Hydration characteristics of cement-bonded composites made from rattan cane and coconut husk

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Abstract-This work examines the effect of CaCl2 on the hydration of rattan (Laccosperma secundiflorum) and coconut (Cocos nucifera) husk particles mixed with Portland cement. Hydration tests were conducted in sealed thermally insulated containers using an aggregate/cement/water ratio of 15 g : 200 g : 90.5 ml. CaCl₂ was added at four concentrations (by weight of cement): 0 (control) 1, 2 and 3% for the rattan and coconut husk particles, and at 0 (control) and 3% for a 50:50 mixture (by weight) of rattan and coconut husk. Hydration temperature was monitored on-line over a period of 23 h. The compatibility of both aggregates and their 50:50 mixture with Portland cement was assessed using the parameters of time to maximum hydration temperature, maximum hydration temperature, inhibitory index, and rate of heat generation. Findings showed that without CaCl₂ both aggregates exhibited relatively low level of compatibility with Portland cement, with the rattan particles exhibiting relatively higher degree of inhibition. The addition resulted in reduced setting time (about 60%), increased hydration temperature (50-80%), lower inhibitory index and higher rate of heat generation in all the aggregate/cement mixtures. Recommendations for further research include the identification of the cement-inhibitory chemicals present in coconut husk and rattan and investigations on the mechanism of CaCl₂ interaction with rattan/cement and coconut husk/cement systems.

Key words: Rattan; coconut husk; calcium chloride; cement hydration.

INTRODUCTION

Cement-bonded particleboard (CBP) is a lightweight material combining small pieces of lignocellulose aggregate with cement and water. In the past, wood was the main aggregate employed in CBP. However, economic and environmental pressures have led to other lignocellulosics being considered for use. A range of substitute materials, such as agricultural and wood processing residues, tree barks and weeds,

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has been tested. Examples include rice straw and giant ipil-ipil [1], bagasse [2, 3], oil palm shell [4] and cork granules [5]. Several other candidate materials are available, including rattan cane and coconut husk. These materials are present as waste in West Africa.

Coconut is one of the most economically useful palms in tropical Asia and Africa [6]. All parts of the palm are useful and local people use it for lumber and as a source of food. Coconut husk, however, is of limited commercial use at present. For this reason, some work has been conducted on the use of the husk of two coconut species (*Orbignya species* I and *Cocos nucifera*) for CBP production [2, 3, 7]. While Oyagade [2] studied the dimensional stability of CBP manufactured from *Cocos nucifera* husk, Fabiyi [3] examined both the dimensional stability and strength properties of boards made from this material. Almeida *et al.* [7] reported that CBPs made using the shell of *Orbignya species* show some inhibitory effects on cement curing and that the addition of CaCl₂ improved its compatibility. No work has been conducted to examine the effects of calcium chloride on the hydration characteristics of *C. nucifera* husk mixed with Portland cement.

Rattan, a non-timber forest product used for manufacturing cane furniture, grows as a spiny climber in the tropics and sub-tropics. The three species most endemic to Africa are Laccosperma (Syn. Ancistrophyllum), Eresmophatha (Mann. and Wendl.) and Oncocalamus (Wendl.). However, Laccosperma secundiflorum (P. Beauv.) Kuntze is most widespread [8]. Following harvesting, rattan is stripped of its spines and leaf sheaths before drying. Unfortunately much of the cane material becomes discoloured by staining fungi during this part of the process. This material cannot be processed for furniture and is considered waste. At present 20-30% of processed rattan is waste [9]. Since staining fungi utilise low-molecular-weight sugars present in freshly harvested canes, thereby reducing the quantities of sugars present [10], this material may be better for CBP manufacture. This is because low molecular weight sugars retard cement hydration [10-13]. Olorunnisola and Adefisan [14] investigated the effect of CaCl₂ on strength and water absorption properties of CBP produced using rattan furniture waste (in form of strands). However, no work has been undertaken to investigate hydration behaviour of rattan converted into particles and mixed with Portland cement.

Different methods have been used to assess wood-cement compatibility for manufacture of CBPs. Although monitoring the hydration characteristics of the wood/cement mixture is a common approach, there is no standard method for interpreting the results. Some of the different methods used are shown in Table 1. However, as noted by Hachmi *et al.* [15], the different criteria used sometimes yield conflicting recommendations regarding the use of candidate lignocellulosics. For this reason it is worth employing a number of different schemes to assess compatibility.

This work examines the effect of CaCl₂ on the hydration of rattan *L. secundiflorum* and coconut (*C. nucifera*) husk particles mixed with Portland cement. If these

Table 1.

