PRE-TREATMENT EFFECTS ON HYDRATION BEHAVIOUR OF CEMENT BONDED BOARDS MADE FROM EREMOSPATHA MACROCARPA AND LACCOSPERMA SECUNDIFLORUM CANES

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ABSTRACT

The effects of pre-treatments on the setting time (t_{max}), maximum hydration temperature (T_{max}) and time ratio (t_R) of two rattan species (*Eremospatha macrocarpa* and *Laccosperma secundiflorum*) particles mixed with Portland cement were investigated. The untreated *E. macrocarpa* and *L. secundiflorum* had t_{max}, T_{max} and t_R of 12.4 and 11.9 h, 59.1 and 58.3°C, 1.1 and 1.1, respectively while cold water treated *E. macrocarpa* and *L. secundiflorum* had t_{max}, T_{max} and t_R of 10.7 and 10.0 h, 63.3 and 65.1°C, 1.0 and 0.9, respectively. $CaCl_2$ pre-treated *E. macrocarpa* and *L. secundiflorum* had t_{max}, T_{max} and t_R were 5.2 and 5.6 h, 83.7 and 78.6°C, 0.5 and 0.5, respectively. Therefore, $CaCl_2$ pre-treatment significantly served as an accelerator to improve cement hydration parameters better than cold water. Findings showed that *L. secundiflorum* inhibited cement setting more than *E. macrocarpa* due to its higher sugar content.

KEYWORDS:

Rattan canes; pre-treatment; cement bonded boards; compatibility indices

INTRODUCTION

Lignocellulosic materials such as wood particles, maize husks, groundnut husks, rice stalks etc. have been incorporated in cement mixes over the years to produce environmentally friendly building components used for ceiling, flooring, sidings, internal/external wall cladding (Badejo, 1989; 1992; Olorunnisola, 2006). Many of these lignocellulosic materials contain sugars, polyphenolics, proteins and extractives which often impair cement bonds. Therefore, pre-treatment measures like prolonged storage, fungi infestation, carbon dioxide treatment, aqueous extraction and chemical additive treatment are incorporated in the production line to remove or reduce cement setting inhibitors (Badejo, 1989; Olorunnisola *et al*., 2005, Olorunnisola, 2008). Chemical and aqueous pre-treatments are the two common measures adopted in the manufacture of cement bonded composites (CBCs) in Nigeria (Badejo 1989; Olorunnisola 2005). While aqueous extraction (cold or hot water) removes or partially removes soluble inhibitory components that may hinder formation of strong crystalline bond, chemical pre-treatment accelerates the hydration of cement, reduces the setting time, increases the maximum hydration temperature and improves the bonding between particles of the lignocellulosic materials and cement (Sutigno, 2002; Almeida, *et al*., 2002, Fabiyi, 2004). However, Alberto *et al*., (2000) noted that different lignocellulosic materials require different pre-treatments depending on their chemical constituents. Whereas inhibitory materials were found amenable to hot water extraction, the less inhibitory substances were amenable to cold water extraction (Alberto *et al*., 2000).

A major limitation in the production of CBCs is that many of the lignocellulosic materials used as furnish are produced in small quantities in scattered locations. Hence collection and haulage are often difficult and expensive (Olorunnisola, 2006). Therefore, lignocellulosic materials such as rattan canes that are accessible and abundant in forests of western Nigeria (Dahunsi, 2000); are now being considered as candidate furnish for CBCs manufacture (Olorunnisola and Adefisan, 2002). Rattans however contain sugars and extractives (Dahunsi, 2000; Olorunnisola and Adefisan, 2008) which are likely to impair cement interaction; thus pre-treatment with cold water and/or chemical accelerators such as calcium chloride ($CaCl_2$) should be incorporated to enhance formation of strong crystalline bond. Therefore cold water extraction and $CaCl_2$ pre-treatment effects on the compatibility of two rattan species (*Eremospatha macrocarpa* and *Laccosperma secundiflorum*) with cement are examined.

