EFFECTS OF SELECTED PRE-TREATMENTS ON THE SETTING OF CEMENT COMPOSITE FROM TWO RATTAN SPECIES

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Abstract

The effects of aqueous extraction and removal of silified epidermis from rattan canes on the maximum hydration temperature (T_{max}) and setting time (t_{max}) of two rattan cane species, Calamus deerratus and Lacosperma secundiflorum, mixed with Portland cement were investigated. T_{max} ranged from 37.0 to 58.6^{0}C and from 43.1 to 56.8^{0}C for the C. deerratus and L. secundiflorum composites respectively while t_{max} was from 3.7 to 26.5 hrs and 7.4 - 25.7 hrs. Aqueous extraction increased the T_{max} and reduced the t_{max} of the rattan-cement mixes while removal of epidermis generally caused reduction in both T_{max} and t_{max}. The two rattan species seemed more amenable to cold than hot water extraction. Although cement inhibition of the Calamus species was greater than that of Lacosperma species, aqueous extraction improved the t_{max} of the Calamus-cement composite more than Lacosperma-cement composite. Epidermis removal, species, aqueous pre-treatment and their interactions had significant effect on the setting time of the rattan-cement mixes.

Keywords: rattan, cement composite, hydration temperature, setting time

INTRODUCTION

Rattans are numbered among the important commercial non-timber forest products employed in furniture Manufacture in many parts of the tropics. A major problem associated with this mode of utilisation, however, is the level of material wastage involved. As noted by Olorunnisola (2005), over 30% of rattan stems harvested at any particular time for furniture manufacture is wasted. Besides, only about 20% of the over 600 known rattan species produce the most sought-after fine quality canes and are, therefore, of commercial value. The remaining species with low quality canes are not utilised due to inflexibility, tendency to breakage, poor mechanical properties (Dransfield and Manokaran 1993).

One of the alternative potential uses of rattans yet to be fully explored is the production of environmentally friendly and fire-resistant cement-based composites for both structural and non-structural applications in building construction such as production of lightweight concrete blocks, ceiling and floor panels, and roofing sheets. A major advantage of the use of rattan in composite panel production is the possibility of complete material utilisation since there would be no need for pre-sorting and discarding of canes as is done in rattan furniture manufacture (Olorunnisola 2005). The use of rattan canes for Cement-Bonded Composites (CBC) production has been reported by Olorunnisola and Adefisan (2002); Olorunnisola *et al*, (2005) and Olorunnisola (2005).

Rattan canes like other woody materials contain elements which tend to inhibit the setting of cement. Inhibition of Portland cement setting by many lignocellulosics is a major limitation to the development of new Cement-Bonded Composites. The incompatibility is usually ascribed to the presence of sugars, extractives, etc. in such lignocellulosics that obstruct the formation of strong crystalline bonds with cement. Hence, physical, chemical and biological (fungi infestation) pre-treatment methods are adopted to minimise such inhibitions (Lee, 1984; Badejo, 1989; Shi *et al.*, 1999, Sutigno, 2002). Badejo (1989) and Olorunnisola (2005) reported that chemical and aqueous pre-treatment measures are two of the common methods adopted. However, chemical additives tend to be expensive. Therefore, aqueous pre-treatment measures may be preferable.

Aqueous pre-treatment involves removal or partial removal of soluble inhibitory wood components by extraction with cold or hot water. Sutigno (2002) noted that soaking lignocellulosic particles in cold water dissolves tannins, gums, sugars, and colouring matter, while hot water pre-treatment dissolves tannins, gums, sugars, colouring matter and starches. Alberto *et al* (2000) also observed that different lignocellulosics require different aqueous pre-treatments depending on their constituents. It was reported that cold water treatment improved the compatibility of two Mozambican wood species while other species required hot water pre-treatment.

Olorunnisola and Adefisan (2002) reported improved bond strength and dimensional stability between cement and strands obtained from rattan cane furniture wastes pre-treated with hot water and 3% calcium chloride ($CaCl_2$), while Olorunnisola (2005) and Olorunnisola *et al* (2005) found enhanced bond strength and dimensional stability between particles of *Lacosperma secundiflorum* canes treated with 3% (by weight of cement) of $CaCl_2$. However, the effect of different aqueous pre-treatment and epidermis removal on the cement hydration of different rattan species is yet to determined.

