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Effects of pre-treatment of rattan (*Laccosperma secundiflorum*) on the hydration of Portland cement and the development of a new compatibility index

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Abstract

Effects of aqueous extraction, sieving, mixing with coconut husk and addition of calcium chloride on the compatibility of *Laccosperma secundiflorum* rattan with Portland cement, in terms of hydration inhibition, were investigated. Compatibility was assessed based on maximum hydration temperature, time taken to attain maximum temperature and area under the hydration heating rate curve relative to that of neat cement. Alternative cement–lignocellulosics compatibility classification method was also developed. Untreated rattan particles were found inhibitory to cement hydration. However, sieving coupled with extraction in cold water for 60 min greatly improved the compatibility. Hot water extraction had no advantage over cold water extraction for the species. Adding cold water-extracted coconut husk to the species also improved its compatibility. The proposed compatibility index, based on the ratio of the setting times of wood–cement mixture and neat cement, yielded satisfactory results comparable to propositions from previous researchers.

Keywords: Rattan; Cement; Composite; Hydration; Compatibility

1. Introduction

Wood–cement composite panels are employed in building construction for diverse purposes including flooring, siding, and ceiling. The woody part of these composites is obtainable from different sources including fast growing (plantation) tree species, wood and agricultural residues, and non-commercial or low-value tree species [1–5]. These panel products appear to have the potential to satisfy established cultural preference in the tropics for low-cost cement-based construction materials [6]. They are specially noted for their relatively high fire and decay resistance, low water absorption, and good dimensional stability [2].

As observed by Olorunnisola et al. [7] and Olorunnisola [8,9] rattan cane, particularly *Laccosperma secundiflorum* (P. Beauv.) Kuntze and coconut husk (*Cocos nucifera*) represent two of the relatively abundant lignocellulosics in Africa and Asia that could form the basis of a viable wood–cement composite industry. However, like other lignocellulosics, both materials tend to inhibit cement setting as they contain ethanol- and hot water-soluble extractives [7]. It has been well established that the degree of compatibility of woody materials with Portland cement is a function of the amount of wood components such as soluble sugars, carbohydrates, and other wood extractives [10–12].

The major inhibitory effect of wood on cement delayed is setting. Other manifestations of inhibition include relatively lower hydration temperature and prolonged time to attain maximum temperature [11,13,14]. Different means have thus been devised for minimizing these effects. They include use of wood preservatives, prolonged storage of the wood material, hot and/or cold water extraction of the soluble sugars, blending of different wood aggregates,

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and use of inorganic compounds (chemical additives or accelerators) in dilute solutions such as sodium hydroxide (NaOH), sodium silicate (Na_2SiO_3), calcium chloride ($CaCl_2$) and aluminum sulphate Al_2 $(SO_4)_3$) [2,15–17]. Moslemi et al. [15] reported substantial reduction in cement inhibition when a combination of hot water extraction and addition of accelerator was employed on wood samples. Irle and Simpson [18] also observed that the compatibility of some plant materials improved with washing with cold water, hot water or calcium hydroxide solution.

Another variable of importance in cement inhibition during hydration is wood particle size. It has been reported that the inhibition of cement setting increases with increasing fineness of wood particles [19]. While Olorunnisola et al. [7] also reported that a combination of the effect of aggregate mixture and $CaCl_2$ addition had some positive effect on the hydration behaviour of composites from rattan and coconut husk, no work has been reported on the effect of particle size and water extraction with or without additive use on the compatibility of rattan with Portland cement.

Different schemes have been proposed for evaluating the compatibility of wood with cement based on hydration test. A review of these methods presented by Olorunnisola et al. [7] indicated that while many of them lack consistency in the classification lignocellulosics, they also involve cumbersome computations which ought to be further simplified for practical utility. Two key parameters that have featured prominently in most previous compatibility indices are maximum hydration temperature and time to reach maximum hydration temperature. However, four out of the six indices reviewed took the setting time of cement and wood–cement mixtures into account in one way or the other, suggesting that setting time is a critical parameter in assessing wood compatibility with cement. Also most indices classified species into three categories, i.e., compatible/suitable, moderately compatible and incompatible/inhibitory.

