Carbon Neutral Off-White Rice Husk Ash as a Partial White Cement Replacement

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Abstract: Large amounts of rice husk ash (RHA) are produced every year worldwide, and difficulties related to its disposal may cause this product to become an environmental hazard. Owing to its high amorphous silica content, RHA has shown to be a valid supplementary cementing material in the production of concrete. This paper presents the physical and chemical properties of a new generation RHA that is off-white in color, is carbon neutral, and has no crystalline SiO_2 and toxic metals. The effects on mechanical properties of a mixture using off-white rice hull ash (OWRHA) as partial replacement of white cement were also investigated. The OWRHA-blended concrete has higher compressive and splitting tensile strengths at various ages compared with the control mixture. It is shown that up to 15% of OWRHA could be advantageously blended with white cement to enhance white concrete performance without modifying the aesthetics of the final product.

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Introduction

Rice husk, as an agricultural residue, constitutes the largest (about 20%) and most worthless by-product of the rice milling industry. About 70-million tons of rice husk ash (RHA) is produced annually worldwide (FAO 2009), and since the husk does not biodegrade or burn easily (Beagle 1978), it is considered as an environmental nuisance. When burned, the ash of rice husk contains the highest proportion of silica among all the plant residues, nearly 20% silica in amorphous form (Nair et al. 2008). Under controlled combustion conditions, this by-product can produce amorphous silica with high reactivity [P. K. Mehta, "Siliceous ashes and hydraulic cements prepared therefrom," U.S. Patent 4105459 (1978)]. Spire et al. (Spire et al. 1999) studied the properties of mortars containing different percentage of RHA and noted not only an increase in compressive strength but also a reduction in water absorption characteristics and in oxygen permeability. Cost reduction, performance, durability, and environmental concerns are the primary characteristics that can make RHA a valid alternative to partially substitute Portland cement (Mehta 1979; Jauberthie et al. 2000; Ganesan et al. 2008).

Until the 1970s, production of RHA was by uncontrolled combustion, and for this reason the product was usually crystalline and poor in pozzolanic properties (Mehta 1992). After Mehta [P. K. Mehta, "Siliceous ashes and hydraulic cements prepared therefrom." Belgium Patent 802909 (1973)] described the effect of pyroprocessing parameters on the pozzolanic reactivity of RHA, Pitt [N. Pitt, "Process for preparation of siliceous ashes." U.S. Patent 3959007 (1976)] designed a fluidized-bed furnace for controlled combustion of RHA. Controlling temperature and atmosphere, a highly reactive RHA was obtained. Today, RHA generated by the processes that are on the market contains 3% or more with graphitic carbon which determines the dark pigmentation of the material, restricting its utilization in architectural applications where color is the driver, and leads to excessive demand from water and chemical admixtures in order to maintain appropriate slump and air content (Zhang et al. 1996; Yu et al. 1999). There have been some attempts to produce amorphous SiO_2 from rice husk using different techniques, but none of these methods has successfully produced commercial-scale off-white amorphous SiO_2 with less than 1% amorphous carbon (Vempati et al. 2006; Boateng et al. 1991; Savant et al. 1996). Vempati et al. (2006) developed a new continuous production process of manufacturing RHA in which the rotary tube furnace was maintained in aerobic conditions and temperature at 700°C with a residence time of 40 min to obtain off-white RHA with a carbon content of less than 0.3%. This carbon neutral ash is off-white color with no graphitic carbon, no crystalline SiO_2 and toxic metals, and therefore considered environmentally friendly. This process generates 17 MJ/kg of rice husk of energy which could be used to power other industrial processes. The energy generation and the reduction of carbon in Portland cement concrete are not the only reasons that make this material sustainable. Based on the U.S. Green Building Council certification practice, the LEED Green Building Rating System for New Construction and Major Renovation considers that the reflective quality of white surfaces may help to improve lighting efficiency and reduce temperature fluctuations,

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Table 1. Chemical Composition (% by Mass)

Element composition	White Portland cement type I	OWRHA (600°C for 180 min)
SiO_2	23.06	94.8
Al_2O_3	4.46	0.52
Alkalis	2.37	2.92
C	—	0.24
P_2O_5	—	1.09
Fe_2O_3	0.25	0.13
MnO	—	0.39
CaO	66.6	—
SiO_3	3	—
MgO	0.26	—

resulting in lower heating and cooling of the structure and related energy costs. The off-white rice husk ash (OWRHA) can be combined with white cement to obtain off-white concrete for sustainable constructions.

