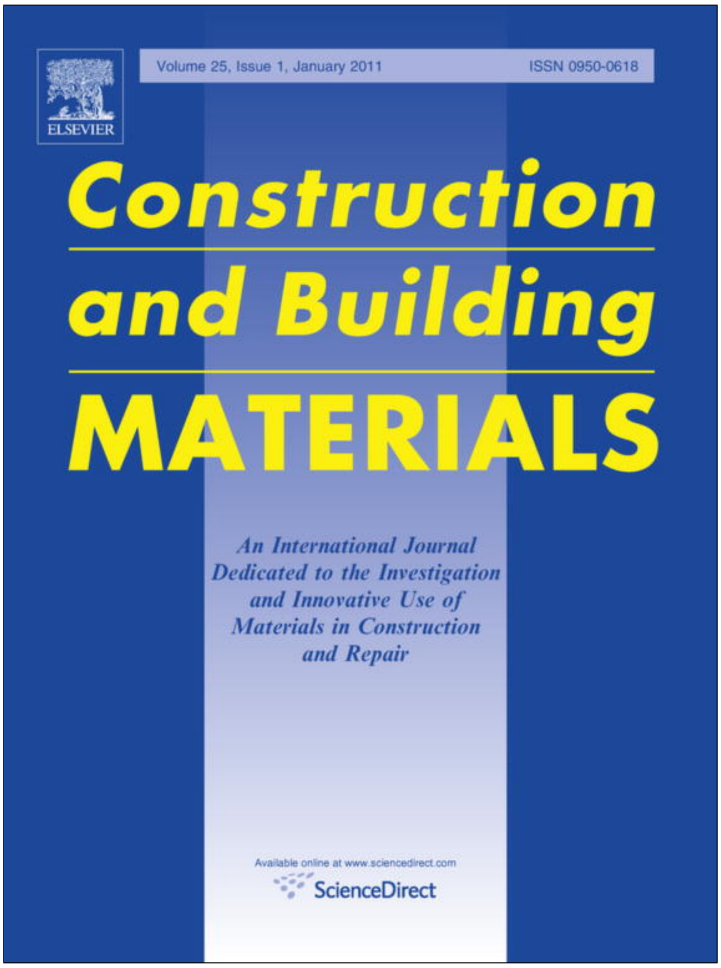
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Effect of off-white rice husk ash on strength, porosity, conductivity and corrosion resistance of white concrete

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A B S T R A C T

Rice husk ash (RHA) is one of the promising pozzolanic materials that can be blended with Portland cement for the production of durable concrete and the reduction of the environmental impact of the cement industry. Commercially available RHA contains 3% or more graphitic carbon which determines the dark pigmentation of the material. Recent studies have led to the production of carbon neutral rice ash named OWRHA (Off-White Rice Husk Ash), with no graphitic carbon, no crystalline SiO_2 and toxic metals, so legitimately considered environmental friendly. This paper presents the results of an experimental investigation on the strength, porosity, corrosion resistance and thermal conductivity of white concrete blended with OWRHA. The results show that the compression strength of the concrete increases with replacement level of OWRHA, while the porosity decreases. Accelerated corrosion tests demonstrate that the use of OWRHA increases the corrosion resistance at all levels of replacement. The results on mortal samples showed that also thermal conductivity and density decrease with the increase in OWRHA and with age. With the intention of reducing the carbon footprint in the cement industry, this study evidences encouraging results for the use of OWRHA in sustainable construction.

1. Introduction

The need of reducing the carbon footprint associated to cement production drove much research towards the study of by-products to be used as supplementary cementitious materials (SCMs). Significant research has been directed towards the utilization of rice husk ash (RHA) as SCM [1,2]. Rice husk is a major agricultural waste, constituting the largest (about 20%) and most worthless by-product of the rice milling industry. About 70 million tons of RHA is produced annually worldwide [3], and since the husk does not biodegrade or burn easily [4], it is considered as an environmental nuisance. The burned ash of rice husk contains the highest proportion of silica among all the plant residues: nearly 20% silica in amorphous form [5], and under controlled combustion conditions, this by-product can produce an amorphous silica with high reactivity [6]. Spire et al. [7] studied the properties of mortars containing different percentages of RHA and noted an increase in compressive strength, and a reduction in absorption characteristics and in oxygen permeability. Cost reduction, performance, durability, and environmental concerns are the primary characteristics that can make RHA a competitive additive to Portland cement [8–11].

