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Material Characterization Studies on Low- and High-Carbon Rice Husk Ash and Their Performance in Portland Cement Mixtures

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ABSTRACT: In this study, low- and high-carbon rice husk ash (RHA) in their as-received and ground forms were characterized by means of different methods in order to evaluate their performance in Portland cement mixtures. RHA-cement pastes and mortars, at three different RHA replacement levels of 0 %, 10 %, and 20 %, were prepared at a constant water/cement ratio of 0.485. Results from this investigation indicate that the material characteristics of low- and high-carbon RHA were significantly different in most of the tests conducted. In the as-received condition, the high-carbon RHA had a greater bulk density than low-carbon RHA, but their bulk densities were comparable after grinding. The low-carbon RHA was more effective in its pozzolanic reaction than high-carbon RHA in both ground and as-received conditions. The microstructures of both lowcarbon and high-carbon RHA cement pastes were denser than those of control pastes. At a given dosage of RHA and superplasticizer, the ground and the as-received low-carbon RHA mixtures performed significantly better than the high-carbon RHA mixtures. Similarly, mixtures with low-carbon RHA showed significantly higher strength activity indices than those with high-carbon RHA at both dosage levels investigated. Grinding RHA was found to be beneficial in all the tests conducted, as the ground RHA mixtures depleted more calcium hydroxide, registered higher flow values, and possessed greater strength than unground RHA mixtures at all replacement levels. Thus, the grinding process significantly helps in utilizing both high- and low-carbon RHA in concrete.

KEYWORDS: low-carbon rice husk ash, high-carbon rice husk ash, sustainability, calcium hydroxide depletion

Introduction

Rice husk ash (RHA), like other pozzolans such as fly ash, silica fume, slag, etc., has been identified by many investigators as important for promoting sustainability in concrete [1–5]. This is primarily because of the beneficial use of large quantities of agro-waste material ($\sim 6.7 \times 10^6$ tons of rice husks as of 2009 [6]) to partially replace cement for construction. RHA is produced by burning rice husks, typically at high temperatures, for specified durations. When the incineration temperature or duration is excessive, RHA containing crystalline silica and a high graphitic carbon content. This happens most often with the open heap burning of rice husks or for use in power plants,

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where, as there is no proper control of the temperature, the duration of combustion, or the ambient atmospheric conditions [1]. Open heap burning is also prohibited in many developed countries because it produces objectionable gases resulting in environmental pollution and other problems related to human health [1,2].

During the past three decades, different techniques have been developed to incinerate rice husks in a controlled atmosphere; among them are the fixed bed reactor [5], fluidized bed reactor [2–4], torbed reactor [7], and rotary tube furnace [8] techniques. Of these techniques, only a few were successful in producing a low-carbon RHA economically and through an environmentally friendly process [7,8]. Temperatures between 500°C and 900°C have been employed by researchers to produce amorphous silica in RHA with a low carbon content [7,9–12]. Though some research conducted in the past has suggested that temperatures above 700°C yield crystalline RHA [9,10], other studies have shown that amorphous silica can be produced even at high temperatures of up to 800°C if a shorter incineration duration is used [7,12]. A recent study clearly demonstrated that amorphous silica can be produced from RHA by incinerating rice husks at low temperatures for a longer duration or at high temperatures for a shorter duration [13]. The temperature and duration used during RHA combustion have been found to significantly change the carbon content, and the nature of silica present in RHA, thereby changing the material characteristics of RHA.

The oxide compositions of RHA obtained by different researchers are shown in Table 1. As this table shows, silica is the principle oxide present in RHA, the quantity of which varies typically from 80 % to 95 %. Past research has shown that the quality and quantity of silica in RHA can significantly alter the properties of concrete [9,16,34]. In addition to silica, unburned carbon is typically present in RHA in quantities ranging from <1 % to 7.67 %. The unburned carbon imparts a dark color to RHA when present in significant amounts [35,36]. From the investigations reported previously, the color of RHA has been found to change substantially from black to gray or gray to white when its carbon content is approximately below 2.70 %, and thus this limit may be considered for distinguishing between high- and low-carbon RHA [7,9,14–33]. In addition, the carbon content or loss of ignition value of RHA can be expressed as an inverse function of its silica content as shown in Figs. 1(*a*) and 1(*b*); a decrease in the carbon content of RHA results in an increase in its silica content. This might be because carbon is the principle oxide present in rice husks and tends to remain unburned in the ash or escapes into the atmosphere after incineration, depending upon the combustion methods and parameters used.

Significant research has been conducted in the past with high-carbon RHA and low-carbon RHA in their ground forms, but little is known about the influence of coarser, unground RHA in Portland cement mixtures. Thus there is a need to perform detailed investigations to establish the limitations associated with using unground RHA in Portland cement mixtures. Moreover, the beneficial effects of grinding RHA on specific properties of mixtures containing RHA are not thoroughly understood or quantified, and it is extremely important that knowledge of these effects be obtained before the use of RHA in Portland cement concrete is promoted.

In this study, detailed characterization studies were conducted with RHA obtained from two different incineration processes: (a) a controlled combustion process developed by Vempati et al. [8], which produced low-carbon RHA, and (b) the uncontrolled combustion of rice husks in an agrielectric power plant, which produced high-carbon RHA. A comparative investigation of the performance of the unground and as-received low- and high-carbon RHA as a supplementary

