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Suitability of Portland Cement and Rice Husk Ash Pozzolan Systems for Cement Bonded Composites Production

James S. Fabiyi
Department of Forestry and Wood Technology, Federal University Technology, PMB 704, Akure, Ondo State, Nigeria.

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* Corresponding author: E-mail: jsfabiyi@gmail.com

Abstract
The suitability of substituting ordinary Portland cement (OPC) with rice husk ash (RHA) pozzolan for the production of cement bonded composites (CBCs) was investigated using the hydration technique. Hydration parameters such as the maximum hydration temperature, setting time to reach maximum hydration temperature, the time ratio index and inhibitory index for wood particle-hybrid cement-water systems were considered. The hydration characteristics observed for the wood-cement-water mixtures of different hybrid cement containing 0, 10 and 20% RHA pozzolan showed considerable differences in the hydration reaction. The treatment of wood particles with calcium chloride considerably improved the compatibility of the various hybrid cements with wood particles. The inhibitory index results showed that chemical treatment of Obeche wood approach is not necessary in the production of well consolidated CBCs from the hybrid cement.

Keywords: Rice husk ash, pozzolan, hybrid cement, hydration, inhibitory index

1. Introduction
The challenges facing wood industries in Nigeria are basically due to the problems of over-exploitation and deforestation of both natural and plantation forests in the country. Much exploitation and deforestation have been done in order to meet the increasing demand of the teeming population for wood and wood products, agricultural land, industrial establishment sites and housing estates [1]. These activities have left country's forests with young and small diameter trees which are mostly composed of a high proportion of juvenile wood as well as lesser known or underutilized timber species. The depletion of the forest resource base has made it difficult for wood based industries to meet the wood products demand. Therefore production of wood composites of which cement bonded composites (CBCs) belong could serve as one of the ways to meet such demand. Unfortunately, the production of CBCs is becoming expensive because of the increasing cost of Portland cement procurement. Reducing the cost of CBCs production is of great interest and this can be done by finding an alternative way of substituting Portland cement with natural pozzolana cement. A pozzolan has been defined as a siliceous or aluminosiliceous material that, in finely divided form and in the presence of moisture, chemically reacts with the calcium hydroxide released by the hydration of Portland cement to form calcium silicate hydrate and other cementitious compounds [2]. Therefore, natural pozzolana cement could be produced from any biomaterials with high silica content. An example of such biomaterials is rice husk, which is also a very common agricultural food crop. It was reported that approximately 100 million tons of rice husks are generated annually worldwide while about 2 million are generated in Nigeria annually [3]. Out of the total rice produced, the husks constitute approximately 20%. It is expected that the generation of rice husks would increase due to the increasing world population, campaign for food security and high mechanization. Unfortunately, recycling of agricultural residues like rice husks may not be commensurate with the increase in rice husks generation. Large quantity of the rice

husks generated in developing countries are normally burnt off in the rice farm/mills or dumped in landfills, while the insignificant portion is used as fuel. Burning in the open air is likely to cause environmental pollution as carbon dioxide (CO_2) and carbon-monoxide (CO) would be released into the atmosphere as a result of insufficient oxygen in the heap of the rice husk. Also, the aerobic decomposition of rice husks in the landfills could be a major source of methane emissions. The release of these gases into the atmosphere changes the climate, thereby resulting in global warming, which is now one of the greatest threats to our world. Blending pozzolan with Portland cement could be referred to as "green cement" because it reduces carbon dioxide emissions and also encourages the use of waste materials in the case of the recycling of wastes [4]. Bilodeau and Malhotra [5] estimated that the production of 1 tone of OPC led to the release of 1 tone of CO_2, a major contributor to the greenhouse effect and the global warming of the planet. Decrease in the amount of CO_2 released into the atmosphere during Portland cement production can be achieved with the use of natural pozzolans. Based on this fact, using rice husks for the production of pozzolan would help reduce CBC production cost, minimize environmental pollution, enhance municipal solid wastes management, and also contribute positively to the economic growth of our society.

Cisse and Laquerbe [6] reported that uncontrolled temperature burning of rice husk tends to produce crystalline silica and consequently poor pozzolanic properties. Crystalline silica causes a very serious health hazard especially when inhaled and may result in a disease known as silicosis [7]. However, biomass ash production at temperatures below 1000°C yield high amorphous silica content, which does not contain too many harmful forms of silica. So the conversion of rice husk to ash for the purpose of pozzolan production can be done below 1000°C. Olamide and Oyawale [8] concluded that during conversion of rice husk to RHA, optimal silica was produced at temperature of 700°C.

