Fundamental Investigations into Performance of Carbon-Neutral Rice Husk Ash as Supplementary Cementitious Material

K. V. Harish, P. R. Rangaraju, and R. K. Vempati

Although the benefits of rice husk ash (RHA) as a pozzolan have been known for a long time, its use in concrete has been limited. Past efforts to produce RHA have yielded ashes, some with high carbon content and others with some crystalline silica, neither of which is desirable for use of RHA as a pozzolan. Within the past decade, the production of off-white-colored RHA containing high levels of amorphous silica and very little carbon has become possible through innovations in incineration techniques. Results are presented from a study conducted to ascertain the properties of a low-carbon-content RHA and evaluate it as a supplementary cementing material. In addition, the as-obtained RHA was ground to a smaller particle size to study the effect of finer ash on properties of cementitious mixtures. Specific tests conducted include pozzolanic reactivity and microstructure of RHA and other properties of cementitious pastes and mortars. These include flow, initial and final setting time, compressive strength, flexural strength, and split tensile strength. In addition, other tests such as water absorption, effective porosity, and rapid chloride ion permeability were conducted to ascertain the improvements offered by RHA in enhancing durability of cementitious mixes. Comparative studies were conducted with silica fume mixes. Results from this study are very encouraging and suggest that RHA produced with a consistent quality (i.e., high amorphous silica content and low carbon content) can serve as an environmentally friendly and sustainable supplement for cementitious material needs in future; it can reduce the carbon footprint of concrete.

Rice hull (rice husk) remains as a major agricultural waste, and it is estimated that approximately 130 million tons of it is produced worldwide from an annual rice grain production of 659 million tons as of 2007 (*1*). It is estimated that the annual rice production in the United States is about 9 million metric tons, out of which 1.8 million metric tons are rice hulls (*1, 2*). Rice hulls have poor nutritional value for use as animal feed and find little application as raw material for production of other value-added materials. At present, disposal of rice hull involves land-filling, incineration, and other applications, such as animal bedding material, soil enhancer, and fuel for power generation (*3, 4*). Disposal of huge quantities of biomass through some of these traditional methods, such as open-heap burning, have raised some environmental concerns (*5*). Furthermore, the dust and smoke created by such burning processes have been known to cause health problems in local residents (*3, 6*). Special incinerators have been developed to burn rice hulls to control the emissions of particulate matter and produce rice hull ash (RHA) that is enriched in silica content (*7–10*); also, a process for the preparation of siliceous ash was patented.

The rice hulls contain only 25% to 30% silicon dioxide by mass, however, on incineration, the silica content of the RHA increases to 80% to 95% silicon dioxide, depending on conditions in the incinerator (*7–10*). Previous research studies have shown that RHA produced under controlled conditions contained significant amount of amorphous silica. Furthermore, other research studies have shown RHA containing amorphous silica to be pozzolanic (*11, 12*). The pozzolanic behavior of RHA, that is, the ability of RHA to react with calcium hydroxide at ambient temperature in presence of moisture to form cementitious hydration products, is an important characteristic of RHA that can be beneficially tapped for its use as a supplementary cementitious material (SCM) in portland cement concrete.

The performance of RHA as an effective pozzolan depends on two factors: quantity of silicon dioxide and the nature of silicon dioxide (amorphous and crystalline). From a practical standpoint, the quantity of unburnt carbon present in RHA can also affect its use in concrete as an SCM. Attempts have been previously made to commercially produce RHA as a pozzolan with desirable characteristics (*7–10*). However, few methods have been commercially successful in producing RHA with low unburnt carbon content, using environmentally friendly processing techniques. Typical carbon content of RHA reported in literature ranged from 0.41% to 5.91% (*13–15*). It is known from past research studies that high unburnt carbon content in SCMs leads to excessive demand for water and chemical admixtures, to maintain appropriate slump, air content, and other properties in concrete (*11–15*).

Recently, Vempati et al. developed a new method of producing RHA using a rotary kiln furnace in which oxidizing conditions and controlled incineration of rice hulls at ~412°C for 4 h. This continuous production process yielded RHA with carbon content less than 0.3% (*3*). This paper focuses on studying the material properties of RHA produced using the procedure developed by Vempati et al., along with establishing its performance as a pozzolanic additive in portland cement mixtures. Further, the influence of grinding RHA to a smaller particle size on performance of RHA as an SCM was investigated in this study. Studies were also conducted to ascertain

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TABLE 1 Chemical Composition (% by mass)

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	Alkali $(Na_2O)_{eq.}$ (%)	SO_3	MgO	MnO	P_2O_5	Carbon
Cement	19.78	4.98	3.13	61.84	0.82	4.15	2.54	—	—	—
RHA	94.8	0.52	0.13	—	1.92	—		0.39	1.09	0.24
SF	93	0.5	2.1	0.8	0.1	0.2		—	—	—