Researcher(s)	Reference	Oxide Composition by Mass, %								
		SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	Na ₂ O	Total Alkalis, Na ₂ O _{eq}	Carbon	LOI
Mehta, 1977	[9]	80-95	_	_	_	1.00-2.00	_	_	5.91	8.55
Cook and Suwanvitaya, 1983	[14]	79.50				3.75	0.07	3.80		14.30
Al-Khalaf and Yousif, 1984	[15]	85-88	1.12-1.43	0.36-0.47	0.16-0.19	3.76-4.10	1.05-1.22	4.60	_	1.89-3.49
James and Rao, 1986	[16]	95	0.441	_	0.713	1.048	0.415	1.32	<1	_
Hwang and Wu, 1989	[17]	88-95.40	1.16-2.56	_	0-0.74	1.30-6.50	0.70 - 1.10	_	_	_
Boetang and Skeete, 1990	[18]	88.04-94.55	0.06-0.91	_	_	0.62-2.81	0.04-0.30	_	2.71-7.67	2.87-8.11
Ikpong and Okpala, 1992	[19]	82.13	0.16	4.27	0.38	0.14	1.23	0.95	_	8.60
Zhang et al., 1996	[20]	87.20	0.55	0.15	0.16	3.68	1.12	4.42	5.91	8.55
Ismail and Waliuddin, 1996	[21]	80	3.82	3.93	0.41	1.45	0.67	1.89	_	8.56
Qijun Yu et al., 1999	[22]	91.90	0.38	0.25	0.41	2.78	0.05	2.81	0.41	2.93
Sensale and Molin, 1999	[23]	93	0.59	0.3	0.06	3.4	0.62	3.81	_	2.04
Sugitha et al., 1999	[24]	91.90	0.38	0.25	0.41	2.78	0.05	2.81	0.41	2.93
Nehdi et al., 2003	[7]	89-94.60	0.40-0.60	0.10-0.50	0.04-0.30	1.00 - 1.60	0.20 - 1.00	_	1.00-5.10	1.80-7.00
Mahmud et al., 2004	[25]	88.82	0.67	0.46	0.67	2.91	0.12	2.99	_	4.81
Santos, 2005	[26]	95.85	0.35	_	0.42	3.15	_	_	_	_
Bui et al., 2005	[27]	86.98	1.40	0.84	0.73	2.46	0.11	2.53	_	5.14
Sensale, 2006	[28]	87.20	0.55	0.15	0.16	2.20	0.70	2.66	_	8.10
Sakr, 2006	[29]	87	2.50	1.75	2.50	2.50	_	_	_	_
Gastaldini et al., 2007	[30]	96.26	0.76	0.41	0.22	1.44	0.03	1.46	_	4.49
Saraswathi et al., 2007	[31]	92.95	0.53	0.31	0.26	2.06	0.08	2.11	_	1.97
Giaccio et al., 2007	[32]	87.20	0.55	0.15	0.16	3.60	1.12	4.34	_	6.55
Ganesan et al., 2008	[33]	87	0.48	0.22	0.28	3.14	1.02	3.81	_	2.10

 TABLE 1—Major oxide contents of RHA used by various investigators.

Note: LOI, loss on ignition.

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Note: The number shown within brackets near the data point represent the corresponding published literature from the reference section

FIG. 1-Relationship between carbon content or loss of ignition of RHA and its silica content.

cementing material was also conducted in order to understand the limitations involved in using unground RHA particles. In addition, some tests were performed using ground low- and highcarbon RHA particles in order to understand the benefits of grinding RHA to finer fractions as discussed previously.

Experimental Investigations

Materials and Mixture Proportions

The materials used to prepare cementitious pastes, mortars, and concretes containing RHA included Portland cement, low-carbon RHA (carbon content < 0.54 %), high-carbon RHA (carbon content > 5 %), sand, and coarse aggregate. A high-alkali ASTM C150 [37] Type I cement having an alkali content of 0.82 % NaO_{eq} was used. The chemical compositions of the cement, low-carbon

TABLE 2—Chemical compositions of cement and pozzolan.							
Oxide or Compound	Cement	LC-RHA	HC-RHA				
SiO ₂	19.78	92.46	88.50				
Al ₂ O ₃	4.98	0.26	0.02				
Fe ₂ O ₃	3.13	0.30	0.86				
CaO	61.84	1.63	0.48				
Alkali (Na ₂ O _{eq} , %)	0.82	1.24	1.19				
SO ₃	4.15	0.11	0.02				
MgO	2.54	0.38	0.34				
Carbon	_	0.54	>5.00				
Tri-calcium silicate (C ₃ S)	63.49	_	_				
Di-calcium silicate (C ₂ S)	8.81	_	_				
Tri-calcium aluminate (C ₃ A)	7.96	_	_				
Tetra-calcium alumino ferrite (C ₄ AF)	9.52	—	_				

Notes: LC-RHA, as-received low-carbon rice husk ash; HC-RHA, as-received high-carbon rice husk ash.

	Quantity of Material, g								
Mixture Description	Cement	RHA San		Coarse Aggregate	Water SP		w/cm		
Control	500	0	0	_	242	RQ	0.485		
RHA	500 (1 – <i>x</i> /100)	500 (<i>x</i> /100)	0	_	242	RQ	0.485		
Control	500	0	1375	—	242	RQ	0.485		
RHA	500 (1 – <i>x</i> /100)	500 (<i>x</i> /100)	1375	—	242	RQ	0.485		
	Mixture Description Control RHA Control RHA	Mixture Description Cement Control 500 RHA 500 (1 - x/100) Control 500 RHA 500 (1 - x/100) RHA 500 (1 - x/100)	Mixture Description Cement RHA Control 500 0 RHA 500 (1 - x/100) 500 (x/100) Control 500 0 RHA 500 (1 - x/100) 500 (x/100) RHA 500 (1 - x/100) 500 (x/100)	Quantity of M Mixture Description Cement RHA Sand Control 500 0 0 RHA 500 (1 - x/100) 500 (x/100) 0 Control 500 0 1375 RHA 500 (1 - x/100) 500 (x/100) 1375	Quantity of Material, g Mixture Description Cement RHA Sand Coarse Aggregate Control 500 0 0 - RHA 500 (1 - x/100) 500 (x/100) 0 - Control 500 0 1375 - RHA 500 (1 - x/100) 500 (x/100) 1375 -	Mixture Description Cement RHA Sand Coarse Aggregate Water Control 500 0 0 — 242 RHA 500 (1 – x/100) 500 (x/100) 0 — 242 Control 500 0 1375 — 242 RHA 500 (1 – x/100) 500 (x/100) 1375 — 242	Quantity of Material, g Mixture Description Cement RHA Sand Coarse Aggregate Water SP Control 500 0 0 - 242 RQ RHA 500 (1 - x/100) 500 (x/100) 0 - 242 RQ Control 500 0 1375 - 242 RQ RHA 500 (1 - x/100) 500 (x/100) 1375 - 242 RQ		

TABLE 3—Mixture proportions of paste and mortar.

Notes: SP, superplasticizer; RQ, required quantity to achieve a desired flow; x, percent RHA replacement level for cement.

RHA (LC-RHA), and high-carbon RHA (HC-RHA) are provided in Table 2. The specific gravities of the LC-RHA and HC-RHA were 2.15 and 2.19, respectively. Sand used in this study was natural river sand from a local source with a specific gravity of 2.50. The coarse aggregate used in this study was a granitic gneiss crushed stone from Liberty Quarry in Liberty, SC. A high-range water reducer with a poly-carboxylate base (superplasticizer) was used at a dosage between 0 % and 0.75 % by weight of cementitious material in all the tests except the flow test. In the flow test, the dosage of superplasticizer was varied up to a maximum value of 2.5 % by weight of cementitious material in order for us to understand the limitations involved when using RHA in cementitious mixtures.

The mixture proportions of pastes and mortars used in this study are provided in Table 3. The mixture or powder designation IDs provided for low-carbon unground RHA, low-carbon ground RHA, high-carbon unground RHA, and high-carbon ground RHA were LC-URHA, LC-GRHA, HC-URHA, and HC-GRHA, respectively. All the cementitious mixtures were investigated at a constant water-to-cement (w/cm) ratio of 0.485. The replacement levels of RHA in most of these tests were 0 %, 10 %, and 20 % by mass of cement, but in some tests a 15 % RHA replacement level was used.

