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A methodology for assessing the chemical and physical potential of industrially sourced rice husk ash on strength development and early-age hydration of cement paste



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HIGHLIGHTS

• A model to quantify the contribution of chemical and physical parameters in RHA reactivity is proposed.

Good correlation between compressive strength and heat release was observed.

 \bullet $\varphi\text{-values}$ have advantages because it considers RHA characterization and CH availability.

• RHA is a promising SCM even considering high variability from industrial suppliers.

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ABSTRACT

Among supplementary cementitious materials (SCMs), rice husk ash (RHA) is a by-product of the agricultural industry that is recognized by its high reactivity and ability to enhance performance of cementbased materials. The dependence of RHA reactivity on burning conditions and grinding procedures makes it sensitive to uncontrolled combustion/production. This study assesses 5 RHAs from industrial sources to understand how uncontrolled combustion impacts the reactivity of RHA. A fundamental model is proposed that combines physical (specific surface area) and chemical (amorphous silica content) characteristics of RHA with hydration and strength gain, considering the relative contribution of each one over time. Isothermal calorimetry was used to continuously assess strength development at early ages. The results show that the model is well correlated with strength development at different ages and allows the computation of an equivalent water-to-cement ratio considering solely the characteristics of a RHA sample, and prior to producing and testing specimens.

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1. Introduction

The effects of SCMs can be analysed as two simultaneous, nondivisible contributions occurring in a cementitious system:

1. Chemical contribution, referred as the *pozzolanic effect*. This is the formation of new hydration products by reaction of portlandite (CH, produced during cement hydration) and the aluminosilicate phases present in supplementary cementitious

http://dx.doi.org/10.1016/j.conbuildmat.2017.05.187 0950-0618/© 2017 Elsevier Ltd. All rights reserved. materials [1,2]. The rate of reaction depends on the specific surface available to react and the amount of CH, aluminosilicate, and water.

2. Physical contribution, referred to as the *filler effect*, is related to changes in the particle spacing and surface availability for nucleation of hydration products [2–4].

Rice husk ash (RHA) is obtained from agricultural waste and has been widely investigated owing to its potential as an SCM [5–7]. World rice production rose to approximately 740 billion tonnes in 2014 [8]. The main uses of rice husks are as fuel for the paddy milling process [9] and electric power plants [10]. RHA is obtained by combustion of the rice husks, which releases a large proportion of the organic matter contained, leaving a residue with high silica content [11,12]. The effects of rice husk ash on concrete strength [5,6,9,12–15] and durability [13,16–20] are well documented in

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the literature. The high reactivity observed in RHA is attributed to its high amorphous silica content and its high specific surface area.

The amorphous silica content in RHA is impacted mainly by the burning temperature and time of the husks [11,12,21]. While higher burning temperatures are more efficient at removing organic matter quickly, this may also lead to crystallization of the silica phases [22]. Della et al. [11] found that burning rice husk at 700 °C for 6 h produced RHA with 95% amorphous silica content. Pre-treatment of rice husk with hydrochloric acid prior to the burning process has also been shown to be effective to stabilize the pozzolanic activity but also to reduce the sensitivity of the RHA composition to the burning conditions [23].

It has been shown in previous studies that in addition to chemical composition, grinding is a controlling factor to produce highreactivity RHA [13,24]. The high specific surface area of RHA results from both its particle size [25] and mesoporous structure [26–28]. Cordeiro et al. [25] found that grinding RHA to particle sizes up to 10 μ m decreases the specific surface area and then increases it. Finer SCMs enhance hydration owing to the filler effect [3,29]. Furthermore, it has been shown that even crystalline RHA blended concrete exhibits an increase in compressive strength owing to this physical interaction [9]. However, Mehta [30] established that grinding RHA to finer particle sizes should be avoided to preserve its mesoporous structure, which explains its high reactivity.

Whereas the fundamental variables controlling the performance of RHA blended mixtures have been identified in previous studies, the relationship between them and their relative contribution to hydration and strength development has not been established in a way that would allow mixture design based on RHA characterization. Furthermore, most of the studies present results with ashes obtained in laboratory-controlled conditions, which differ significantly from the feasible industrial applications of this material. This study focuses on the characterization of five industrially sourced RHA samples from a chemical and physical perspective and relates these parameters to the observed strength development of blended mixtures.

