Reactivity Assessment of Residual Rice-Husk Ashes

J. Hoppe Filho¹; M. R. Garcez²; M. H. F. Medeiros³; L. C. P. Silva Filho⁴; and G. C. Isaia⁵

Abstract: The production of rice-husk ash without controlled burning results in pozzolans with different physicochemical characteristics. The amorphous content of residual rice-husk ash (RRHA) is related to the temperature and exposure time during burning, whereas the BET specific surface area depends on the siliceous structure formed during plant growth, in addition to burning and grinding methods. In this study, amorphous content was quantified by X-ray diffraction (Rietveld refinement) in three RRHA samples randomly collected from rice mills located in southern Brazil, in which the burning process was not controlled. Pozzolanic activity evolution of RRHA samples in lime paste was monitored by thermogravimetry for 182 days in order to determine the influence of the lime:pozzolan ratio, BET specific surface area, and average particle size. The results show that lime consumption is not significantly affected by the BET specific surface area or average particle size of RRHA samples with a high amorphous content (>66%). The reaction rate of RRHA depends on the lime:pozzolan ratio. Lime consumption per unit surface area of RRHA declines with an increase in the BET specific surface area. Because the proposed shape factor connects the BET specific surface area and average particle size of RRHA and indirectly includes the amorphous content of RRHA samples, it can therefore be used as an indicator of RRHA reactivity. DOI: 10.1061/(ASCE)MT.1943-5533.0001820. © 2017 American Society of Civil Engineers.

Author keywords: Rice-husk ash; Pozzolanic activity; Amorphous content; BET specific surface area.

Introduction

In the rice industry, typical thermal treatment produces ash that contains a large amount of silica and residual carbon (Antiohos et al. 2014). The degree of crystallization of the amorphous siliceous structure and organic matter content of the rice husk depend on the temperature, exposure time and oxygen availability during burning (Nair et al. 2008; Cordeiro et al. 2009; Wansom et al. 2010; Zain et al. 2011). Cellular amorphous structures subjected to high temperatures gradually convert into crystalline structures in the form of quartz and/or cristobalite (Antiohos et al. 2013; Pavía et al. 2014), reducing the BET specific surface area (Feng et al. 2004; Chandrasekhar et al. 2006). Residual rice-husk ash (RRHA) samples produced without controlling the burning temperature exhibit different physicochemical characteristics, which are partially modified by comminution (Rukzon et al. 2009). Grinding changes particle size distribution and, to a lesser degree, the BET specific surface area (Cordeiro et al. 2011; Rêgo et al. 2015b). The low added value of this pozzolan and its abundance in southern Brazil enhance its potential use as a mineral addition to portland cement. Residual carbon content and amorphous content variability, as well as their influence on pozzolanic activity, are the main barriers to the use of rice-husk ash in cementitious composites (Antiohos et al. 2014).

The lack of information on RRHA reactivity and the durability of concretes produced by partial replacement of cement with this pozzolan has prompted extensive research (Chandrasekhar et al. 2006; Cordeiro et al. 2009; Sensale 2010; Cordeiro et al. 2011, 2012; Zerbino et al. 2011, 2012; Antiohos et al. 2013, 2014; Rêgo et al. 2015b, a). Results show that, even with low amorphous content, finely ground RRHA consumes a considerable amount of lime (Rêgo et al. 2015b). Portland cement replaced with RRHA by mass at 15% exhibits similar mechanical properties and durability to reference concrete (Zerbino et al. 2011), except for the high carbonation (Antiohos et al. 2013).

The pozzolanic activity and BET specific surface area of RRHA with a high amorphous content ground for at least 1 h are not significantly influenced by particle size (Cordeiro et al. 2011; Rêgo et al. 2015b). On the other hand, there is insufficient information regarding how the BET specific surface area of different RRHA samples with a high amorphous content and similar particle size distribution influences their ability to consume lime. Studies investigating the variation of BET specific surface area by grinding showed no direct influence on the pozzolanic activity of RRHA (Cordeiro et al. 2011; Rêgo et al. 2015b), although the physical effect (filler and extra nucleation points) increases the compressive strength of the cementitious matrix (Rukzon et al. 2009).

The main objective of this paper is to investigate the evolution of pozzolanic activity in RRHA samples in lime paste, simulating 15 and 25% partial replacement of portland cement, by mass, in order to determine the influence of the lime:pozzolan ratio, amorphous content, BET specific surface area, average particle size, and RRHA shape factor.