Until the 1970s, the production of RHA was by uncontrolled combustion, and for this reason the product was usually crystalline and with a considerable amount of unburned carbon (∼30%), resulting in a poor pozzolanic activity [12]. After Mehta [12] described the effect of pyroprocessing parameters on the pozzolanic reactivity of RHA, Pitt [13] designed a fluidized-bed furnace for controlled combustion of RH, which allowed for the control of temperature and atmosphere, and to obtain a highly reactive RHA. Nowadays, RHA produced with commercially available equipment contains 3% or more of graphitic carbon, which determines the dark pigmentation of the material. This level of carbon content restricts its utilization to architectural applications where color is the driver, and leads to excessive demand from water and chemical admixtures in order to maintain appropriate slump and air content [14,15]. There have been some attempts to produce amorphous SiO_2 from rice husk using different burning techniques, but none of these methods has successfully produced commercial-scale off-white amorphous SiO_2 with less than 1% amorphous carbon [16–19]. In 2006, Vempati [16] developed a new continuous production process to manufacture RHA. The rotary tube furnace was maintained in aerobic conditions and at a temperature of 700 °C with a residence time of 40 min to obtain off-white RHA with carbon content less than 0.3%.

The purpose of this study is to characterize the RHA produced as per Vempati [16] and to examine its effect on blended concrete. This RHA is off-white in color, with no graphitic carbon, no crystalline SiO_2 and toxic metals, and is legitimately considered environmen-

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tally friendly. Furthermore, the absence of graphitic carbon allows the off-white appearance of the product and opens the door to architectural applications and sustainable constructions where the reflective propriety of the concrete surface can improve the building energy performance. In this work, Off-White Rice Husk Ash (OWRHA) was characterized and blended with white Portland cement (WPC) in different ratios to evaluate strength, porosity, corrosion resistance and thermal conductivity. The knowledge of such parameters is fundamental for possible applications of OWRHA in construction and in particular in the decorative and sustainable construction industry.

2. Experimental investigation

The study was developed in two stages. In the first stage, physical and chemical properties of OWRHA were studied in order to define the pozzolanic activity of the material. X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetric Analysis (TGA), Particle Size Analysis, and scanning electron microscopy (SEM) were performed on OWRHA samples. In the second stage, concrete specimens with different percentage of OWRHA were tested. In the second stage, the effect of OWRHA dosages over varied curing periods on mechanical proprieties, porosity, corrosion resistance and thermal conductivity of OWRHA blended concrete were analyzed.

The combination of the two stages is needed to demonstrate the pozzolanic propriety of the novel byproduct, and so the validity to be employed as SCM in the second stage.

2.1. Stage I

2.1.1. OWRHA and WPC

The off-white rice husk ash was manufactured by burning rice husk procured from a rice mill plant located at Jonesboro, AK. The off-white RHA with <0.3% amorphous carbon was produced as per Vempati et al., and details of the process can be found elsewhere [17]. Unground OWRHA was used after determining that its particle size was comparable to that of white Portland cement (WPC). Generally, reactivity of cementitious materials is favored by increasing the fineness [20–22]; however, Mehta [6] demonstrated that a high degree of fineness of RHA's grinding should be avoided since this material gains its pozzolanic activity mainly from the internal surface area of the particles. OWRHA's specific gravity is 2.2 and its main chemical components are summarized in Table 1 together with those of WPC. The total percentage composition of iron oxide (Fe_2O_3 = 0.13%) silicon dioxide (SiO_2 = 94.8%) and aluminum oxide (Al_2O_3 = 0.52) was found to be 95.45%. The value is considerably above the required value of 70% minimum for pozzolans (ASTM C 618).