Infrared spectroscopy was used to monitor the chemical structure produced during the chemical reactions that occur as a result of the interactions among the mixes of cement, water and untreated or pre-treated lignocellulosic material. The versatility of IR spectroscopy is due to the fact that it requires minimum sample preparation, it is not tedious and not time consuming. However, its use for monitoring the changes that occur when cement, lignocellulosic fibres and chemical additives are mixed together is limited in literature. This study therefore aimed at investigating the effects of cold water extraction and incorporation of $CaCl_2$ on the hydration behaviour as well as the infrared spectroscopy of *E. macrocarpa* and *L. secundiflorum* mixed with Portland cement.

MATERIALS AND METHODS

Matured stems of *E. macrocarpa* and *L. secundiflorum* canes were harvested from Gambari Forest Reserve located between longitude 50° 44' E and latitude 7° 14' N in Ibadan, Oyo state, Nigeria. The canes were converted into billets of about 6 cm and hammer milled. The milled particles were sieved using a set of 1.18 mm, 0.85 mm and 0.60 mm sieves. Particles that passed through the 0.85 mm sieve and were retained in the 0.60 mm sieve were collected and dried to 10% moisture content. These particles were divided into three sets with the first set mixed with cement but no additive incorporation (henceforth called untreated) while the second set pre-treated with $CaCl_2$ before mixed with cement. The third set was soaked in distilled water for 30 minutes, drained and dried to 10% moisture content.

HYDRATION TEST

For the hydration tests, 15 g of the rattan particles, 200 g of Portland cement (purchased at a local hardware store, Moscow, Idaho state, USA) and 93 ml of distilled water were mixed in a polyethylene bag to form homogeneous slurry following the method developed by Adefisan and Olorunnisola (2007). The neat cement was mixed with 90 mL of distilled water while $CaCl_2$ used as chemical accelerator was added by dissolving in the distilled water at two concentration levels: 0% (untreated) and 3% (by weight of cement). However, untreated and 30 min. cold water extracted samples were mixed with the neat cement and 90 mL of distilled water only prior hydration characterisation. Hydration characterisation was performed in a set of well insulated thermos flasks. The temperature rise was monitored for 24 h using thermocouples (J-type) connected to an 8-channel datalogger (USB TC-08, Pico Technology). Three sample specimens of each mixture were prepared. The compatibility of *E. macrocarpa* and *L. secundiflorum* with cement was assessed using the compatibility indices (Table 1).

CHEMICAL CHARACTERISATION

Specimens for the chemical analysis were obtained from each of the untreated and pre-treated rattan/cement mixes and ground into fine powder using pestle and mortar. Fourier transform infrared (FTIR) spectra were measured directly from the powder (1% w/w basis) thoroughly dispersed in KBr (99% w/w basis). Spectra were recorded using a Thermo Scientific Nicolet 8700 spectrometer equipped with a DTGS detector. Each spectrum was taken as an average of 64 scans at a resolution of 4 cm^{-1}.

RESULTS AND DISCUSSION

Setting time of rattan-cement composites

The results of the hydration tests are presented in Table 2. The setting times of the *E. macrocarpa* and *L. secundiflorum* cement mixes without pre-treatment were 12.4 and 11.9 h, respectively. Based on the classification of Hofstrand *et al.* (1984), the untreated rattan particles were very suitable for CBB production. $CaCl_2$ and 30 min cold water extraction pre-treatments significantly reduced setting times of the rattan composites when compared with the untreated from 12.4 to 5.2 h and 12.4 to 10.7 h for the *E. macrocarpa*, respectively while for *L. secundiflorum* cement mixes, it reduced from 11.9 to 5.6 h and 11.9 to 10.0 h, respectively. Olorunnisola (2008) reported the setting time of 10.3 h for 30 min cold water extracted *L. secundiflorum* (sieved) mixed with cement. This is agreement with 10.0 h obtained in this study for same condition. Composites treated with $CaCl_2$ had significantly lower t_{max} than those pre-treated with cold water. This implies that $CaCl_2$ incorporation was more effective than cold water extraction in minimizing the effects of cement setting inhibitors in the rattan-cement mixes. The preponderance of parenchyma cells (containing starches and tannins) in *E. macrocarpa* canes compared to *L. secundiflorum* (Dahunsi, 2000) may account for the difference in compatibility. Although the choice of rattan species did not significantly affect the t_{max}, pre-treatment however significantly affected the setting times of the rattan-cement mixes (Table 2). What this suggests is that the rattan particles should be pre-treated prior to CBC manufacture.