Materials

Residual Rice-Husk Ash

Three samples of RRHA were randomly collected from rice mills located in southern Brazil and ground in a ball mill for 60 min (RHA 1, RHA 2, and RHA 3a). Additionally, in order to assess the influence of particle size on RRHA pozzolanic activity, one of the samples (RHA 3b) was ground for 15 min. Fig. 1 shows the particle size distribution of the RRHA samples.

Table 1 shows the chemical composition, obtained by X-ray fluorescence (XRF), and some physical characteristics of the RRHA samples.

X-ray diffraction (Rietveld refinement) was used to identify the crystalline phases and amorphous content of the RRHA samples. Lithium fluoride p.a. (10% by mass) was added to the samples as an internal standard (Gobbo et al. 2007). X-ray diffraction patterns were obtained using a Rigaku Ultima IV X-ray diffractometer (Rigaku, Tokyo, Japan) over a range of 5°–75° 2θ and steps of 0.02° for 3 s, with Cu Kα radiation, 40 kV/30 mA, and 1° divergence slit. Fig. 2 shows the X-ray diffraction patterns of the RRHA samples, with crystalline phases and the amorphous halo at 22° 2θ.

Results of the quantitative analysis using Rietveld refinement are shown in Table 2. All the RRHA samples exhibited a high amorphous content (around 71%) and were composed of amorphous silica (approximately 62%), a considerable amount of cristobalite (approximately 29%), and quartz.

The particle size distribution, BET specific surface area, and amorphous content of the RRHA samples are shown in Fig. 3. The average particle size (D50) of the samples ground for 60 min was similar, but higher in those ground for 15 min. The BET specific surface area of the samples varied considerably. In samples RHA 3a and RHA 3b, it was observed that longer grinding times increased the BET specific surface area. Despite its variation, amorphous content can be considered high for all the RRHA samples.

The similar D50 exhibited by samples ground for 60 min enabled the evaluation of the influence of BET specific surface area on RRHA pozzolanic activity. On the other hand, for samples RHA 3a and RHA 3b, which were subjected to different grinding times, variations in D50 and slight differences in BET specific surface area were used to assess the influence of particle size on RRHA reactivity.

Calcium Hydroxide

Calcium hydroxide (CH; analytical reagent) was characterized by thermogravimetry (TG/DTG) using a METTLER-TOLEDO (Greifensee, Switzerland) Star® SW 9.30 thermogravimetric analyzer, and contains 1.50% water, which was completely volatilized by 350°C, and 8.97% calcium carbonate. The calcium hydroxide content in the reagent is 86.93% (by mass).

Methods

Mix Proportion

Mix proportions of the pozzolanic pastes (lime + RRHA) were defined according to the portlandite produced by Brazilian cement CP V—ARI, which is similar to portland cement Type III, HESC—high early strength cement [ASTM C150-12 (ASTM 2012)], considering 15 or 25% replacement by mass.

Replacement was defined based on the optimum replacement levels presented in the literature, which vary from 20 to 30% by mass (Ganesan et al. 2008; Zerbino et al. 2011; Antiohos et al. 2014). Susceptibility to alkali-silica reaction (ASR) when replacement level exceeds 15%, which depends on average RRHA particle size and the portland cement used, was also considered (Zerbino et al. 2012). Based on the chemical reaction involved in the formation of C—S—H, Jamil et al. (2013) suggested that the maximum contribution of rice-husk ash pozzolanic activity occurred at a replacement level of approximately 15% by mass, regardless of the type of cement.

The CH content produced by portland cement after 91 days in a nonvolatile base is approximately 32% (Hoppe Filho 2008). As shown in Fig. 4, a 15% partial replacement of portland cement by RRHA (64% CH + 36% RHA)—normalized), whereas 25% partial replacement produces 24.0% CH + 25.0% RHA (49% CH + 51% RHA—normalized). Table 3 shows the normalized mix proportions and CH:RHA ratios. Pastes were mixed in 0.7 water:solid ratios, molded into polymer containers and stored in sealed bottles at ambient temperature in the laboratory, until reaching test age.

Table 1. Chemical Composition and Physical Properties of the RRHA Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Grinding time (min)</th>
<th>Designated</th>
<th>Chemical composition (%)</th>
<th>Physical properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>SiO2</td>
<td>K2O</td>
</tr>
<tr>
<td>1</td>
<td>60</td>
<td>RHA 1</td>
<td>89.1</td>
<td>4.8</td>
</tr>
<tr>
<td>2</td>
<td>60</td>
<td>RHA 2</td>
<td>86.2</td>
<td>6.7</td>
</tr>
<tr>
<td>3</td>
<td>60</td>
<td>RHA 3a</td>
<td>94.8</td>
<td>1.5</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>RHA 3b</td>
<td>95.0</td>
<td>1.4</td>
</tr>
</tbody>
</table>

Note: DMAX = maximum particle size; LOI = loss on ignition.
Calcium Hydroxide p.a. Consumption in Normal Hydration Condition

Sample preparation—At different ages (7, 14, 28, 56, 91, 119, and 182 days), over a period of 6 months, samples were removed from the sealed bottles, placed in a chamber at −30°C to stop hydration, and freeze dried.