The main objective of this work was to examine the effects of combined pretreatment measures on the compatibility of rattan cane particles with Portland cement, using previously reported compatibility indices. A secondary objective was to explore the possibility of developing an alternative classification method for measuring the compatibility of cement with various lignocellulosics.

2. Materials and methods

2.1. Material collection and preparation

Mature freshly harvested rattan stems were obtained from wild forests through rattan harvesters at Sapele, Delta State Nigeria and subsequently identified in the Herbarium of the Department of Botany, University of Ibadan, Nigeria, as those of *L. secundiflorum*. Coconut (*Cocos nucifera*) husks were collected from processors at Badagry, Lagos State, Nigeria. Both materials were air-dried for 21 days, hammer-milled and air-dried for another 21 days.

2.2. 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). Cement was stored in air-tight containers. $CaCl_2$ powder (technical grade) was used as an accelerator. Distilled water at room temperature (20 ± 2 °C) was used.

2.3. Pre-treatment measures on rattan particles

Pre-treatment measures employed on the rattan particles are as shown in Table 1. They included sieving, aqueous extraction and $CaCl_2$ addition. 'As-received' rattan particles were those obtained after hammer milling the rattan canes. For sieved samples, a set of sieves comprising 2.4 mm, 1.2 mm, 850 μm and 600 μm were used. Only particles retained on 600 μm sieve were used for hydration tests. Aqueous extraction involved soaking some samples in cold water (existing at room temperature) and others in hot water maintained at 50 °C, using a water bath, for durations ranging from 30 to 120 min as indicated in Table 1 and then draining and washing off the soluble extractives with distilled water. For samples pre-treated with $CaCl_2$, the additive was dissolved in distilled water used for the hydration experiments at 1% concentration (by weight of cement).

2.4. Hydration tests

The hydration test method used was the same as described by Okino et al. [3], Olorunnisola et al. [7], Mos-

Table 1
Pre-treatments measures performed on rattan particles

Treatment	Description
Controls	Neat cement
	Untreated rattan
	Untreated coconut husk
	Untreated rattan + untreated coconut husk (50:50 by weight)
Pre-treatment measure I	
	Soaking in cold water for 30 min
	Soaking in cold water for 60 min
	Soaking in hot water for 60 min
Pre-treatment measure II	
	Sieving + soaking in cold water for 30 min
	Sieving + soaking in cold water for 60 min
	Sieving + soaking in cold water for 120 min
	Sieving + soaking in hot water for 30 min
	Sieving + soaking in hot water for 60 min
	Sieving + soaking in hot water for 120 min
Pre-treatment measure III	
	Sieving + soaking in cold water for 60 min + 1% $CaCl_2$
	Sieving + soaking in hot water for 60 min + 1% $CaCl_2$
Pre-treatment measure IV	
	Sieving + mixing with coconut husk + soaking in cold water for 60 min
	Sieving + mixing with coconut husk + soaking in hot water for 60 min

lemi and Lim [11] and Weatherwax and Tarkow [19]. Each rattan–cement composite, replicated thrice, comprised 15 g of aggregates, 200 g of Portland cement, and 90.5 ml of distilled water. For each composition, 15-gram 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.

The hydration tests were conducted in sealed thermally insulated containers (Dewar flasks) in which T-type thermocouples, connected to a multipoint data recorder, were inserted. Temperature measurements for each test were taken at 1-min interval over a 23-h period. Time expired to achieve the maximum temperature was assessed. The ambient room temperature and relative humidity were kept constant at 20 °C ± 2 °C and 65%, respectively, throughout the experiment.

2.5. Data analysis and interpretation

The compatibility levels of the rattan and coconut husk samples with Portland cement were assessed using the three schemes shown in Table 2 as proposed by Hofstrand et al. [12], Sandermann and Kohler [20], and Hachmi [21]. The three schemes were developed based on the assumption that hydrated neat cement and wood–cement-water mixtures exhibit the same hydration behaviour except that the hydration rate of wood–cement mixtures tends to be generally lower. Hence compatibility of wood with cement is assessed by comparing hydration rates (i.e., setting time, the maximum hydration temperature, and the area under the hydration heating rate curve) of aggregate–cement–water mixtures with that of neat cement.