Rice husk has been identified as one of the agriculture residues that has high silicon content, which is the cementious or binding agent in biomass ash [9]. The main composition of rice husk are SiO_2, lignin, cellulose, protein, fat and other nutrients with 18.80-22.30, 9-20, 28-38, 1.90-3.0, 0.30-0.80 and 9.30-9.50% on weight basis. Further chemical analysis showed that silicon content is different at various regions in rice husk with 45.16, 2.27 and 27.27% for the husk external surface, husk interior and husk internal surface, respectively [9]. It must be noted that these chemical composition values may vary depending on location where rice is grown.

The production of an hybrid cement from the mixture of Portland cement and RHA pozzolan is not a new discovery. Previous studies had considered its uses for sandcrete blocks production and concrete [10-13]. However, information on its suitability for the production of cement bonded composites has not been reported in published articles. Therefore, the main objective of this study was to investigate the suitability of Portland cement and rice husk ash pozzolan for the production of cement bonded composites.

2. Material and methods

2.1 Raw materials procurement

Tricplochiton scleroxylon (Obeche) wood particles used in this study was collected from a local sawmill located at Akure, Ondo State, Nigeria. The wood particles were subjected to hot water treatment (maintained at 80°C for about 2 h) to remove water-soluble extractives. The wood particles were then air-dried to approximately 12% moisture content. The dried wood particles were stored in a sealed polythene bags to maintain the 12% moisture content prior to board production. Two cementious materials used were Ordinary Portland cement (OPC) and rice husk ash (RHA) pozzolan. The OPC was purchased from a local cement distributor in Akure. The pozzolan was produced from rice husk (RH), which was collected from a rice mill located at Akure, Ondo State, Nigeria. The RH was processed by air drying prior to conversion to ash. It was ash at 800°C in an enclosed barrel until the entire husk turned to ash (≈ 2 h). After the production of the pozzolan from RH, it was added to OPC at the mixing proportions of 0, 10 and 20% of total OPC (i.e. 100:0, 90:10 and 80:20 of OPC:RHA pozzolan) known as hybrid cement A, B and C.

2.2 Compatibility testing

For the hydration test, 15 g of wood particles, 200 g of hybrid cement (PC:RHA of 100:0, 90:10 and 80:20%) and 93 ml of distilled water were measured and then mixed in a polyethylene bag to form homogenous slurry following the method developed by Adefisan and Olorunisola [14]. The neat cement or hybrid cement was mixed with 90 ml distilled water while $CaCl_2$ which was used as chemical accelerator was added by dissolving it in the distilled water at 3% (by weight of cement). The test is performed in well insulated thermos flasks. The temperature rise is monitored for 24 h using thermocouples (J-type) connected to an 8-channel data logger (USB TC-08, Pico Technology). Three specimens of each mixture were tested. The compatibility of Obeche wood particles with the hybrid cement (combination of OPC and RHA pozzolan) was accessed based on the compatibility indices namely maximum hydration temperature (T_{max}), setting time to reach maximum hydration temperature (t_{max}), time ratio index (t_R) and inhibitory index (I, %) developed by Sandermann and Kohler [15], Hofstrand *et al*. [16], Olorunnisola [17] and Weatherwax and Tarkow [18], respectively. Among these compatibility indices, only t_R and I base suitability judgment on the relationship that exists between the time to reach the maximum temperature of wood-cement-water system and neat cement. The equations for calculating the t_R and I developed by Olorunnisola [17] and Weatherwax and Tarkow [18] as expressed in equations 1 and 2, respectively were employed in this study:

$$\text{Time ratio index}, t_R = \frac{t_{max\_wc}}{t_{max\_nc}} \quad (1)$$

$$\text{Inhibitory index}, I = \frac{t_{max\_wc} - t_{max\_nc}}{t_{max\_nc}} \times 100 \quad (2)$$

where
t_{max_wc} = time required for wood-hybrid cement-water system to attain its maximum hydration temperature;
t_{max_nc} = time required for the uninhibited Portland cement (no pozzolan) to reach its maximum hydration temperature.

3. Result and discussion

The hydration curves of the mixes of Portland cement, rice husk ash pozzolan and wood particles of Obeche which was conducted in order to determine its suitability for cement bonded composites production are shown in Figures 1 and 2. Various hydration parameters like maximum hydration temperature (T_{max}), maximum setting times (t_{max}) and time ratio (t_r) were calculated from Figures 1 and 2.