2.1.2. X-ray diffraction (XRD)

The mineralogy of OWRHA was obtained with a Philips X'pert System diffractometer (Philips Electrical Co., Almelo, Netherlands). Using about 150 mg, the XRD patterns were recorded with a diffraction angle of 5–40 °2θ at a scan rate of 3° min^{-1} using Ni-filtered Cu Kα (1.584 Å) radiation. The X-ray diffraction pattern (Fig. 1) showed a pattern which is typical for amorphous solids [23]. The broad smooth hump between 15(°2θ) and 35(°2θ) indicates that combustion converted the ordered crystalline cellulose structure into a random amorphous structure. Previous studies have shown that RHA with most of its silica in an amorphous form, as in this case, is very reactive and can considerably improve the strength and durability of concrete [5].

Table 1
Chemical compositions of white Portland cement and off-white rice husk ash (by Mass).

Compound	WPC (%)	OWRHA (%)
SiO_2	23.06	94.80
Al_2O_3	4.46	0.52
CaO	66.60	N/A
MgO	0.26	N/A
SO_3	3.00	N/A
MnO	N/A	0.39
Fe_2O_3	0.25	0.13
Alkalis	2.37	2.92
C	N/A	0.24
P_2O_5	N/A	1.09

N/A = not available.

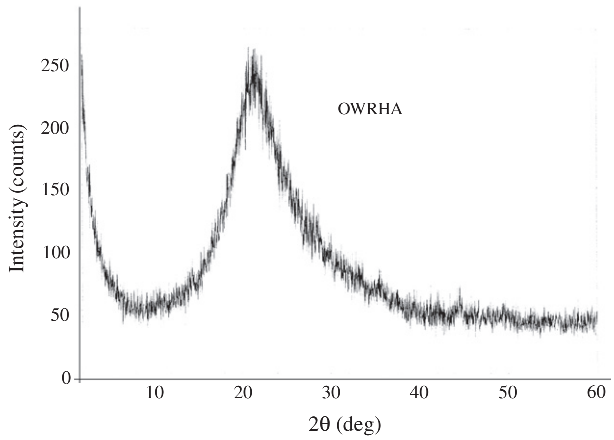

Fig. 1. X-ray diffractogram of off-white rice husk ash.

2.1.3. Fourier transformation infrared spectroscopy (FTIR)

The off-white rice husk ash was characterized by infrared spectroscopy. The FTIR spectrum of OWRHA was collected in the transmission mode using a Perkin Elmer 2000 (Perkin Elmer, Massachusetts, USA). Three hundred milligrams of KBr was mixed with 6 mg of the sample, and 128 scans were collected and averaged. The FTIR spectrum of OWRHA is presented in Fig. 2. The FTIR study showed the presence of –O–Si–O– vibrations attributable to asymmetrical stretching bands which occurred around 1220 cm^{-1} and 1080 cm^{-1} in the form of shoulder and broad bands, respectively. Symmetrical stretching band occurred at 773 cm^{-1}, and Si–O bending band at 466 cm^{-1}. Free water bands were observed at 3436 cm^{-1}. The position of these bands confirmed that the material was amorphous SiO_2.

2.1.4. Thermo-gravimetry analysis (TGA)

Thermogravimetric analysis of OWRHA was conducted using a TGA-HP (TA Instruments, Delaware, USA). The analysis was carried out by heating the sample from ambient to 950 °C at a rate of 20 °C per minute while under a nitrogen atmosphere. The total weight loss over the temperature range was then calculated using Universal Analysis software (TA Instruments, Delaware, USA). The TGA indicated that the loss of ignition of OWRHA is ~0.03% (Fig. 3), which demonstrates the absence of residual combustible carbon, reducer of the pozzolanic activity.

2.1.5. Particle size distribution

Particle size distributions of WPC and OWRHA were determined using a laser diffraction particle size analyzer (Malvern Instruments, Worcestershire, UK). The particle size distribution curves shown in Fig. 4 suggest that WPC and OWRHA have comparable particle size distribution. The particle density was 3.15 g/cm^3 for both materials and the specific surface area is 0.43 m^2/g for OWRHA and 0.49 m^2/g for WPC. Previous study showed that the high fineness of RHA had a greater pozzolanic reaction and the small particles could also fill in the voids of the mortar mixture, thus increasing the compressive strength and durability of the mortar [24,25]. Therefore, if unground OWRHA can increase the properties of the concrete, then finer particles (higher grinding time) may further improve the final product.