Table 1 - Cement compatibility assessment schemes

S / No.	Parameters	Classification Index	Reference
1	Setting time (time to reach maximum temp., t_{max})	Suitable (<15hr) Unsuitable (> 20hr)	Hofstrand *et al.* (1984)
2	Maximum hydration temp.	Suitable (T_{max} > 60°C) Intermediately Suitable (T_{max} = 50 - 60°C) Unsuitable (T_{max} < 50°C)	Sandermann and Kohler, (1964)
3	Time ratio (t_R): ratio of setting time of wood/cement mix to neat cement i.e. $t_R = t_{WC}/t_{NC}$	$1 \leq t_R \geq 1.5$ (Suitable) $1.5 < t_R \geq 2.0$ (Acceptable) $t_R > 2.0$ (Inhibitory)	Olorunnisola (2008)

MAXIMUM HYDRATION TEMPERATURE OF THE RATTAN-CEMENT COMPOSITES

The maximum hydration temperatures (T_{max}) attained by the rattan/cement mixes were 59.1 and 58.3°C for untreated *E. macrocarpa*/cement mix and *L. secundiflorum*/cement mix, respectively (Table 2). Based on the Sandermann and Kohler (1964) index, the untreated rattans could be classified as 'intermediately suitable' for the production of cement bonded board. Pre-treatment enhanced the T_{max} of the rattan composites. The T_{max} for $CaCl_2$ and 30 min. cold water extraction pre-treated *E. macrocarpa*/cement mix were 83.7°C and 63.7°C, respectively. In addition, T_{max} for $CaCl_2$ and 30 min. cold water extraction pre-treated *L. secundiflorum*/cement mix were 78.6°C and 65.1°C, respectively (Table 2). Composites treated with $CaCl_2$ had significantly higher T_{max} than those pre-treated with cold water. This indicates that $CaCl_2$ was more effective than cold water in reducing cement inhibitors present in the rattan particles. Additionally, it suggests that cold water treatment only removes limited amounts of the inhibitors from the canes. Most importantly, the mechanism of $CaCl_2$ actually differs from that of water; it results in the formation of hydrated cement materials containing chlorides (Taylor, 1997).

Generally, composites made from *E. macrocarpa* particles recorded higher T_{max} than those from *L. secundiflorum*. This may indicate that *L. secundiflorum* inhibited cement setting more than *E. macrocarpa* particles. Dahunsi (2000) observed that *L. secundiflorum* canes had the highest sugar content (over 70%) out of the three rattan species available in western Nigeria. The higher sugar contents in the *L. secundiflorum* canes may therefore contribute to its low T_{max}. In addition, the rattan species (*E. macrocarpa* and *L. secundiflorum*) did not significantly affect the T_{max} of the cement composites but pre-treatments had significant effect on the maximum hydration temperature (Table 3).