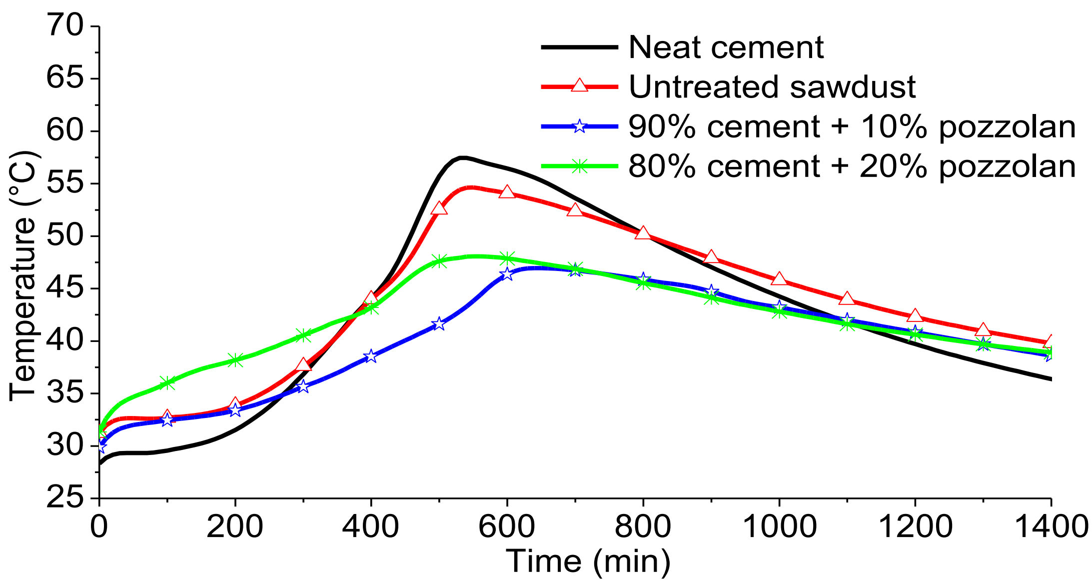

Figure 1. Exothermic reactions (temperature versus time) for neat cement and untreated *Triplochiton scleroxylon* wood- hybrid cement (ordinary Portland cement and rice husk ash pozzolan)-water systems.

3.1 Maximum hydration temperature

From Table 1, the maximum hydration temperature (T_{max}) for untreated wood particles/'hybrid cement A' mix, untreated wood particles/'hybrid cement B' mix and untreated wood particles/'hybrid cement C' mix were 54.5, 46.9 and 48.1°C, respectively. For the 3% $CaCl_2$ treated wood particles, treated wood particles/'hybrid cement A' mix, treated wood particles/'hybrid cement B' mix and treated wood particles/'hybrid cement C' mix were 63.3, 56.2 and 53.4°C, respectively.

Sandermann and Kohler [15] already developed three suitability classes based on maximum hydration temperature that any lignocellulosic materials with T_{max}<50°C, T_{max}=50-60°C and T_{max}>60°C are unsuitable, intermediately suitable and suitable, respectively for CBC production. Therefore, untreated wood particles/'hybrid cement A' is intermediately suitable while untreated wood particles/'hybrid cement B' and untreated wood particles/'hybrid cement C' are not suitable for the production of CBC. However, treated wood particles/'hybrid cement A' is suitable while treated wood particles/'hybrid cement B' and treated wood particles/'hybrid cement C' are intermediately suitable for the production of CBC.