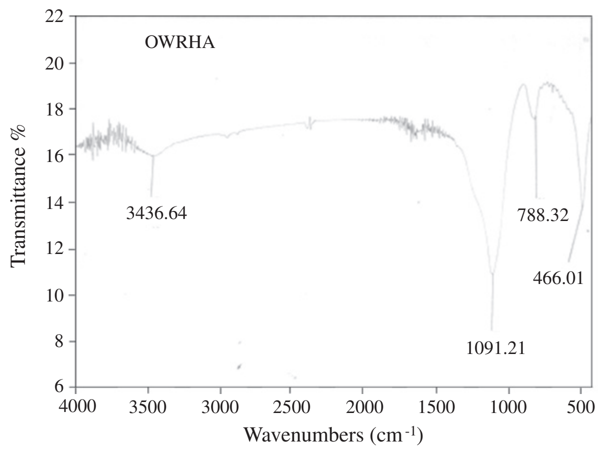

Fig. 2. Fourier transformation infrared spectroscopy of off-white rice husk ash.

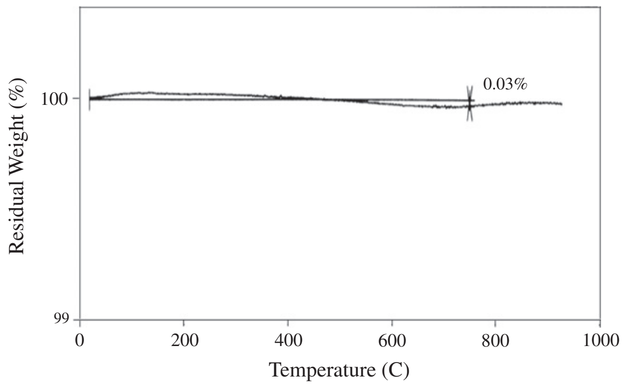

Fig. 3. Thermo-gravimetric analysis of off-white rice husk ash.

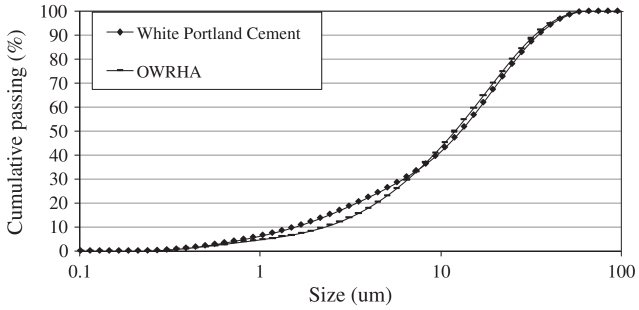

Fig. 4. Particle size distribution of off-white rice husk ash and white Portland cement.

2.1.6. Scanning electron microscopy (SEM)

The SEM analysis was performed on WPC and OWRHA to study the microgeometry of the material. The particles were imaged using an Environmental SEM (ESEM) system (Philips XL30 ESEM-FEG, Philips Electrical Co., Almelo, Netherlands). Scanning electron micrographs of the material were taken in the variable pressure secondary electron (VPSE) mode with the following instrument parameters: electron beam energy and probe current were 5 kV and 170 pA, respectively, with a variable working distance. The SEM image reveals that OWRHA consists of very irregular-shaped particles with porous cellular surface (Fig. 5) typical of unground RHA [26]. The basic cellular structure comes from the organic material which OWRHA is derived from and is responsible for the high surface area of the material even when the particles are not small in size [14]. Considering the porous nature of the OWRHA grains and the high internal surface area, the process of grinding to form smaller particles is not expected to make a significant difference in the total surface area. However, the large size of the grains together with their shape will likely results in remnants of unreacted particles in hardened concrete that will serve as pockets of excess porosity.

