EFFECTS OF KENAF BAST FIBRES ON HYDRATION BEHAVIOUR OF CEMENT

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AMEL BA, PARIDAH MT, RAHIM S, OSMAN Z, ZAKIAH A & AHMED SH. 2014. Effects of kenaf bast fibres on hydration behaviour of cement. The compatibility between cement and kenaf bast fibre and its improvement with various types of accelerators were investigated by observation and analysis on hydration behaviour in terms of hydration characteristics, namely; maximum hydration temperature and required time to reach maximum temperature. Five extraction methods (crude, water retting, decortication, NaOH retting and benzoate retting), four accelerators (CaCl2, AlCl3, Na2SO4, CaO), three concentrations (2, 4 and 6%) and three particle sizes (0.5, 0.8 and 4.0 mm) were used. The hydration behaviour of mixtures demonstrated that NaOH and benzoate were unsuitable with cement. Meanwhile, CaCl2 and CaO were found to be effective accelerators for restraining inhibitory influences. In addition, 2% accelerator was available and acceptable for quick-curing cement. Particle sizes of 0.5 and 0.8 mm required addition of accelerators to reach maximum cement setting.

Keywords: Accelerators, particle size, cement hydration reaction

INTRODUCTION

Hydration rate is measured based on the ion concentration and pH value of the mixture. Cement setting is critical in kenaf–cement-bonded composites because the process of Portland cement hydration, which leads to crystallisation and crystal growth, is an exothermic reaction. Higher reaction temperatures give a better crystalline formation of cement. It is possible to determine the extent of cement hydration (setting and hardening) and the strength values of kenaf–cement-bonded composites by measuring the reaction temperature of kenaf–cement–water mixtures during the exothermic reaction. Some researchers used this theory to research the hydration behaviour of wood–cement–water mixtures to estimate the inhibitory characteristics of wood species (Hachmi et al. 1990, Wei et al. 2000). Since hydration rate influences the extent of cement setting, the compatibility of components with cement is very critical. Generally, hydration characteristics of Portland cement may be affected when other components are added to the cement admixtures.

Cement hydration is a complex reaction process requiring water, heating and an alkaline environment (Jennings et al. 2002). One of the key issues in determining the compatibility of wood species for manufacturing wood–cement-bonded composites is the effect of wood on the hydration (setting) rate of the ordinary Portland cement (OPC) (Yi et al. 2000, Jorge et al. 2004). The problem of compatibility between cement and wood arises due to the presence of wood extractives, especially sugars, which appear to inhibit the setting of cement (Wei 2002). Wood mainly consists of three main components, namely, cellulose, hemicellulose and lignin. During treatment under high temperature, high pressure destructuring process, the degradation of wood components such as hemicellulose is...
unavoidable. The severe treatment conditions cause more polysaccharide degradation, releasing degraded carbohydrates with lower polymerisation into water or alkali solution. Theoretically during hydration, the Portland cement is cured once the mixture is dried and through chemical reactions between water and calcium silicate (the main ingredient in cement) (Jennings et al. 2002). However, the hydration of Portland cement will be affected once it is mixed with wood fibres due to the nature of wood such as extractives and heat capacity.

The presence of inhibitory substances such as starches, sugars, hemicelluloses and extractives may delay the hydration of the Portland cement (Vaickelioniene & Vaickelionis 2006, Noor Azrieda et al. 2009). The authors concluded that lignin appeared to have minimal effect on cement setting. Alkali-soluble extractives and sugars contribute to reducing the release of heat and prolonging the setting time of Portland cement as measured by the heat of hydration given off during cement setting. Wood fibre that delays hydration will be classified as not compatible with OPC. The compatibility property indicator is based on the maximum temperature reached at a minimum time. This level of suitability is expressed by the hydration rate. There are three classifications on the degree of hydration rate (Yi et al. 2000): (1) least inhibitory species whereby the maximum temperature is > 50 °C and the time is < 10 hours, (2) moderate inhibitory whereby the maximum temperature is > 40 °C and the time is < 15 hours and (3) highly inhibitory whereby the maximum temperature is < 40 °C and the time is > 15 hours. If the wood fibre is recorded as highly inhibitory, it is considered as unsuitable for the manufacture of wood–cement-bonded composite as removal of extractives from the wood is necessary prior to manufacturing.