Different compatibility indices have been used in the classification of lignocellulosics for CBC manufacture (Hachmi, *et al.*, 1990; Karade *et al.*, 2003; Pereira *et al.*, 2006 etc.). Most of these compatibility indices only provide a quantitative means of assessing the behaviour of wood-cement mixes. Also, they lack precision and involve cumbersome computations which ought to be simplified (Pereira *et al.*, 2006; Olorunnisola, 2008). However, Jorge *et al.*, (2004) noted that the maximum hydration temperature (T_{max}) and the time to attain it (t_{max}) are the two predictors of the general inhibitory properties of lignocellulosics when used for CBC production.

The objectives of this work were to investigate the effects of aqueous extraction and removal of silified epidermis on the maximum hydration temperature (T_{max}) and setting time (t_{max}) (i.e. time to attain the maximum hydration temperature) of cement composites from two rattan cane species, viz: *Calamus deerratus* and *Lacosperma secundiflorum*.

MATERIALS AND METHODS

Matured stems of *Calamus deerratus* and *Lacosperma secundiflorum* rattan canes were harvested from Gambari forest reserve Ibadan, Oyo state, Nigeria. These were identified through comparison with stocks kept in the herbarium of the Department of Botany and Microbiology, University of Ibadan. The harvested canes were separated into two parts. One part was manually peeled to remove the silified epidermis, while the other part was left intact. Both the peeled and the unpeeled canes were converted into billets of about 6 cm and hammer milled. The milled particles were collected and sieved using a set of 1.18 mm, 0.85 mm and 0.60 mm sieves. Particles retained in the 0.60 mm sieve were collected.

Particles from both peeled and unpeeled canes collected were separated into two portions i.e. those to be used 'as is' and those to be subjected to pre-treatment. Pre-treatments involved soaking different portions of the particles in de-ionised cold (25^{0}C) and hot water (80^{0}C) for 30 minutes respectively, draining and re-washing with de-ionised water to remove soluble extractives, and air drying for 14 days to an average moisture content of 10%. Phytochemical screening of the aqueous extracts of the rattan canes was accordance with procedures adapted from Harborne (1991) and Evans (2006).

For the hydration tests, 15g of the rattan particles, 200g of ordinary Portland cement and 93ml of de-ionized water were mixed in a polyethylene bag to form homogeneous slurry. For the control experiment, 200g of neat cement was mixed with 90ml de-ionized water as in Hachmi *et al*. (1990). The tests were performed in a set of well insulated thermos flasks. Temperature rise was monitored for 27 hours using a T-type thermocouple. Three replicates of each mixture were prepared. The compatibility was assessed using the compatibility indices shown in Table 1.

Table 1: Compatibility Indices

S / No.	Parameter	Classification Index	Reference
1	Time to Maximum Temperature	Suitable (<15hr) Unsuitable (> 20hr)	Hofstrand *et al.* 1984
2	Maximum Hydration Temperature	Suitable (T_{max} > 60^{0}C) Intermediately Suitable (T_{max} = 50 - 60^{0}C) Unsuitable (T_{max} < 50^{0}C)	Sandermann and Kohler, 1964

RESULTS AND DISCUSSIONS

Phytochemical Properties of Extracts

The results of the screening carried out on the cold and hot water extracts of peeled and unpeeled samples of *Calamus deerratus* and *Lacosperma secundiflorum* are shown in Tables 2 and 3. Phytochemical analyses revealed saponins, tannins, terpenes, alkaloids and carbohydrates in the cold and hot water extracts of both *C. deerratus* and *L. secundiflorum*. Anthraquinones were absent. Tannins, terpenes, alkaloids and carbohydrates are extractives and sugars in lignocellulosics, the presence of which could inhibit setting in composites.

Cold and hot water extracts of unpeeled *L. secundiflorum* indicated stronger presence of alkaloids, terpenes and saponins than those of the peeled extracts. On the contrary, stronger presence of alkaloids, terpenes and saponins were observed in the cold water extracts of peeled *C. deerratus* than in the unpeeled samples. The hot water extracts of the peeled *Calamus* species indicated stronger presence of alkaloids and terpenes than the unpeeled samples.

Table 2: Phytochemical Screening of Aqueous Extracts of *C. deerratus*

Phytochemical Constituents	Hot Water Extracts		Cold Water Extracts	
	Peeled	Unpeeled	Peeled	Unpeeled
Alkaloids	Positive*	Positive	Positive*	Positive
Anthraquinoes	Negative	Negative	Negative	Negative
Tannins	Positive	Positive	Positive	Positive
Terpenes	Positive*	Positive	Positive*	Positive
Carbohydrates	Positive	Positive	Positive	Positive
Saponins	Positive	Positive	Positive*	Positive