NOTE: SiO_2 = silicon dioxide, Al_2O_3 = aluminum oxide, Fe_2O_3 = iron oxide, CaO = calcium oxide, $(Na_2O)_{eq}$ = sodium oxide equivalent, SO_3 = sulfur trioxide, MgO = magnesium oxide, MnO = manganese oxide, P_2O_5 = phosphorus pentoxide, — = not present.

the relative performance of RHA and ground RHA (GRHA) with that of silica fume (SF). The findings reported in this paper highlight the role of RHA and GRHA as effective pozzolans that increase strength and durability of portland cement mixtures and that the performance of RHA and GRHA is very comparable to that of SF.

RESEARCH SIGNIFICANCE

SCMs such as slag, SF, and fly ash are typically obtained as by-products from operations that generate significant amounts of irrecoverable CO_2 emissions (i.e., greenhouse gas). However, RHA can be considered as a carbon-neutral SCM, for the net carbon dioxide produced in the combustion of rice hulls is compensated for by the consumption of carbon dioxide by the rice plant in the photosynthesis process required for its growth. Also, since a paddy crop is grown multiple times within a given year, and rice is a staple food for the majority of the world's population, RHA can be produced in a sustainable manner. From these considerations, RHA can be categorized as both a sustainable and green SCM that can truly reduce the carbon footprint of portland cement concrete. In addition, pozzolanic RHA can provide an economic incentive for the paddy farming community that is currently finding it difficult to dispose of rice hulls economically.

OBJECTIVES

The objectives of this study are to

- Study the properties of RHA produced using the rotary furnace;
- Establish the potential of RHA as a pozzolanic admixture in portland cement mixtures;
- Determine the effect of grinding of RHA on its pozzolanic properties, as well as its influence on fresh and hardened properties of pastes and mortars; and
- Compare the behavior of ground and unground RHA with SF as an SCM for use in portland cement mixtures.

EXPERIMENTAL INVESTIGATION

Materials

The materials used for the study include ASTM Type I cement, standard Ottawa sand, water, and superplasticizer in appropriate proportions specific to individual tests. The specific gravity of sand was 2.5. The pozzolanic materials used in this study are unground rice hull ash (URHA), GRHA, and SF. The URHA used in this study was produced by the process developed by Vempati et al., and details of the process can be found elsewhere (*3*). The GRHA was prepared by grinding URHA in a Retsch vibratory disc mill at 100 rpm for 15 min. The specific gravity of URHA, GRHA, and SF is equal to 2.2, and chemical compositions are presented in Table 1. The particle size distribution of URHA, GRHA, and SF was determined with a Malvern Mastersizer 2000 laser particle analyzer, and results are shown in Figure 1. The measuring capability of this instrument is 0.02 μ to 2,000 μ. The Malvern particle size results are an average of two measurements each of three specimens of each material. Samples were prepared by dispersing a small amount of powder in 10 mL of deionized water with one drop of TAMOL 1254 added as a dispersant. The samples were then agitated for 5 min in an ultrasonic bath to disperse the material. Measurements were performed in deionized water using the amorphous silica setting (refractive index 1.45; absorption 0.1).

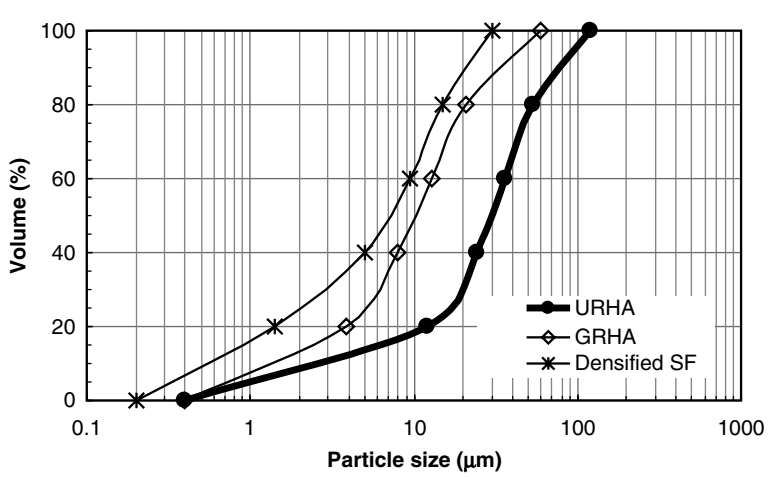


FIGURE 1 Particle size distribution of pozzolonic materials used in study.

From the distribution, it was found that the median particle size of URHA, GRHA, and densified SF grains were 29.9 μm, 10.2 μm, and 7.37 μm, respectively, and it was found that URHA is coarsely graded compared with GRHA and SF. The typical size of individual SF grain is on the order of only 0.1 μm; however, commercial SF is supplied in densified condition, and hence the median size of the SF granule is larger.