Table 2 - Hydration parameters (± standard deviation) of *Laccosperma secundiflorum* and *Eremospatha macrocarpa* cement mixes

Pre-treatment	Setting time maximum (t_{max}, h)	Maximum hydration (temperature T_{max}, °C)	Time ratio index (t_R)
		L. secundiflorum	
None	11.9±0.2	58.3±2.4	1.1±0.04
3% $CaCl_2$	5.6±0.4	78.6±0.3	0.5±0.04
Cold Water	10.0±0.2	65.1±3.0	0.9±0.0
		E. macrocarpa	
None	12.4±0.5	59.1±1.8	1.1±0.06
3% $CaCl_2$	5.2±0.2	83.7±5.7	0.5±0.04
Cold Water	10.7±0.4	63.3±0.9	1.0±0.03
		Rattan species	
E. macrocarpa	9.4±3.3	68.7±11.8	0.9±0.31
L. secundiflorum	9.2±2.8	67.3±9.2	0.8±0.25

TIME RATIO INDICES OF THE RATTAN-CEMENT COMPOSITES

The time ratio indices (t_R) of the rattan/cement mixes are presented in Table 2. The time ratio indices for both untreated rattan species cement mixes were the same (1.1). Similarly, the t_R for $CaCl_2$ pre-treated *E. macrocarpa*/cement mix and *L. secundiflorum* and cement mix were the same (0.5) while it was 1.0 for cold water pre-treated *E. macrocarpa*/cement mix and 0.9 for *L. secundiflorum*/cement mix. Based on the classification of Olorunnisola (2008), the untreated rattans are suitable for CBB production. Pre-treatment significantly improved the t_R of the rattan/cement mixes. The greater improvement again was observed for composites pre-treated with $CaCl_2$ (Table 2). The implication is that $CaCl_2$ improved the compatibility of the rattan/cement mixes more than cold water treatment. Pre-treatment significantly affected the time ratio indices of the rattan-cement mixes but rattan species do not.

CHEMICAL CHARACTERISATION

Figures 1 and 2 show the FTIR spectra of *E. macrocarpa* and *L. secundiflorum* (untreated and pre-treated with and without cement). Infrared bands observed in the various treatments with their peak assignments and structural polymers for *E. macrocarpa* and *L. secundiflorum* are presented in Tables 3 and 4, respectively. There are similarities and a few dissimilarities in the positions and intensities of the infrared spectra of the two rattan species investigated. Peaks numbered 2, 6, 8, 10, 14, 15, 17 and 20 have the same band frequencies for both *E. macrocarpa* and *L. secundiflorum* (Tables 3 and 4). However, the band frequencies for peaks numbered 3, 4, 11, 12, 13, 17, 18, 21, 23, 24 and 25 for *E. macrocarpa* differed from that of *L. secundiflorum*. Majorly, the carbonyl band for xylan differed by 3 cm^{-1} with *E. macrocarpa* occurring at 1737 cm^{-1} and *L. secundiflorum* at 1734 cm^{-1}.

The lignin assigned peak for *E. macrocarpa* and *L. secundiflorum* occurred around 1506/1608 cm^{-1}. However, some peaks occurred at higher frequency for *E. macrocarpa* than *L. secundiflorum*. Examples of these are peaks at 698, 1113, 1162, 1463 and 3412 cm^{-1} for *E. macrocarpa* compared with *L. secundiflorum* at 695, 1110, 1161, 1457 and 3407 cm^{-1}. The band at 3540 – 3000 cm^{-1} which was assigned to O-H groups in the rattans emerged due to the combination of cellulose, hemicelluloses and lignin (Fax 1992). This band is broader with higher intensity for *L. secundiflorum* than *E. macrocarpa*. Likewise the intensities for peaks numbers 10, 11, 12, 13, 15, 17, 19 and 20 are higher for *L. secundiflorum* than *E. macrocarpa*. Generally, there is no difference in the chemical functional groups that were present in both rattan species. Therefore, the peak intensity, band width and band frequency from the spectra can only be used to differentiate the two rattan species under investigation.