The compatibility levels indicated in the three schemes were set based on the assumptions that the setting (time to reach maximum hydration temperature) of neat cement typically takes about 8 h for Scheme 1; the maximum hydration temperature tends to exceed 60 °C for Scheme 2; and that the area under hydration heat rate curve within a time interval of 3.5 and 24 h is a good indicator of cement hydration behaviour for Scheme 3 [2,20,21].

3. Results and discussion

3.1. Maximum temperature attained by the composites

The maximum temperatures (T_{max}) attained by the different aggregate–cement mixtures are shown in Table 3. The values ranged from 35.3 °C for the un-treated 50:50 rattan–coconut husk–cement mixture to 75.1 °C for neat cement. An increase in T_{max} was observed in all pre-treated samples. Samples subjected to a combination of pre-treatment measures involving sieving, soaking in cold water for 60 min and adding 1% $CaCl_2$ attained the highest hydration temperature of 59.5 °C. Sieving and soaking in hot water for 60 min had the least positive effect on T_{max}. This result is close to 61.4 °C reported by Olorunnisola et al. [7] for rattan–cement composites pre-treated with only 2% $CaCl_2$.

A closer look at Table 3 also shows that sieving of the rattan particles had an appreciable effect on T_{max}. The noticeable enhancement in T_{max} was perhaps because by sieving, part of the lignified rattan covering which is an insulating material had been removed. Both the temperature of the water and the soaking duration had no appreciable effect on T_{max} in most cases (<5%) except for samples sieved and soaked in hot water where there was about 29% increase in T_{max} between samples soaked for 60 minutes and those soaked for 120 minutes. Combination of soaking with the addition of $CaCl_2$ had a more notable effect on hot water pre-treated samples, resulting in about 45% increase in hydration temperature, while samples soaked in cold water recorded about 17% rise in temperature. What these results suggest is that the cement hydration inhibitors present in the rattan samples were readily soluble in cold water. This is contrary to the findings of Irle and Simpson [18] who reported that hot water extraction was very effective pre-treatment for wood, flax and linseed fibres.

Addition of un-treated coconut husk to un-treated rattan resulted in a hydration temperature lower than those attained by cement composites from either material. However, soaking the mixed particles in cold and hot water,

Table 2
Methods employed to assess aggregate/cement compatibility

S. no	Parameter	Classification index	Equation	Reference
1	Setting time	Suitable ($t_{max} \leqslant 15$ h)	–	[12]
		Intermediately suitable (15 h > t_{max} < 20 h)		
		Unsuitable ($t_{max} \geqslant 20$ h)		
2	Maximum hydration temperature	Suitable ($T_{max} > 60$ °C)	–	[20]
		Intermediately suitable (50 > $T_{max} \leqslant 60$ °C)		
		Unsuitable ($T_{max} \leqslant 50$ °C)		
3	Area ratio (C_A), i.e., area under the hydration heat rate curve for neat and inhibited cement	Compatible ($C_A \geqslant 68\%$)	$C_A = (A_{wc}/A_{nc})100$	[13,21]
		Moderately compatible (68% > C_A > 28%)		
		Not compatible ($C_A \leqslant 28\%$)		

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} = maximum temperature attained by aggregate–cement mixture.
A_{wc} and A_{nc} are areas under the hydration heat rate curve from 3.5 to 24 h of particle–cement mixture and neat cement, respectively.