Adding an accelerator into a mixture of cement–water can hasten the hydration process. Xu and Stark (2005) found that the accelerator CaCl₂ caused early hydration of wood–cement-bonded composites.

The objective of this study was to observe the hydration rate of five types of kenaf bast fibres which were extracted using different methods. The second objective was to classify kenaf bast fibres as affected by the extraction method. The study also evaluated the influences of accelerators (CaCl₂, AlCl₃, Na₂SO₄ and CaO), concentration (0, 2, 4 and 6%) and particle size (0.5, 0.8 and 4 mm) on the mixture of kenaf bast fibre–cement–water.

**MATERIALS AND METHODS**

Kenaf, sp.V36, was collected from Taman Pertanian Universiti Malaysia. The kenaf bast fibres were extracted using different extraction methods (crude, decortication, water retting, NaOH retting and benzoate retting), ground using Universal cutting mill and screened with different sieve sizes (0.5, 0.8 and 4 mm). The kenaf bast fibres were dried in an oven and kept in sealing plastic bag prior to hydration test (ASTM 1983). The OPC was obtained from Negeri Sembilan, Malaysia. The chemicals, i.e. aluminium chloride (AlCl₃), calcium chloride (CaCl₂), sodium sulphate (Na₂SO₄), calcium oxide (CaO) used were of analytical grade. The hydration test was carried out at the Forest Research Institute Malaysia. The machine used for the test was a hydration test kit (Figure 1). It consisted mainly of (1) 12-point temperature time recorder and plotter, (2) insulating container (thermos flask filled with insulating material such as cotton and mineral wool) and (3) platinum thermocouple. Accessories included a pipe sleeve, copper pipe and plastic container.

The OPC mixture for the test was divided into three different mixtures: (1) OPC neat (control), (2) OPC mixed with kenaf bast fibre (KBF) and water and (3) OPC mixed separately with KBF, water and accelerators (AlCl₃, CaCl₂, Na₂SO₄ and CaO) at three different concentrations (2, 4 and 6%). The mixture of OPC–KBF–water was carried out according to Rahim (1996). To evaluate the index of compatibility between KBF and cement of the hydration reaction, the maximum temperature (T_max) of hydration and the time (t_max) required to reach the temperature were used. Approximately 200 g of cement and 15 g of oven-dried KBF were mixed and kneaded with 90.5 ml of distilled water for about 4 min and then the admixtures were placed in a plastic bag which was stored in a thermoflask (Rahim 1996). A thermocouple was soaked into the mixture which was placed in a thermoflask covered with a lid and the thermocouple was connected to the hydration test machine using Type J wire (Figure 1). Temperature at 5 s interval was recorded automatically by the machine as the mixture was left overnight. Time
and temperature readings were plotted to obtain the exothermic hydration curve. The hydration data were used to calculate the inhibitory index, \( I \). The results were analysed using Statistical Analyses System (SAS). The least significant difference test at 95% level of confidence was used to detect differences between means.

RESULTS AND DISCUSSION

Effects of extraction method of kenaf bast fibres on hydration rate

Figure 2 shows that the hydration of Portland cement is an exothermic reaction. Neat cement attained maximum hydration temperature (MHT) of at least 76.5 °C within a hydration time of 8 hours. However, in the presence of crude bast fibre, the MHT was reduced to 53.2 °C and the hydration time to MHT was delayed to 20 hours. This indicated that the presence of crude bast fibre in Portland cement had some retardation effect on the hydration properties of cement. Figure 2 also shows that in the presence of decorticated, water-retted, NaOH-retted and benzoate-retted bast fibres in cement, the MHT of cement was reduced to below 54, 57, 34 and 32 °C respectively, while the hydration time to MHT delayed further to 14 hours (decorticated and water) and 20 hours (NaOH and benzoate). Three categories of compatibility between wood species and cement has been classified before any wood fibre is considered for use in wood cement composite manufacture (Sanderman et al. 1960). They reported that wood cement admixtures which recorded MHT ≥ 60 °C as compatible, 50–60 °C as intermediate compatible and ≤ 50 °C as incompatible. Based on this classification, our results showed that crude, decorticated and water-retted bast fibres were intermediate compatible while NaOH-retted and benzoate-retted bast fibres were incompatible with Portland cement. Figure 2 shows that the hydration curves of all kenaf-based mixtures were easily distinguished from that of neat cement. The worst was found in NaOH- and benzoate-retted fibres.