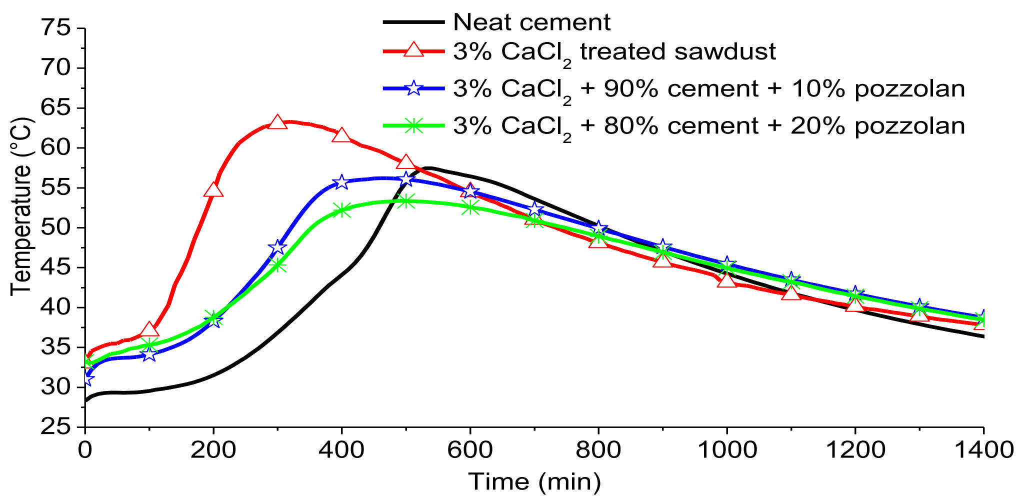

Figure 2. Exothermic reactions (temperature versus time) for neat cement and treated *Triplochiton scleroxylon* wood- hybrid cement (ordinary Portland cement and rice husk ash pozzolan)-water systems.

These findings revealed that incorporation of RHA pozzolan to the OPC would reduce its compatibility with both untreated and treated Obeche wood particles on the basis of maximum hydration temperature. However, the compatibility of the hybrid cement and wood particles improved with the incorporation of $CaCl_2$. This is not surprising because $CaCl_2$ has been identified as a good accelerator that helps cement setting [19]. The practical implication is that the hybrid cement at 10 and 20% RHA pozzolan incorporation into the OPC are suitable for CBC production. For both untreated and treated Obeche wood, increase in the pozzolan proportion caused decrease in the T_{max}. According to Zhou and Kamdem [20], the T_{max} reduction may be caused by a reduced value of cement hardening or by the presence of a determined mass of lignocellulosic material which did not contribute to heat generation but, on the contrary, absorbed it. In this study, the possible reason could because of the reduced value of cement hardening since same mass of wood was used.

Oyagade [21] explained that chemical composition of cement which may vary from country to country and from one manufacturer to the other dictates the maximum hydration temperature attainable. Therefore, absolute comparison of wood-cement-water systems solely on the maximum hydration temperature for compatibility classification by researcher who did not purchase cement from the same source where Sandermann and Kohler [15] bought their own may be misled. More importantly, judging the suitability of hybrid cement (combination of OPC and RHA pozzolan) based on maximum hydration temperature alone may not be enough reason for rejecting it since incorporation of RHA pozzolan reduced the maximum hydration temperature relative to control (0% RHA pozzolan). Heat is needed for bond formation and at low temperature, the composite mix may not form strong bond [22]. Practically speaking, the maximum hydration temperature for an admixture of cement, lignocellulosic material and water may be equal or greater than 40°C and still be suitable for CBC production [23]. This implies that it should not be a hard rule to set minimum allowable maximum hydration temperature to above 50°C suggested by Sandermann and Kohler [15].

3.2 Setting time to reach maximum hydration temperature

The maximum setting times (time to reach maximum temperature, t_{max}) for the untreated wood particles/'hybrid cement A', untreated wood particles/'hybrid cement B' and untreated wood particles/'hybrid cement C' were 9.2, 10.9 and 9.4 h, respectively. For 3% $CaCl_2$ treated wood particles, the maximum setting times were 5.3, 7.7 and 8.2 h for treated wood particles/'hybrid cement A', treated wood particles/'hybrid cement B' and treated wood particles/'hybrid cement C', respectively. Increase in the RHA pozzolan proportion in the hybrid cement caused increase in the maximum setting time for the untreated and treated wood particles. Based on Hofstrand *et al.* [16] who classified suitability index into two (suitable when t_{max}<15 h) and unsuitable when t_{max}> 20 h), all the hybrid cement with untreated and treated Obeche wood particles were suitable for the production of CBC. Unlike maximum hydration temperature classification of wood-cement-water system for CBC suitability, maximum setting times revealed that increasing the proportion of RHA pozzolan in the hybrid cement enhance considerable improvement on their suitability both for untreated and treated wood particles.

The use of maximum hydration temperature or setting time alone to judge suitability of raw material for cement bonded composite production without reference to the degree of exothermic reaction attainable by the wood-cement-water system may be misleading [21]. Therefore, the most likely appropriate quantitative measures for determining suitability of wood with cement are time ratio and inhibitory index.

Table 1. Hydration parameters of *Triplochiton scleroxylon* wood - cement (Portland cement and rice husk ash pozzolan)-water systems.

