer around the partially hydrated cement grains or forming a chelate complex with the cations present in the hydrated cement (Jo and Chakraborty 2015). In addition, it was reported that hemicellulose and lignin are soluble in the alkali solution (John and Anandjiwala 2008; John and Thomas 2008). They can be decomposed when subjected to the alkaline environment of concrete (Wei and Meyer 2014). The decomposition products can also delay the hydration of cement (Bilba et al. 2003; Vaickelionis and Vaickelioniene 2006; Xie et al. 2016). Therefore, in order to mitigate the delay effect of plant-based biomass powders on cement hydration, it is necessary to conduct treatment prior to its application.

Up to now, many treatments have been applied to plant-based biomass wastes, of which alkaline treatment is the most commonly used. It was reported that alkaline treatment can remove the hemicellulose, lignin, pectin, and wax of plants (Lu and Oza 2011; Sawpan et al. 2011). Jo and Chakraborty (2015) reported that a mild alkali treatment on jute was demonstrated to reduce the set delay effect on cement hydration compared with untreated jute. Quiroga et al. (2016) compared three different treatment methods, including water extraction, alkaline hydrolysis, and retention of inhibitory substances, on wood and concluded that alkaline hydrolysis was the most effective method for minimizing the set delay effect on cement hydration. Although there are very limited reports on the application of treated plant-based powders in construction materials, alkaline treatment is expected to be a practical method due to the alkalinity of the cement-based materials.

In this study, the residuals of hemp products were ground into powders and then treated by a saturated lime (calcium hydroxide) solution. Untreated and alkaline-treated hemp powders were used to partially replace cement, respectively. The purpose of this study is not only to identify the effect of untreated and alkaline-treated hemp powders on the hydration of cement paste, but also to figure out the effect of replacement rate, treatment method, and hemp powder size on cement hydration. Moreover, the microstructure of cement paste without and with hemp powders was also examined.

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(a) (b) (c)

Fig. 1. (a) Hemp leaves; (b) fine hemp powders; and (c) coarse hemp powders.

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Materials and Experiments

Hemp Powder Preparation

Hemp powders were prepared from flowering materials (flowers and leaves) and used to partially replace cement in this study. These flowering materials were provided by Green Remedy (Louisville, Kentucky). Cannabidiol (CBD) was extracted from them prior to hemp powder preparation. To prepare the hemp powders, oven-dried flowering materials [Fig. 1(a)] were firstly chopped and blended into powders by using a high-speed blender and then sieved through a No. 200 sieve. The powders passing through the sieve were called fine hemp powders, as shown in Fig. 1(b), and those retained on the sieve were called coarse hemp powders, as shown in Fig. 1(c).

Chemical Treatment

The fine and coarse hemp powders were chemically treated by using the same procedure as introduced subsequently. Saturated calcium hydroxide solution (saturated lime water) was prepared in a beaker at room temperature. Hemp powders were introduced to the prepared solution (hemp-to-solution mass ratio = 0.1) and stirred by using an AREX-6 Digital PRO (a kind of hot plate stirrer) (Velp Scientifica, Usmate, Italy) to ensure good dispersion. The beaker was then covered to avoid water evaporation. After 24 h, a centrifuge was used to filter out hemp powders from the calcium hydroxide solution. A part of the treated hemp powders was collected without washing. The remaining part of the treated hemp powders was further washed by distilled water several times until the pH was close to neutral. The collected wet hemp powders (both washed and nonwashed) were dried in an oven for 24 h at 40°C. Finally, the dried hemp powders were ground by using a mortar and pestle to eliminate clumping. Based on the hemp powder size and treatment method, six types of hemp powders were obtained, as listed in Table 1.

Table 1. Classification of hemp powders

Sample	Specification
RU	Untreated hemp powders retained on No. 200 sieve
PU	Untreated hemp powders passing through No. 200 sieve
RTN	Nonwashed treated hemp powders retained on No. 200 sieve
PTN	Nonwashed treated hemp powders passing through No. 200 sieve
RTW	Distilled-water-washed treated hemp powders retained on No. 200 sieve
PTW	Distilled-water-washed treated hemp powders passing through No. 200 sieve

Note: P = passing; R = retained; U = untreated; T = treated; N = nonwashed; and W = washed.

Cement Paste Preparation

The chemical compositions and mineral clinker compounds of the Type I/II cement used are given in Tables 2 and 3 (Shang and Sun 2019). The water/binder ratio was kept at 0.50, and hemp powders were used to replace 5.0% and 10.0% of cement by weight. The corresponding mix proportion is given in Table 4. The samples are named after the hemp powder type and dosage. For example, RTW10 denotes the paste with 10% of cement replaced by distilled-water-washed treated hemp powders that were retained on a No. 200 sieve.

Isothermal Calorimetry Test

In order to evaluate the hydration of cement, an isothermal calorimetry (IC) test was conducted on cement paste according to ASTM C1702 (ASTM 2009). A TAM Air (TA Instruments, New Castle, Delaware), a commercial calorimeter, was adopted

Table 2. Chemical composition of Type I/II low alkaline (LA) portland cement

Chemical compound	Weight (%)
CaO	63.30
SiO_2	19.70
Al_2O_3	5.00
Fe_2O_3	3.47
MgO	3.59
SO_3	2.50
Na_2O	1.55
K_2O	0.45
Loss on ignition	0.54

Table 3. Major compounds of Type I/II LA portland cement

Clinker phase	Weight (%)
C_3S	59.32
C_2S	11.81
C_3A	7.39
C_4AF	10.55

Table 4. Mix proportions of cement paste

Sample	Water	Cement (Type I/II)	Hemp powder	Specification
Control	0.5	1	0	Cement paste with no hemp powders
PU5	0.5	0.95	0.05	Cement paste with 5% PU
RTN5	0.5	0.95	0.05	Cement paste with 5% RTN
PTN5	0.5	0.95	0.05	Cement paste with 5% PTN
RTW5	0.5	0.95	0.05	Cement paste with 5% RTW
PTW5	0.5	0.95	0.05	Cement paste with 5% PTW
PU10	0.5	0.90	0.10	Cement paste with 10% PU
RTN10	0.5	0.90	0.10	Cement paste with 10% RTN
PTN10	0.5	0.90	0.10	Cement paste with 10% PTN
RTW10	0.5	0.90	0.10	Cement paste with 10% RTW
PTW10	0.5	0.90	0.10	Cement paste with 10% PTW

Note: P = passing; R = retained; U = untreated; T = treated; N = nonwashed; W = washed; 5 = 5%; and 10 = 10%.

in this study (Sun et al. 2017). It is an eight-channel isothermal heat conduction calorimeter with an operating temperature range of 5°C–60°C. Before the testing, the equipment was carefully calibrated based on the calibration procedures specified by the manufacturer's manual. The energy change during hydration was collected and registered by an automated data-acquisition program. The energy value was calculated based on the unit weight of cementitious materials' mass.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was conducted on each type of hemp powder under a nitrogen atmosphere with a flow rate of 40 mL/min from 25°C to 600°C, with a heating rate of 10°C/min. Around 10 mg of hemp powders were used for each run. The purpose of this step was to study the decomposition of hemp powders within the temperature range and thus evaluate the effectiveness of calcium hydroxide treatment on the removal of hemicellulose.