Thermogravimetric analysis—The dried samples were ground to 150–75 μm and analyzed by thermogravimetry in nitrogen atmosphere. The heating rate was 10°C/min, applied from 30 to 1,000°C.

Mass losses occurred between 30 and 400°C owing to decomposition of C─S─H [chemically bound water (CBW)], from 400 to 520°C owing to decomposition of portlandite, and between 520 and 1,000°C owing to volatilization of the carbon dioxide (CO₂) in calcium carbonate. Mass losses were corrected considering a nonvolatile base (Taylor 1997).

Table 2. Phase Composition of Residual RRHA—Rietveld Method

<table>
<thead>
<tr>
<th>Samples</th>
<th>SiO₂TOTAL</th>
<th>Quartz</th>
<th>Cristobalite</th>
<th>Amorphous</th>
<th>SiO₂AMORPHOUS</th>
</tr>
</thead>
<tbody>
<tr>
<td>RHA 1</td>
<td>89.1</td>
<td>0.1</td>
<td>23.2</td>
<td>76.7</td>
<td>65.8</td>
</tr>
<tr>
<td>RHA 2</td>
<td>86.2</td>
<td>0.1</td>
<td>29.1</td>
<td>70.8</td>
<td>57.0</td>
</tr>
<tr>
<td>RHA 3a</td>
<td>94.8</td>
<td>0.8</td>
<td>29.5</td>
<td>69.7</td>
<td>64.5</td>
</tr>
<tr>
<td>RHA 3b</td>
<td>95.0</td>
<td>1.6</td>
<td>32.3</td>
<td>66.1</td>
<td>61.1</td>
</tr>
</tbody>
</table>

SiO₂TOTAL = from chemical analysis (XRF).
SiO₂AMORPHOUS = SiO₂TOTAL − SiO₂QUARTZ − SiO₂CRISTOBALITE.

Fig. 2. X-ray diffraction patterns of the RRHA samples (C: cristobalite; Q: quartz; F: lithium fluoride—internal standard)

Fig. 3. Particle size distribution, BET specific surface area, and amorphous content of RRHA samples
Results and Discussion

Pozzolanic Activity Evolution of Residual Rice-Husk Ash Samples

Chemically Bound Water in the Form of Calcium Silicate Hydrate (C–S–H)

Fig. 5 shows the evolution of CBW in the form of calcium silicate hydrate (C–S–H) in the pozzolanic systems over a period of 6 months. The lime:pozzolan ratio and BET specific surface area do not directly influence CBW content when the D₅₀ of the RRHA samples are similar. The higher D₅₀ of sample RHA 3b reduced CBW content, regardless of its high BET specific surface area (44.4 m²/g).

The greater availability of lime in the system increased CBW content per unit mass of RRHA in samples with similar D₅₀, as shown in Fig. 6. The BET specific surface area does not significantly influence hydrate content per unit mass of RRHA. The lower fineness of sample RHA 3b reduced CBW content to the same levels observed in samples with a low lime addition rate, despite the greater availability of lime.

Lime Consumption Capacity

The lime content consumed by pozzolanic activity, shown in Fig. 7, was similar in all the samples regardless of the lime:pozzolan ratio. As such, changes in the D₅₀ of RRHA samples do not interfere significantly in lime consumption capacity when amorphous content is high, corroborating the findings of Rego et al. (2015b). The different BET specific surface areas observed in RRHA samples did not affect portlandite consumption. Fixed lime content (lime consumption) at 182 days varied between 27 and 30%, maintaining considerable alkalinity in the pastes for continued pozzolanic activity.