Casting and Curing of Test Specimens

The ingredients were weight batched and mixed using a specific procedure similar to that stated in ASTM C192 [38]. Accordingly, a portion of the RHA was added to the mix water along with an adequate amount of high-range water reducer, and the remaining portion of RHA was added to the cement. After each portion of RHA had been mixed separately, the (cement + URHA) mixture was added to the fine aggregates and dry mixed well, and the (URHA + superplasticizer + water) mixture was finally added to this dry mixture. The entire quantity was thoroughly mixed to obtain a wet, homogeneous concrete mixture, which was cast in specimen molds and cured for 24 h. After demolding, the specimens were subjected to moist curing until testing, unless otherwise stated in the description of specific tests.

Experimental Test Methods

In order to study the influence of the carbon content and fineness of RHA on the properties of paste and mortars, a total of ten different tests were conducted, the procedures of which are discussed in this section. For investigations with mortars, tests were performed with three specimens, and the average values are reported.

X-ray Diffraction

X-ray diffraction (XRD) was used as a qualitative tool to identify the crystalline phases in the ashes. The XRD procedure involved grinding the RHA samples to a very fine powder using a Retsch grinding mill and subsequently mounting the powder to a leveled surface in an aluminum holder. A Scintag 2000 system with a germanium detector and a seven-position automatic sample changer was used for powder diffraction. The test parameters included a 2θ angle range from 5° to 70° (Cu K_{α} radiation) with a scan rate of 0.01° per second. The analysis of the peak intensities obtained from the XRD, performed in order to identify the best possible match for a crystalline phase, was done using the Inorganic Crystal Structure Database, NIST Crystal Data File, and Powder Diffraction File electronic databases of the PDML software.

Scanning Electron Microscopy

The microstructures of the RHA powder, paste, and mortar samples were determined using a variable pressure Hitachi S-3400 scanning electron microscope.

Malvern Laser Particle Size Analysis

The particle size distributions (PSDs) of the RHA particles were determined using a Malvern Mastersizer 2000. A small quantity of the powder was dispersed in 10 ml of de-ionized water containing a drop of dispersing agent. The sample was then agitated for 5 min in an ultrasonic bath to disperse the material. The measurements were performed in de-ionized water using a refractive index of 1.55 and an absorption of 0.10. The results in this study were based on the percentage volume passing, and each value represents the average of four measurements.

Retsch Planetary Ball Mill Grinding

The grinding of RHA particles was carried out using a Retsch planetary ball mill (Model PM 200). In order to optimize the grinding regime (speed and duration), the low-carbon RHA was ground in this ball mill using two different grinding speeds, 100 rpm and 250 rpm. At each speed, the grinding time was maintained for 0, 5, 15, 30, and 60 min to obtain RHA particles of different sizes. The average particle size of the ground RHA particles was then determined using the Malvern Mastersizer 2000. Parallel studies were also conducted on as-obtained RHA, which is identified as unground RHA in this paper.

Bulk Density Test

The bulk density of both unground and ground RHA samples was determined by means of weight and volume measurements of loose and vibrated powders, as described below. A cylindrical measure of known weight (W) and volume (V) was filled with RHA, and its weight in loose and vibrated conditions (W_1 and W_ν) was determined. In these studies, a vibrating table with a frequency of 3600 rpm was used. The bulk density (BD) of the samples in the loose and vibrated conditions could then be calculated using the formulas

$$BD_1 = \frac{W_1}{V} \tag{1}$$

$$BD_{\nu} = \frac{W_{\nu}}{V} \tag{2}$$

Packing Density of the Blended Cementitious (Cement + RHA) Powders

The procedure followed to determine the packing density of dry cementitious (cement + RHA) powder was similar to that described previously. After we had measured the weight of the blended sample in the vibrated condition (W_s), the volume of the sample (V_s) was calculated by dividing

 W_s by the specific gravity of the sample. The packing density (PD) of the blended cementitious powders was determined using the following formula:

$$PD = \frac{V_s}{V}$$
(3)

Thermo-Gravimetric Analysis

To ascertain the pozzolanic reactivity of RHA, thermo-gravimetric analysis (TGA) was conducted on RHA cementitious (cement + RHA) pastes at different RHA replacement levels. After curing for a specified duration, the paste was dried and ground in a protected environment to prevent carbonation before being introduced into the TGA unit. The TGA was performed using a Thermal Analysis 2950 Thermo-Gravimetric Analyzer. The samples were uniformly heated from ambient temperature to 800°C at a ramp rate of 10°C/min, with N₂ as a purging gas. The Ca(OH)₂ content was observed between 400°C and 450°C in all the paste samples and was quantified using Thermal Advantage software. The normalized Ca(OH)₂ contents in all the samples were calculated by considering the Ca(OH)₂ content in the control paste samples as 100 %. TGA was conducted on both unground and ground RHA pastes.

Flow Studies

The flow studies on RHA cementitious mortars were conducted based on ASTM C230 [39] and ASTM C1437 [40]. In this method, the wet cementitious mortars were prepared and poured inside the standard mold on the flow table in three layers, with each layer rodded 25 times. The top surface of the filled mold was leveled off, the mold was lifted vertically upward, and the flow-table crank was jolted 25 times in 15 s. The flow diameter was measured in six different directions to determine the average flow diameter from which the percentage flow value was calculated using the specified formula.

Strength Activity Index

The strength activity index (SAI) test was conducted based on ASTM C311 [41] in order to determine the percentage strength-over-control values of RHA mortar cubes (50 mm by 50 mm by 50 mm in size). In this test, the strength value registered by the Portland cement mortar specimen was considered as 100, and the other mixtures containing RHA at specified replacement levels were evaluated with respect to the control. This test was used to determine the effectiveness of RHA as a successful pozzolan, and the ASTM C311 specification for a minimum SAI value of 75 % was used to qualify strength criteria.

Results and Discussion

Characterization of Low- and High-Carbon Rice Husk Ash

The XRD analyses conducted on LC-RHA and HC-RHA particles are shown in Fig. 2. As Fig. 2 shows, the HC-RHA displayed well-defined peaks at 2θ angles of 22° , 27° , and 36° , indicating that the nature of silica in it was crystalline. By using the compound search database of the PDML software, these peaks were found to represent the crystalline phases of cristobalite and quartz. The presence of these crystals in the HC-RHA indicates that the husk had undergone burning temperatures higher than 900°C during uncontrolled combustion [12,16]. In the XRD patterns of LC-RHA, a broad hump centered at a 2θ angle of 22° can be observed, which is indicative of



FIG. 2—X-ray diffractograms of low- and high-carbon rice husk ash.

siliceous glass. As there are no well-defined peaks, the nature of silica present in the LC-RHA is assumed to have been amorphous. Such amorphous silica has been produced by other investigators using temperatures similar to those used in the current study (between 750°C and 850°C) [7].