Methods employed to assess aggregate/cement compatibility S

S	Parameter	Classification index	Equation	Reference
1	Time to maximum tem- perature	Suitable (<15 h) Unsuitable (>20 h)	_	[20]
2	Maximum hydration temperature	Suitable ($T_{\text{max}} > 60^{\circ}$ C) Intermediately suitable ($T_{\text{max}} = 50-60^{\circ}$ C) Unsuitable ($T_{\text{max}} < 50^{\circ}$ C)	_	[21]
3	Inhibitory index (<i>I</i>) cal- culated based on setting time, maximum tempera- ture, gradient of tempera- ture–time curve	Compatible ($I < 50$) Highly inhibitory ($I > 50$)	$I = 100 \times (t_{\text{max}} - t'_{\text{max}})/$ $t'_{\text{max}}(T'_{\text{max}} - T_{\text{max}})/$ $T'_{\text{max}}(S' - S)/S'$	[20]
4	Area ratio (C_A) , i.e., area under the hydration heat rate curve for neat and inhibited cement	Compatible ($C_A > 68\%$) Moderately compatible (C_A 28–68%) Not compatible ($C_A < 28\%$)	$C_{\rm A} = (A_{\rm wc}/A_{\rm nc}) \times 100$	[15, 22]

 t_{max} = time required for the aggregate/cement mixture to attain maximum hydration temperature, i.e., setting time of the inhibited aggregate/cement mixture.

 t'_{max} = time required for cement to attain maximum hydration temperature, i.e., setting time of neat cement.

 T'_{max} = maximum temperature attained by neat cement.

 T_{max} = maximum temperature attained by aggregate/cement mixture.

S' = temperature-time slope of neat cement.

S = temperature–time slope of inhibited cement.

 $A_{\rm wc}$ and $A_{\rm nc}$ are areas under the hydration heat rate curve from 3.5 h to 24 h of particle/cement mixture and neat cement, respectively.

materials are suitable for CBP manufacture, it may provide economic and environmental benefits to local communities in West Africa.

MATERIALS AND METHODS

Rattan and coconut husk collection and preparation

Rattan stems of *L. secundiflorum* were obtained from harvesters at Sapele, Delta State Nigeria, while coconut (*Cocos nucifera*) husks were collected from processors at Badagry, Lagos State, Nigeria. The longer coconut husk fibres were separated from the shorter ones and discarded. Both materials were air-dried for three weeks, hammer-milled, sieved and further air-dried for 3 weeks. Only particles that passed through a 850 μ m sieve and were retained on a 600 μ m sieve were used for hydration tests.

Specifications of cement, additives and water

Fresh Portland cement was used (class strength 32.5 R grade, graded in accordance with BS EN 197-1:2000 [16]). Cement was stored in airtight containers. CaCl₂ powder (technical grade) was used as an accelerator. Distilled water at room temperature ($20 \pm 2^{\circ}$ C) was used.

Determination of moisture and extractive contents of the aggregates

The oven dry moisture contents of the rattan and coconut husk particles were determined at a temperature of $103 \pm 2^{\circ}$ C, in accordance with British Standard BS 812-109:1990 [17]. Three replicate samples were used for each determination and the average values are reported. The extractive contents of the samples were determined using an ethanol/toluene mixture, ethyl alcohol (96%) and distilled water as solvents, according to ASTM D 1105-96 [18]. Extractive contents of two replicates were used.

Hydration tests

Hydration tests were conducted in sealed thermally insulated containers (Dewar flasks). The aggregate/water/cement mixtures comprised 15 g aggregate particles, 200 g Portland cement and 90.5 ml distilled water (as in Moslemi and Lim [11] and Weatherwax and Tarkow [19]). Each 15-g aggregate sample was dry-mixed with 200 g of cement in a polythene bag, then wetted with distilled water and mixed until homogenous mixture was obtained. CaCl₂ was added by dissolving in the distilled water before use at four concentrations (by weight of cement), 0 (control) 1, 2 and 3%, respectively, for the rattan and coconut husk, and at 0 (control) and 3 for the 50 : 50 mixture (by weight) of rattan and coconut husk.

The mixture was transferred to a Dewar flask and a thermocouple was inserted to enable temperature measurement at 1-min intervals over a 23-h period. The time expired to achieve the maximum temperature was assessed. Three replicates were used. The ambient room temperature and relative humidity were kept constant at $20 \pm 2^{\circ}$ C and 65%, respectively, throughout the experiment.

Data analysis and interpretation

The compatibility of the rattan and coconut husk samples with Portland cement was assessed using the four compatibility test parameters shown in Table 1.

RESULTS AND DISCUSSION

Moisture and extractive contents of the materials

The average moisture and extractive contents of the aggregates are shown in Table 2. The average moisture content of the rattan particles was 10.9%, while that of the

Table 2.