Fig. 4. Portlandite content in Brazilian portland cement Type CP V—ARI and portlandite content (theoretical), when 15 or 25% of cement is replaced, in weight, by residual RHA

Fig. 5. Chemically bound water as calcium silicate hydrate (C–S–H) in pozzolanic systems with different lime:RHA ratios

Fig. 6. Chemically bound water as C–S–H by unit mass of RRHA samples

Fig. 7. Portlandite consumption by pozzolanic activity of RRHA samples with different lime:addition ratios

Table 3. Mixture Proportions

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Residual RHA samples used</th>
<th>Calcium hydroxide (%)</th>
<th>Rice-husk ash (%)</th>
<th>CH:RHA ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix 01a</td>
<td>RHA 1 X RHA 2 X RHA 3a X RHA 3b X</td>
<td>64</td>
<td>36</td>
<td>1.78 g CH/g RHA</td>
</tr>
<tr>
<td>Mix 02a</td>
<td>RHA 1 X RHA 2 X RHA 3a X RHA 3b X</td>
<td>49</td>
<td>51</td>
<td>0.96 g CH/g RHA</td>
</tr>
</tbody>
</table>

*aWater:solids ratio of mixtures = 0.70.*
Lime consumption per amorphous unit mass, presented in Fig. 8, indicates that consumed lime content depends on the lime:pozzolan ratio. The greater availability of portlandite increases the reaction rate of RRHA, a fact also observed by Quarcioni et al. (2015). Although high $D_{50}$ affects CBW content, it did not influence lime consumption. Therefore, the C–S–H formed from larger RRHA particles tends to incorporate less CBW, although lime consumption is similar to that observed in samples with smaller particles. This means that the hydrated products formed exhibit varying stoichiometry.

Fig. 9 shows the influence of BET specific surface area on lime consumption capacity. Portlandite consumption per unit mass is not directly related to BET specific surface area. Correction of portlandite consumption as a function of amorphous content, quantified by Rietveld refinement, shows that RRHA produced without controlled burning displays high reactivity, with lime consumption greater than 1,000 mg Ca(OH)$_2$/g of amorphous fraction when the lime:RHA ratio is 1.78.

Variations in the BET specific surface area do not significantly affect lime consumption per unit mass of RRHA, as shown in Fig. 9. Thus, the larger the BET specific surface area, the smaller the consumed lime content per unit area (Fig. 10). Values of $R^2 \geq 0.90$ after 14 days indicate a statistical correlation between the results and the proposed mathematical model. Thus, increased RRHA BET specific surface area enables better distribution of C–S–H formed by pozzolanic activity in the hydrated matrix microstructure.

**Influence of Physical Characteristics on RRHA Reactivity**

The physicochemical characteristics of RRHA depend primarily on factors such as cellular amorphous structure formed during plant development, rice husk burning process (temperature, exposure time, and oxygen availability), and grinding. Burning conditions affect the crystallization of the cellular amorphous structure comprising the rice husk, forming tridymite and/or cristobalite and/or quartz. The gradual formation of the crystalline structure slowly reduces the BET specific surface area of RRHA (Chandrasekhar et al. 2006). As such, samples with a low amorphous content tend to exhibit a lower BET specific surface area. Grinding after burning alters RRHA particle size distribution, reduces average particle size, increases Blaine specific surface area (Rukzon et al. 2009; Cordeiro et al. 2011), and, to a lesser degree, changes the BET specific surface area. Excessive grinding tends to reduce RRHA BET specific surface area when amorphous content is high; however, almost no changes are observed when amorphous content is low (Rêgo et al. 2015b). The alveolar microstructure of RRHA particles, influenced by the degree of crystallinity, characterizes a nonmonolithic material that, after grinding, does not follow the inverse relationship between average particle size and BET specific surface area, meaning that variables become independent (Cordeiro et al. 2011). This characteristic justifies results reported in the literature, particularly in the event of high RRHA amorphous content.

The ability of RRHA to consume lime by pozzolanic activity depends on the $D_{50}$ and BET specific surface area, which is directly related to RRHA amorphous content. As such, it is important to consider the relationship between these characteristics and lime consumption. Because the proposed shape factor, showed in Eq. (1), incorporates the BET specific surface area and average particle size of RRHA and indirectly includes the amorphous content of RRHA samples, it can be used as an indicator of RRHA reactive capability.

$$\text{Shape factor } (Mm/g) = \frac{\text{BET specific surface area } (m^2/g)}{\text{Average particle size } (D_{50})(\mu m)}$$  (1)
Lime consumption as a function of shape factor, presented in Eq. (2), considers the influence of the physical–chemical characteristics of the particles on RRHA reactivity by pozzolanic activity.

\[
\text{Lime consumption} = \frac{1,000 \times \text{Lime consumption} [\text{mg Ca(OH)}_2/\text{g}]}{\text{Shape factor (Mm/g)}}
\]

The proposed shape factor was used to analyze RRHA reactivity with data obtained from the literature, based on the lime consumption results quantified by the modified Chapelle test (Raverdy et al. 1980). Table 4 presents RRHA pozzolanic activity data, adapted from recently published studies, in addition to the shape factor and lime consumption, estimated by Eqs. (1) and (2), using data collected from literature.