The scanning electron microscopy (SEM) analyses conducted on HC- and LC-RHA samples are shown in Figs. 3(a) and 3(b), respectively. Figure 3(a) shows that the HC-RHA particles were vesicular in nature, and their porous microstructure is clearly visible from the grain edges. The microstructure of the LC-RHA particle shown in Fig. 3(b) appears to be similar to that of the HC-RHA particle.

Figure 4(*a*) shows the particle size distribution of LC- and HC-RHA particles. As the figure shows, the average particle sizes (D_{50}) of the as-received LC- and HC-RHA were found to be ~29.9 μ m and ~38 μ m, respectively. The average particle size of both ashes in the as-received condition appears to be larger than that of conventional pozzolans such as fly ash and silica fume. Because the presence of larger particles of pozzolan in cement paste has been found to delay the pozzolanic reaction, thereby resulting in a lower compressive strength than expected [7,42], grinding of RHA was considered as an essential and important step in this study to improve the performance of unground RHA particles in portland cement systems.

Investigations on the Effect of Grinding Low-carbon Rice Husk Ash on Its Particle Size Distribution

In order to understand the effect of grinding on the PSD of RHA, laser particle size analysis was employed. Detailed studies on the PSD were performed only with LC-RHA, and the optimum grinding regime finalized based on this study was employed for further investigations with LC- and HC-RHA.

Figures 4(b) through 4(e) show several graphs to explain the effect of grinding on the PSD of RHA. Specifically, Figs. 4(b) and 4(c) show the variation in the PSD of RHA with different grinding times using grinding speeds of 100 and 250 rpm, respectively. As can be seen, the average particle



(a) Microstructure of a single high-carbon RHA grain



(b) Microstructure of a single low-carbon RHA grain

FIG. 3—SEM analysis of single high- and low-carbon RHA particles.

size of RHA decreased with the increase in grinding time. The PSD curves of RHA powders obtained using different grinding regimes can be observed to be uniform and parallel.

Figure 4(d) shows a comparison of the PSDs of ground RHA powders obtained via two different grinding regimes, 250 rpm for 15 min and 100 rpm for 45 min. As the figure shows, these powders appear to have the same PSD curve, indicating that a specific PSD of RHA can be obtained by suitably reducing the grinding speed and subsequently increasing the grinding duration, or vice versa. A slight deviation in their PSDs is noticeable in the graph beyond a cumulative volume of 90 %, which might have been due to the presence of limited coarser RHA particles. This small deviation has less prominence and can be neglected, as the PSD curves are similar up to a cumulative volume of 90 %.

Figure 4(e) shows the effect of grinding time and speed on the average particle size of RHA. As expected, the average particle size decreased with increases in both grinding speed and grinding



(a) Particle size distribution of unground low- and high- carbon rice husk ash



ground RHA powders having same D₅₀ but produced from 100 rpm and 250 rpm

e) Effect of grinding time and speed on the average particle size, D₁₀ of RHA

FIG. 4—Effect of grinding of low-carbon RHA on its particle size distribution.

duration. The minimum average particle size of RHA obtainable was $\sim 6 \,\mu$ m, using a 250 rpm speed and 60 min duration. The average particle size of RHA tended to level off after a specific grinding time and speed, beyond which significant changes in the particle size were not observed, indicating that very high energies might be required in order to reduce particle sizes still further.

However, such high grinding energies are not usually required, as the RHA derives its pozzolanic reactivity primarily from its cellular structure [42].

From the studies conducted, three grinding regimes were chosen to study in detail the effect of grinding RHA on specific properties of RHA and cement + RHA blends: regime 1 (as-received or unground condition), regime 2 (250 rpm for 5 min), and regime 3 (250 rpm for 60 min). The average particle sizes of RHA obtained using regimes 1, 2, and 3 were 29.9 μ m, 11.9 μ m, and 5.74 μ m, respectively. From these results, it can be seen that the RHA grains are fairly soft, and the continued reduction in particle size beyond the initial 15 min is not very significant.

Comparison of Bulk Densities of Low- and High-Carbon Rice Husk Ash in the As-received (Unground) and Ground Conditions

The loose and vibrated bulk densities of both LC- and HC-RHA particles obtained using grinding regimes 1, 2, and 3 are shown in Fig. 5. As this figure shows, the unground RHA particles from regime 1 had a lower bulk density than the RHA particles obtained from regimes 2 and 3. Such a trend was observed with both HC- and LC-RHA and for both loose and vibrated conditions. In addition, the vibrated bulk density of RHA was found to be slightly greater than its loose bulk density.

In the case of HC-RHA, a steady increase in the bulk density with increasing grinding effort was not observed. For example, the loose bulk density of unground HC-RHA was found to improve slightly from 351 kg/m³ to 359 kg/m³ upon regime 2 grinding, indicating that regime 2 was not effective in grinding HC-RHA. When regime 3 was employed, a steep increase in the bulk density of RHA from 359 kg/m³ to 503 kg/m³ was observed, indicating that grinding substantially improved the bulk density. In the case of LC-RHA, the loose bulk density increased steadily from 215 kg/m³ to 436 kg/m³ and 436 kg/m³ to 574 kg/m³ when grinding regimes 2 and 3 were employed, respectively, indicating that significant breakdown of the RHA particles occurred progressively with increasing grinding effort.



FIG. 5-Comparison of loose and vibrated bulk densities of low- and high-carbon RHA.

Overall, because the bulk density of unground HC-RHA was higher than that of unground LC-RHA, the grinding process was found to be more effective in increasing the bulk density of the latter than of the former.

Comparison of Packing Density of Dry Cementitious Powder Containing Low- and High-Carbon Rice Husk Ash

The concept of packing density in the mixture proportioning of concrete has been employed by many investigators primarily to reduce the quantity of cementitious material (in order to achieve economy) and to eliminate excess air voids by increasing the density of concrete [43,45]. In this concept, different aggregate gradations are usually filled in a known volume of the measure, and their packing density is determined from their measured weights. The aggregate gradation that has the maximum packing density requires the minimum cementitious material in order to fill the voids.

The packing density of dry cementitious material (cement + pozzolan) is not usually determined by investigators, as the variation in the binder proportions due to changes in the replacement level of pozzolan tends to cause very little effect. There are two reasons for this: (a) the PSDs of cement and pozzolan are approximately similar, and (b) the bulk density of cement (\sim 1400 to 1600 kg/m³) is approximately twice that of conventional pozzolans such as fly ash and slag (\sim 600 to 850 kg/m³), primarily because of the high apparent specific gravity of the former (\sim 3.15) relative to that of latter (\sim 2.2 to 3.0). However, this change in the specific gravity and the corresponding change in bulk density do not significantly affect the overall bulk density of the cementitious material at nominal replacement levels of pozzolans. At higher replacement levels, this deviation can be significant, especially when one of the components significantly differs from the other in bulk specific gravity, because of internal porosity as observed with RHA. In addition, the cementitious material does not produce significant internal air voids in the wet paste, and thus knowledge of the packing density of dry cementitious material has very little significance.