* Strong Positive Presence

Table 3: Phytochemical Screening of Aqueous Extracts of *L.secundiflorum*

Phytochemical Constituents	Hot Water Extracts		Cold Water Extracts	
	Peeled	Unpeeled	Peeled	Unpeeled
Alkaloids	Positive	Positive*	Positive	Positive*
Anthraquinoes	Negative	Negative	Negative	Negative
Tannins	Positive	Positive	Positive	Positive
Terpenes	Positive	Positive*	Positive	Positive*
Carbohydrates	Positive	Positive	Positive	Positive
Saponins	Positive	Positive*	Positive	Positive*

* Strong Positive Presence

Saponins were however more pronounced in the hot water extracts of unpeeled than in the peeled samples. What the foregoing observations suggest is that more inhibitory compounds were found at the periphery of the stems of *L. secundiflorum* than in the core and, more in the core of the stems of the *Calamus* species than at the periphery. Apkofure (1992) had noted that extractive contents have patterns. They may decrease from periphery to the centre of the heartwood; increase from the pith to the heartwood, or increase from sapwood to heartwood. Cladius (2006) also reported that the concentration of extractives in mangrove tree (*Rhizophora racemosa*) decreased from bark to inner bark (bast) to the stem depending on the locality, time of the year, height and age of the tree.

Effect of Peeling on the Hydration Behaviour of Rattan-Cement Mixtures

As shown in Tables 4 and 5 maximum hydration temperature and the setting time of the *Calamus*-cement mixtures ranged from 37.0 to 58.6^{0}C and 3.7 to 26.5 hrs respectively, compared with those of the *Lacosperma*-cement mixtures (43.1 – 56.8^{0}C and 7.4 - 25.7 hrs respectively). Based on the classifications of Hofstrand *et al.*, 1984 and Sandermann and Kohler (1964), the two rattan species were unsuitable for CBC production without pre-treatment. However, visual examination showed that cement mixes containing particles of the *Lacosperma* species and unpeeled *Calamus* without aqueous pre-treatments formed hardened pastes while those of the peeled *Calamus* did not form cohesive bonds after 27 hours of experimentation. This observation is in line with that of Blankenhorn *et al.* (1994) who noted that some lignocellulosics contain sugars and extractives (phenolics) which will not only retard cement hydration but also degrade cement bonds.

The differences in the observed behaviour may be attributed to anatomical differences between the two rattan species. Lucas and Dahunsi (2004) had noted differences in the anatomy between *Calamus deerratus* and *Lacosperma secundiflorum* species grown in western Nigeria. While the *Calamus* species tested contained higher proportions of parenchyma cells (storage tissues) than sclerenchyma cells (strengthening tissues), the *Lacosperma* species had higher proportions of sclerenchyma than parenchyma cells. The higher proportions of parenchyma cells in the *Calamus* species may affect the hydration behaviour when mixed with cement.

Table 4: Effects of Aqueous Pre-treatments and Pre-processing on the Maximum Hydration Temperature of Rattan-cement Mixtures (n=3)

	T_{max} (^{0}C)			
	C. deerratus		*L. secundiflorum*	
Aqueous Pre-treatments	Unpeeled	Peeled	Unpeeled	Peeled
None	46.6^{B}(2.29)	37.0^{C}(0.59)	43.1^{B}(3.18)	45.1^{B}(2.31)
Cold Water	57.9^{A}(6.31)	57.1^{A}(0.21)	56.8^{A}(3.30)	58.0^{A}(0.85)
Hot Water	58.6^{A}(4.39)	57.7^{A}(0.78)	56.6^{A}(3.77)	55.5^{A}(3.85)

Means with the same letters are not statistically different, Standard deviation in parentheses

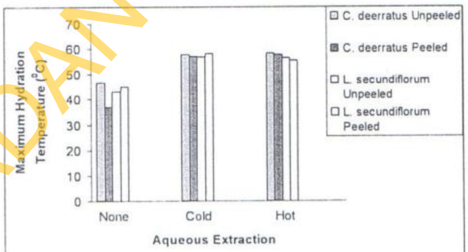

Fig. 1: Effects of Pre-processing and Aqueous Extraction on the Maximum Hydration Temperature of Rattan-Cement Mixtures

Table 5: Effects of Aqueous Pre-treatments and Pre-processing on the Setting Time of Rattan-cement Mixtures (n=3)

	t_{max} (Hrs)			
	C. deerratus		*L. secundiflorum*	
Aqueous Pre-treatments	Unpeeled	Peeled	Unpeeled	Peeled
None	26.5^{A}(0.89)	3.7^{F}(0)	25.7^{AB}(1.68)	24.7^{B}(0.58)
Cold Water	7.8^{CDE}(0.75)	8.5^{CD}(0)	8.6^{C}(0.17)	7.4^{ED}(0.17)
Hot Water	7.2^{E}(0.25)	8.6^{CD}(0.12)	8.3^{CDE}(0.15)	7.7^{CDE}(0.05)