TGA was also conducted on 7-day paste mixtures under a nitrogen atmosphere, with a flow rate of 40 mL/min from 25°C to 1,100°C. The temperature profile includes increasing from room temperature to 105°C by 20°C/min and then keeping the temperature at 105°C for 12h to remove the evaporable water in the sample. Subsequently, the sample was heated from 105°C to 1,100°C by 20°C/min, and then the temperature was kept at 1,100°C for 6 h to extract all chemically bound water (CBW). Around 20 mg of paste sample was used for each run. The TGA tests were also conducted on pure cement powder and each type of hemp powder by adopting the same test procedure in order to correct the weight loss between 105°C and 1,100°C for chemically bound water calculation.

For cement paste without hemp powders, the calculation of CBW is regularly defined as the weight loss of the tested cement paste between 105°C and 1,100°C, corrected for the loss on ignition (LOI) of the dry cement powder itself (Cao et al. 2015; Feng et al. 2004). However, for cement pastes with hemp powders, the calculation of chemically bound water is more complicated and needs to be additionally corrected to account for the decomposition of hemp powders involved. In this study, the detailed procedures of the CBW correction and the corresponding calculation of degree of hydration (DoH) for cement pastes with hemp power are given in the Appendix.

Fourier Transform Infrared Spectroscopy Analysis

Fourier transform infrared spectroscopy (FTIR) testing was conducted on pure cement powder and 7-day cement pastes by using a PerkinElmer Spectrum 100 series spectrometer (Shelton, Connecticut). The sampling technique of attenuated total reflection (ATR) was adopted to enable samples to be examined directly without further preparation (State of Connecticut 2005). The background spectrum was collected at ambient atmosphere, and then the samples were directly analyzed. The spectra were recorded in the range of 650–4,000 cm^{-1} with a resolution of 4 cm^{-1} (Horgnies et al. 2013). The purpose of the FTIR test is to evaluate the effect of hemp powders on cement hydration qualitatively by observing the change of functional groups of cement pastes.

Scanning Electron Microscopy Analysis

Scanning electron microscopy (SEM) analysis was conducted on the powdered cement pastes using a NOVA NANOSEM 600 provided by FEI (Hillsboro, Oregon). Before testing, in order to suppress charging effect, gold sputtering was applied to the surface of specimens. During testing, the Everhart Thornley detector (ETD) in the secondary electron mode was adopted. The beam current and voltage were 0.32 nA and 10 kV, respectively. The purpose of SEM analysis is to observe the microstructure of cement pastes with hemp powders and then identify the effect of hemp powders on cement hydration.

Results and Discussion

Thermogravimetric Analysis on Hemp Powders

The TGA test has been applied to various types of hemp powders as listed in Table 1. The TGA results on fine hemp powders (PU, PTN, and PTW) and coarse hemp powders (RU, RTN, RTW) are shown in Figs. 2(a and b), respectively. For each figure, the weight loss and weight-loss rate [denoted as derivative weight (%/min)] are plotted.

In both figures, it can be seen that there is a large weight loss between 200°C and 300°C, which is indicative of high hemicellulose content (Lv et al. 2010; Yang et al. 2007). Even a clear shoulder peak at 200°C–300°C exists in DTGA curves for both figures, which is mainly attributed to the decomposition of hemicellulose (Wang et al. 2016). Between 200°C and 300°C, the weight losses of PU, PTN, and PTW are 22.53%, 17.35%, and 16.04%, respectively, and the weight losses of RU, RTN, and RTW are 21.87%, 17.00%, and 15.18%, respectively, as indicated in Table 5. Compared with untreated hemp powders, for chemically treated hemp powders, the hemicellulose content decreases, and also the shoulder peaks in DTGA curves become less obvious. In addition, both PTW and RTW have much smoother DTGA curves at 200°C–300°C than PTN and RTN. This can be attributed to the washing process after the chemical treatment that further removes hemicellulose. The weight loss of fine hemp powders is slightly more than that of the coarse hemp powders, which may be because fine hemp powders with the larger specific surface area are much easier to be decomposed than coarse hemp powders.

From both figures, a significant peak can be seen between 300°C and 400°C on the derivative. weight curve, which is mainly due to the decomposition of cellulose (Yang et al. 2007). In this temperature range, the weight losses of PU, PTN, and PTW are 18.68%, 21.12%, and 23.40%, respectively, and the weight losses of RU, RTN, and RTW are 19.03%, 23.92%, and 26.25%, respectively, as indicated in Table 5. It can be seen that the weight loss of cellulose correlates to the weight loss of hemicellulose. That is because the chemical treatment and washing process remove some hemicellulose, resulting in the increased relative content of cellulose.

Bilba et al. (2003) reported that hemicellulose could be decomposed into saccharides in an alkali medium that can have a negative impact on cement hydration. The TGA results indicate that the

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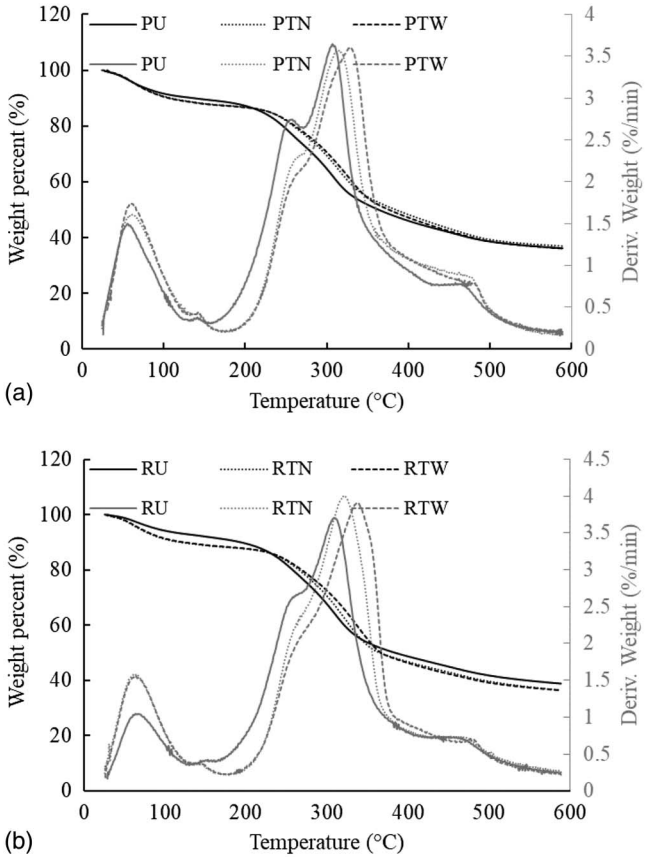

Fig. 2. TGA and DTGA curves of (a) fine; and (b) coarse hemp powders.