The objective of this paper is to provide information on the utilization of the OWRHA as a partial cementitious binder substitute with reference to pozzolanic activity and strength of hardened concrete. In order to evaluate the favorable percentage of replacement of white Portland cement (WPC) with OWRHA, different mixtures were tested. The results are also compared to experimental values of RHA available in the literature.

Experimental Work

The study was developed in two phases. In the first phase, physical and chemical properties of OWRHA were studied in order to define the pozzolanic activity of the material produced using the procedure developed by Vempati et al. (2006). The investigation included X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, thermogravimetric analysis (TGA), and scanning electron microscopy (SEM). In the second phase, studies on concrete specimens were conducted. This phase was mainly concerned with the effect of different OWRHA dosage in varied curing periods on the compressive and splitting tensile strength.

Phase I

Materials

OWRHA OWRHA was manufactured by burning rice husk procured from a rice mill plant located at Jonesboro, Ark. The continuous process of manufacturing off-white RHA with <0.3% amorphous carbon is summarized as follows: a rotary tube furnace was used which was maintained in an aerobic condition and temperature set at 700°C, and the residence time of the rice hulls in the furnace was 40 min. At this stage of the project, unground OWRHA was used after determining that its particle size was comparable to that of WPC. Generally, reactivity of cementitious materials is also favored by increasing the fineness of the pozzolanic material (Kiattikomol et al. 2001; Paya et al. 1995, 1997); however, Mehta [Mehta P. K. (1978). "Siliceous ashes and hydraulic cements prepared therefrom." U.S. Patent 4105459.] demonstrated that a high degree of fineness of RHA's grinding should be avoided since this material gains its pozzolanic activity mainly from the internal surface area of the particles. The main chemical components of OWRHA as used in this project are summarized in Table 1. The total percentage composition of iron oxide (Fe_2O_3 =0.13%), silicon dioxide (SiO_2=94.8%), and aluminum oxide (Al_2O_3=0.52) was found to be 95.45%. The value is considerably above the required value of 70% minimum for pozzolans (ASTM C 618).

XRD

The mineralogy of OWRHA was obtained with a Philips X'pert System diffractometer (Philips Electrical Co., Almelo, Netherlands). Using about 150 mg, the XRD patterns were recorded with a diffraction angle of 5–40°2θ at a scan rate of 3° min^{-1} using Ni-filtered Cu Kα (1.584 Å) radiation. The X-ray amorphous diffraction pattern (Fig. 1) showed a pattern which is typical for amorphous solids (Kamath and Proctor 1998). The broad smooth hump between 15°2θ and 35°2θ indicates that pyrolysis converted the ordered crystalline cellulose structure into a random amorphous structure. Previous studies have shown that RHA with most of its silica in an amorphous form, as in this case, is very reactive and can considerably improve the strength and durability of concrete (Nair et al. 2008).

FTIR

OWRHA was characterized by infrared spectroscopy. The FTIR spectrum of OWRHA was collected in the transmission mode using a Perkin Elmer 2000 (Perkin Elmer, Cambridge, Mass.). Three hundred milligrams of KBr was mixed with 6 mg of the sample, and 128 scans were collected and averaged. The FTIR