In the case of cementitious material containing unground or as-received RHA, the packing density is important because its bulk density is very low (\sim 215 kg/m³), approximately one-seventh of that of Portland cement. Thus, nominal replacement levels of RHA for cement can be expected to cause significant changes in the packing density of the cementitious material. In addition, the increased average particle size of RHA relative to cement and the cellular microstructure of RHA can cause significant changes in the physico-mechanical arrangement of these particles when combined. Thus, the packing density of cementitious material can provide some useful information regarding the volume changes that occur in the paste as a result of the addition of a pozzolan, like RHA, having a very low bulk density.

The packing density of dry cementitious material containing unground LC- and HC-RHA at different RHA replacement levels for cement in loose and vibrated conditions is shown in Fig. 6. The *x*-axis of this figure represents the RHA replacement level, varying from 0 % (indicating cement only) to 100 % (indicating unground RHA only). As this figure shows, the loose or vibrated packing density of dry cementitious material significantly decreased with increasing RHA replacement levels from 0 % to 50 %. At RHA replacement levels beyond 50 %, there was very little decrease in the packing density. In order to evaluate the percentage decrease in the packing density, two different replacement levels of 15 % and 50 % were considered. These values represent approximately the optimum and maximum replacement levels for cement as found in the literature [7,23,25,29,33,46,47]. It can be observed from this figure that the loose packing density of cementitious material decreased by 26 % and 33 % for HC- and LC-RHA, respectively, at a 15 %



FIG. 6-Effect of RHA replacement level on the packing density of low- and high-carbon RHA.

replacement level. At a 50 % RHA replacement level, the percentage decrease in the packing density of cementitious material decreased by 59 % and 78 % for HC- and LC-RHA, respectively. Despite having a lower average particle size, the LC-RHA registered a lower packing density than HC-RHA at any given replacement level, probably because of the lower specific gravity and carbon content of the former. The trend observed with vibrated packing density was similar to that observed with loose packing density, except that the former registered slightly higher values than the latter.

The significant decrease in the packing density observed at the optimum replacement level of unground LC-RHA is a clear indication that significant voids exist during loose or vibrated packing. Such an effect can cause significant changes in the volume of the concrete mixture, especially during wet mixing, though these changes will be less pronounced than in dry mixing because of the interactions of aggregates and water. This effect tends to be particularly exacerbated when unground RHA is used at higher replacement levels. Thus, it is critical to determine the experimental density of concrete containing unground LC-RHA after thorough mixing of the ingredients. This experimental density can be used to obtain the yield of concrete, which in turn can be used to back-calculate the accurate mixture proportions of concrete.

This test was not conducted on dry cementitious powders containing ground RHA, as its bulk density is significantly greater than (approximately three times) that of unground RHA and comparable to that of conventional pozzolans such as densified silica fume. In addition, because the average particle size of ground RHA is approximately similar to that of cement, its addition was expected to have much less of an effect on the packing density of the dry binder.

Microstructure of Rice Husk Ash Cementitious Pastes

The microstructures of the control, HC-URHA, and LC-URHA pastes after a 28-day moist curing period are shown in Fig. 7. In the RHA paste specimens, the dosage of RHA used was a 15 % mass replacement of cement.

Figures 7(*a*i) through 7(*a*iii) show the microstructure of the paste specimens at a low magnification of $40 \times$. From these figures, it is evident that all the three paste specimens contained some air



FIG. 7—Microstructure of cementitious paste specimens after a 28-day moist curing period.

voids and unhydrated cement grains, along with some remnants of partially hydrated RHA grains. Both RHA pastes were characterized by the presence of irregular-shaped and coarse remnant grains of RHA particles, seen as non-uniform gray-colored particles well distributed in the matrix. In addition, some micro-cracks in the RHA pastes were observed, most appearing as a result of artifacts during the specimen preparation process.

Other important features that can be compared in the SEM images shown in Figs. 7(bi) through 7(biii) taken at a magnification of 600X, are the amount of unhydrated cement grains and the dense

nature of the cement paste. The microstructure of the hydrated paste in the LC-URHA and HC-URHA mixtures was found to be denser than that of the control paste, probably because of the pozzolanic reaction between the RHA particles and the cement hydration products. The control paste was found to contain numerous residual unhydrated cement particles, seen as large, bright grains, indicating their high backscatter coefficient. In the case of HC-URHA pastes, most of the residual unhydrated cement grains were smaller in size than those of the control paste. In the case of LC-URHA pastes, even fewer residual unhydrated cement grains were observed, suggesting more complete hydration of the cement grains. Zhang et al. [20] conducted investigations to quantify the amount of unhydrated cement particles in control and RHA pastes. The results from their investigation suggest that the amount of unhydrated cement particles in the RHA paste was only slightly less than that in the control paste. The significant decrease in the amount of hydrated cement grains relative to that observed in the control paste in the present study clearly disagrees with some previous findings [20]. This aspect needs to be researched further, and the benefits of using RHA in the internal curing of cement paste should be explored and quantified.

The capillary porosity in the matrices of the control, HC-URHA, and LC-URHA paste specimens was observed at two magnifications ($600 \times$ and $1000 \times$) as shown in Figs. 7(*c*i) through 7(*c*vi). As seen in Figs. 7(*c*i) through 7(*c*iii), the control paste was characterized by the presence of significant capillary porosity in its matrix; however, both HC-URHA and LC-URHA paste specimens showed a denser matrix that embedded the remnant RHA grains. These particles, being porous, create significant internal porosity. Because the presence of such internal porosity can negatively affect the strength of the matrix, grinding RHA to a finer particle size is warranted. However, the internal porosity in RHA pastes was also found to be discrete and isolated, and thus it is not expected to increase the permeability of the overall matrix. In our previous studies, mixtures containing LC-URHA at cement replacement levels of 10 % and 20 % showed very low rapid chloride ion permeability results relative to control mixtures [47]. In addition, the porosity of the RHA pastes (excluding the internal porosity) in their matrix region appeared to be significantly lesser than that of the control paste, as can be observed in Figs. 7(*c*iv) through 7(*c*vi).

Comparison of Normalized Calcium Hydroxide Contents of High- and Low-carbon Rice Husk Ash Paste

In order to understand the effect of the addition of RHA on the pozzolanic reactivity and to compare the performance of HC- and LC-RHA pastes, the 28-day normalized $Ca(OH)_2$ contents of control and RHA pastes were determined and are shown in Fig. 8. Because all the pastes had a constant RHA dosage of 10 % cement replacement level, a decrease in the normal $Ca(OH)_2$ content of 10 % was considered as the dilution limit, above which any further decrease was considered as a definite indication of pozzolanic reactivity.