Table 3
Influence of rattan pre-treatment on hydration of cement

Pre-treatment measures	Parameters			Level of compatibility		
	t_{max} (h)	T_{max} (°C)	C_A (%)	CL^1	CL^2	CL^3
Controls						
Neat cement	7.1	75	100			
None (un-treated rattan)	23.0	37	55	U	U	MC
None (un-treated coconut husk)	17.5	39	63	U	U	MC
Mixing untreated rattan with untreated coconut husk (50:50 by weight)	20.4	35	61	U	U	MC
Pre-treatment measure I						
Soaking in cold water for 30 min	9.6	59	94	S	IS	C
Soaking in cold water for 60 min	11.3	49	79	S	U	C
Soaking in hot water for 60 min	10.9	48	77	S	U	C
Pre-treatment measure II						
Sieving + soaking in cold water for 30 min	10.3	51	85	S	IS	C
Sieving + soaking in cold water for 60 min	9.5	51	83	S	IS	C
Sieving + soaking in cold water for 120 min	9.9	53	85	S	IS	C
Sieving + soaking in hot water for 30 min	9.9	54	89	S	IS	C
Sieving + soaking in hot water for 60 min	13.2	41	67	S	U	MC
Sieving + soaking in hot water for 120 min	9.8	53	86	S	IS	C
Pre-treatment measure III						
Sieving + soaking in cold water for 60 min + 1% $CaCl_2$	8.3	60	94	S	S	C
Sieving + soaking in hot water for 60 min + 1% $CaCl_2$	8.6	59	94	S	IS	C
Pre-treatment measure IV						
Sieving + mixing with coconut husk + soaking in cold water for 60 min	12.1	47	79	S	U	C
Sieving + mixing with coconut husk + soaking in hot water for 60 min	13.6	45	77	S	U	C

CL^1 = compatibility level based on setting time [12].
CL^2 = compatibility level based on maximum hydration temperature [20].
CL^3 = compatibility level based on area ratio [13,21].
C = compatible; S = suitable; U = unsuitable; IS = intermediately suitable; MC = moderately compatible.

respectively, resulted in higher temperatures with cold water-treated samples again exhibiting higher temperatures. It is reasonable to assume, therefore, that the cement inhibitors in the coconut husk were also more soluble in cold water. A practical implication of this observation is that in actual industrial production no extra cost would be required to generate hot water for extraction during actual composite fabrication.

Based on the classification scheme developed by Sanderman and Kohler [20] (Table 2), all the pre-treatment measures, i.e., sieving, soaking in water and addition of 1% $CaCl_2$ generally resulted in rattan–cement–water mixtures that ranged between 'intermediately suitable' and 'suitable' for wood–cement composite production.

3.2. Setting times for the composites

The setting times are presented in Table 3. The setting time for the rattan–cement composite was 23 h as compared with 7.1 h for the neat cement. Pre-treating the rattan particles using different methods, reduced the setting time by between 9.4 and 14.7 h. Olorunnisola et al. [7] had reported a similar degree of reduction in setting time for *L. secundiflorum* particles pre-treated with between 1% and 3% $CaCl_2$. It can be inferred, therefore, that aqueous extraction is as effective as $CaCl_2$ in reducing the inhibitory effects of *L. secundiflorum* on Portland cement.

The combination of sieving, soaking in cold water for 60 minutes, and adding 1% $CaCl_2$ had the greatest effect in setting time reduction, while mixing with un-treated coconut husk had the least effect. Except for samples sieved and soaked in hot water for 60 minutes, combining water soaking with the addition of $CaCl_2$ resulted in only minimal improvement (about 1 h reduction) in the setting times of the mixtures. What this suggests in practical terms is that where cost of $CaCl_2$ might be a major deciding factor in composite production, aqueous extraction alone might be sufficient for enhancing compatibility.

Fig. 1 shows a relatively high positive correlation ($R^2 = 0.72$) between setting time and maximum hydration temperature for all the rattan–cement–water mixtures. However, using the setting time parameter alone as specified by Hofstrand et al. [12] (Table 2), all the pre-treatment measures resulted in materials that could be considered 'suitable' for wood–cement composite production.

3.3. Area under the hydration heating rate curves for the composites

As proposed Hachmi et al. [21], the inhibitory effects of a lignocellulosic can be measured by comparing the area under the hydration heating rate curve of neat cement with

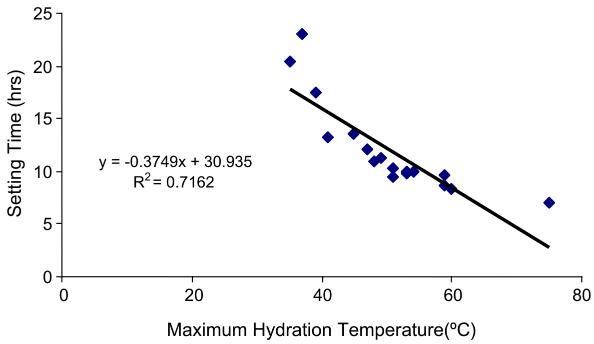

Fig. 1. Correlation between setting time and maximum hydration temperature.

that of wood–cement–water mixture. The resulting compatibility measure is known as the C_A factor. The C_A factors for the various rattan–cement mixtures are presented in Table 3. They varied from 55% for the un-treated rattan–cement mixture to 94% for composites produced with rattan particles sieved, soaked in cold/hot water for 60 min to which 1% $CaCl_2$ was added. These results also indicated varying degrees of improvement in compatibility with the adoption of different pre-treatment measures.