Material	Moisture content (%)		Extractive content (%)					
	Average	SD	Ethanol extract		Hot water extract			
			Average	SD	Average	SD		
Rattan Coconut husk	10.9 16.8	0.14 0.07	5.9 21.2	2.76 4.67	5.9 30.3	0.35 5.23		

Moisture and extractive contents of the rattan and coconut husk particles

Table 3.

Influence of CaCl₂ on the hydration behaviour of the aggregates

Particle/cement mixture	Parameters				Level of compatibility			
	<i>t</i> (h)	T_{\max} (°C)	I (%)	$C_{\mathrm{A}}\left(\% ight)$	CL^1	CL^2	CL ³	CL ⁴
Rattan only	23.0	37.4	119.6	55	U	U	HI	MC
Rattan + 1% CaCl ₂	14.0	53.2	27.4	84	S	IS	С	С
Rattan + 2% CaCl ₂	9.5	61.4	5.2	97	S	S	С	С
Rattan + 3% CaCl ₂	9.0	53.3	6.4	84	S	IS	С	С
Coconut husk only	17.5	38.9	75.8	63	U	U	HI	MC
Coconut husk $+ 1\%$ CaCl ₂	10	55.0	10.2	89	S	IS	С	С
Coconut husk $+ 2\%$ CaCl ₂	8.4	59.3	3.6	94	S	IS	С	С
Coconut husk $+ 3\%$ CaCl ₂	7.4	55.3	2.0	87	S	IS	С	С
Rattan/coconut husk mixture only	20.4	35.3	110.5	60	U	U	HI	MC
Rattan/coconut husk mixture with 3% CaCl ₂	7.0	64.5	0.5	99	Se	S	С	С
Neat cement	6.4	74.5						

t = time to maximum temperature.

 $T_{\text{max}} = \text{maximum temperature attained by wood/cement mixture.}$

I =inhibitory index.

 CL^1 = compatibility level based on time to maximum temperature [20].

 $CL^2 =$ compatibility level based on maximum hydration temperature [21].

 CL^3 = compatibility level based on inhibitory index [20].

 CL^4 = compatibility level based on area ration [15, 22].

C = compatible; S = suitable; U = unsuitable; IS = intermediately suitable; MC = moderately compatible; HI = highly inhibitory.

coconut husk particles was 16.8%, indicating the latter to be more hygroscopic. The ethanol- and hot water-soluble extractive contents of the coconut husk particles were much higher than those of the rattan. The nature of these extractives was not determined.

Time to maximum temperature for aggregate-cement mixtures

The times required for aggregate/cement systems to attain maximum temperature are presented in Table 3. For individual aggregates, 17.5 h was taken by the coconut husk/cement mixture without $CaCl_2$ to attain maximum temperature and

23 h for the rattan/cement mixture without $CaCl_2$. The equivalent time to maximum temperature for neat cement (without coconut husk or rattan) was 6.4 h. Using the time to maximum temperature parameter alone as measure of compatibility, the rattan, coconut husk and a 50:50 mixture of rattan and coconut husk are unsuitable for CBP, since all took more than 15 h to attain maximum temperature. Aggregate/cement/water systems that attain maximum hydration temperature in less than 15 h are considered suitable, while those that require more than 20 h are considered inhibitory [20].

The addition of CaCl₂ resulted improved the time to maximum temperature of the mixtures. The greatest improvement in time was observed in the rattan-cement mixture with the addition of 3% CaCl₂. In this case the time to maximum temperature reduced by 14 h (from 23 h (0% CaCl₂) to 9 h (3% CaCl₂). Examination of results presented in Table 3 shows that CaCl₂ was more effective in reducing the time to maximum temperature of Portland cement mixed with rattan than that mixed with coconut husk. This might be due to the relatively high extractive content of the coconut husk particles used.

Maximum temperatures of the mixtures

The maximum temperatures (T_{max}) of the different aggregate-cement mixtures are shown in Table 3. The values ranged from 35.3°C for the 50:50 rattan/coconut husk/cement mixture without CaCl₂ to 74.5°C for neat cement. Using T_{max} as the criterion for compatibility, both materials are 'unsuitable' for CBP since they reduced hydration temperature of cement to below 50°C, the threshold value recommended by Sanderman and Kohler [21].

An increase in the T_{max} for both aggregates mixed with cement was observed as CaCl₂ was added, particularly when 50:50 rattan/coconut husk was used. In this case, T_{max} was raised by about 82%, from 35.3°C (0% CaCl₂) to 64.5°C (3% CaCl₂). The highest T_{max} values were attained by the rattan/cement and coconut husk/cement mixtures at 2% CaCl₂. The reason for this is unclear. However, in describing the accelerating mechanism of chlorides, Zhengtian and Moslemi [12] observed that chlorides tend to enhance the solubility of cement in water and, hence, the intensity of the exothermic reaction at the initial stage. Therefore, it seems the addition of chlorides only tends to result in initial rise in temperature.