As seen in Fig. 8, the normalized $Ca(OH)_2$ contents of the RHA pastes were found to be lower than that of the control paste. Among the RHA pastes, the normalized $Ca(OH)_2$ contents of those containing LC-RHA were found to be lower than in those containing HC-RHA, indicating that the former was more reactive than the latter. In addition, the normalized $Ca(OH)_2$ content of the HC-URHA paste was equal to 10 %, indicating that the depletion was primarily due to dilution effects and no significant pozzolanic reaction had occurred. Also, the normalized $Ca(OH)_2$ content of the HC-GRHA paste was found to be slightly below the dilution effect.

In both unground and ground RHA pastes, the $Ca(OH)_2$ content of the ground RHA was found to be significantly lower, indicating the beneficial effects of grinding the RHA particles.



FIG. 8—Comparison of normalized calcium hydroxide contents of high- and low-carbon RHA pastes at a 10% replacement level.

The percentage reduction in the normalized $Ca(OH)_2$ content due to the grinding of RHA was found to be 3 % and 9 % for HC-RHA and LC-RHA pastes, respectively. These percentage reduction values clearly indicate that grinding was more effective in the LC-RHA paste than in the HC-RHA paste. In addition, the percentage reduction in the normalized $Ca(OH)_2$ content of the RHA pastes due to the variation in the carbon content of RHA was found to be between 15 % and 20 %, depending on whether or not the RHA was ground.

Comparison of Flow Behavior of High- and Low-Carbon Rice Husk Ash Mortars

Figure 9(a) shows the effect of the superplasticizer dosage on the flow values of RHA mortars at a 15 % replacement level, and Fig. 9(b) shows the effect of the RHA replacement level on the superplasticizer dosage.

As Fig. 9(*a*) shows, the flow values of all the RHA mortars increased with increasing superplasticizer dosage, as expected. For a given level of superplasticizer, the unground RHA mortars registered a lower flow value than the ground RHA mortars, probably for two reasons: Firstly, the coarse grains of RHA particles can absorb more water and superplasticizer because of their cellular and porous microstructure, resulting in an unavailability of water or superplasticizer in the mixture. Secondly, the bulk density of unground RHA particles is significantly lower (\sim 215 kg/m³) than that of ground RHA particles (\sim 584 kg/m³), and thus for a given RHA replacement level by mass, the bulk volume of paste containing unground RHA is higher, resulting in a corresponding increase in the porosity of concrete. Thus, high amounts of superplasticizer are necessary to meet a flow requirement. In the case of ground RHA mortars, the particles are finer and therefore do not trap moisture and superplasticizer, unlike the unground grains.

At any given superplasticizer dosage, the HC-RHA mortars registered lower flow values than the LC-RHA mortars. Previous studies have suggested that greater carbon contents in concrete result in a greater demand for chemical admixtures [7]. It is likely that the unburned carbon content in the RHA grains acts as activated carbon that can adsorb organic compounds out of the pore solution, making higher superplasticizer dosages necessary.



(a) Effect of superplasticizer dosage on flow values of RHA mortars at 15% replacement level



(b) Effect of RHA replacement level on superplasticizer dosage

FIG. 9—Flow studies on RHA cementitious mortars.

The horizontal limit line in Fig. 9(a) indicates the standard flow of 110 % as specified by ASTM C311 [41], and the vertical limit line indicates the maximum superplasticizer dosage of 1.25 % [48] by weight of the cementitious material that is recommended for the poly-carboxylate-based superplasticizer provided by the manufacturer. The results shown in Fig. 9(a) indicate that LC-URHA and HC-URHA mixtures did not meet the standard flow requirement corresponding to a superplasticizer dosage of 1.25 %. However, the LC-GRHA and HC-GRHA mixtures met the standard flow requirement, indicating the beneficial effects of grinding RHA.

From Fig. 9(b), it is evident that the superplasticizer demand of all the mortars increased with increasing RHA replacement levels up to 20 %. As discussed previously, the ground RHA mortars registered higher flow values than unground RHA mortars, and the LC-RHA mortars registered higher flow values than HC-RHA. The horizontal limit line in the figure shows the maximum superplasticizer dosage (1.25 %) recommended for poly-carboxylate-based superplasticizer. As can be seen from this figure, the maximum cement replacement levels that can be recommended for the HC-URHA and LC-URHA mixtures based on the flow requirement are 3 % and 10 %,

respectively. In the case of ground RHA mixtures, the recommended replacement levels can be up to 20 % or beyond depending on the carbon content.

Comparison of Strength Activity Index of Mortars Containing High- and Low-Carbon Rice Husk Ash

Figure 10 shows the 28-day SAIs of RHA mortars. It can be observed from this figure that an increase in the RHA replacement level from 0 % to 20 % steadily increased the SAI of LC-RHA mortars, whereas the SAI of HC-RHA mortars decreased. This trend clearly indicates that the LC-RHA mortars performed better and the HC-RHA mortars performed worse than the control mortars at any given replacement level. The improved performance of LC-RHA mortars is probably due to the presence of high amounts of amorphous silica and the low carbon content in the RHA. The amorphous silica in the LC-RHA tends to actively participate in the pozzolanic reaction, converting the calcium hydroxide to high-strength hydrates (C–S–H) and resulting in greater strength [16,34]. Similarly, for unground and ground HC-RHA mortars, the percentage decreases in the SAI at a constant RHA replacement level of 10 % were found to be 25 % and 20 %, respectively.

In addition, the SAI of mortars containing ground RHA was slightly higher than that of mortars containing unground RHA. For example, the percentage increases in the 28-day SAI due to the grinding of LC- and HC-RHA at a replacement level of 10 % were found to be only 3.57 % and 6.85 %, respectively. Such an increase is primarily due to the reduction in the RHA particle size and the presence of large amounts of small particles. Unlike larger particles, smaller particles tend to disperse well in the wet mixture and participate completely in the pozzolanic reaction.

Considering the minimum SAI requirement of 75 % as indicated in ASTM C311 [41], it can be observed from these data that all mortars satisfied the minimum strength criteria at a 10 % RHA replacement level, with LC-RHA mortars performing better than HC-RHA mortars. However, at a higher RHA replacement level of 20 %, all mortars except the unground HC-RHA mortars satisfied the strength criteria. Thus, in situations when only an HC-RHA is available, grinding may be considered as an effective strategy to improve its performance in mortars. However, in situations when



FIG. 10-Strength activity index of RHA cementitious mortar after a 28-day curing period.

Copyright by ASTM Int'l (all rights reserved); Tue Jun 25 11:49:02 EDT 2013 Downloaded/printed by Clemson University Libraries pursuant to License Agreement. No further reproductions authorized. only an LC-RHA is available, grinding is not absolutely necessary, as the performance of LC-URHA is only slightly poorer than that of LC-GRHA.

