



# An investigation on mechanical and durability properties of mortars containing nano and micro RHA



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## HIGHLIGHTS

- Use of nano-RHA and micro-RHA in mortars.
- Compressive strength, capillary absorption, chloride and electrical resistivity.
- Incorporation of nano-RHA and micro-RHA demonstrated better performance at higher ages.
- Nano-RHA enhanced the durability and strength at early ages due to high surface area.

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## ABSTRACT

This study investigated the effects of nano rice husk ash (NRHA) and micro rice husk ash (MRHA) on chloride permeability, electrical resistivity, capillary absorption and compressive strength of mortars. It was found that the incorporation of NRHA and MRHA enhanced mortar performance in the long term, whereas NRHA contributed to considerable enhancements at both early and long-term ages. Results proved that the strength and durability properties of mortars containing NRHA up to 7.5% levels of replacement by weight of cement gradually enhanced in comparison with control mix. However, mortars incorporating 2.5% NRHA together with 12.5% MRHA demonstrated the best improvements for durability and strength development at the age of 90 days.

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## 1. Introduction

Aggressive agents of chloride ions penetration into the concrete reinforcement is one of the most usual corrosive attacks in harsh environments that leads to several destructive consequences such as reduction of serviceability and strength. Cracks are the facilitator of the penetration process and certain routes for accession of moisture and oxygen to the reinforcement. Furthermore, the presence of enough chloride ions would cause the deterioration of passive layer adhered to steel rebar [1].

It is highly accepted that the incorporation of a pozzolanic material can reduce the permeability of concrete and enhance its resistance against chloride ions penetration [2]. The usage of supplementary cementitious materials such as rice husk ash has not

only reduced the fuel demand for cement production and environmental pollution but also improved the mechanical properties and durability of concrete especially in aggressive environments [3].

Rice approximately covers 1% of land area of the earth and it is one of the main sources of food for billions of people [4]. One of the main agriculture industry residues is rice husk, which contains high amount of amorphous silica up to 85–95%. Rice husk ash (RHA) is a solid residue of rice husk controlled combustion, which shows the highest pozzolanic behavior among all plant residues productions [5].

Many of the previous studies have concentrated on investigating the optimum conditions for rice husk controlled combustion such as burning time and temperature, oxidizing procedures and heating rate in order to produce reactive ash [5]. In this regard, several studies [6,7] have concluded that incineration of rice husk at temperatures below 500 °C is imperfect and appreciable amount of unburned carbon is not expelled yet in this circumstances, which can result in adverse effects on ash pozzolanic activities.

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On the other hand, burning at temperatures higher than 700 °C can lead to producing RHA with crystalline pozzolanic activity instead of amorphous one which is not preferable due to the reduction in reactivity [8–11].

Uniform clusters of calcium-silicate-hydrates (C-S-H) are formed through the chemical pozzolanic reactions between the calcium hydroxide produced by the cement hydration and amorphous silica of RHA that result in a higher densification of the matrix [12]. The addition of well pulverized RHA to cement paste could provide an environment for RHA particles to fill the voids between cement grains both physically and chemically and result in the reduction of porosity and enhancement of bond between cement and aggregates [13,14].

Researchers [13–19] have proven that the incorporation of RHA in mortars can enhance the compressive strength in the long-term. However, they concluded that this ameliorative effect is limited to optimum replacement level of 20% by weight of cement. On the other hand, few other studies have found that the durability properties of the RHA blended mortars could face great enhancement against chloride ions penetration with higher dosages of replacement in comparison with compressive strength [20,21].

In the last few decades, by the advent of nanotechnology, several studies devoted to investigating the effects of mineral nanoparticles on cementitious concretes and mortars [22–38]. The results showed that a large surface area provided by nanoparticles hasten the rate of pozzolanic reactions and cement hydration. In this regard, researchers have found that the binary blended mortars containing RHA and nano-SiO<sub>2</sub> with extremely fine amorphous silica particles would lead to prodigious enhancement of the compressive strength [39–43]. Results show that the increase in long-term strength is mainly dependent on RHA, whereas the nano-SiO<sub>2</sub> particles can extraordinary enhance the high early age strength. The addition of nano-alumina helped the compressive strength of mortars to enhance even when 30% RHA was used [40]. Unfortunately, only limited studies are concerned with durability properties of mortars containing RHA and nanoparticles such as nano-TiO<sub>2</sub> or nano-SiO<sub>2</sub> [41–44]. Results show that the incorporation of nanoparticles and RHA can contribute to chloride resistivity enhancement in early and long-term age, respectively. However, the study of nano-RHA addition into cementitious materials is still untouched.