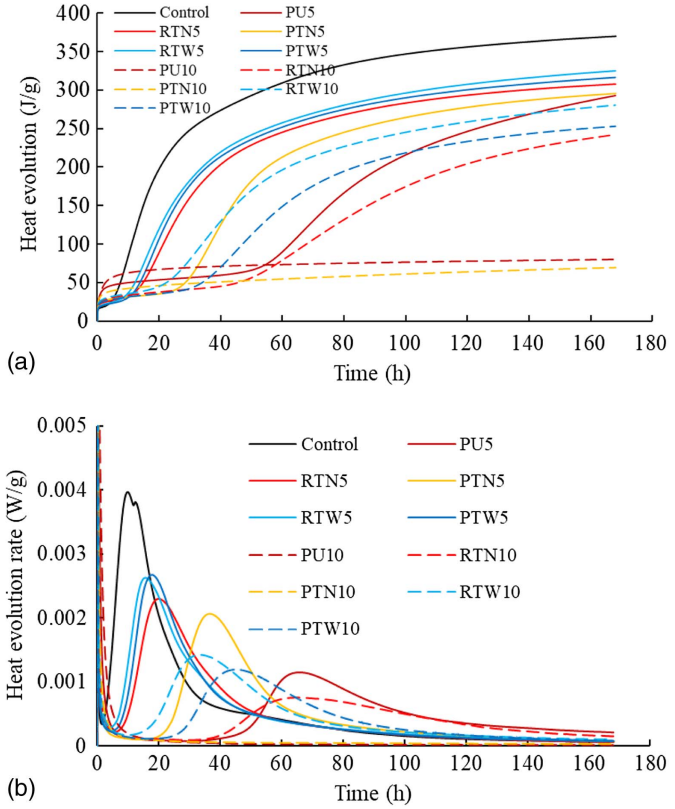

Fig. 3. (a) Heat evolution; and (b) heat evolution rate during hydration of portland cement paste without and with hemp powders.

Table 5. Weight loss (W_L) of all types of hemp powders

Sample	W_L (%)	
	200°C–300°C	300°C–400°C
PU	22.53	18.68
PTN	17.35	21.12
PTW	16.04	23.40
RU	21.87	19.03
RTN	17.00	23.92
RTW	15.18	26.25

chemical treatment and further washing process can assist the removal of hemicellulose, which is helpful to minimize the set delay effect of the hemp powders. By comparison of the weight losses, it is expected that the descending order of set delay effect is PU, PTN, and PTW for fine hemp powders, and RU, RTN, and RTW for coarse hemp powders. In addition, the set delay effect on the hydration of RU, RTN, and RTW is expected to be less than that of PU, PTN, and PTW, respectively, due to the smaller specific surface area.

Isothermal Calorimetry and Thermogravimetric Analysis on Cement Pastes

Based on the calorimetric analysis, the heat evolution and heat evolution rate during hydration of portland cement pastes without and with hemp powders are shown in Figs. 3(a and b), respectively. The hydration time corresponding to the peaks in Fig. 3(b) is listed in Table 6. The DoH of 7-day cement pastes is calculated according to the procedure introduced in the Appendix and summarized in Fig. 4.

Table 6. Hydration time corresponding to peaks of heat evolution rate curves

Sample	Peak 1 (h)	Peak 2 (h)
Control	9.91	12.45
PU5	65.72	—
RTN5	20.01	—
PTN5	36.64	—
RTW5	15.74	—
PTW5	18.01	—
PU10	—	—
RTN10	65.15	—
PTN10	—	—
RTW10	33.56	—
PTW10	45.37	—

Note: M-dashes indicate no peak.

From Fig. 3(b), two peaks next to each other [corresponding to C_3S (first peak from the left side) and C_3A hydration (second peak), respectively] of the control sample can be clearly seen; however, for any other sample, the second peak disappears, showing the C_3A hydration and ettringite conversion are much affected. This may be because saccharides from hemp powders prevent the formation of ettringite. It has been reported that saccharides influence the morphological evolution of hydrating aluminate particles, such as C_3A (Smith et al. 2011). Moreover, the microstructure analysis introduced in this study shows the formation of needlelike ettringites is much influenced by hemp powders.

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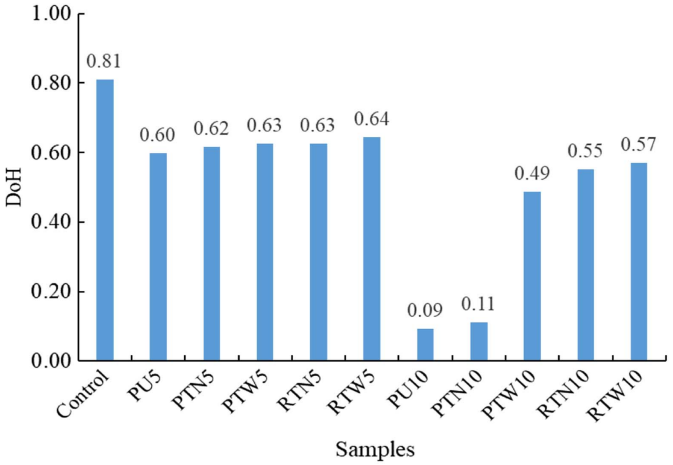

Fig. 4. Degree of hydration of 7-day cement paste samples.

Compared to the control, Figs. 3(a and b) show that the heat evolution and heat evolution rate of other samples with hemp powders are all reduced, and Table 6 indicates that the hydration time corresponding to the maximum peak of heat evolution rate curve increases (except for PU10 and PTN10). That indicates the addition of hemp powders in cement delays the hydration, which is supported by the reduced DoH at 7 days, as shown in Fig. 4. For PU10 and PTN10, the heat evolution amount [Fig. 3(a)] is very low, and no peaks exist in their heat evolution rate curve [Fig. 3(b)]. The calculated DoH values in Fig. 4 are very small. When 10% of the cement powder is replaced, the delay effect of hemp powders is so significant that the dormant period of the samples is very much extended.