Inhibitory index

The inhibitory index (I) is a quantitative measure of the compatibility of an aggregate mixed with cement that takes into account setting time, $T_{\rm max}$ and $T_{\rm max}$ /time slope. The I values for different aggregates with and without CaCl₂ are shown in Table 3. The I values ranged from 75.8% for the coconut husk particles to 119.6% for rattan. Since I values are greater than 50% for both aggregates alone and as a mixture, they can be considered highly inhibitory to cement setting. Any aggregate that gives an inhibitory index greater than 50%

is considered highly inhibitory [11, 19]. The rattan-cement mixture that had the longest time to maximum temperature and lowest hydration temperature value also had the highest I value.

The addition of $CaCl_2$ resulted in reductions in inhibitory index for all mixtures, the greatest reduction being observed in the rattan/coconut husk mixture, where the inhibitory index fell from 110.5% (0% $CaCl_2$) to 0.5% (3% $CaCl_2$). The rate of reduction in inhibitory index with increasing levels of $CaCl_2$ was greater in the rattan/cement mixture than the coconut husk/cement mixture, suggesting that $CaCl_2$ was a more effective accelerator for rattan.

Heat generated by mixtures

The area ratio method developed by Hachmi et al. [15] compares the area under the hydration heating rate curve for the time interval of 3.5 to 23 h for aggregate/cement/water mixture with that of neat cement. The resulting compatibility measure is known as the C_A factor. C_A factors for the various aggregate-cement mixtures are presented in Table 3. The C_A factors for the mixtures without CaCl₂ vary from 55 for the rattan-cement mixture to 63 for the coconut husk-cement mixture. Using the C_A factor criterion, it can be said that both aggregates are moderately compatible with Portland cement without the addition of CaCl₂, with coconut husk being more compatible than rattan. This result follows a similar pattern to the other assessments. However, the C_A factor is expected to decrease as the extractive content increases. In this case, the untreated coconut husk with higher extractive content had a higher C_A factor, just as it had a much lower inhibitory index, than the rattan cane. This calls for a thorough investigation of the chemical position composition of the extractive contents of both materials. As noted by Hachmi and Moslemi [22], often the chemical composition rather than the percentage inclusion of extractives plays a vital role in wood-cement compatibility.

The addition of $CaCl_2$ again resulted in improved compatibility between aggregate and cement with greater C_A values obtained using 2% CaCl₂ than at 3%.

CONCLUSIONS

The findings of this study have shown that:

- 1. Rattan (*L. secundiflorum*) and coconut husk (*C. nucifera*) are not very suitable as aggregates for wood/cement particleboard production without pre-heating the wood particles as they prolong the setting time and reduce the hydration temperature. This hydration behaviour is similar to many hardwoods that are generally not compatible with Portland cement without treatment.
- 2. Coconut husk is most suitable and a mixture of rattan and coconut husk particles more suitable than rattan. This probably results from the presence of more low-molecular weight sugars in the rattan than the coconut husk.

- 3. CaCl₂ enhances the compatibility of rattan cane and coconut husk particles with Portland cement.
- 4. The addition of $CaCl_2$ at 2% concentration seems to result in higher compatibility of rattan cane and coconut husk particles with Portland cement than at 3% concentration.
- 5. The different compatibility assessment parameters adopted, i.e., setting time, maximum hydration temperature, inhibitory index and the heat generated by the mixtures, all indicated that the aggregates were incompatible with Portland cement without pre-treatment.

RECOMMENDATIONS FOR FURTHER STUDIES

In view of the findings of this study the following areas of further research are recommended:

- 1. Identification of the cement-inhibitory chemicals present in coconut husk and rattan.
- 2. Investigations on the actual mechanism of CaCl₂ interaction with rattan/cement and coconut husk/cement systems.
- 3. Evaluation of the compatibility of blue-stained rattan canes with Portland cement. Since most rattan waste is generated as a result of the presence of fungal stain that removes low-molecular-weight sugars, then stained rattan may provide lower inhibition.
- 4. Evaluation of the effects of varied particle sizes and other pre-treatment measures, e.g., hot and cold water, prolonged storage and other chemical accelerators on the hydration behaviour of rattan/cement and coconut husk/cement systems.
- 5. Fabrication and evaluation of actual boards produced from rattan *L. secundiflorum* and *C. nucifera* husk.
- 6. Evaluation of other rattan species for particleboard production.

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