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Evaluation of alkali–silica reaction in concretes with natural rice husk ash using optical microscopy

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highlights

- Alkali–silica reaction in concretes with natural RHA was studied.

- Concretes with RHA, under field conditions, were evaluated up to four years.

- Microscopy observations were done to identify reaction products and internal damage.

- In presence of alkalis RHA produces severe damage in concrete, but no gel was found.

article info

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ABSTRACT

The use of rice husk ash as a supplementary cementing material is of great interest to many developing countries where rice production is in abundance. A highly reactive pozzolan is obtained when rice husk ash is burnt under controlled conditions. Previous work showed that it is possible to use residual rice husk ash "as nature" (natural rice husk ash). Nevertheless, it was observed that the incorporation of natural rice husk ash implies risks of expansions and mechanical degradation due to the reactions with alkalis. Based on the performance of slab prototypes placed outdoors during more than 3 years, this work analyzes the causes and the damage processes involved in the development of expansions in presence of natural rice husk ash. Optical microscopy observations on thin sections; crack patterns of the slabs and the strength and surface strain evolution along the time were evaluated. Visual and microscope observations showed clear signs of damage due to expansive reactions in concretes incorporating natural rice husk ash when high alkalis contents are available; although not gel was found, numerous cracks and voids were observed close to unburned rice husk particles, as well as high expansions and significant decreases in strength and stiffness. In concretes with alkalis contents lower than 3 kg/m^3 , even with the same percentages of natural rice husk ash, there were no significant decreases in the mechanical properties.

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1. Introduction

Rice husk, an agricultural waste with low nutritional properties for animals, constitutes about one fifth of the 500 million metric tons of rice produced annually in the world. Considering that 20% of the grain is husk, and 20% of the husk after combustion is converted into ash, a total of 20 million tons of ash can be obtained. Due to the growing environmental concern, and the need to conserve energy and resources, efforts have been made to burn the husks at controlled temperature and atmosphere, and to utilize

the ash so produced as a building material. In the last decades, the use of this residue in civil construction, especially in addition to concrete, has been subject of many researches.

Rice-husk ash (RHA) is a pozzolanic material and its reactivity and particle size depend on the burning and grinding conditions under which it is produced. In general, the average particle size ranges from 5 to 10 μ m, and the specific surface area ranges from 20 to 50 m^2/g [\[1\]](#page--1-0). The use of RHA as a supplementary cementing material is of great interest to many developing countries where rice production is in abundance. A highly reactive pozzolan is obtained when RHA is burnt under controlled conditions. In other conditions a ''residual RHA'' is produced with a lower quality, but it can be improved by grinding.

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Zerbino et al. [\[2\]](#page--1-0) showed that it is possible to use residual RHA ''as nature'' adopting a mixing process with the coarse aggregates of concrete to reduce the particle size. In that paper results of fresh and mechanical properties as well as water permeability of concretes replacing cement by residual RHA previously grinded (GRHA), RHA without grinding (NRHA) and a Control concrete without ashes were presented. When compared with Control concrete (without ashes) concrete replacing 15% of the cement by NRHA achieved similar mechanical and durability properties. However better results were obtained with GRHA. Regarding other particular properties, it was shown that matrix–aggregate bond strength improved when GRHA or NRHA were incorporated. No significant effects on the stress–strain behavior in compression or in the residual properties after high temperature exposure were found. These results definitively prove that the incorporation of natural RHA in concrete represents a good alternative for disposal of this residue, obtaining some technical benefits, even without the previous optimization through a grinding process, with a significant ecological positive impact.

Nevertheless as RHA contains siliceous vitreous minerals and cristobalite, deleterious reactions, such as alkali–silica reactions (ASR), with portland cement can take place when alkalis and certain environmental conditions are present. Damage due to ASR in concrete was first recognized by Stanton [\[3\]](#page--1-0) in USA and has since been observed in many other countries. Most researchers agree that is a reaction between certain forms of silica present in the aggregates and the hydroxide ions (OH^{-}) in the pore solution of the concrete $[4]$. The three major factors to develop the reaction are: alkalis in the pore solution, reactive silica, present in certain aggregates such as volcanic glass, cryptocrystalline silica (opal, trydimite, cristobalite, and chalcedony), strain quartz and the presence of water. Other factors can play a significant role, such as environmental relative humidity, porosity of the concrete and presence of mineral admixtures. Analytical techniques such as scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) and/or X-ray diffraction (XRD) together with standard petrographic examination are widely used methodologies in the identification of damaging processes in concrete structures [\[5\].](#page--1-0)

Microstructural analysis represents a very important tool to evaluate ASR in concrete structures, especially when complemented by macroscopic observations of cracks and other signs of reaction. Microstructural analysis includes examination of plane polished sections, examination of thin sections in a polarization microscope and occasionally scanning electron microscopy (SEM) analysis, as well. The crack patterns are of particular importance in the ASR diagnosis. Many petrographers only use microstructural analyses (i.e. analysis of thin sections and SEM) in their examinations. These analyses give a very good view of details in the interior of a structure, but they are less suited to give a sufficient overview of the extent of cracking in the concrete. The experiences have shown the importance and advantages of using larger samples to describe the crack intensity and crack pattern in drilled concrete cores by analysis of plane polished sections. Thus, a better correlation to what is observed in the field might be obtained. However, the presumption is that an experienced person performs the field survey, in addition to selecting representative sampling locations on the structures. It is also important to carry out a comprehensive visual examination of the drilled cores when they arrive in the laboratory as a basis for detail planning of the laboratory program [\[6\].](#page--1-0)