Table 4. Validation of Shape Factor and Lime Consumption, Estimated by Eqs. (1) and (2), Using Data Collected from Literature

<table>
<thead>
<tr>
<th>Sample</th>
<th>Grinding time (min)</th>
<th>Density (g/cm³)</th>
<th>LOI (%)</th>
<th>SiO₂ (%)</th>
<th>D₉₀ (μm)</th>
<th>BET (m²/g)</th>
<th>Amorphous content (%)</th>
<th>Pozzolanic activity (mg CaO/g)</th>
<th>Shape factor (Mm/g)</th>
<th>Lime consumption (μg Ca(OH)₂/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RHA “A”</td>
<td>60</td>
<td>2.11</td>
<td>8.02</td>
<td>87.00</td>
<td>25.91</td>
<td>33.24</td>
<td>—</td>
<td>676.3</td>
<td>Rêgo et al. (2015b)</td>
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</tr>
<tr>
<td></td>
<td>180</td>
<td>2.11</td>
<td>7.94</td>
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<td>19.58</td>
<td>32.49</td>
<td>—</td>
<td>685.5</td>
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<td>2.12</td>
<td>8.03</td>
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<td>RHA</td>
<td>120</td>
<td>2.29</td>
<td>11.90</td>
<td>82.60</td>
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<td>33.67</td>
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<td>477.1</td>
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<td>Cordeiro et al. (2011)</td>
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<td>86.00</td>
<td>12.00</td>
<td>19.00</td>
<td>—</td>
<td>704.6</td>
<td>Barata et al. (2006)</td>
<td>1.58</td>
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<td>RHA “A”</td>
<td>—</td>
<td>—</td>
<td>98.94</td>
<td>2.00</td>
<td>260.00</td>
<td>Amorphous</td>
<td>—</td>
<td>850.0</td>
<td>Souza et al. (2000)³</td>
<td>130.00</td>
</tr>
<tr>
<td>RHA “B”</td>
<td>—</td>
<td>—</td>
<td>99.69</td>
<td>0.60</td>
<td>480.00</td>
<td>—</td>
<td>—</td>
<td>940.0</td>
<td>—</td>
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</tr>
<tr>
<td>RHA “C”</td>
<td>—</td>
<td>—</td>
<td>99.22</td>
<td>1.50</td>
<td>290.00</td>
<td>—</td>
<td>—</td>
<td>880.0</td>
<td>—</td>
<td>193.33</td>
</tr>
</tbody>
</table>

Note: Pozzolanic activity = modified Chapelle test (Raverdy et al. 1980).
³RHA samples were pretreated with acid solutions before burning.

Fig. 11. Portlandite consumption obtained from modified Chapelle test as a function of the shape factor.
because reactivity is influenced by the lime:RRHA ratio (Quercioni et al. 2015). After 182 days, lime consumption for the paste with a lime:RRHA ratio of 1.78 was similar to that obtained using the modified Chapelle test; however, reactivity declined when the lime:RRHA ratio decreased to 0.96.

Conclusions

Results of RRHA pozzolanic activity in lime paste indicated that lime consumption capacity is not significantly dependent on BET specific surface area and average particle size in samples with a high amorphous content.

Residual rice-husk ash particle size distribution influences chemically bound water in C–S–H, meaning that the stoichiometry of the hydrated product depends on particle size.

At a certain age, the RRHA reaction rate increases with greater lime availability in the pozzolanic system. Lime consumption per unit area of RRHA declines as the BET specific surface area rises.

The shape factor proposed in the present study is directly related to the lime consumption capacity of RRHA because it incorporates BET specific surface area and average particle size as well as amorphous content of RRHA samples. As such, samples with similar shape factors tend to exhibit similar reactivity regardless of differences in BET specific surface area and average particle size.

Maximum lime consumption was 0.84 g Ca(OH)$_2$/$g$ RHA, increasing to 1.30 g Ca(OH)$_2$/$g$ amorphous RRHA when corrected for amorphous content. Lime consumption decreases when RRHA content rises in the pozzolanic system.

Reactivity of RRHA, despite the high residual carbon content, is higher than the minimum proposed (330 mg CaO/g) by Ravera et al. (1980) to be classified as pozzolanic addition.

A decline in the lime:RRHA ratio, signifying higher cement replacement, reduces amorphous silica consumption by pozzolanic activity, thus increasing the potential for alkali-silica reaction.

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