Pre-treatment	Setting time max. (t_{max}, h)	Remark	Max. temp. (T_{max}, °C)	Remark	Time ratio index (t_R)	Remark	Inhibitory index (*I*)	Remark
Neat cement	9.0±0.0	S	57.3±1.1	MS	-	-	-	-
0%Pozzolan	9.2±0.0c	S	54.5±0.7bc	MS	1.0±0.0c	S	2.2	LI
10%Pozzolan	10.9±0.1a	S	46.9±0.2d	U	1.2±0.0a	S	21.2	MI
20%Pozzolan	9.4±0.1b	S	48.1±1.1d	U	1.0±0.0c	S	4.4	LI
3%$CaCl_2$	5.3±0.0f	S	63.3±1.6a	S	0.6±0.0e	S	-41.2	LI
10%Pozzolan + 3$CaCl_2$	7.7±0.0e	S	56.2±1.2b	MS	0.9±0.0d	S	-14.4	LI
20%Pozzolan + 3$CaCl_2$	8.2±0.0d	S	53.4±0.6c	MS	1.1±0.0b	S	-8.9	LI

LI = low inhibition, MI = medium inhibition, MS = moderately suitable, S = suitable and U = unsuitable

3.3 Time ratio index

The time ratio (ratio of setting time of wood/cement mix to neat cement, t_R) for untreated and treated wood particles/hybrid cement/water mixes are presented in Table 1. The time ratio for the untreated wood particles/'hybrid cement A', untreated wood particles/'hybrid cement B' and untreated wood particles/'hybrid cement C' were 1.0, 1.2 and 1.0, respectively. The highest time ratio was observed with 10% RHA pozzolan in the hybrid cement when mixed with untreated wood particles. Time ratios for 3% $CaCl_2$ treated wood particles were 0.6, 0.9 and 1.1 for treated wood particles/'hybrid cement A', treated wood particles/'hybrid cement B' and treated wood particles/'hybrid cement C', respectively. Time ratio increased with increase in the proportion of the RHA pozzolan in the hybrid cement when mixed with treated wood particles. For the 3% $CaCl_2$ treated wood particles, increase in the proportion of the pozzolan by 20% of the hybrid cement caused increase in the time ratio from 0.6 to 1.1. The results of hydration testing for RHA pozzolan confirmed good reactivity between OPC and

RHA pozzolan used in this study. For classification of lignocellulosic materials' suitability with cement mix in relation to time ratio, Olorunnisola [17] divided them into three groups. Suitable, acceptable and inhibitory are those with $1 \leq t_R \geq 1.5$, $1.5 < t_R \geq 2.0$ and $t_R > 2.0$, respectively. Based on this classification, all the hybrid cement and Obeche wood particles mixes are suitable for CBC production. Similar to the maximum setting times classification of wood-cement-water system for CBC suitability, ratio of setting time of wood: cement mix to neat cement revealed that increasing the proportion of RHA pozzolan in the hybrid cement contribute significantly to the improvement of their suitability both for untreated and treated wood particles.

3.4 Inhibitory index

The inhibitory indices calculated for the untreated and treated wood mixed with the hybrid cement and water under investigation are presented in Table 1. The inhibitory indices ranged from 2 to 21 and -41 to -9 for the untreated and treated wood particles, respectively. The compatibility between Obeche wood particles and hybrid cement was classified in accordance with Okino *et al*. [24]. When the inhibitory index, $I \leq 10$, $10 < I \leq 50$, $50 < I \leq 100$ and $I > 100$, such raw material is graded as having low, medium, high and extreme inhibition, respectively [24]. Comparing the inhibitory indices obtained in this study with the values assigned to different inhibitory indices graded by Okino *et al.* [24], the mixtures of the hybrid cement with the untreated and treated wood particles are highly suitable for CBC production The inhibitory index results showed that chemical treatment of Obeche wood approach is not necessary in the production of well consolidated CBCs from the hybrid cement.

Conclusion

From this study, incorporation of rice husk ash pozzolan at certain proportions of ordinary Portland cement when mixed Obeche wood particles showed that pozzolan supplementary is suitable for cement bonded composites (CBCs) production based on hydration testing conducted. The classification of the suitability of the hybrid cement for the production of CBC based on the maximum setting times and ratio of setting time of wood: cement mix to neat cement are more favourable than using the maximum hydration temperature. Both the maximum setting times and ratio of setting time of wood: cement mix to neat cement revealed that increasing the proportion of RHA pozzolan in the hybrid cement enhance considerable improvement on their suitability both for untreated and treated wood particles. Inhibitory index showed that any proportion (at least 0 to 20%) of RHA pozzolan in the hybrid cement is highly suitable for the production of CBCs whether Obeche wood is treated not untreated.

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