The delay effect of hemp powders on cement hydration could be mainly due to the fact that some saccharides are possibly leached out (Vaickelionis and Vaickelioniene 2006). The delaying mechanism of saccharides for cement hydration has not been identified clearly; however, some possible reasons are proposed. One reason is that saccharides can form a complex with the metal ions present in the cement through chelation (Chakraborty et al. 2013a). A protective layer around the partially reacted cement grain is formed to prevent further hydration of cement, which can delay the nucleation and growth of the hydrated products (Jo and Chakraborty 2015). The second reason may be that some saccharides are unstable in cement paste and can be degraded into some sugar acids, which can inhibit cement hydration (Kochova et al. 2017). The third possible reason can be that organic retarders have strong calcium binding capacity that can prevent the formation of calcium–silicate–hydrate (C-S-H) (Kochova et al. 2017).

Effect of Replacement Content on Cement Hydration

In this study, 5% and 10% hemp powders were used to replace cement, respectively. Because for cement pastes with any type of hemp powders, the effect of replacement content on cement hydration is similar, RTN-incorporated cement pastes are taken as an example for analysis. The heat evolution and heat evolution rate of RTN-incorporated cement paste with different replacement contents are shown in Figs. 5(a and b), respectively. It can be seen that compared with RTN5, the heat evolution and heat evolution rate of RTN10 is lower. Also, Table 6 and Fig. 4 indicate that with the increase of replacement content, the hydration time corresponding to the maximum peak of heat evolution rate curve increases, and the DoH value decreases. This is not only because higher replacement content can dilute cement and thus undoubtedly reduce the heat evolution, but also more hemp powders can be possibly decomposed to generate more saccharides in cement paste, inhibiting cement hydration.

Effect of Treatment Method on Cement Hydration

In this study, different treatment methods were applied to hemp powders. The cement pastes with 5% fine hemp powders are taken as an example for analysis. Figs. 6(a and b) plot the heat evolution and heat evolution rate of the pastes with 5% fine hemp powders, respectively. PTN5 and PTW5 both have a higher heat evolution amount and heat evolution rate than those of PU5, and PTW5 has the highest amount among the three. Moreover, the hydration time corresponding to the maximum peak of the heat evolution rate curve is reduced, and the corresponding DoH value is improved, as reported in Table 6 and Fig. 4, respectively. This indicates that compared with untreated hemp powders (PU), the addition of treated hemp powders (PTN or PTW) to cement paste can mitigate the delay effect on the hydration of cement. The reason can be after calcium hydroxide treatment, much hemicellulose, lignin, and other extractives that can be decomposed into saccharides are removed from hemp powders (Lu and Oza 2011; Sawpan et al. 2011). This is also confirmed by the TGA test on hemp powders in this research.

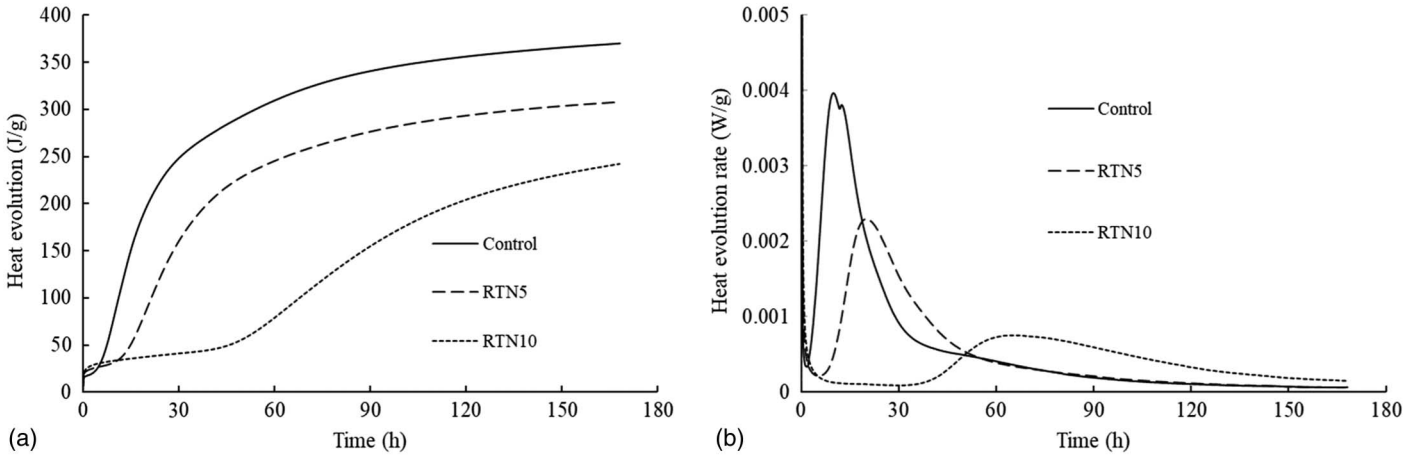

Fig. 5. (a) Heat evolution; and (b) heat evolution rate during hydration of RTN-incorporated cement paste.

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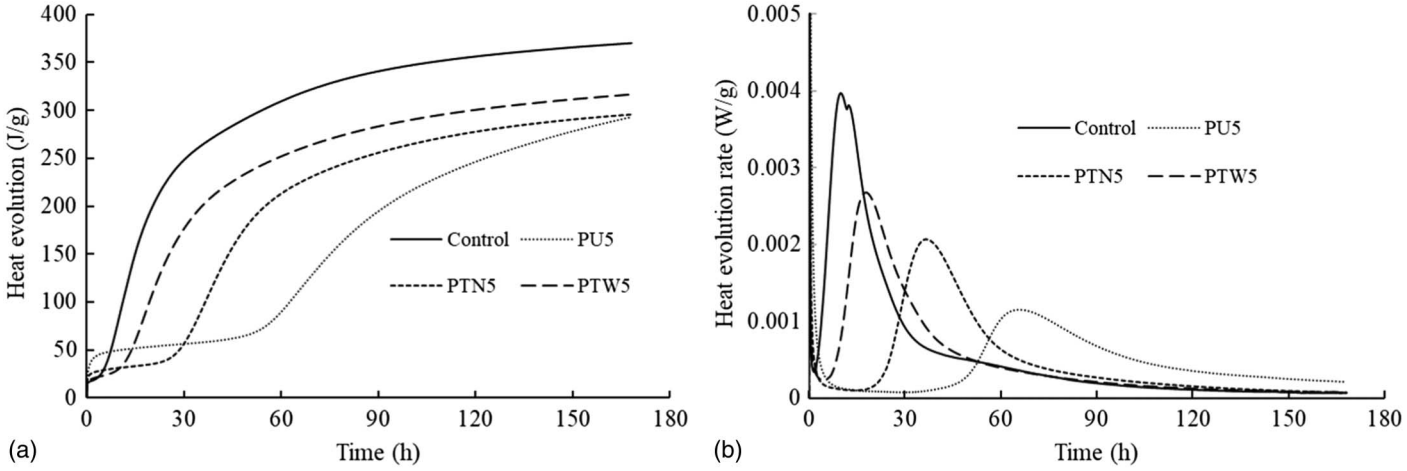

Fig. 6. (a) Heat evolution; and (b) heat evolution rate during hydration of portland cement paste with 5% fine hemp powders.