Zerbino et al. [\[7\]](#page--1-0) studied the development of ASR in mortars and concretes prepared with NRHA and GRHA. The experimental work included accelerated and long term expansion tests, mechanical characterization, microscopic observations and studies on prototypes. It was observed that the incorporation of RHA in concrete implies risks of expansions and mechanical degradation by reactions with alkalis. Accelerated tests (ASTM C 1260) in mortars showed that NRHA increases the expansion, while previously ground ash (GRHA) can lead to inhibition or exacerbation of the ASR, depending on the percentage used. It was proved that, as expected, the damage levels strongly depend on the cement used. Thus, when using NRHA the cement must be carefully selected, since some cements can enhance the risk of ASR. It was also observed that the presence of pozzolans can minimize the risk of reaction when NRHA is incorporated. Concretes incorporating GRHA showed expansions (ASTM C 1293) below the limit of 0.040%, while drastic expansions were found in concretes with NRHA. Although the use of NRHA leads to a residual mechanical behavior characteristic of damaged concretes, no typical signs of ASR as the presence of gels were found. Finally, the performance under field conditions of slab prototypes incorporating GRHA and NRHA and different alkalis contents was studied; it was found that when external alkalis were added the degradation of concretes incorporating NRHA was in accordance with the behavior expected from mortar and concrete prisms results. When alkalis were supplied only by the cement, prisms tests and field measurements indicated admitted grades of expansions but a few small fissures were observed on the slab with higher content of cement ($Na₂O_{eq}$ equal to 2.86 kg/m^3).

After four years, cores were extracted from those slabs in order to identify reaction products and to analyze the microstructural characteristics. The objective of this work is to discuss the causes and the damage processes involved in the development of expansions in presence of NRHA. The study is based on optical microscopy observations on thin sections and was complemented with the evaluation of the crack pattern of the slabs and the results of strength and surface strain evolution along the time.

2. Experiences

2.1. Materials

A residual rice husk ash (generated by uncontrolled burning conditions), from Rio Grande do Sul State, Brazil, was used ''as nature'' without previous grinding. After the burning process, the ash is only dried, homogenised, and packed to ease transport to the laboratory. As explained in a previous paper [\[2\]](#page--1-0) the weak burnt particles of the natural rice husk ash (NRHA) can be ground during concrete production by adopting an adequate sequence during the mixing process, where the NRHA and the coarse aggregates are mixed together during a certain time before the rest of the component materials are incorporated. As a result, the size of NRHA particles is significantly reduced being the mean size near to 150 lm. Details about the chemical analysis and how the ashes were obtained are presented in $[2]$. For comparison, an optimized ground rice husk ash (GRHA) obtained by grinding the NRHA for one hour in a ball mill was also used. The Blaine specific surface area of the GRHA was 750 m^2/kg and the particles mean size ranged between 4 and 6 lm, with a few particles as large as 100μ m. [Table 1](#page--1-0) presents the physical characteristics of the ashes, including size analysis of NRHA after 10 min mixing together with the coarse aggregates in a concrete mixer.

[Fig. 1](#page--1-0) shows the X-ray diffraction (XRD) analysis of GRHA and NRHA. As expected the results of both ashes are very similar. It can be seen a clear peak of cristobalite (c), abundant amorphous siliceous material, evidenced by the elevation of the base line of the diagram between 2 θ angles 20° and 30° and some quartz attributed to the presence of impurities.

[Fig. 2](#page--1-0) shows the results corresponding to the DSC–TGA (differential scanning calorimetry and thermogravimetric analysis). It can be seen in both samples, a total weight loss near to 6% and near 4% when considering from 100 \degree C. This percentage is attributed to unburned material (see [Table 1](#page--1-0)).

Two Ordinary Portland Cements identified as OPC1 and OPC2 were used; their respective total alkali contents $Na₂O_{eq}$ are 0.66% and 0.80%.

Non-reactive aggregates, siliceous natural sand and granitic crushed stone, were used. The coarse aggregate is a granitic crushed stone 19 mm maximum size that when tested in accordance with ASTM C 1293 gave expansions of 0.006% at 52 weeks. The structures in which it has been used show excellent performance both in strength and durability. The fine aggregate is a siliceous natural sand obtained from the east coast of the La Plata River (fineness modulus 2.36, density 2.63); it has rounded particles composed of quartz (>70%), lithic granitic fragments, feldspars (plagioclase), pyroxenes and a very small amount (0.5%) of chalcedony. Expansion tests according to ASTM C 1293 and ASTM C 1260 indicate expansions of 0.038% at 1 year and 0.080% at 16 days, respectively.

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