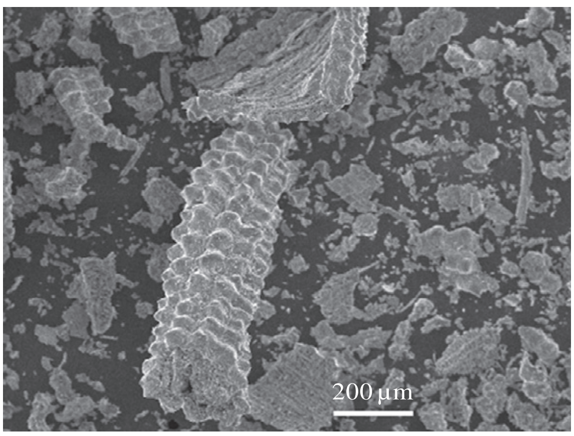

Fig. 5. Scanning electron microscopy of off-white rice husk ash.

2.1.7. Stage I conclusions

From the characterization carried in Stage I, it can be stated that OWRHA has proven to be an effective pozzolanic material, with high amorphous silica content and high specific surface area.

2.2. Stage II

The effect of OWRHA as supplementary cementitious material for white Portland cement both in cement and mortar is evaluated in Stage II.

2.2.1. Materials

Clean river sand with specific gravity 2.55 and fineness modulus of 2.49 was used as fine aggregate; these proprieties were evaluated according to respectively ASTM C128-07a and ASTM C125-11a. Locally available (South Florida) well ground limestone of size greater than 4 mm and less than 12 mm, with specific gravity of 2.6 evaluated as per ASTM C127-07, was used as coarse aggregate.

White Type I Portland cement conforming to ASTM C 150-07 was utilized. The cement had a specific gravity of 3.15 and a fineness of 408 m^2/kg. The main chemical components of the WPC used are presented in Table 1.

High-range water reducing admixture (Adva 140 M, Grace Construction Product, Maryland, USA), was used to maintain a consistent workability, expressed as a constant slump without any additional amount of mixing water. This superplasticizer is based on polycarboxylate technology and meets the requirements of ASTM C494 as a Types A and F, and ASTM C1017 Type I.

Following the industry practice, a typical slump for ordinary decorative application would be in the 100–130 mm range, and this value was adopted as the target in the study of the different mixtures tested.

2.2.2. Mix and specimen characteristics

Three mixtures with different proportions, including the control one, were prepared with a water/cement ratio of 0.44. The mixtures were designed following the information from literature review [28,29]. Blended cements were prepared by replacing WPC with OWRHA in dry conditions, they were thoroughly homogenized and kept in polyethylene containers. The concrete proportions and the designations are presented in Table 2.

Plain concrete cylinders 100 mm in diameter and 200 mm in height were used to determine the compressive and splitting tensile strengths of each batch. The specimens were cast in two layers, compacted and cured according to ASTM C 192.

For the durability test, plain concrete blocks 100 by 100 by 50 mm were used for this investigation. The samples were cast, compacted using a vibrating table, and moist cured at 20 °C with a RH of 95%.

For the corrosion test, concrete prism 51 by 51 by 152 mm, with an embedded #2 (6 mm) steel rebar 152 mm long were used. The bar was positioned in the prism as shown in Fig. 6. The exposed area of the steel bar was protected with an epoxy resin coating. The concrete prisms were cast in two layers, vibrated and covered for 24 h with a plastic sheet. Following this period, the specimens were demolded and cured in water.

The thermal conductivity test was performed on mortar specimen with binder/sand ratio 1:2 and water/cementitious ratio 0.44; three batches were prepared replacing respectively, 0%, 7.5%, and 15% by weight of WPC with OWRHA. For each batch, 18 disc samples with diameter 54 mm and nine 50 mm cubes were prepared and used respectively for the thermal conductivity and compressive testing. Each conductivity test is performed on two identical discs of different nominal thickness, 12 and 24 mm. After vibrating and trowel finishing, the molds were covered with a plastic sheet for 24 h and stored in water at 20 °C until testing. After curing, the samples were dried in an oven at 110 °C and weighted at 24 h intervals until the loss of weight did not exceed 1% in a 24 h interval (ASTM C332); then, tested for thermal conductivity. In order to reduce the error due to the surface contact between the coupon and the testing plates, the samples were polished with sandpaper before testing. Three different sandpaper grits were used 60, 120, and 150 (following the Coated Abrasive Manufacturers Institute CAMI classification) in the same order as listed. Samples were tested after 7, 28, and 90 days. Three cubes for each batch and age were tested in compression following the ASTM C109.