Compared with nonwashed treated hemp powders (PTN), the washing process after chemical treatment can further mitigate the delay effect on the hydration of cement, resulting in a higher degree of hydration for washed treated hemp powders (PTW). This can be attributed to the further removal of hemicellulose during the washing process. This can also be due to the removal of some remaining saccharides during washing.

Effect of Hemp Powder Size on Cement Hydration

To study the size effect, cement pastes with 10% washed treated hemp powders are compared. The heat evolution and heat evolution rate of portland cement paste with 10% washed treated hemp powders is shown in Figs. 7(a and b), respectively. It can be seen that compared with RTW10, PTW10 can lower the heat evolution amount and heat evolution rate. Moreover, Table 6 and Fig. 4 demonstrate that PTW10 has an increased hydration time corresponding to the maximum peak of the heat evolution rate curve and a lower DoH value, respectively. This indicates that the addition of fine hemp powders (PU, PTN, and PTW) can intensify the delay effect on the hydration of cement compared with coarse hemp powders (RU, RTN, and RTW). The possible reason may be that finer hemp powders (PU, PTN, and PTW) have higher specific surface area than coarse hemp powders (RU, RTN, and RTW), and they can be much easier to be dissipated in cement paste to produce saccharides, delaying the hydration of cement.

FTIR Analysis on Cement Pastes

The FTIR spectra of original portland cement (OPC) and hydrated cement pastes at 7 days are shown in Fig. 8. There are four important regions, which include a water region (>1,600 cm^{-1}), carbonate region (1,400–1,450 cm^{-1}), sulfate region (1,100–1,150 cm^{-1}), and silicate region (<1,000 cm^{-1}) (Mollah et al. 2000). The FTIR spectral data of each region are given in Table 7.

Water Region

From Fig. 8(a), it is shown that for OPC, there are no significant peaks in the water region. However, for all hydrated cement pastes [Figs. 8(b–l)], three important peaks appear in this region, and the corresponding spectral data are provided in Table 7. The first peak appears between 3,612 and 3,645 cm^{-1}, which corresponds to the OH band from $Ca(OH)_2$ (Mollah et al. 2000; Ylmén et al. 2009). This is attributed to the hydration of C_3S and C_2S. The second peak appears between 3,409 and 3,435 cm^{-1}, which corresponds to hydrogen-bonded OH species (OH–OH) adsorbed on the surfaces (Mollah et al. 2000). This is due to the symmetric and asymmetric stretching vibration (υ_1 and υ_3) of H_2O in the hydrated cement

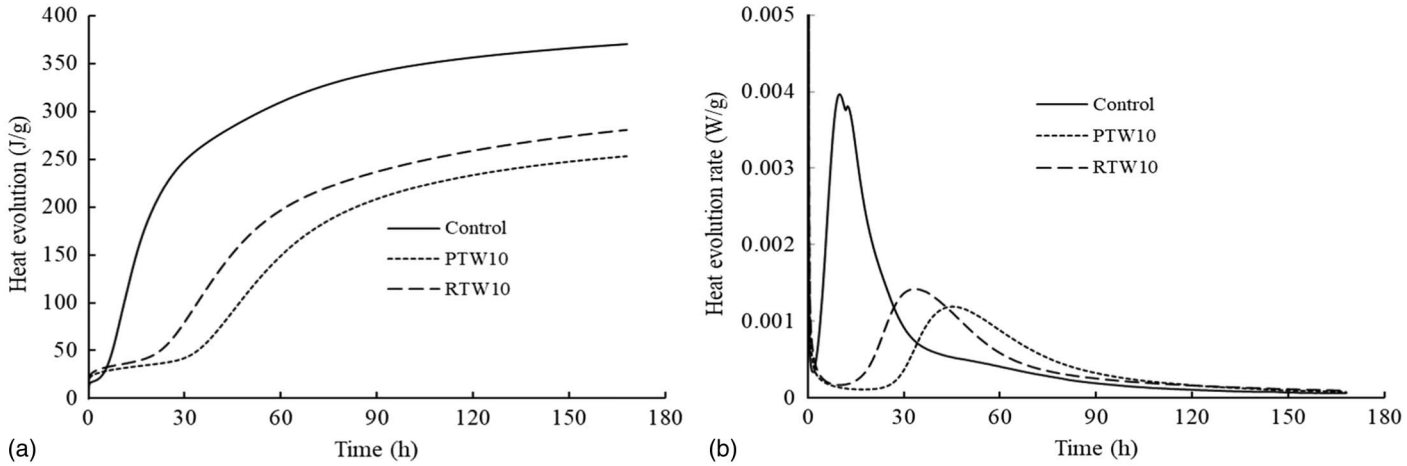

Fig. 7. (a) Heat evolution; and (b) heat evolution rate during hydration of portland cement paste with 10% washed treated hemp powders.

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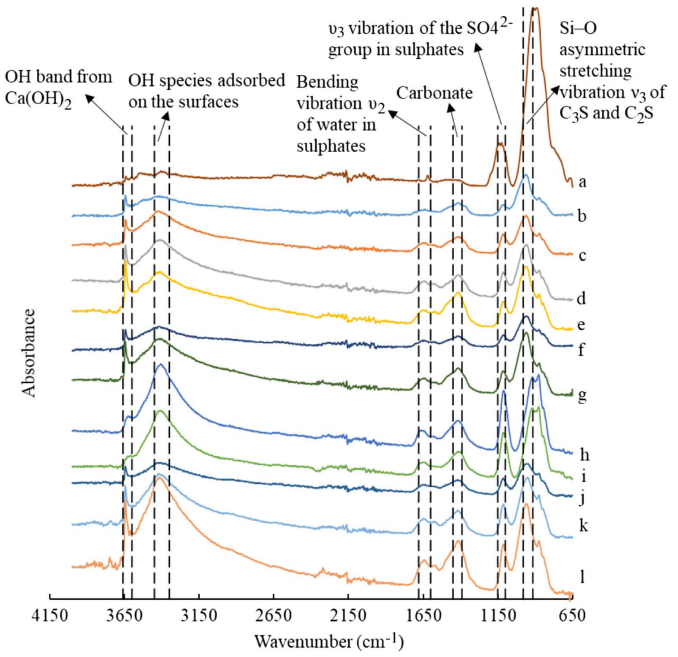

Fig. 8. FTIR spectra of (a) OPC; (b) control; (c) PU5; (d) PTN5; (e) PTW5; (f) RTN5; (g) RTW5; (h) PU10; (i) PTN10; (j) PTW10; (k) RTN10; and (l) RTW10.