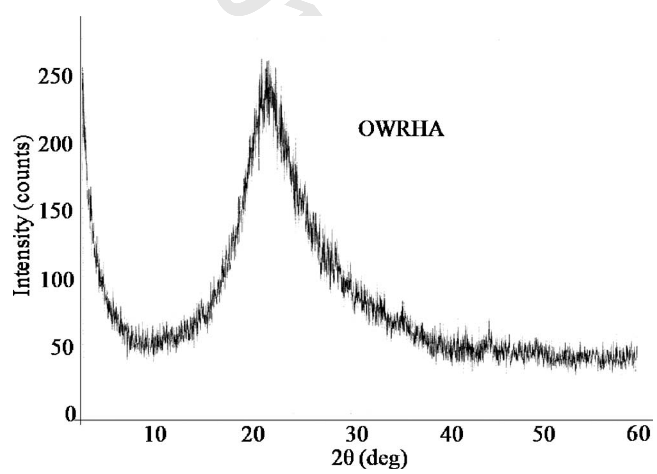

Fig. 1. X-ray diffractogram of OWRHA

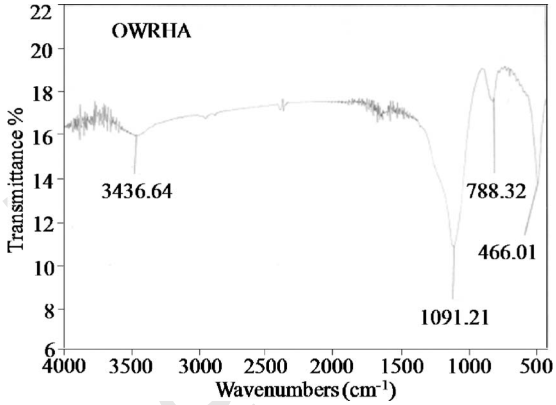

Fig. 2. FTIR of OWRHA

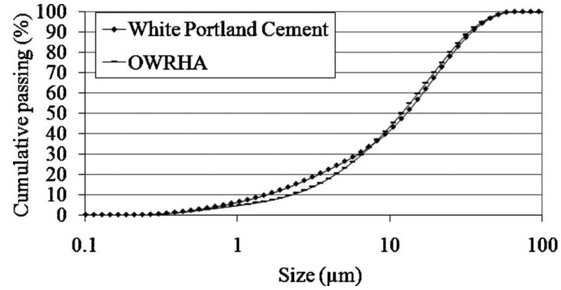

Fig. 4. Particle size distribution of OWRHA and WPC

spectrum of OWRHA is presented in Fig. 2. The FTIR study showed the presence of -O-Si-O-vibrations attributable to asymmetrical stretching bands which occurred around 1,220 and 1,080 cm^{-1} in the form of shoulder and broad bands, respectively. Symmetrical stretching band occurred at 773 cm^{-1}, and Si-O bending band at 466 cm^{-1}. Free water band was observed at 3,436 cm^{-1}. The position of these bands confirmed that the material was amorphous SiO_2.

Thermogravimetry

TGA of OWRHA was calculated using a TGA-HP (TA Instruments, New Castle, Del.). The analysis was carried out by heating the sample from ambient to 950°C at a rate of 20°C/min while under a nitrogen atmosphere. The total weight loss over the temperature range was then calculated using Universal Analysis software (TA Instruments, New Castle, Del.). The TGA indicated that the loss of ignition of OWRHA is ~0.03% (Fig. 3), which demonstrates the absence of residual combustible carbon, reducer of the pozzolanic activity.

Particle Size Distribution

Particle size distribution of WPC and OWRHA was determined using a laser diffraction particle size analyzer Malvern (Malvern Instruments, Worcestershire, U.K.). The particle size distribution curves shown in Fig. 4 suggest that WPC and OWRHA have comparable particle size distribution. The particle density was 3.15 g/cm^3 for both materials, and the specific surface area is 0.43 m^2/g for OWRHA and 0.49 m^2/g for WPC. Previous study showed that the high fineness of RHA had a greater pozzolanic reaction, and the small particles could also fill in the voids of the mortar mixture, thus increasing the compressive strength and durability of the mortar (Rukzon and Chindaprasirt 2006; Rukzon et al. 2009). Therefore, if unground OWRHA can increase the properties of the concrete, then finer particles (higher grinding time) may further improve the final product.