2.2.3. Compressive and splitting tensile strengths

Compression and splitting tensile tests were performed on three cylinders for each batch and age. Even though the sample size is limited and does not allow for a complete statistical analysis, it remains valid for relevant considerations on properties trends. The compressive strength of each batch was determined in accordance with ASTM C 39 after 7, 28 and 90 days of moist curing at 20 °C and relative humidity of 95%. Splitting tensile strength tests were conducted as per ASTM 496M after 28 days of moist curing at 20 °C and RH of 95%. A SATEC MKIII-C with 690 MPa capacity was used for both tests.

Table 3 reports the average and the normalized value with respect to the 0% OWRHA batch, of compressive strength of the three batches. The experimental results reveal that the incorporation of either 7.5% or 15% of OWRHA positively affects the strength of concrete. Similarly as reported in Table 3 the 15% OWRHA blended

Table 2
Mix proportions.

Code	OWRHA (%)	Quantities (kg/m^3)					Water/cementitious ratio
		Cement	OWRHA	Sand	Coarse aggregates	Water	
W0	0	471	0				
W7.5	7.5	435	35.2	1193	468	211	0.44
W15	15	398	68.8				

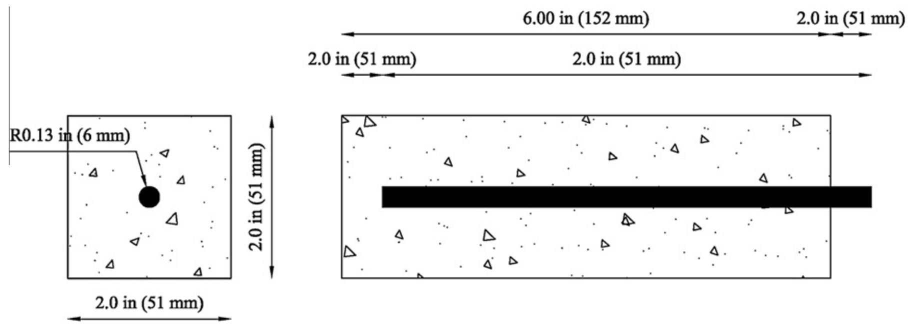

Fig. 6. Geometry of the accelerated corrosion test with impressed voltage sample.

Table 3
Average concrete compressive and tensile strengths at different ages.

Code	Average compressive strength									Average splitting tensile strength		
	7 days			28 days			90 days			28 days		
	MPa	Normalized	COV[a] (%)	MPa	Normalized	COV[a] (%)	MPa	Normalized	COV[a] (%)	MPa	Normalized	COV[a] (%)
W0	27.6	100	3	36.0	100	2	37.9	100	0.4	4.5	100	4
W7.5	28.0	101	1	40.9	113	1	44.3	117	1	4.6	103	2
W15	29.2	106	1	41.6	115	1	45.6	120	1	4.9	109	1

[a] Coefficient of variation.

concrete shows an increase of the splitting tensile strength of ~9% with respect to the control concrete. Only very low increase (~2%) was demonstrated for the 7.5% blend.

2.2.4. Effective porosity

The percentage of water absorption is considered a measure of the pore volume (porosity) in hardened concrete, which is occupied by water under saturated condition. The values of the water absorption of the three batches were measured 28 days of moisture curing, following the ASTM C642.

For each batch, three samples were tested. The porosity in all batches decreases with age as a result of the hydration of cementitious materials. Fig. 7 shows the values of the effective porosity of the three batches. The effective porosity decreases with the increase in percentage of OWRHA. This reduction is probably due to the porous microstructure of the RHA also observed with the SEM analysis. A more impermeable concrete delays the permeation of Cl^- ions, reducing the corrosion current and extending the time of damage occurrence [28,29].