Means with the same letters are not statistically different, Standard deviation in parentheses

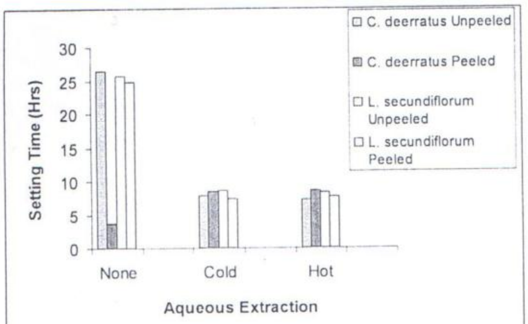

Fig. 2: Effects of Pre-processing and Aqueous Extraction on the Setting Time of Rattan-Cement Mixtures

Generally, the unpeeled *Calamus* had higher maximum hydration temperature and lower setting time than the peeled. On the contrary, the peeled *Lacosperma* generally had higher maximum temperature and lower setting time than the unpeeled (Tables 4 and 5) (Figs. 1 and 2). This may suggest that peeling has negative effect on the maximum hydration temperature and setting time of the *Calamus* species but it is positive in the case of *Lacosperma* species. Higher quantities of extractives (alkaloid, terpenes and saponins) in the unpeeled *Lacosperma* (Table 3) may have resulted in higher setting time than in the peeled *Lacosperma*-cement composite. This observation again suggests that cement inhibitors are concentrated at the periphery in the *Lacosperma* species and at the core of the *Calamus* species.

Analysis of variance revealed no significant differences ($P < 0.05$) between the maximum hydration temperature of unpeeled and peeled *Lacosperma* composites while the hydration temperature of the peeled *Calamus* composite was significantly different from the unpeeled. Also, significance differences existed between the setting time of unpeeled and peeled rattan composites (Tables 6 and 10). What this implies is that whereas pre-processing did not affect the maximum hydration temperature of the *Lacosperma*-cement mixes, it negatively affected the hydration temperature of the *Calamus* species and resulted in reduced setting time in the rattan-cement mixes.

Table 6: Duncan's Multiple Comparison of the Effects of Pre-processing on the Behaviour of Rattan-Cement Mixtures (n=9)

Pre-Processing	*C. deerratus* T_{max}	*L. secundiflorum* T_{max}	*C. deerratus* t_{max}	*L. secundiflorum* t_{max}
Unpeeled		2^{AB}(7.39)	13.8^{CD}(9.52)	14.2^{C}(8.66)
Peeled	50.6^{B}(10.19)	52.8^{AB}(6.36)	6.9^{E}(2.42)	13.3^{D}(8.56)

Means with the same letters are not statistically different, Standard deviation in parentheses

Effect of Aqueous Pre-treatments on the Hydration Temperature and Setting Time of Rattan-Cement Mixtures

Aqueous pre-treatments enerally resulted in improved hydration temperature and reduced setting time in the rattan-cement mixes. Composites from the *Calamus* species subjected to hot water extraction had higher hydration temperature and lower setting time than those from the *Calamus* particles soaked in cold water. However, cold water extraction resulted in higher hydration temperature and lower setting time in the *Lacosperma*-cement mixes than hot water (Table 7). This may indicate that while hot water extraction improved the compatibility of the *Calamus* species, particles of the *Lacosperma* species were more amenable to cold water extraction. Differences in the anatomical structures between the two rattan species again may be attributable for this variability.

Significant differences ($P<0.05$) existed in the hydration temperatures and setting times of the untreated rattans and those subjected to aqueous extraction. However, there was no significant difference between the maximum hydration temperatures and setting times of composites from the two rattan canes subjected to cold and hot water extraction (Tables 7, 8 and 10). What this suggests is that aqueous extraction improved the hydration temperatures and setting times of the rattan-cement composites. Although cold water extraction is usually less efficient than hot water in the production of cement composites, the two rattan species were more amenable to cold water extraction in line with the findings of Alberto *et al.* (2000). The results obtained from the phytochemical screening of the aqueous extracts (Tables 2 and 3) tallied with this observation that the extractives of the two rattan species were readily soluble in cold water.