On the other hand, Table 1 shows that the extraction method has a significant influence on inhibitory index and pH of fibre. Crude, decorticated and water-retted bast fibres gave moderate inhibitory indexes (Table 2; -0.43, -0.16 and -0.09 respectively) with maximum temperature values respectively 53.2, 54.0 and 57.0 °C and time 20, 14 and 14 hours. Both NaOH- and benzoate-retted bast fibre showed highly inhibitory (-3.63 and -3.68 respectively) with maximum temperatures of 34 and 32 °C respectively and time to MHT of more than 18 hours. It could be concluded that the inhibitory effects of NaOH- and benzoate-retted bast fibres on cement setting increased the maximum time (\( t_{\text{max}} \)) and decreased MHT values. The inhibitory effect of these extracts on cement setting could be noted by the increase in \( t_{\text{max}} \) and decrease in \( T_{\text{max}} \) values.
It was found that NaOH- and benzoate-retted bast fibres was unsuitable for the manufacturing of cement-bonded kenaf board. It is worth noting that acidity also has significant influence on the hydration rate of cement (Nasser et al. 2011). These results were supported by other findings (Zhengatian & Moslemi 1989, Mohamed 2004).

**Effects of extraction method, accelerator and concentration on hydration properties**

The effects of extraction method (crude, decorticated and water-retted), accelerator (\(\text{CaCl}_2\), \(\text{AlCl}_3\), \(\text{Na}_2\text{SO}_4\) and CaO) and concentration (2, 4 and 6 %) on Figures 3 to 5. The MHT of Portland cement was reduced from 82.7 to 54.6, 58.0 and 60.0 hours.

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**Table 1** Summary of ANOVA for effects of extraction methods on inhibitory index and pH

<table>
<thead>
<tr>
<th>Source</th>
<th>Df</th>
<th><strong>p value</strong></th>
<th><strong>Inhibitory index</strong></th>
<th><strong>pH</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>Extraction method</td>
<td>4</td>
<td>&lt; 0001***</td>
<td>&lt; 0001***</td>
<td></td>
</tr>
</tbody>
</table>

***Significant difference at \(p \leq 0.01\)

**Table 2** Inhibitory index and pH of kenaf bast fibres extracted using different methods

<table>
<thead>
<tr>
<th>Extraction method</th>
<th>Inhibitory index (%)</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crude BF</td>
<td>-0.43 b</td>
<td>6.55 c</td>
</tr>
<tr>
<td>Decorticated BF</td>
<td>-0.16 ab</td>
<td>6.50 c</td>
</tr>
<tr>
<td>Water-retted BF</td>
<td>-0.09 a</td>
<td>6.50 c</td>
</tr>
<tr>
<td>NaOH-retted BF</td>
<td>-3.63 c</td>
<td>10.03 a</td>
</tr>
<tr>
<td>Benzoate-BF</td>
<td>-3.68 c</td>
<td>6.71 b</td>
</tr>
</tbody>
</table>

Values within the same column with the same letter are not significantly different (\(p \leq 0.01\)); BF = bast fibres
59.0 °C when mixed with crude, decorticated and water-retted kenaf bast fibres respectively (Figure 2). However, the MHT for all samples ranged from 10–20 hours which was longer than that of the control (8 hours). Based on MHT, effects of accelerators on hydration process were generally grouped into (1) two groups (one at very short time which is < 4 hours while the other at long time > 18 hours) for the crude fibres (Figure 3) (2) three groups for decorticated fibres (Figure 4) and (3) two groups for water-retted fibres (Figure 5). However, the lines look identical in their behaviour for decorticated and water-retted fibres.