Table 7. FTIR spectral data for original portland cement and hydrated cement pastes

	Water region			Carbonate region	Sulfate region	Silicate region
Sample	Peak 1	Peak 2	Peak 3	Peak 1	Peak 1	Peak 1
OPC	—	—	—	—	1,143 and 1,126	917
Control	3,644	3,419	1,641	1,424	1,118	962
PU5	3,643	3,422	1,650	1,420	1,119	961
PTN5	3,644	3,419	1,648	1,424	1,117	960
PTW5	3,644	3,419	1,658	1,420	1,120	965
RTN5	3,644	3,427	1,657	1,425	1,115	961
RTW5	3,645	3,418	1,649	1,420	1,118	964
PU10	3,626	3,409	1,670	1,424	1,114	917
PTN10	3,612	3,414	1,653	1,419	1,114	923
PTW10	3,645	3,435	1,655	1,422	1,115	964
RTN10	3,645	3,423	1,650	1,421	1,114	954
RTW10	3,645	3,419	1,643	1,416	1,114	959

Note: M-dashes indicate no peak.

paste (Chakraborty et al. 2013a; Choudhary et al. 2015; Ylmén et al. 2009). The third peak appears between 1,641 and 1,670 cm^{-1}, which may be attributed to the bending vibration υ_2 of water in sulfates, mainly gypsum (Ylmén et al. 2009).

Carbonate Region

For OPC, Fig. 8(a) does not show any significant peaks in the carbonate region. However, for all hydrated cement pastes [Figs. 8(b–l)], a peak between 1,416 and 1,425 appears, which is due to the reaction of atmospheric CO_2 with the calcium hydroxide produced by cement hydration (Liu and Sun 2013).

Sulfate Region

The sulfates present in OPC are gypsum ($CaSO_4 \cdot 2H_2O$), hemihydrate ($CaSO_4 \cdot 0.5H_2O$), and anhydrite ($CaSO_4$), and the sulfate region in the FTIR spectrum is located between 1,100 and 1,150 cm^{-1}, corresponding to the υ_3 vibration of the SO_4^{2-} group in sulfates (Choudhary et al. 2015; Ylmén et al. 2009). Fig. 8(a) shows that two peaks (1,143 and 1,126 cm^{-1} listed in Table 7) appear in the sulfate region of OPC, which may be because different forms of sulfates lead to different peaks. Upon hydration, these two peaks become one peak that is between 1,114 and 1,120 cm^{-1} for all hydrated cement pastes, as indicated in Figs. 8(b–l) and Table 7. The shift of the sulfate band toward a lower wave number is due to the formation of ettringite (Mollah et al. 2000).

Silicate Region

Fig. 8(a) shows that OPC has a significant peak at 917 cm^{-1} in the silicate region, which is due to Si–O asymmetric stretching vibration (ν_3) of C_3S and C_2S phases (Choudhary et al. 2015). For hydrated cement pastes (except for PU10 and PTN110), this peak significantly shifts toward a higher wave number that is between 954 and 965 cm^{-1} as indicated in Table 7. The shifting of the Si–O stretching vibration is indicative of the polymerization of silicate units (SiO_4^{2-}), with the formation of C-S-H phase as a result of cement hydration (Govin et al. 2006; Mollah et al. 2000; Peschard et al. 2004). However, Fig. 8(h) shows the silicate peak of PU10 does not shift, and Fig. 8(i) shows the peak of PTN10 only shifts 6 cm^{-1}, which may be because the degree of hydration for these two samples is so low that very little C-S-H is formed. This is confirmed by IC and TGA tests that PU10 and PTN10 are still in the dormant period after 7 days.

Microstructure

In order to examine the effect of untreated and chemically treated hemp powders on the microstructure of cement paste, the control, PU10, PTN10, and PTW10 samples at 7 days are taken as an example for analysis. In addition, the SEM image on unhydrated cement powders is also adopted to assist analysis. For unhydrated cement powders, some large irregular particles can be clearly seen, as shown in Fig. 9(a). For the control cement paste, Fig. 9(b) shows that some needlelike ettringites and C-S-H are formed after cement hydration. For PU10, some large unhydrated cement particles and rodlike ettringites can be seen in Fig. 9(c). This could be due to large amounts of decomposition products (most possibly much saccharides) of untreated hemp powders delaying the hydration of cement, as confirmed by TGA and IC tests in this study.

Moreover, the seriously delayed cement hydration prevented the further development of rodlike ettringites into needlelike ettringites. It has been reported that at a very early stage, ettringites are formed as hexagonal rods, and the relatively slender ettringites can be observed with the hydration progress (Liu 2014). Also, Cody et al. (2001) reported that the presence of sugars can lead to the morphology of ettringites to be short thick hexagonal prisms rather than needles.

For PTN10, many unhydrated cement particles are clearly shown in Fig. 9(d). Compared with PU10, PTN10 has some small needlelike ettringites instead of rodlike ettringites on the surface, which indicates that some needlelike ettringites started to form. The reason may be chemically treated hemp powders have fewer saccharides inside, which makes the hydration of cement less delayed. For PTW10, although a small quantity of unhydrated cement particles can still be identified, they are not obvious in Fig. 9(e). Compared with PU10 and PTN10, more hydration products are formed, and the hydration of PTW10 seems to be the least delayed.