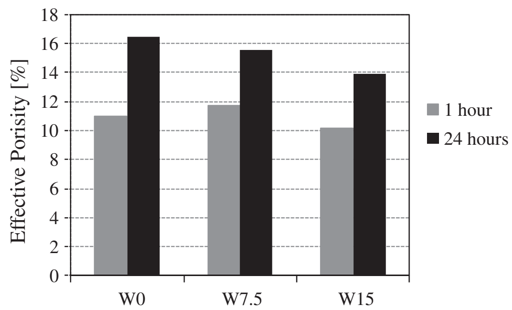

Fig. 7. Effective porosity test results after 28 days.

2.2.5. Coefficient of water absorption

The coefficient of water absorption is used as a measure of water permeability [27]. Following ASTM C642, this parameter is evaluated through the rate of water uptake of dry concrete in a period of 60 min. The test was conducted on three samples for each batch and age.

Table 4 presents the coefficient of water absorption of the OWRHA blended concrete specimens at 7, 28 and 90 days. In addition to the coefficient of water absorption, the table shows the normalized values with the respect to the 0% OWRHA concrete and the coefficient of variation (COV). It is observed that the coefficient of water absorption for the OWRHA blended concrete up to 15% replacement is less than that of the 0% benchmark concrete. The coefficient slightly decreases with time, and at 90 days, for all three mixtures, the values are about 25% smaller than the values at 7 days. This confirms that prolonged curing results in a low reduction of permeable voids for the OWRHA concrete (with replacement level up to 15%). The low porosity reduction can be caused by unreacted grains that create porosity in the hardened concrete; this porosity becomes a medium for the movement of the water in the concrete that reduces the effectiveness in the controlling of the water absorption.

2.2.6. Accelerated corrosion test with impressed voltage (ACTIV)

After 28 days of water curing, the prisms were subjected to accelerated corrosion testing with impressed voltage. The impressed voltage technique is an accelerated corrosion conditioning method that can provide valuable information on the permeation characteristics of the concrete [28,29]. The reinforced concrete specimens illustrated in Fig. 6 were immersed in a 5% NaCl solution. The embedded steel rebar acts as the anode with respect to an external stainless steel cathode when applying a constant potential of 12 V to the system (Fig. 8). The condition of the

Table 4
Coefficient of water absorption of concrete at various percentage of off-white rice husk ash replaced concrete.

Code	Average coefficient of water absorption								
	7 days			28 days			90 days		
	$\times 10^{-11}$ m^2/s	Normalized	COV* (%)	$\times 10^{-11}$ m^2/s	Normalized	COV* (%)	$\times 10^{-11}$ m^2/s	Normalized	COV* (%)
W0	4.29	100	1	1.05	100	3	1.04	100	5
W7.5	4.00	93	4	1.04	98	2	0.97	90	2.5
W15	4.12	96	2	1.06	101	4.5	0.96	89	5.5

* Coefficient of variation.

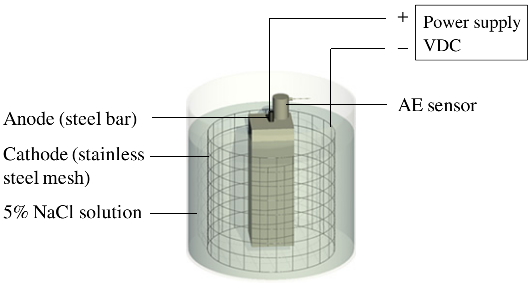

Fig. 8. Accelerated corrosion test with impressed voltage test set up.

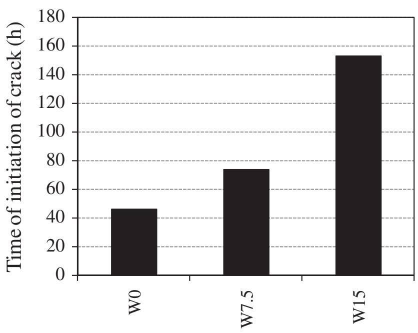

Fig. 9. Time of initiation of crack of mortars subjected to accelerated corrosion test with impressed voltage.

sample was continuously monitored and the time of development of the first crack was recorded. The time of first crack was identified with the time when the first visible crack of width of approximately 0.05 mm appears [30].