SEM

The SEM analysis was performed on WPC and OWRHA to study the microgeometry of the material. The particles were imaged using environmental SEM (ESEM) system (Philips XL30 ESEM-FEG, Philips Electrical Co., Almelo, Netherlands). Scanning electron micrographs of the material were taken in the variable pressure secondary electron mode with the following instrument parameters: electron beam energy and probe current were 5 kV and 170 pA, respectively, with a variable working distance. The SEM image reveals that OWRHA consists of very irregular-shaped particles with porous cellular surface (Fig. 5) typical of unground RHA (Jauberthie et al. 2003). The basic cellular structure comes from the organic material which OWRHA is derived and is responsible for the high surface area of the material even when the particles are not very small in size (Zhang and Malhotra, 1996).

Phase I Conclusions

From the characterization of OWRHA carried in phase I, it can be stated that this product has proven to be an effective pozzolanic material, with high amorphous silica content and high specific surface area. The effect of OWRHA as supplementary cementitious material for WPC is evaluated in phase II.

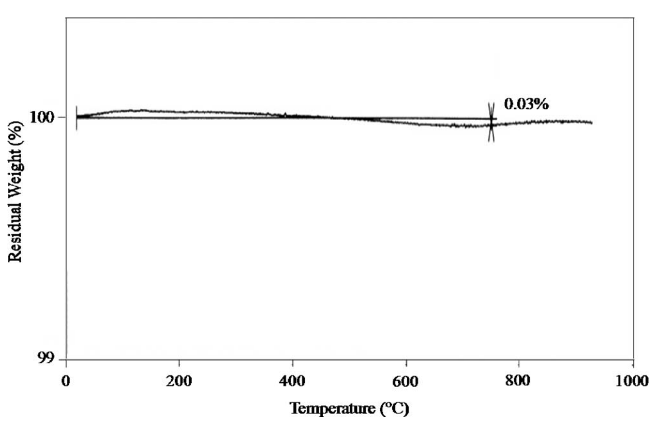

Fig. 3. TGA of OWRHA

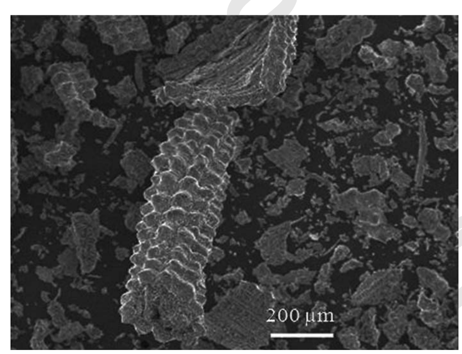

Fig. 5. SEM of OWRHA

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Table 2. Mix Proportions

Mix design	OWRHA (%)	Quantities (kg/m^2) Cement	OWRHA	Sand	Coarse aggregates	Water	Water/cement
W0	0	470	0				
W7.5	7.5	435	35				
W15	15	400	70	1,193	468	211	0.45

Table 3. Average Concrete Compressive Strength of Tested Concrete at Different Ages

		Average compressive strength			Average compressive strength			Average compressive strength		
		MPa	Normalized	COV (%)	MPa	Normalized	COV (%)	MPa	Normalized	COV (%)
Mix Number	Symbol	7 days			28 days			90 days		
1	W0	27.6	100	3	36.1	100	2	38.0	100	0.4
2	W7.5	28.0	101	1	41.0	113	1	44.4	117	1
3	W15	29.2	106	1	41.6	115	1	45.6	120	1

Phase II

Materials

Aggregates Clean river sand with a specific gravity of 2.55 and a fineness modulus of 2.49 was used as a fine aggregate. Locally available well ground limestone of size greater than 4 mm and less than 12 mm, with a specific gravity of 2.6, was used as a coarse aggregate.