Conclusions

In this research, untreated and calcium hydroxide–treated hemp powders were used to partially replace cement. Compared with

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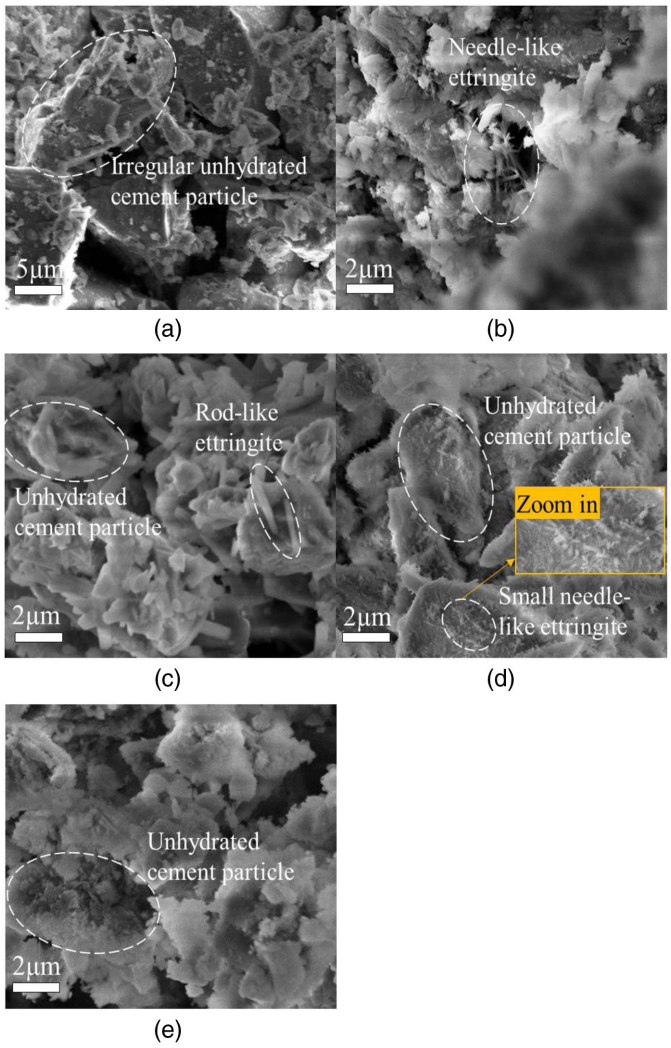

Fig. 9. Microstructure of (a) unhydrated cement powder; (b) control; (c) PU10; (d) PTN10; and (e) PTW10.

pure cement paste, IC testing shows that the incorporation of hemp powders in cement paste can reduce heat evolution amount and slow down heat evolution rate; also, DoH calculated based on TGA testing matched the IC results. This confirms the delayed effects of hemp powders on cement hydration. SEM analysis showed that the formation of hydration products are significantly influenced if hemp powders involved.

The research also shows that the 10% replacement content delays the hydration of cement significantly. Particularly, for PU10 (untreated fine powders) and PTN10 (treated but nonwashed fine powders) samples, the hydration is almost completely inhibited, and the degree of hydration is significantly low compared with others (e.g., 5% replacement). This indicates that 10% is the limit replacement if hemp powders are used.

Compared with the pastes with untreated hemp powders, treated hemp powders can reduce the delay effect when used. Also, the washed treated hemp powders had the minimum delay effect on cement hydration. The reduced delay effect may be attributed to the fact that much hemicellulose is removed after calcium hydroxide treatment and the washing process, as confirmed by TGA test in this study.

In addition, the study also showed that fine hemp powders can delay the hydration of cement paste more than coarse hemp powders, which can be attributed to the higher specific surface area of fine hemp powders that can be easily decomposed into saccharides to delay the hydration of cement.

Generally, regardless of replacement content, treatment method, and hemp powder size, the delaying effect of hemp powders on cement hydration is obvious, which can possibly reduce the mechanical properties of cementitious materials significantly. However, hemp powders can be useful in serving as powder-type retardation agents to adjust cement hydration rate as needed, particularly in some extreme occasions where rapid hydration needs to be avoided.

Appendix. Degree of Hydration Calculation Procedure Based on TGA

Correction for Chemically Bound Water Calculation

TGA testing was conducted on pure cement powder, each type of hemp powder, and paste mixtures according to the procedure introduced previously. The weights of pure cement powder at 105°C and 1,100°C are denoted as C_1 and C_2, respectively. The weights of hemp powders at 105°C and 1,100°C are denoted as H_1 and H_2, respectively. The weights of cement paste mixtures at 105°C and 1,100°C are denoted as W_1 and W_2, respectively. The loss on ignition of pure cement powder between 105°C and 1,100°C is $(C_1 - C_2)/C_1$, denoted as L_c. The weight-loss percentage of hemp powders between 105°C and 1,100°C is $(H_1 - H_2)/H_1$, denoted as L_h. The weight loss of the tested cement paste between 105°C and 1,100°C is calculated as shown in Eq. (1)

$$W_t = W_1 - W_2 \tag{1}$$

where W_1 = weight of cement paste at 105°C; and W_2 = weight of cement paste at 1,100°C.

The weight loss of cement paste between 105°C and 1,100°C (W_t) includes several main parts: (1) decomposition of chemically bound water; (2) loss on ignition of cement powder used; (3) decomposition of hemp powders if involved; and (4) calcium carbonate decomposition. Therefore, to calculate chemically bound water, W_t needs to be modified. The calcium carbonate decomposition correction is not considered because it is hard to be exactly quantified in this study. Therefore, the calculated degree of hydration should be a little bit higher than its real value, but it does not affect the comparative analysis of all samples. Other steps for weight correction of W_t are shown as follows.

Hemp Powder Decomposition Correction (W_h)

For cement paste with hemp powders, W_t needs to be modified by considering the decomposition of hemp powders. Assume the total weight of hemp powders used in cement paste is H_t. Based on the weight-loss percentage of hemp powders between 105°C and 1,100°C, L_h, the remaining weight of hemp powders incorporated into cement paste will be $(1 - L_h) \times H_t$ when cement paste is heated to 1,100°C. Because the weight of cement paste at 1,100°C is W_2, the weight of ignited cement will be $W_2 - (1 - L_h) \times H_t$ by excluding the remaining hemp powders. Based on the loss on ignition of pure cement powder, L_c, the total weight of cement powder used will be deduced as $[W_2 - (1 - L_h) \times H_t]/(1 - L_c)$. If the replacement percentage of cement powder with hemp powders is denoted as r, then the total weight of hemp powders used will be $[W_2 - (1 - L_h) \times H_t]/[(1 - L_c) \times (1 - r)]$.

Finally, an equilibrium can be achieved as shown in Eq. (2), by which H_t can be solved [Eq. (3)]. The total weight of cement powder used can be calculated by substituting H_t into $[W_2 - (1 - L_h) \times H_t]/(1 - L_c)$, as shown in Eq. (4), which is denoted as C_t. If no hemp powders are used in cement paste, the replacement content r is 0, then C_t will be simplified into $W_2/(1 - L_c)$

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$$\frac{[W_2 - (1 - L_h) \times H_t] \times r}{(1 - L_c) \times (1 - r)} = H_t \tag{2}$$

$$H_t = \frac{W_2 \times r}{(1 - L_h) \times r + (1 - L_c) \times (1 - r)} \tag{3}$$

$$C_t = \frac{W_2 \times (1 - r)}{(1 - L_h) \times r + (1 - L_c) \times (1 - r)} \tag{4}$$

where H_t = total weight of hemp powders used in cement paste; C_t = total weight of cement powder used in cement paste; W_2 = weight of cement paste at 1,100°C; r = replacement percentage of cement powder with hemp powders; L_h = weight-loss percentage of hemp powders between 105°C and 1,100°C; and L_c = loss on ignition of pure cement powder (between 105°C and 1,100°C).