Table 7: Duncan's Multiple Comparison of the Effect of Aqueous Pre-treatments on the Behaviour of Rattan-Cement Mixtures (n=6)

	T_{max}		t_{max}	
Aqueous Pre-treatments	*Calamus species*	*Lacosperma species*	*Calamus species*	*Lacosperma species*
None	41.8^{B}(5.43)	44.1^{B}(2.7)	15.1^{D}(12.5)	25.2^{C}(1.26)
Cold Water	57.5^{A}(4.02)	57.4^{A}(2.25)	8.1^{E}(0.62)	8.0^{E}(0.68)
Hot Water	58.2^{A}(2.86)	56.0^{A}(3.47)	7.9^{E}(0.75)	8.0^{E}(0.34)

Means with the same letters are not statistically different, Standard deviation in parentheses

Comparison of Composites from the two Rattan Species

Though no significant differences existed between the maximum hydration temperatures of the two rattan species, the setting times of the species were significantly different with the *Lacosperma* species having higher mean setting time (Table 10). This may indicate that aqueous extraction improved the setting time of the *Calamus* species more than the *Lacosperma* species. Also, the interactions of aqueous pre-treatment and pre-processing (removal of silified epidermis) had significant effects on the setting time of the *Calamus* species. This suggests that the setting time of the *Calamus* composite was influenced by the interactions of aqueous pre-treatment and pre-processing (Table 9).

Table 8: Analysis of Variance of the Effect of Aqueous Pre-treatment on Behaviour of the two Rattan–cement Mixtures (n=3)

		Mean Square Values							
		T_{max}				t_{max}			
		C.deerratus		*L.Secundiflorum*		*C.deerratus*		*L.secundiflorum*	
Source	Df	Unpeeled	Peeled	Unpeeled	Peeled	Unpeeled	Peeled	Unpeeled	Peeled
Aqueous Pre-treatments	2	136.51*	414.42*	183.18*	140.67*	361.21*	23.36*	297.04*	292.49*
Error	6	21.45	0.33	11.72	7.01	0.047	0.004	0.96	0.12

- Significant at 0.05 level of probability

Table 9: Analysis of Variance of the Effect of Aqueous Pre-treatments and Pre-processing on the behaviour of Rattan-Cement Mixtures (n=6)

		Mean Square Values			
		T_{max}		t_{max}	
Source	df	*Calamus*	*Lacosperma*	*Calamus*	*Lacosperma*
Aqueous Pre-treatments (T)	2	513.23*	320.19*	100.43*	589.39*
Pre-processing (P)	1	63.09*	2.14	214.94*	4.01*
T x P	2	37.70	3.67	284.15*	0.144
Error	12	10.86	9.37	0.238	0.541

- Significant at 0.05 level of probability

Table 10: Multiple comparison of the effects of Pre-processing, Species and Aqueous pre-treatments on the behaviour of Rattan-cement Mixtures

Factors	T_{max}	t_{max}
Pre-Processing (n=18)		
Unpeeled	53.3^{A}	14.0^{A}
Peeled	51.7^{A}	10.1^{B}
Aqueous Pre-treatments(n=12)		
None	43.0^{B}	20.1^{A}
Cold Water	57.4^{A}	8.1^{B}
Hot Water	57.1^{A}	8.0^{B}
Species (n=18)		
C. deerratus	52.5^{A}	10.4^{B}
L. secundiflorum	52.5^{A}	13.7^{A}

Means with the same letters are not statistically different

Table 11: Analysis of variance of the effects of Aqueous pre-treatments, Pre-processing and Species on the behaviour of Rattan-cement Mixtures

Source	df	Mean Square Value T_{max}	t_{max}
Aqueous Pre-treatments (T)	2	818.75*	588.09*
Pre-processing (P)	1	21.0	138.85*
Species (S)	1	0.007	101.67*
P x S	1	44.22*	80.10*
T x P	2	12.67	143.87*
T x S	2	14.67	101.73*
T x P x S	2	28.71	140.42*
Error	24	10.13	0.39

* Significant at 0.05 level of probability

CONCLUSIONS

The effects of aqueous extraction and removal of epidermis on the maximum temperature and setting time of cement composites from *Calamus deerratus* and *Lacosperma secundiflorum* rattan cane species were investigated. The following conclusions were drawn from the findings:

(i) The *Calamus* species inhibited cement setting more than the *Lacosperma* species due to differences in the anatomical structure.

(ii) The removal of silified epidermis negatively affected the setting time of the two rattan species and the hydration temperature of the *Calamus* species.

(iii) Aqueous extraction improved the maximum hydration temperatures and the setting times of the rattan-cement mixes compared to the untreated samples.

(iv) The rattan particles were more amenable to cold water extraction than hot water extraction with a practical implication of low capital investment during Cement bonded board production.

(v) Aqueous extraction positively enhanced the setting time of *Calamus* species more than the *Lacosperma* species

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