Chemical changes that occurred due to the blending of untreated, 30 min. cold water extracted and $CaCl_2$ pre-treated *E. macrocarpa* or *L. secundiflorum* mixed with cement after the hydration testing was terminated are illustrated in Figures 1 and 2. The addition of Portland cement caused the appearance of some peaks in the spectra (numbers 1, 5, 7, 22 and 26). The formation of the band at 3643 cm^{-1} in the untreated, 30 min. cold water extracted and $CaCl_2$ treated rattan/cement mixes remained unchanged; indicating that its emergence is due to the chemical constituent of cement. It was due to the metal-bonded hydroxide (Mollah, *et al.* 1992). This indicates the OH band from $Ca(OH)_2$. The band at 3407/3414 cm^{-1}, which was assigned to O-H groups in lignocellulosic materials, was affected by the pre-treatment and incorporation of cement to the rattan species. The blending of the 30 min. cold water extracted and $CaCl_2$ pre-treated rattan particles with cement caused the band to be become narrower than in the ordinary rattan particles. In addition, peaks 23 and 24 at 2853 and 2926-2918 cm^{-1}, respectively drastically decreased due to the addition of cement. This implies that the cement suppressed the methylene and methyl stretching frequencies that occurred in the rattan species.

The lignin assigned peaks (1506/1507 and 1608/1609 cm^{-1}) did not appear in the rattan/cement mix masked due to the presence of broader band with peaks at 1426 and 1457 cm^{-1}. In addition, the peak at 1047 cm^{-1} disappeared when cement was mixed with rattan particles. There were many bands that disappeared due to the addition of cement namely: peaks numbers 21, 15, 14, 13 and 6 which are xylan in hemicelluloses, cellulose, syringyl, guaiacyl and arabinogalactan, respectively (Tables 3 and 4). Addition of $CaCl_2$ to *L. secundiflorum* particles helped expose cellulose significantly to advance participation probably by enlarging its surface area for interpenetration networking with cement than *E. macrocarpa*. Chemical reactivity of this peak (number 17) is rattan species dependent. The region between 1750 cm^{-1} and 750 cm^{-1} was drastically affected (reduced the peaks' intensities) by the incorporation of $CaCl_2$ while 30 min. cold water extraction had little effect on this region for *E. macrocarpa*. However, 30 min. cold water extraction drastically reduced the peaks' intensities in the region between 1510 and 1300 cm^{-1} for *L. secundiflorum*.

