



Hydration and phase formation of blended cementitious systems incorporating chemically transformed rice husk ash



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ABSTRACT

Rice husk ash (RHA) has been used as a supplementary cementing material (SCM). However, because RHA contains a cellular, honeycomb-like morphology, the use of RHA in concrete results in reduced workability and increased water requirements. Reduced workability and increased water requirements can be mitigated by reducing the RHA particles. Smaller RHA particles can be obtained with mechanical grinding. However, mechanical grinding is inefficient because significant energy is required. A recent study assessed a chemical transformation process (“chemical grinding”) and reported that using this process results in reduced RHA particle sizes and an altered cellular morphology. This paper provides results on the early-age hydration and phase formation of the cementitious systems containing chemically transformed RHA (t-RHA). Results indicate that the hydration and phase formation of the t-RHA system is different from the as-received RHA (AR-RHA).

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1. Introduction

Rice husk ash (RHA) is an agricultural waste product and is produced from incineration of rice husks for energy. This incineration is an efficient process to remove organic substances of rice husks, leaving behind inorganic substances – silica ash. Mehta [1] reported that after incineration, approximately 20% by mass of rice husk remains as RHA. The Food and Agriculture Organization (FAO) of the United Nations [2] reported that approximately 70 million metric tons of the RHA are produced worldwide each year. Although millions of tons of RHA are produced worldwide each year, the commercial applications to utilize this material have been reported to be limited [3,4]. This leads to serious challenges with disposal [5]. Da Costa et al. [4] and Rodriguez de Sensale [6] reported that the most common method of disposing RHA is dumping in landfills or into rivers. This disposal method can create environmental challenges and is not sustainable over the long term. Broadening the utilization of RHA is needed to mitigate these challenges.

RHA has a potential to be used as a supplementary cementing material (SCM). Replacing cement with RHA results in an increase in sustainability by reducing the cement content of concrete

mixtures. The Portland Cement Association (PCA) [7] reported that cement production accounts for approximately 5% of the global carbon dioxide (CO₂) emissions. Reducing cement content by substitution of the RHA can lead to the reduction of CO₂ emissions from cement production.

When burnt under controlled incineration conditions, RHA is composed of mostly amorphous silica. The amorphous silica can be up to 90% by mass of the RHA. This RHA can be used to provide additional C–S–H (pozzolanic) products in cementitious systems [8,9]. Taylor [10] reported that systems containing RHA not only perform similar to systems containing silica fume, but the RHA systems also exhibit higher pozzolanic activity. This higher pozzolanic activity leads to improved performance characteristics of the systems. Thus, the RHA blended cementitious systems can exhibit good early- and later-age characteristics. Replacement of cement with RHA is reported to lead to improved mechanical properties of concrete [11–14], good resistance to chloride penetration and sulfate attack [15,16], good freeze–thaw performance [17–19], good resistance to alkali–silica reaction (ASR) expansion [20], and improved resistance to deicing salt scaling [21].

The use of RHA in cementitious systems not only provides economic and environmental benefits but also can improve mixture performance. However, owners and contractors are not accepting RHA as a viable SCM product. This is especially the case when other SCMs are locally available. This is because RHA has high water absorption characteristics. These high water absorption

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characteristics occur because RHA contains a cellular, honeycomb-like morphology and has high surface area. RHA having these characteristics when used in concrete results in a reduction of flow [6]. Reduced flow can be mitigated by increasing the amount of water. However, increasing the amount of water in the mixtures results in a reduction of strength and durability. Thus, in addition to reduced flowability, the reduction of strength and durability also limits the use of RHA in cement and concrete systems.

Many studies [22–24] have investigated mechanical grinding methods (e.g., dry vibratory- and ball-milling) to reduce RHA particle size and eliminate the cellular, honeycomb-like morphology. The reduction of RHA particle size and elimination of its honeycomb morphology results in an increase of flowability and reduction of water demand [22–24]. Reducing RHA particle size and elimination of the honeycomb morphology can make RHA a more widely accepted SCM. To do this, alternatives to mechanical grinding processes should be identified [1,25]. Additional research is required to identify more economical and more efficient alternatives to mechanical grinding of RHA such that this material can be a more widely accepted SCM.

A RHA chemical transformation process has been reported to reduce the honeycomb morphology and reduce RHA particle size [26]. The chemically transformed RHA here is defined as “t-RHA.” The process also resulted in larger amounts of silicate ions dissolved in the t-RHA slurry [26]. This is reportedly due to an alkali extraction from amorphous silica of RHA. The larger amounts of silicate ions and the smaller RHA particles could affect the early-age hydration rates and setting behavior of the RHA blended cement products.