This parameter is used as measurement of the specimen's relative resistance to reinforcement corrosion.

Fig. 9 shows the results of the ACTIV test in terms of time of the first crack. The first cracking for the WPC samples starts after about 46 h. The use of 7.5% OWRHA concrete extends this time to 74 h, which further increases to 153 h when using 15% OWRHA concrete.

2.2.7. Thermal conductivity and density

To study the influencing mechanism of OWRHA on mortar three series of specimens with different percentage of OWRHA (0%, 7.5%, and 15%) were fully sutured at 20 °C and tested at 7, 28 and 90 days after mixing. The samples were prepared for the thermal conductivity test and density was measure as indicated by ASTM C332. Following the ASTM C 518 the thermal conductivity of OWRHA blended paste was measured by a FOX50 heat flow meter (LaserComp Inc., Livonia, USA). A set of two samples of different thickness for each sample type are tested in order to apply the 'Two-Thickness procedure' which eliminates air interface error from the results [30–32]. The measurement range of this device is 0.1–10 W/mK with an error of about ±3.5%. The results of this test are summarized in Table 5; this table allows to understand the influencing mechanism of thermal conductivity on the age of the mortar and the amount of cement replaced by OWRHA. The results show that OWRHA (up to 15% replacement of WPC) reduces the thermal conductivity of the mortar. This behavior can be associated to the fact that the density of the mortar decreases with increase in cement replaced by OWRHA (up to 15%). As previous studies indicated [33–37], the incorporation of supplementary cementitious materials in mortars increases the air content. However, since air has a conductivity 25 times lower than water, it is clear that when the pore are filled with air instead of water, the mortar has a lower conductivity. Additionally, other studies also reported that the thermal conductivity increases with density [38]. Thus, the thermal conductivity of the mortar decreases with the age and the amount OWRHA replaced, this is due to a reduction of the density and an increase in air content.

Table 5
Mortar properties.

Properties	OWRHA Replacement level (%)								
	0			7.5			15		
Age (days)	7	28	90	7	28	90	7	28	90
Thermal conductivity (W/mK)	1.423	1.215	1.201	1.395	1.102	1.060	1.281	0.990	0.971
Density (kg/m^3)	2099	2049	2041	2080	2060	2031	2053	2010	1985
Compressive strength (MPa)	33.2	45.9	46.8	34.5	47.8	49.5	34.9	49.2	52.4

The compressive strength was tested according to ASTM C 109 and the results are in agreement with the ones obtained for concrete.

3. Conclusions

An experimental study was conducted to determine the effect of OWRHA as a supplementary cementitious material for white concrete production, for values of replacement of WPC of 7% and 15% by weight. Based on the test results, the following conclusions are drawn:

1. Due to the high amorphous silica content and specific surface area, OWRHA is an effective pozzolanic material.
2. A percentage of WPC as high as 15% by weight can be replaced with OWRHA without any adverse effect on strength properties. In particular, both compressive and tensile strength of OWRHA blended white concrete increases with cement replacement percentage and specimen age.
3. The open porosity and the coefficient of water absorption of the concrete minimally decrease with the increase in percentage of OWRHA (up to 15%).
4. The resistance to corrosion under ACTIV conditioning is improved in comparison to that of all Portland cement concrete. The pore refinement and the impermeability of the concrete (responsible of the permeation of Cl^- ions) can play an important role in the corrosion resistance of the RC incorporating OWRHA.
5. The replacement of white cement with 7.5% and 15% of OWRHA in mortar was effective for decreasing the thermal conductivity of the mortar. Also the density of the mortar was found to decrease with the increase in OWRHA percentage.
6. The measurements of porosity, coefficient of water absorption and thermal conductivity indicate that the replacement of OWRHA in concrete reduces the open porosity that relates to the ingress of water in the concrete, but increases the closed porosity (because of the RHA structure) resulting in a reduction of thermal conductivity.

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