WPC White type I Portland cement conforming to ASTM C 150-07 was used. The cement had a specific gravity of 3.15 and a fineness of 408 m^2/kg. The main chemical components of the WPC used are presented in Table 1.

Superplasticizer High-range water reducing admixture (Adva 140M, Grace Construction Product, Frederick, Md.) was used to maintain a constant workability expressed as a constant slump without any additional amount of mixing water and without any direct effect on the compressive strength of the concrete. This superplasticizer is based on polycarboxylate technology and meets the requirements of ASTM C494 as types A and F and ASTM C1017 type I.

Following the industry practice, a typical slump for ordinary decorative application would be in the 100–130-mm range, and this value was adopted as the target in the study of the different mixtures tested.

Mix Proportion and Casting of Concrete Specimens

For the mechanical characterization of the concrete, three mixtures with different WPC/OWRHA proportions, including the control one, were prepared and tested. The water to binder ratio of 0.44 was maintained constant in order to focus on the effect of OWRHA. Different OWRHA contents of 0, 7.5, and 15% by mass of cement replaced an equal weight of cement in the mix. Blended cements were prepared by replacing WPC with OWRHA in dry conditions. The mixtures were thoroughly homogenized and kept in polyethylene containers. The three batches were

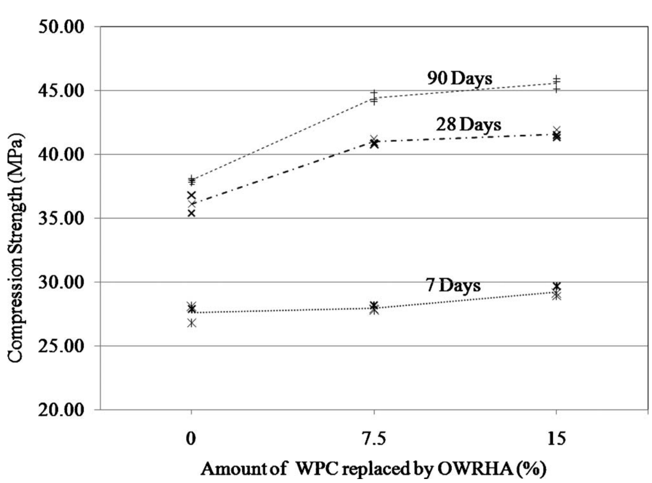

Fig. 6. Compressive strength (megapascals) versus percentage of OWRHA at different ages

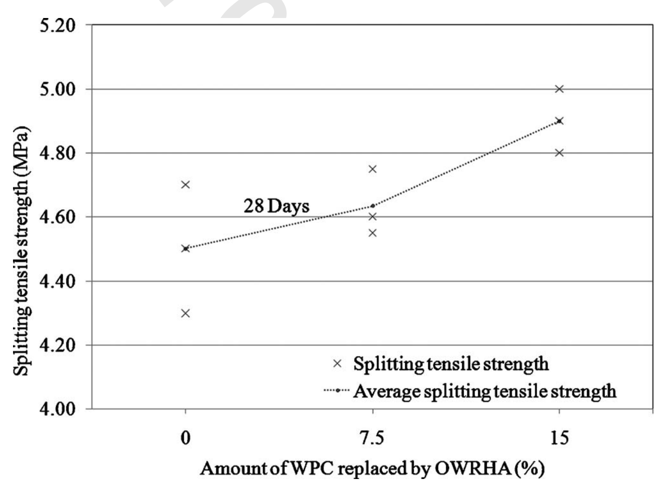

Fig. 7. Splitting tensile strength (megapascals) versus percentage of OWRHA at 28 days

Table 4. Average Splitting Tensile Strength of Tested Concretes at 28 Days

		Average splitting tensile strength		
		MPa	Normalized	COV (%)
Mix Number	Symbol	28 days		
1	W0	4.5	100	4
2	W7.5	4.6	103	2
3	W15	4.9	109	1

coded as W0, W7.5, and W15, where the digits represent the percentage of OWRHA in the blended cement. The mix proportions are presented in Table 2.