Conclusions

The studies conducted for the material characterization of LC-RHA and HC-RHA for use in concrete yielded the following findings:

- (1) From the XRD studies, it was observed that the controlled combustion of rice husks can be used to produce amorphous silica with a low carbon content.
- (2) Both LC- and HC-RHA particles had a porous and cellular microstructure with approximately similar PSDs. The bulk density of unground HC-RHA was found to be greater than that of unground LC-RHA. Grinding RHA significantly increased the bulk density of both types of RHA and decreased their particle size.
- (3) The packing density of blended cement powder containing LC-RHA was found to be less than that of powder containing HC-RHA. At nominal unground RHA replacement levels of 15 %, there was a significant drop in the dry packing density of the RHA blended powders relative to the control mixtures.
- (4) The microstructure of the pastes containing LC-RHA and HC-RHA showed that their cement grains were more hydrated than the cement grains in the control pastes.
- (5) Ground RHA was found to deplete more Ca(OH)₂ than unground RHA at a given replacement level in both HC-RHA and LC-RHA.
- (6) The flow of mortars containing LC-RHA was found to be higher than that of HC-RHA mortars. Also, the mortars containing ground RHA exhibited higher flow than mortars containing unground RHA. Similarly, the superplasticizer demand increased with an increase in the RHA replacement level. The LC-RHA mortars needed less superplasticizer in order to obtain the standard flow of 110 % than did HC-RHA mortars, and the ground RHA needed less superplasticizer than the unground RHA.
- (7) The SAI of LC-RHA mortars increased with an increase in the RHA replacement level from 0 % to 20 %, respectively, whereas that of the HC-RHA mortars decreased over the same range of RHA replacement levels. Whereas the unground HC-RHA mortars did not meet the strength criteria at 10 % and 20 % replacement levels, the ground HC-RHA mortars satisfied the minimum SAI requirement of 75 %. Both unground and ground LC-RHA mortars met the strength criteria.

Overall, it can be concluded that the performance of LC-RHA was better than that of HC-RHA in most of the tests conducted. The use of unground HC-RHA in cementitious systems may not be recommended because of concerns regarding workability. However, after it has been ground to a finer powder, the HC-RHA can still be used at RHA replacement levels up to 20 % to meet both strength and flow criteria. In the case of unground RHA a maximum RHA replacement level of 10 % can be used to improve the strength and microstructure of concrete. However, if the LC-RHA is ground, replacement levels in excess of 20 % cement replacement can be used.

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References

[1] Mehta, P. K. and Pitt, N., "Energy and Industrial Materials From Crop Residue," *Resource, Recovery and Conservation*, Vol. 2, 1976, pp. 23–38.

- [2] Pitt, N., "Process for the Preparation of Siliceous Ash," U.S. Patent No. 3,959,007 (1978).
- [3] Boateng, A. A., Fan, L. T., Walawender, W. P., and Chee, C. S., "Morphological Development of Rice-Hull-Derived Charcoal in a Fluidized-Bed Reactor," *Fuel*, Vol. 70, 1991, pp. 995–1000.
- [4] Savant, N. K., Snyder, G. H., and Datnoff, L. E., "Silicon Management and Sustainable Rice Production," *Advances in Agronomy*, Vol. 58, 1997, pp. 151–199.
- [5] Chakerverty, A. and Kaleemullah, S., "Conversion of Rice Husk into Amorphous Silica and Combustible Gas," *Energy Convers. Manage.*, Vol. 32, 1991, pp. 565–570.
- [6] Food and Agriculture Organization of the United Nations, 2007, "FAO: Food and Agriculture Organization of the United Nations, for a World Without Hunger," http://www.fao.org (Last accessed 31 July 2009).
- [7] Nehdi, M., Duquette, J., and Damatty, A., "Performance of Rice Husk Ash Produced Using a New Technology as a Mineral Admixture in Concrete," *Cem. Concr. Res.*, Vol. 33, 2003, pp. 1203–1210.
- [8] Vempati, R. K., "Composition and Method of Forming Low-Carbon, Amorphous Siliceous Ash From Siliceous Waste Material," U.S. Patent No. 6444186 B1 (2002).
- [9] Mehta, P. K., "Siliceous Ashes and Hydraulic Cements Prepared Therefrom," Belgian Patent No. 802909 (July 1973)/U.S. Patent No. 4105459 (August 1978).
- [10] Chopra, S. K., Ahluwalia, S. C., and Laxmi, S., "Technology and Manufacture of Rice-husk Ash Masonry (RHAM) Cement," *Proceedings of ESCAP/RCTT Workshop on Rice Husk Ash Cement*, New Delhi, India, 1981.
- [11] Nair, D. G., Fraaij, A., Klaassen, A. A. K., and Kentgens, A. P. M., "A Structural Investigation Relating to the Pozzolanic Activity of Rice Husk Ashes," *Cem. Concr. Res.*, Vol. 38(6), 2007, pp. 861–869.
- [12] Yeoh, A. K., Bidin, R., Chong, C. N., and Tay, C. Y., "The Relationship Between Temperature and Duration of Burning of Rice-Husk in the Development of Amorphous Rice-Husk Ash Silica," *Proceedings of UNIDO/ESCAP/RCTT, Follow-up Meeting on Rice-Husk Ash Cement*, Alor Setar, Malaysia, 1979.
- [13] Muthadhi, A. and Kothandaram, S., "Optimum Production Conditions for Reactive Rice Husk Ash," *Mater. Struct.*, Vol. 43(9), 2010, pp. 1303–1315.
- [14] Cook, D. J. and Suwanvitaya, P., "Properties and Behavior of Lime Rice Husk Ash Cements," ACI-SP 79-45, American Concrete Institute, Farmington Hills, MI, 1983, pp. 831–842.
- [15] Khalaf, M. N. and Yousif, H. A., "Use of Rice Husk Ash in Concrete," *International Journal of Cement Composites and Lightweight Concrete*, Vol. 6(4), 1984, pp. 241–248.
- [16] James, J. and Rao, S., "Reactivity of Rice Husk Ash," Cem. Concr. Res., Vol. 16(3), 1986, pp. 296–302.
- [17] Hwang, C. L. and Wu, D. S., "Properties of Cement Paste Containing RHA," Fly Ash, Silica Fume, Slag and Natural Pozzolans in Concrete—Proceedings of the Third International Conference, ACI SP-114, V. M. Malhotra, Ed., American Concrete Institute, Detroit, MI, 1989, pp. 733–762.
- [18] Boateng, A. A. and Skeete, D. A., "Incineration of Rice Hull for Use as a Cementitious Material: The Guyana Experience," *Cem. Concr. Res.*, Vol. 20, 1990, pp. 795–802.
- [19] Ikpong, A. A. and Okpala, D. C., "Strength Characteristics of Medium Workability Ordinary Portland Cement Rice Husk Ash Concrete," *Build. Environ.*, Vol. 27(1), 1992, pp. 105–111.
- [20] Zhang, M. H., Lastra, R., and Malhotra, V. M., "Rice Husk Ash Paste and Concrete: Some Aspects of Hydration and the Microstructure of the Interfacial Zone between the Aggregate and Paste," *Cem. Concr. Res.*, Vol. 26(6), 1996, pp. 963–977.