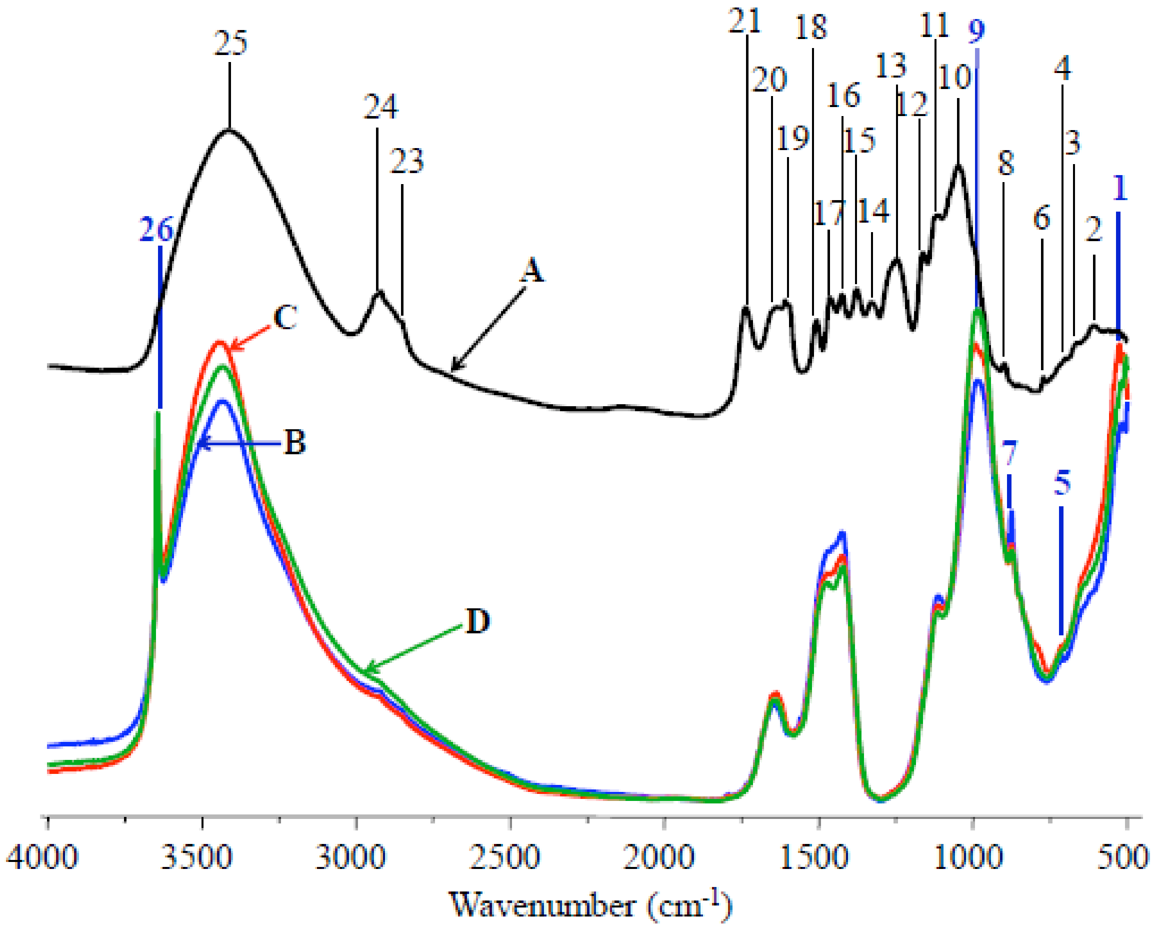

Figure 1 - FTIR spectra of (A) *Eremospatha macrocarpa*, (B) *E. macrocarpa*/cement mix, (C) *E. macrocarpa*/cement/$CaCl_2$, and (D) *E. macrocarpa*/cement/30 min cold water extraction. Each spectrum is an average of spectra from two specimens. Peak position and assignment as well as structural polymer for each peak are presented in Table 3.