Eighteen cylinders of 100-mm diameter and 200-mm height were cast from each batch and used for compressive strength testing (9) and splitting tensile strength testing (9). For each batch and age three cylinders were tested in compression and three in splitting. Even though the sample size is limited and does not allow for complete statistical analysis and generalization, it remains valid for relevant considerations on properties trends. The specimens were cast, compacted, and cured according to the ASTM C 192. Compressive strength was determined as per ASTM C39M after 7, 28, and 90 days of moist curing at 20°C and relative humidity (RH) of 95%. Splitting tensile strength tests were conducted as per ASTM 496M after 28 days of moist curing at 20°C and RH of 95%. A SATEC MKIII-C with 100,000-psi capacity was used for both tests.

Mechanical Properties

Table 3 shows the average compressive strength of concrete mixed with different percentages of OWRHA (0, 7.5, and 15% by weight) at different ages. Fig. 6 shows the data of each sample tested, and the average value is reported with a dashed line. The compressive strength of OWRHA-blended concrete containing 7.5 and 15% is comparable and higher than the control mix at all ages.

The test data indicate that the splitting tensile strengths of control mix and concrete incorporating OWRHA are comparable (Fig. 7). As reported in Table 4, at 28 days, the 15% OWRHA-blended concrete shows an increase in the splitting tensile strength of ~9% with respect to the control concrete. Only very low increase (~2%) was demonstrated for the 7.5% blend. Table 5 shows normalized experimental values for compression and tensile strength of rice hull ash (RHA) blended concrete in the literature (Saraswathy and Song 2007; Ganesan et al. 2008; Sakr 2006; Zhang and Malhotra 1996; Rodríguez de Sensale 2006). The literature was selected by choosing mix design with about the same w/c ratio (~0.4) and the same percentage of replaced cement of the one tested in this research, but with a high-*r* carbon content (-CC) in RHA. For the batch W7.5 it was not possible to find any previous work with the same percentage of replacement, so the comparison was made using 10% RHA blended cement. The comparison shows that white cement, blended with OWRHA, has mechanical properties comparable to concrete made with RHA with carbon content higher than 0.03.

Conclusions

From the results obtained the following can be concluded:

1. Because of the high amorphous silica and specific surface area, OWRHA is an effective pozzolanic material, employable as supplementary cementing material.
2. A percentage of WPC as high as 15% by weight can be replaced with OWRHA without any adverse effect on strength properties.
3. The compressive strength of OWRHA-blended white concrete increases with cement replacement percentage and specimen age.
4. The comparison of the results obtained from OWRHA-blended white concrete with literature related to RHA demonstrates that the compressive and splitting tensile strengths are not negatively affected by using this by-product.

Future Studies and Recommendations

For manufacturing off-white rice hull ash, a rotary tube furnace was used which was maintained in an aerobic condition and temperature set at 700°C, and the residence time of the rice hulls in the furnace was 40 min. Because of its whiteness, OWRHA can be combined with white cement and used for architectural concrete applications. Additional studies related to durability, color, and reflectivity of the white concrete are needed. Ongoing efforts at the University of Miami are addressing these additional research needs.

Table 5. Comparison of the Results of the Present Study with Experimental Values of RHA in the Literature

		Normalized compressive strength			Normalized splitting tensile strength
% of cement replaced with RHA	Investigator	7 days	28 days	90 days	28 days
10	Saraswathy and Song (2007)	112	103	—	104
	Ganesan et al. (2008)	103	111	117	103
	Sakr (2006)	108	111	114	—
	Zhang and Malhotra (1996)	108	106	111	130
	Rodríguez de Sensale (2006)	—	—	—	102
7.5	Present study	101	113	117	103
15	Saraswathy and Song (2007)	116	103	—	110
	Ganesan et al. (2008)	108	113	119	109
	Sakr (2006)	118	112	115	—
	Zhang and Malhotra (1996)	—	—	—	—
	Rodríguez de Sensale (2006)	—	—	—	—
	Present study	106	115	120	109

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