2. Materials and methods

Five samples of RHA from different industrial suppliers (four in Brazil and one in China) were obtained and used in this study to assess the effect of variability in calcination conditions and control of the manufacturing process in RHA reactivity. No attempts to modify the RHA samples were made, and they were all used as received. Type I OPC was used for the entire experimental programme conducted. The chemical composition was determined by X-ray fluorescence (XRF), and the results are shown on Table 1 along with phase composition of OPC determined by quantitative X-ray diffraction (QXRD). In addition, relevant information provided by the suppliers regarding calcination temperature is included along with the specific gravity (SG) of the raw materials. No information about calcination time and heating rates was obtained, which have been found to be relevant parameters [31] to optimize amorphous silica content by avoiding crystallization along with temperature [11,21,32]. Therefore, this would be assumed as part of the intrinsic variability of the industrially sourced ashes.

The loss on ignition (LOI) is an indicative of the content of volatiles present in the sample, particularly carbon. Another factor that significantly affects the residual carbon content of RHA is calcination time [11,12,32], which was not reported by the suppliers. Therefore, calcination temperatures should be taken as referential parameters of the expected LOI because lower LOI values may be obtained by calcination at lower temperatures for longer periods of time.

2.1. Chemical characterization

2.1.1. X-ray diffraction and Rietveld refinement (QXRD)

QXRD analysis was performed to determine the mineral composition of the RHA samples. Testing was performed between 10 and 75° 2 θ at a 0.02° per second sampling rate. Commercially available software (EVA and TOPAS) was used for phase identification and Rietveld refinement. Fluorite was selected as the internal standard to consider the amorphous fraction and correct the Rietveld results, based on the procedure proposed by Chancey et al. [33]. Because this method allows for an estimation of the total amount of amorphous materials in the sample, XRF results can be used in combination with QXRD to estimate the amount of amorphous silica (SiO_{2-Amorphous}) in the different RHA samples. The calculation is performed by subtracting the sum of the amounts of crystalline silica polymorphs identified (SiO_{2-Crystalline}) from the total abundance of SiO₂ in each of the samples determined by XRF, according to Eq. (1):

$$SiO_{2-Amorphous} = SiO_{2-XRF} - \sum SiO_{2-Crystalline}$$
(1)

2.1.2. Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra were obtained using a Shimadzu IRAffinity spectrophotometer, with the RHA samples prepared by mixing 1 mg of each one with 3000 mg of KBr. Spectral analysis was conducted at a resolution of 6 cm^{-1} , over the range between 4000 and 400 cm⁻¹. A total of 45 measurements were made for each of the prepared samples.

2.2. Physical characterization

2.2.1. Particle size distribution (PSD)

Particle size distributions (PSD) of the five RHA samples considered and OPC were measured using a Malvern Mastersizer 2000 laser diffractometer. Isopropanol (refractive index 1.378) was used as a dispersant, and the PSD was measured for 20 s while stirring at 2700 rpm. For PSD calculations, 1.544 and 1.68 were considered refractive indexes for the RHA samples and OPC, respectively. It should be noted that PSD analysis neglects the mesoporous structure of RHA. It has been

Table 1

Chemical composition of OPC and RHA samples, phase composition of OPC and calcination temperatures of RHA. Chemical composition data is presented as percentage by wt.

	OPC	RHA-1	RHA-2	RHA-3	RHA-4	RHA-
SiO ₂	21.59	83.22	73.12	78.78	80.01	93.95
Al ₂ O ₃	3.82	0.00	0.00	0.00	0.00	0.53
Fe ₂ O ₃	3.05	0.07	0.12	0.29	0.08	0.35
CaO	64.34	0.65	0.52	0.66	0.54	0.91
Na ₂ O	0.54	0.65	0.07	0.12	0.09	0.21
K ₂ O	0.46	1.14	0.42	0.63	0.52	1.26
MnO	0.06	0.22	0.10	0.17	0.15	0.12
TiO ₂	0.33	0.00	0.01	0.01	0.00	0.03
MgO	1.84	0.37	0.33	0.40	0.36	0.61
P_2O_5	0.19	0.39	0.43	0.60	0.57	1.09
SO ₃	2.87	0.00	0.00	0.04	0.00	0.00
LOI	1.60	14.26	24.73	19.51	17.69	2.35
Calcination T° (°C)	_	450-500	800-1000	650	700	750
C₃S	66.47	-	-	-	-	-
C ₂ S	17.35	-	-	-	-	-
C ₃ A	5.54	-	-	-	-	-
C ₄ AF	3.37	-	-	-	-	-
Sp. gravity (g/cm ³)	3.122	2.348	2.336	2.492	2.861	2.382

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