In this study, the effects of micro-RHA (MRHA) and nano-RHA (NRHA) addition into mortars on the compressive strength, chloride resistivity, capillary absorption and electrical resistance at various replacement percentages were studied. Finally, the proportions of micro and nano-RHA to obtain minimum chloride penetration and optimal compressive strength was determined.

## 2. Materials

According to ASTM C150 [45] requirements, type II-425 Portland cement was used for all mortar mixtures. Its chemical and physical properties are shown in Table 1.

Rice husk was provided from Mazandaran rice fields and was burnt at a temperature of 650 °C and time duration of 1 h. After combustion, the rice husk ash was pulverized to nanoparticles by means of planetary ball mill in order to attain particle sizes smaller than 100 nm. Based on former studies, particles with dimensions lesser than 100 nm can be considered as nanoparticles [46]. Using trial and error method, three attempts with different time durations of 7, 10 and 13 h were performed to achieve optimum grounding time duration. In the final operation, RHA particles with the mean size of 35.86 nm were achieved by grounding time of 13 h with the speed of 400 rpm and the zirconium-balls/RHA mass ratio of 10:1. A micronizer

**Table 1**

Chemical and physical characteristics of type II Portland cement and RHA.

Chemical component	Cement (%)	RHA (%)
SiO <sub>2</sub>	22.28	83.74
Al <sub>2</sub> O <sub>3</sub>	4.72	0.29
Fe <sub>2</sub> O <sub>3</sub>	2.75	0.67
CaO	64.12	0.74
MgO	1.23	0.86
SO <sub>3</sub>	1.97	0.87
Na <sub>2</sub> O	0.28	0.091
K <sub>2</sub> O	0.76	2.84
P <sub>2</sub> O <sub>5</sub>	0.26	0.37
MnO	0.11	0.14
Loss of ignition (LOI)	1.12	8.39
C <sub>3</sub> S	50.44	–
C <sub>2</sub> S	25.82	–
C <sub>3</sub> A	7.86	–
C <sub>4</sub> AF	14.36	–
Physical properties	Cement	NRHA
Surface area (cm <sup>2</sup> /g)	3035	804,410
Specific gravity (g/cm <sup>3</sup> )	3.02	2.08

was used to pulverize the RHA to micro size with the mean size of 0.588 µm. In each turn, total of 200 g RHA was ground for 1 min in order to reach MRHA with the approximate size of cement particles. The results of zetasizer analysis for NRHA and MRHA are shown in Fig. 1. The XRF and XRD results of the RHA are also given in Table 1 and Fig. 2, respectively. The mostly low curved peaks of XRD diagram are representative of RHA high amorphous silica content which contribute a lot to enhance the pozzolanic activities.

River sand was graded in accordance with ASTM C778 [47] (see Table 2). The graded sand had a water absorption of 1.00%, fineness modulus of 3.49 and specific gravity of 2350 kg/m<sup>3</sup>.

A polycarboxylate based superplasticizer called ZP with pH of 7 ± 1 and specific gravity of 1.1 ± 0.02 was also used to adjust the desirable flow. Potable water was used for both curing and casting purposes.

## 3. Experimental program

In this study, eight different mortar mixtures were prepared by water/binder ratio of 0.46 and sand/binder ratio of 2.18. The proportion of mixtures containing NRHA and MRHA are presented in Table 3. The preparation procedure involved:

- (1) Materials weighing
- (2) Initial mixture of RHA powders with 30% of the mixing water to produce additive gel
- (3) Adjusting the pH value of the gel up to 9 by adding NaOH to gel in order to control surface charges of the RHA microspheres and better dispersion of the small particles
- (4) Mixing of gel, cement and remained water
- (5) Addition of sand
- (6) Mere mixing
- (7) Performing workability test using flow table according to ASTM C230 [48]
- (8) Addition of superplasticizer and further mixing
- (9) Casting specimens in two layers and vibrating each layer for entrapped air removal using vibration table.

After casting, wet towels were used to cover specimens for 24 h in order to prevent excessive water evaporation. For curing, the mortar specimens were demolded and placed in water (saturated with calcium-hydroxide) at 23 ± 2 °C until the test day.

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