Using the C_A factor criterion, the untreated rattan particles fall under the 'moderately compatible' classification, while virtually all the pre-treated aggregates are 'compatible'. Also, as shown in Figs. 2 and 3, there were relatively strong positive correlation between C_A and T_{max}, on the one hand ($R^2 = 0.89$), and between C_A and t_{max} ($R^2 = 0.88$) on the other hand for the rattan–cement mixtures.

3.4. Comparing the various assessment schemes and a proposal for a new scheme

As shown in Table 3, the three compatibility evaluation methods were not very consistent in characterizing the hydration behaviour of the rattan–cement composites. However, classifications based on setting time and C_A yielded more consistent results. For example samples soaked in both hot and cold water for 60 min, respectively, fell under the 'suitable' and 'compatible' classifications based on t_{max} and C_A, respectively, whereas, these same materials fell under the 'unsuitable' classification, based on T_{max}.

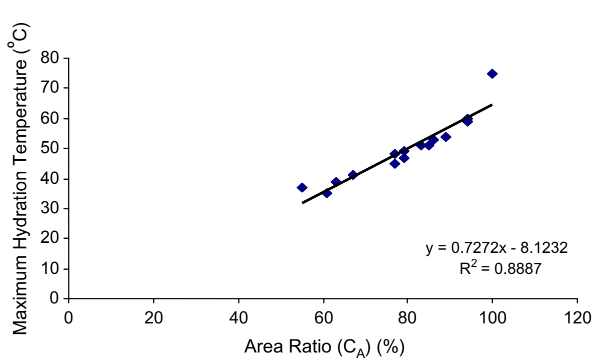

Fig. 2. Correlation between C_A and T_{max}.

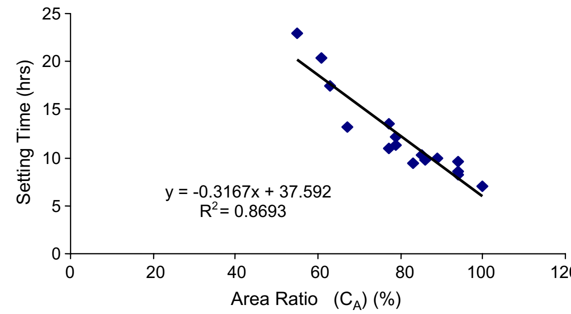

Fig. 3. Correlation between C_A and t_{max}.

What the foregoing indicates is that compatibility assessment should not be based on maximum hydration temperature or the area under the hydration heat rate curve. This is because addition of wood inclusions in cement effectively dilutes the system with respect to cement hydration. This dilution effect lowers the maximum hydration temperature. As noted by Karade [3], direct comparison of the maximum hydration temperatures of neat cement and wood–cement composites could be erroneous because even if there were to be no inhibition, the hydration of a wood–cement mix tends to progress at a lower temperature than that of neat cement.

Comparing the setting time of composites with that of the neat cement from which the composites were manufactured appears to be a more appropriate parameter for judging the suitability of a lignocellulosic for cement composite production. It is a better reflection of the degree of inhibition exhibited by a lignocellulosic against cement setting. Hence, the new assessment scheme being proposed recommends that the compatibility of lignocellulosics be determined using the setting time factor, T_R which is the ratio of setting time of wood–cement composite to that of neat cement.