- [21] Ismail, M. S. and Waliuddin, A. M., "Effect of Rice Husk Ash on High Strength Concrete," *Constr. Build. Mater.*, Vol. 10(7), 1996, pp. 521–526.
- [22] Yu, Q., Sawayama, K., Sugita, M., Shoya, M., and Isojima, Y., "The Reaction Between Rice Husk Ash and Calcium Hydroxide Solution and the Nature of Its Product," *Cem. Concr. Res.*, Vol. 29, 1999, pp. 37–43.
- [23] Sensale, G. R. and Molin, D. C. C. D., "Study of Influence of Rice Husk Ash on Compressive Strength of Concrete at Different Ages," ACI SP-186-13, American Concrete Institute, Farmington Hills, MI, 1999, pp. 213–229.
- [24] Sugitha, S., Yu, Q., Shoya, M., Tsukinaga, Y., and Isojima, Y., "The Resistance of Rice Husk Ash Concrete to Carbonation, Acid Attack and Chloride Ion Penetration," ACI SP-172-2, American Concrete Institute, Farmington Hills, MI, 1999, pp. 29–43.
- [25] Mahmud, H. B., Majuar, E., Zain, M. F. M., and Hamid, N. B. A. A., "Mechanical Properties and Durability of High Strength Concrete Containing Rice Husk Ash." ACI Mater. J., Vol. 221, 2004, pp. 751–765.
- [26] Santos, S. and Prudencio, L. R., "Rice Husk Ash With Low Carbon Content," ACI SP-229-26, 2005, pp. 409–420.
- [27] Bui, D. D., Hu, J., and Stroeven, P., "Particle Size Effect on the Strength of Rice Husk Ash Blended Gap-Graded Portland Cement Concrete," *Cem. Concr. Compos.*, Vol. 27, 2005, pp. 357–366.
- [28] Sensale, G. R., "Strength Development of Concrete With Rice Husk Ash," Cem. Concr. Compos., Vol. 28, 2006, pp. 158–160.
- [29] Sakr, K., "Effects of Silica Fume and Rice Husk Ash on the Properties of Heavy Weight Concrete," J. Mater. Civ. Eng., Vol. 18(3), 2006, pp. 367–376.
- [30] Gastaldini, A. L. G., Isaia, G. C., Gomes, N. S., and Sperb, J. E. K., "Chloride Penetration and Carbonation in Concrete With Rice Husk Ash and Chemical Activators," *Cem. Concr. Compos.*, Vol. 29, 2007, pp. 176–180.
- [31] Saraswathy, V. and Song, H. W., "Corrosion Performance of Rice Husk Ash Blended Concrete," *Constr. Build. Mater.*, Vol. 21(8), 2007, pp. 1779–1784.
- [32] Giaccio, G., Sensale, G. R., and Zerbino, R., "Failure Mechanism of Normal and High-Strength Concrete With Rice-husk Ash," *Cem. Concr. Compos.*, Vol. 29(7), 2007, pp. 566–574.
- [33] Ganesan, K., Rajagopal, K., and Thangavel, K., "Rice Husk Ash Blended Cement: Assessment of Optimal Level of Replacement for Strength and Permeability Properties of Concrete," *Constr. Build. Mater.*, Vol. 22(8), 2008, pp. 1675–1683.
- [34] Mehta, P. K. and Montiero, P. J. M., *Concrete: Microstructure, Properties and Materials*, 3rd ed., McGraw-Hill, New York, 2006.
- [35] Ferraro, R. M., Nanni, A., Vempati, R. K., and Matta, F., "Carbon Neutral Off-White Rice Husk Ash as a Partial White Cement Replacement," *J. Mater. Civ. Eng.*, Vol. 22(10), 2010, pp. 1078–1083.
- [36] Mehta, P. K., "Properties of Blended Cements Made from RHA," ACI J., Vol. 74(9), 1977, pp. 440–442.
- [37] ASTM C150: Standard Specification for Portland Cement, *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2012.
- [38] ASTM C192: Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory, Annual Book of ASTM Standards, ASTM International, West Conshohocken, PA, 2012.
- [39] ASTM C230: Standard Specification for Flow Table for Use in Tests of Hydraulic Cement, *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2008.

- [40] ASTM C1437: Standard Test Method for Flow of Hydraulic Cement Mortar, Annual Book of ASTM Standards, ASTM International, West Conshohocken, PA, 2007.
- [41] ASTM C311: Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete, *Annual Book of ASTM Standards*, ASTM International, West Conshohocken, PA, 2013.
- [42] Mehta, P. K., "The Chemistry and Technology of Cement Made from Rice Husk Ash," Proceedings of the Workshop on Rice Husk Ash Cement, Regional Center for Technology Transfer, Bangalore, India, 1979, pp. 113–122.
- [43] Lange, F., Mortel, H., and Rudert, V., "Dense Packing of Cement Pastes and Resulting Consequences on Mortar Properties," *Cem. Concr. Res.*, Vol. 27(10), pp. 1481–1488.
- [44] Larrard, D. and Sedran, T., "Optimization of Ultra-High Performance Concrete Using a Packing Mode," *Cem. Concr. Res.*, Vol. 25(7), 1996, pp. 1501–1511.
- [45] Chatveera, B. and Lertwattanaruk, P., "Evaluation of Sulfate Resistance of Cement Mortars Containing Black Rice Husk Ash," J. Environ. Manage., Vol. 90(3), 2009, pp. 59–66.
- [46] Rukson, S., Chindaprasirt, P., and Mahachai, R., "Effect of Grinding on Chemical and Physical Properties of Rice Husk Ash," *International Journal of Minerals, Metallurgy and Materials*, Vol. 16(2), 2009, pp. 242–247.
- [47] Harish, K. V. and Rangaraju, P. R., "Fundamental Investigations into the Performance of a Carbon-neutral Rice Husk Ash as Supplementary Cementing Material," *Transportation Research Record.* 2164, Transportation Research Board, Washington, D.C., 2010, pp. 26–35.
- [48] "MSDS Data Sheet for Glenium 7500 High-Range Water Reducing Admixture," BASF Construction Chemicals, http://www.basf-admixtures.com/en/products/highrange/glenium7500/ Pages/default.aspx, Last accessed 12/21/2012.