The use of the t-RHA in blended cementitious systems may influence the hydration. This work evaluates the time-variant concentrations of ions in t-RHA blended cementitious solutions. The control system [100% Portland Cement (PC)] and the systems containing as-received RHA (AR-RHA) were also assessed for comparison. After collecting ion concentration data from the RHA blended cementitious solutions, saturation levels of portlandite, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (gypsum), $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$ (ettringite), and $\text{Ca}_4\text{Al}_2(\text{SO}_4)(\text{OH})_{12} \cdot 6\text{H}_2\text{O}$ (monosulfate) were assessed by calculating the ion activity product (IAP) and dividing by the equilibrium solubility products.

2. Materials and methods

2.1. Materials

Type I/II PC was used for all systems. The chemical composition and Bogue calculations for the cement are shown in Table 1. The PC

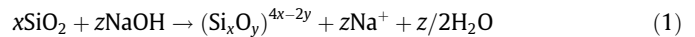
Table 1
Chemical composition and Bogue calculation of PC.

Element	(Mass %)
SiO_2	20.30
Al_2O_3	4.80
Fe_2O_3	3.50
MgO	0.70
SO_3	2.80
CaO	63.9
Loss on ignition	2.60
Insoluble residue	0.11
Limestone	3.20
CaCO_3 in limestone	97.80
Na_{eq}	0.54
<i>Bogue calculations</i>	
Ca_3SiO_5 (alite)	60.6
Ca_2SiO_4 (belite)	12.5
$\text{Ca}_3\text{Al}_2\text{O}_6$ (calcium aluminate)	6.8
$\text{Ca}_4\text{Al}_2\text{Fe}_2\text{O}_{10}$ (calcium aluminate ferrite)	11.9

met ASTM C150, *Standard Specification for Portland Cement* requirements. AR-RHA was procured from an Agrilectic power plant in Lake Charles, LA and the chemical composition is shown in Table 2. The average particle size of the AR-RHA is 192 μm with a standard deviation (SD) in average particle size of 3.15 μm (triplicate tests). ASTM Type II de-ionized (DI) water (1 $\text{M}\Omega \cdot \text{cm}$ at 25 °C) was used for all mixtures and experiments. Pellet sodium hydroxide (NaOH) was American Chemical Society (ACS) grade.

2.2. Preparation of chemical transformation process

The chemical transformation process was performed using NaOH. The t-RHA slurry was prepared by mixing AR-RHA with 2 M NaOH solution for 3 h. Mixing was carried out at 23 °C. The chemical transformation reactions are assumed as:



2.3. Preparation of RHA blended cementitious systems

After obtaining t-RHA slurry, the t-RHA was mixed with PC at 10% and 15% replacement levels. The water–binder ratio (w/b) was 4.0. Mixing of the systems was performed using a magnetic stirrer (at 400 rpm) and continued throughout the test (up to 420 min). The temperature during mixing was 23 ± 1 °C. The control system and the systems containing AR-RHA at 10% and 15% replacement levels were also assessed for comparison.

2.4. Ion concentration

The time elapsed after introducing the cementing materials to the solution will be referred to here as the “hydration time.” Ion concentrations were analyzed at hydration times of 5, 10, 15, 30, 45, 60, 90, 120, 150, 180, 210, 240, 300, 360, and 420 min. Hydroxyl ion concentrations were evaluated using a pH electrode (Metrohm 914). Aluminate and calcium ion concentrations were determined using a Perkin Elmer Analyst 100 flame atomic absorption spectroscopy (FAAS). Sulphate ion concentrations were determined using ion chromatography (IC) (Dionex DX 500 fitted with an AS-4A column and conductivity detector).

At each hydration time, test solution (30 mL) was decanted from the mixing beaker and filtered using a vacuum pump and No. 40 filter paper. Ten mL of filtered solution was used for analyzing aluminate ion concentration and 1 mL of filtered solution was used for analyzing calcium and sulfate ion concentrations. Because high concentrations of calcium and sulfate ions occur at early ages, filtered solutions for analyzing calcium and sulfate ion concentrations were diluted prior to the ion analyses. Filtered solutions for determining calcium and sulfate ion concentrations were

Table 2
Chemical composition of AR-RHA.

Composition	(Mass %) ^a
SiO_2	89.65 – 96.90
C	2.09 – 7.59
Al_2O_3	0.006 – 0.039
Fe_2O_3	0.006 – 0.052
CaO	0.48 – 0.81
MgO	0.13 – 0.53
SO_3	0.018 – 0.24
Na_2O	0.018 – 0.18
K_2O	1.74 – 2.69
Ti_2O_3	0.003 – 0.020
P_2O_5	0.74 – 1.23
Mn_3O_4	0.00 – 0.20

^a Provided by manufacturer; ranges represent variation over 1 year.

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