The proposed scheme is presented in Table 4. It is relatively easy to implement with only minimal computations required. It was developed based on the assumption and practical logic, from the productivity point of view, that wood aggregates that extend the setting time of neat cement by not more than 50% should considered suitable

Table 4
Proposed compatibility assessment scheme

Parameter	Classification index	Recommendation
Ratio of setting time of wood:cement composite to neat cement i.e., $T_R = t_{WC}/t_{NC}$	$1 \leqslant T_R \leqslant 1.5$ (suitable)	(Further) pre-treatment unnecessary
	$1.5 < T_R \leqslant 2.0$ (acceptable)	(Further) pre-treatment recommended
	$T_R > 2.0$ (inhibitory)	(Further) pre-treatment highly recommended

Table 5
Comparison between previous and newly proposed compatibility criteria

Pre-treatment	T_R	Level of compatibility			
		CL[1]	CL[2]	CL[3]	CL[4]
None (un-treated rattan)	3. 2	U	U	MC	I
None (un-treated coconut husk)	2.5	U	U	MC	I
Mixing untreated rattan + untreated coconut husk (50:50 by weight)	2.9	U	U	MC	I
Treatment group A					
Soaking in cold water for 30 min	1.4	S	IS	C	S
Soaking in cold water for 60 min	1.6	S	U	C	A
Soaking in hot water for 60 min	1.5	S	U	C	S
Treatment group B					
Sieving + soaking in cold water for 30 min	1.5	S	S	C	S
Sieving + soaking in cold water for 60 min	1.4	S	S	C	S
Sieving + soaking in cold water for 120 min	1.4	S	S	C	S
Sieving + soaking in hot water for 30 min	1.4	S	S	C	S
Sieving + soaking in hot water for 60 min	1.9	S	S	MC	A
Sieving + soaking in hot water for 120 min	1.4	S	S	C	S
Treatment group C					
Sieving + soaking in cold water for 60 min + 1% $CaCl_2$	1.2	S	S	C	S
Sieving + soaking in hot water for 60 min + 1% $CaCl_2$	1.2	S	IS	C	S
Treatment group D					
Sieving + mixing with coconut husk + soaking in cold water for 60 min	1.7	S	U	C	A
Sieving + mixing with coconut husk + soaking in hot water for 60 min	1.9	S	U	C	A

T_R = ratio of setting time of wood–cement composite to that of neat cement.
CL[1] = compatibility level based on setting time [12].
CL[2] = compatibility level based on maximum hydration temperature [20].
CL[3] = compatibility level based on area ratio [13,21].
CL[4] = newly proposed Compatibility level based on setting time ratio.
A = acceptable; C = compatible; S = suitable; U = unsuitable; I = inhibitory; MC = moderately compatible.

requiring no (further) pre-treatment; those that extend it by between 50% and 100% should be considered acceptable with (further) pre-treatment recommended; while those that extend it beyond 100% should be considered inhibitory, with (further) pre-treatment highly recommended.

The scheme is similar to that proposed by Weatherwax and Tarkow [19] which compares the extent of wood–cement inhibition based on the percentage increase in setting time of neat cement but it does not involve computation of percentages. It also differs from the recommendation of Hofstrand et al. [12] based on specific wood–cement composite setting time frames. Previous findings have established the fact that the setting time of cement and cement composites depends on several factors, including the ambient weather condition, the type (chemical composition) of the cement, and the quantity of water used for mixing [11,13,22]. Besides, Hofstrand et al.'s scheme failed to classify wood–cement systems that have a setting time of between 15 and 20 h. This scheme that directly compares setting time of neat cement with that of the wood–cement composite is more universally applicable.

An illustration of the use of the scheme, based on the findings of this study is presented in Table 5. It is evident from the Table that the proposed scheme clearly and properly identified un-treated rattan as being unsuitable for wood–cement composite production.

4. Conclusions

Untreated and pre-treated particles of rattan (*L. secundiflorum*) were used to produce wood–cement composites. These mixtures were tested to determine the inhibitory effects of rattan on Portland cement using three published assessment schemes. Based on some inconsistencies observed in the outcomes produced by the three schemes, a new, simpler scheme was developed for assessing the inhibition of Portland cement by wood particles.

Based on the findings of this study, the following conclusions were drawn:

(i) *L. secundiflorum* is inhibitory to Portland cement. However, sieving coupled with extraction in cold water for 60 min is sufficient to make the species compatible.
(ii) Hot water extraction had little or no advantage over cold water extraction for the species.
(iii) Adding cold water-extracted coconut husk to the species may also improve its compatibility.
(iv) A new compatibility assessment scheme based on the ratio of setting time of wood–cement composite to that of neat cement yielded satisfactory results.

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