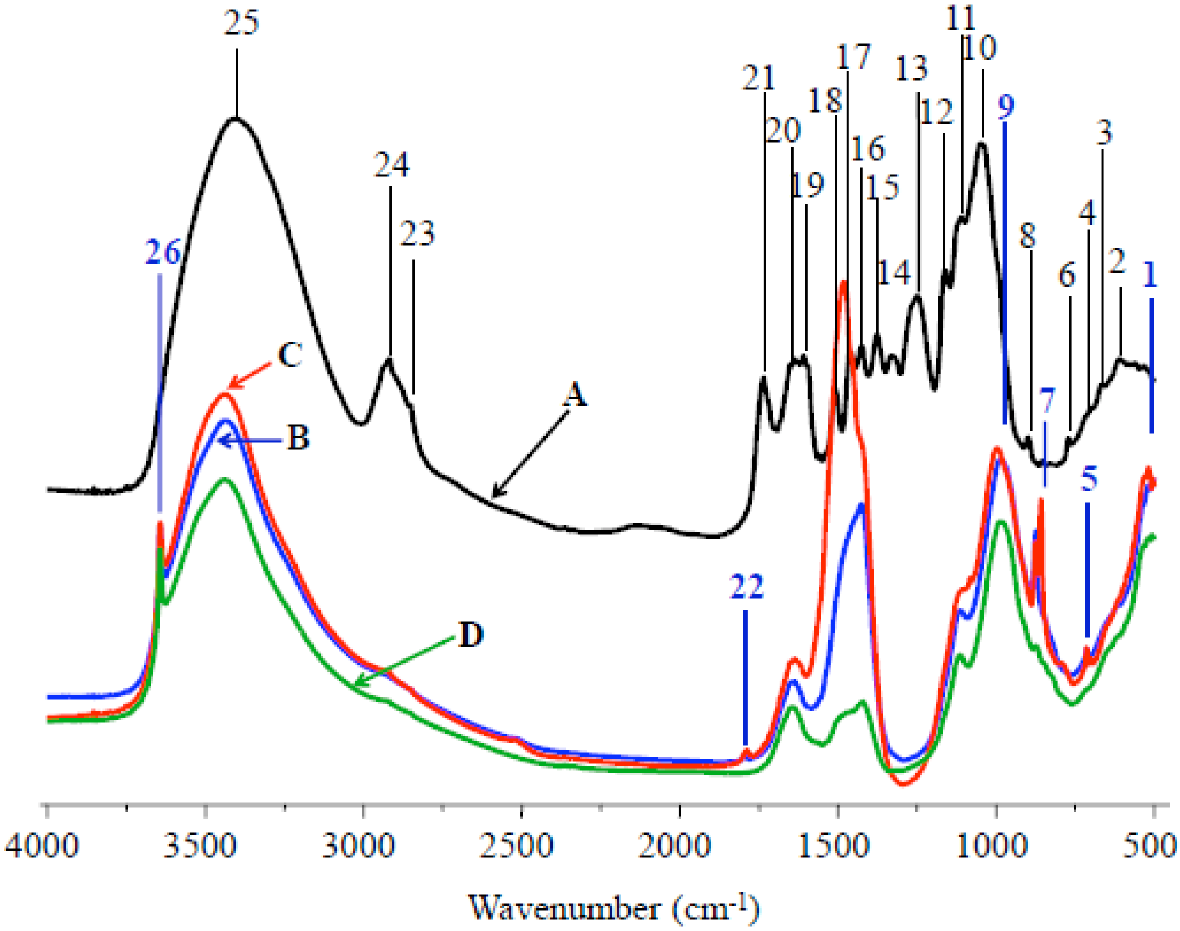

Figure 2 - FTIR spectra of (A) *Laccosperma secundiflorum*, (B) *L. secundiflorum*/cement mix, (C) *L. secundiflorum*/cement/$CaCl_2$, and (D) *L. secundiflorum*/cement/30 min cold water extraction. Each spectrum is an average of spectra from two specimens. Peak position and assignment as well as structural polymer for each peak are presented in Table 4.

CONCLUSIONS

The effects of cold water extraction and $CaCl_2$ on the setting time (t_{max}), maximum hydration temperature (T_{max}), time ratio (t_R) and surface chemistry of *Eremospatha macrocarpa* and *Laccosperma secundiflorum* particles mixed with Portland cement were investigated. Hydration behaviour showed that *E. macrocarpa* and *L. secundiflorum* canes are suitable for CBB production based on their setting time and time ratio index. However, maximum hydration temperature only ranked *E. macrocarpa* and *L. secundiflorum* as "intermediately suitable." Cold water extraction (for 30 min) and $CaCl_2$ improved the hydration behaviour of the rattan cement mixes. *L. secundiflorum* inhibited cement setting more than *E. macrocarpa*. The surface chemistry studied using infrared spectroscopy showed that addition of cement to rattan fibre caused the suppression of methylene and methyl stretching frequencies that occurred in the rattan species. Addition of $CaCl_2$ to *L. secundiflorum* particles helped expose cellulose to advance participation probably by enlarging its surface area for interpenetration networking with cement than *E. macrocarpa*.

Table 3 - Summary of infrared bands observed in *Eremospatha macrocarpa* and the untreated and pre-treated *E. macrocarpa* cement mixes.