Based on the total weight of hemp powders used in cement paste, H_t, and weight-loss percentage of hemp powders between 105°C and 1,100°C, L_h, the weight loss of hemp powders used in cement paste between 105°C and 1,100°C can be obtained as shown in Eq. (5), denoted as W_h. W_h is hemp powder decomposition correction content, which will be subtracted from W_t for calculating chemically bound water if hemp powders are involved

$$W_h = H_t \times L_h \tag{5}$$

where W_h = weight loss of hemp powders used in cement paste between 105°C and 1,100°C; H_t = total weight of hemp powders used in cement paste; and L_h =weight-loss percentage of hemp powders between 105°C and 1,100°C.

Loss on Ignition Correction (W_l)

Based on the loss on ignition of pure cement powder, L_c, the weight loss of cement powder used between 105°C and 1,100°C will be $C_t \times L_c$ as shown in Eq. (6), denoted as W_l. W_l is the loss on ignition correction content, which will be subtracted from W_t for calculating chemically bound water

$$W_l = C_t \times L_c \tag{6}$$

where W_l = weight loss of cement powder used between 105°C and 1,100°C; C_t = total weight of cement powder used in cement paste; and L_c = loss on ignition of pure cement powder.

With the aforementioned correction, the chemically bound water (w_b) of cement paste with hemp powders can be calculated by using Eq. (7). Fig. 10 also schematically plots the correction procedure of weight loss of cement paste

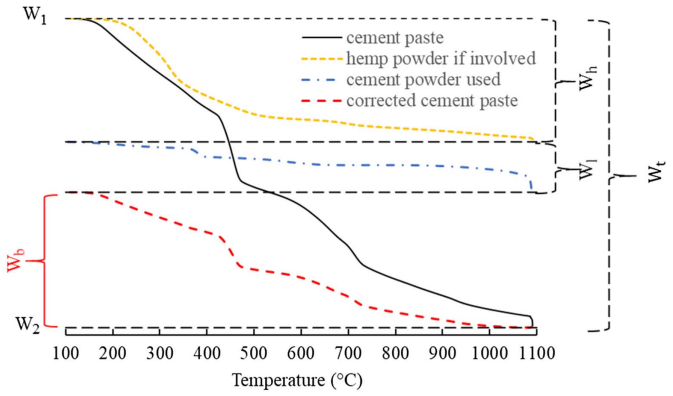

Fig. 10. Schematic of weight-loss correction of cement paste.

$$W_b = W_t - W_l - W_h \tag{7}$$

where W_t = total weight loss of cement paste between 105°C and 1,100°C; W_l = loss on ignition correction, namely the weight loss of cement powder used between 105°C and 1,100°C; and W_h = hemp powder decomposition correction, namely the weight loss of hemp powders used between 105°C and 1,100°C.

Degree of Hydration Calculation

For cement paste with hemp powders, the weight of ignited cement is corrected to be $W_2 - (1 - L_h) \times H_t$ that has been introduced in "Hemp Powder Decomposition Correction" section. The chemically bound water per unit gram of ignited cement then can be calculated by using $W_b/[W_2 - (1 - L_h) \times H_t]$, denoted as W_{bg}. By substituting H_t obtained in Eq. (3) into $W_b/[W_2 - (1 - L_h) \times H_t]$, W_{bg} can be obtained as shown in Eq. (8). If no hemp powders are used in the paste mixtures, the replacement content r is 0, and then W_{bg} will be simplified into W_b/W_2

$$W_{bg} = \frac{W_b \times [(1 - L_h) \times r + (1 - L_c) \times (1 - r)]}{W_2 \times (1 - r) \times (1 - L_c)} \tag{8}$$

where W_{bg} = chemically bound water per unit gram of ignited cement; W_b = chemically bound water of cement paste; W_2 = weight of cement paste at 1,100°C; r = replacement percentage of cement powder with hemp powders; L_h = weight-loss percentage of hemp powders between 105°C and 1,100°C; and L_c = loss on ignition of pure cement powder (between 105°C and 1,100°C).

For completely hydrated Type I portland cement, the chemically bound water per unit gram of ignited cement ranges from 0.23 to 0.25 (Pane and Hansen 2005). In this study, 0.25 is adopted. Therefore, DoH can be calculated by using W_{bg} divided by 0.25, as shown in Eq. (9). If no hemp powders are used in paste mixtures, the replacement content r is 0, and then DoH will be simplified into $W_b/(0.25 \times W_2)$

$$\text{DoH} = \frac{W_b \times ((1 - L_h) \times r + (1 - L_c) \times (1 - r))}{0.25 \times W_2 \times (1 - r) \times (1 - L_c)} \tag{9}$$

where W_b = chemically bound water of cement paste; W_2 = weight of cement paste at 1,100°C; r = replacement percentage of cement powder with hemp powders; L_h = weight-loss percentage of hemp powders between 105°C and 1,100°C; and L_c = loss on ignition of pure cement powder (between 105°C and 1,100°C).

Data Availability Statement

Some or all data, models, or code generated or used during the study are available from the corresponding author by request. The items contain the experimental results of isothermal calorimetry analysis, thermogravimetric analysis, and Fourier transform infrared spectroscopy analysis for this study.

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Notation

The following symbols are used in this paper:

C_t = total weight of cement powder used in cement paste;
C_1 = weight of pure cement powder at 105°C;
C_2 = weight of pure cement powder at 1,100°C;
H_t = total weight of hemp powders used in cement paste;
H_1 = weight of hemp powders at 105°C;
H_2 = weight of hemp powders at 1,100°C;
L_c = loss on ignition of pure cement powder (between 105°C and 1,100°C);
L_h = weight-loss percentage of hemp powders between 105°C and 1,100°C;
r = replacement percentage of cement powder with hemp powders;
W_b = chemically bound water;
W_{bg} = chemically bound water per unit gram of ignited cement;
W_h = weight loss of hemp powders used between 105°C and 1,100°C;
W_l = weight loss of cement powder used between 105°C and 1,100°C;
W_t = weight loss of cement paste between 105°C and 1,100°C;
W_1 = weight of cement paste at 105°C; and
W_2 = weight of cement paste at 1,100°C.

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