The addition of 4 and 6% CaCl₂ as well as 4% AlCl₃ had negative effects on hydration of crude, decorticated and water-retted fibres as they reduced the hydration time to < 4 hours. The same negative effect was also observed with addition of 2% CaCl₂ and 6% AlCl₃ on decorticated and water-retted fibres. However,
Na$_2$SO$_4$ showed negative results with crude fibres. It showed positive effect on decorticated and water-retted fibres. The 2 and 6% for both CaO and AlCl$_3$ greatly increased the hydration time (>20 hours) of crude fibres which was not favoured. Meanwhile 4 and 6% CaO reduced the time for decorticated and water-retted fibres respectively. The best results were observed at 2% CaCl$_2$ and 4% CaO with crude fibres as well as 2% CaO with decorticated and water-retted fibres.

Effects of extraction method and particle size on hydration properties

The MHT of the Portland cement was reduced from 82.7 to 56.3 °C when crude bast fibres were integrated into the system and the hydration time to MHT was delayed to 16 hours (Table 3). This indicates that the presence of crude bast fibres in Portland cement has some retardation effect on the hydration properties of cement. In the presence of the decorticated and water-retted fibres respectively, the MHT was reduced to 58.7 and 60.5 °C and the hydration time to MHT delayed to 10.7 hours for both (Table 3). This result indicated that decorticated and water-retted treatments fulfilled the requirement to reduce the hydration temperatures of Portland cement without significantly affecting its hydration time to reach MHT.

On the other hand, at particle sizes 0.5 and 0.8 mm, the MHT was reduced to 53.6 and 56.3 °C respectively (Table 4). The time to MHT was also delayed to more than 12 hours which might not be favourable for fibre–cement composite manufacture. In the presence of particle size 4 mm, the MHT was reduced to 62.6 °C within 9 hours (Table 4).

CONCLUSIONS

The incorporation of kenaf fibre inhibited the hydration process of the cement. The most affected was when using NaOH-retted followed by benzoate-retted fibres. Both were classified as high inhibition. All untreated (crude), water-retted and decorticated fibres had mild effect and could be classified as intermediate inhibition. Thus, both NaOH and sodium benzoate were not suitable for fibre extraction in the manufacture of cement-bonded kenaf board. The use of CaCl$_2$, AlCl$_3$ and CaO were effective in reducing the hydration time and 2% concentration was found to be sufficient to reach MHT within the time period. Fibres of sizes 0.5 and 0.8 mm required the addition of accelerators to reach their maximum cement setting, while size 4 mm did not. There was no significant difference in hydration properties between decorticated and water-retted fibres.
REFERENCES


Table 3  Effects of extraction methods on the maximum hydration temperature of kenaf bast fibre cement mixture

<table>
<thead>
<tr>
<th>Extraction method</th>
<th>MHT (°C)</th>
<th>Time (hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crude BF</td>
<td>56.3 b</td>
<td>16.0 a</td>
</tr>
<tr>
<td>Decorticated BF</td>
<td>58.7 a</td>
<td>10.7 b</td>
</tr>
<tr>
<td>Water-retted BF</td>
<td>60.5 a</td>
<td>10.7 b</td>
</tr>
</tbody>
</table>

BF = bast fibres; MHT = maximum hydration temperature; means followed by the same letter in the same column are not significantly different at p ≤ 0.05 according to least significant difference test

Table 4  Effects of particle size on the maximum hydration temperature of kenaf bast fibre cement mixture

<table>
<thead>
<tr>
<th>Particle size (mm)</th>
<th>MHT (°C)</th>
<th>Time (hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5</td>
<td>53.6 b</td>
<td>17 a</td>
</tr>
<tr>
<td>0.8</td>
<td>56.3 b</td>
<td>14 b</td>
</tr>
<tr>
<td>4.0</td>
<td>62.6 a</td>
<td>9 c</td>
</tr>
</tbody>
</table>

MHT = maximum hydration temperature; means followed by the same letter in the same column are not significantly different at p ≤ 0.05 according to least significant difference test