Peak No.	Wavenumber (cm^{-1})				Peak assignment	Structural polymer
	Erem	Erem/Cement mix	30 min. cold H_2O	Mix/ $CaCl_2$		
1	-	519	520	525	v_4 of Si-O bending	Cement
2	609	617	-	617	Unknown	
3	662	-	662	650	alkene C-H bending	
4	698	-	-	-	Rocking vibration CH_2	Cellulose
5	-	713	-	713	v_4 of CO_3	Cement
6	771	-	-	-		Arabinogalactan
7	-	875	873	874	v_2 of CO_3	Cement
8	898	-	-	-	C1-H deformation of glucose ring	Cellulose
9	-	986	988	991		
10	1047	-	-	-	C–O stretch	Cellulose and hemicellulose
11	1113	1112	1115	1113	aromatic skeletal and C–O stretch	Polysaccharides and Lignin
12	1162	-	-	-	C–O–C vibration	Cellulose and hemicellulose
13	1247	-	-	-	C–O of guaiacyl ring	Lignin
14	1330	-	-	-	C_1–O vibration	Syringyl
15	1377	-	-	-	C–H deformation	Cellulose and hemicellulose
16	1425	1425	1421	1425	C-H in-plane deformation with aromatic ring stretching	Lignin
17	1463	1472	1472	1472	CH deformation, asymmetry in CH_3 and CH_2	Cellulose
18	1506	-	-	-	aromatic skeletal vibration (C=C), guaiacyl > 5	Lignin
19	1608	-	-	-		Lignin
20	1635	1646	1645	1645		Extractives
21	1737	-	-	-	Conjugated C=O	Xylan in hemicelluloses
22	-	-	-	-		
23	2853	2853	2853	2853		
24	2920	2923	2923	2923	C-H stretching	
25	3412	3433	3438	3445	OH stretching	Rattan polymers
26	-	3644	3643	3643	OH from $Ca(OH)_2$	Cement

Table 4 - Summary of infrared bands observed in *Laccosperma secundiflorum* and the untreated and pre-treated *L. secundiflorum* cement mixes.

Peak No.	Position (cm^{-1})				Peak assignment	Structural polymer
	Laco	Laco/ Cement mix	30 min. cold H_2O	Mix/ $CaCl_2$		
1	-	518	521	518	v_4 of Si-O bending	Cement
2	609	617	617	617	Unknown	
3	664	665	-	-	alkene C-H bending	
4	695	-	-	-	Rocking vibration CH_2	Cellulose
5	-	713	-	713	v_4 of CO_3	Cement
6	771	-	-	-		Arabinogalactan
7	-	874	873	875	v_2 of CO_3	Cement
8	898	-	-	-	C1-H deformation of glucose ring	Cellulose
9	-	984	984	996		
10	1047	-	-	1083	C–O stretch	Cellulose and hemicellulose
11	1110	1113	1114	1108	aromatic skeletal and C–O stretch	Polysaccharides and Lignin
12	1161	-	-	-	C–O–C vibration	Cellulose and hemicellulose
13	1248	-	-	-	C–O of guaiacyl ring	Lignin
14	1330	-	-	-	C_1–O vibration	Syringyl
15	1377	-	-	-	C–H deformation	Cellulose and hemicellulose
16	1425	1426	1420	1420	C-H in-plane deformation with aromatic ring stretching	Lignin
17	1457	1457	1466	1481	CH deformation, asymmetry in CH_3 and CH_2	Cellulose
18	1507	-	-	-	aromatic skeletal vibration (C=C), guaiacyl > 5	Lignin
19	1609	-	-	-		Lignin
20	1635	1636	1641	1636		Extractives
21	1734	-	-	-	Conjugated C=O	Xylan in hemicelluloses
22	-	1793	-	1789	$CaCO_3$	Cement
23	2852	2852	2852	2852		
24	2919	2919	2919	2919	C-H stretching	
25	3407	3438	3433	3444	OH stretching	Rattan polymers
26	-	3643	3643	3643	OH from $Ca(OH)_2$	Cement

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