RHA as received from the production process was off-white in color. From X-ray diffraction investigations, the RHA was found to contain only amorphous silica, unlike other ashes (*3*). The Brunauer-Emmett-Teller surface area measurement indicated that the RHA has a surface area of 35 m^2/g, approximately 1.5 times higher than that of conventional SF (*6*).

The mixture proportions of URHA, GRHA, and SF cementitious pastes and mortars are carried out at a constant water-to-cementitious-material ratio, that is, w/cm = 0.485, and the mixture notations and proportions are presented in Table 2.

Microstructure of Rice Husk Ash

The microstructure of RHA samples was determined using a variable pressure Hitachi S-3400 scanning electron microscope using mounted powder samples and epoxy embedded samples both. The powder samples were mounted on a double-sided carbon tape. Scanning electron microscopy (SEM) photographs of unground and ground samples are shown in Figure 2.

From Figure 2*a,* it is clear that the URHA particles are distributed over a wide size range with varying particle shapes, while GRHA particle size range is more narrowly distributed. From Figure 2*b,* it can be seen that URHA particles have an irregular, vesicular, layered, and porous microstructure wherein the inner and outer portions are clearly visible. In the case of some larger particles, the microstructure of RHA resembles that of clustered fibers. However, GRHA particles have a finer microstructure and are more uniformly sized. Often, the smaller particles of GRHA appear to clump together to form small spongy clusters. The microstructure of GRHA represents a significant breakdown in the URHA structure caused by the grinding process.

Considering the porous nature of RHA grains and the high internal surface area, the process of grinding RHA to form smaller particles is not expected to make a significant difference in the total specific surface area. However, the smaller size of GRHA particles will enable them to more completely participate in pozzolanic reaction, compared with the larger RHA grains. Also, the large grain size of URHA will likely result in remnants of unreacted particles in hardened paste that will serve as pockets of excess porosity in hardened concrete while creating higher water demand in the fresh concrete.

Thermogravimetric Analysis

The pozzolanic reactivity of RHA was ascertained by determining the amount of calcium hydroxide depleted from cementitious pastes at 28 days. Powdered samples for thermogravimetric analysis (TGA) were prepared by grinding hydrated paste specimens and quickly introducing them into the TGA unit to prevent carbonation. TGA was carried out using the Thermal Analysis 2950 thermogravimetric analyzer. The samples were uniformly heated from ambient to 600°C at a ramp rate of 10°C per min, with N_2 as purging gas. The calcium hydroxide decomposition was noticed from 400°C to 450°C for all mixtures, and it was quantified using TA Advantage software. The TGA results from tests conducted on cementitious pastes containing URHA and GRHA at different replacement levels are shown in Figure 3. The calcium hydroxide contents of mixtures are normalized with respect to the control mix.

From Figure 3 there is a clear indication that addition of URHA and GRHA at different replacement levels results in a significant decrease in calcium hydroxide content. The decrease in percentage of calcium hydroxide at any replacement level is due to a combination of dilution and pozzolanic effects. Dilution effect refers to the percentage decrease in calcium hydroxide content due to reduced amount of cement in the paste at a given replacement level. Pozzolanic effect refers to the percentage decrease in calcium hydroxide content beyond the dilution effect. For instance, at 20% replacement level the pozzolanic effect of URHA and GRHA is 25% and 33%, respectively. Generally, GRHA is more effective in pozzolanic reaction than RHA at different replacement levels.

Test Methods

Normal Consistency and Setting Time

Due to the high internal surface area and porosity of RHA, it is essential to determine if the replacement levels and process of grinding affects the normal consistency of the cementitious pastes. Hence, the normal consistency test was carried out according to ASTM C187, whose values were used for the setting time tests according to ASTMC 191 at 7.5% and 15% replacement levels.

Flow Behavior of Pastes

Flow studies were carried out according to the standard ASTM C230 procedure. It was found in trial studies that addition of these pozzolans affected the flow drastically, and hence superplasticizers were needed to enhance the flow. Additional flow studies were conducted to assess the quantity of superplasticizer that is required to achieve a given flow.

TABLE 2 Mixture Proportions Used in Study

Mixture Notation	Replacement Level (%)	Cement (g)	SCM (g)	Sand (g)	Water (mL)	w/cm[a]
Control	0	500	0	1,375	242	0.484
URHA,10%; GRHA,10%; SF,10%	10	450	50	1,375	242	0.484
URHA,20%; GRHA,20%; SF,20%	20	400	100	1,375	242	0.484

NOTE: Sand is completely removed from the mix while performing tests with cement paste.
[a]w/cm = water-to-cementitious-material ratio.

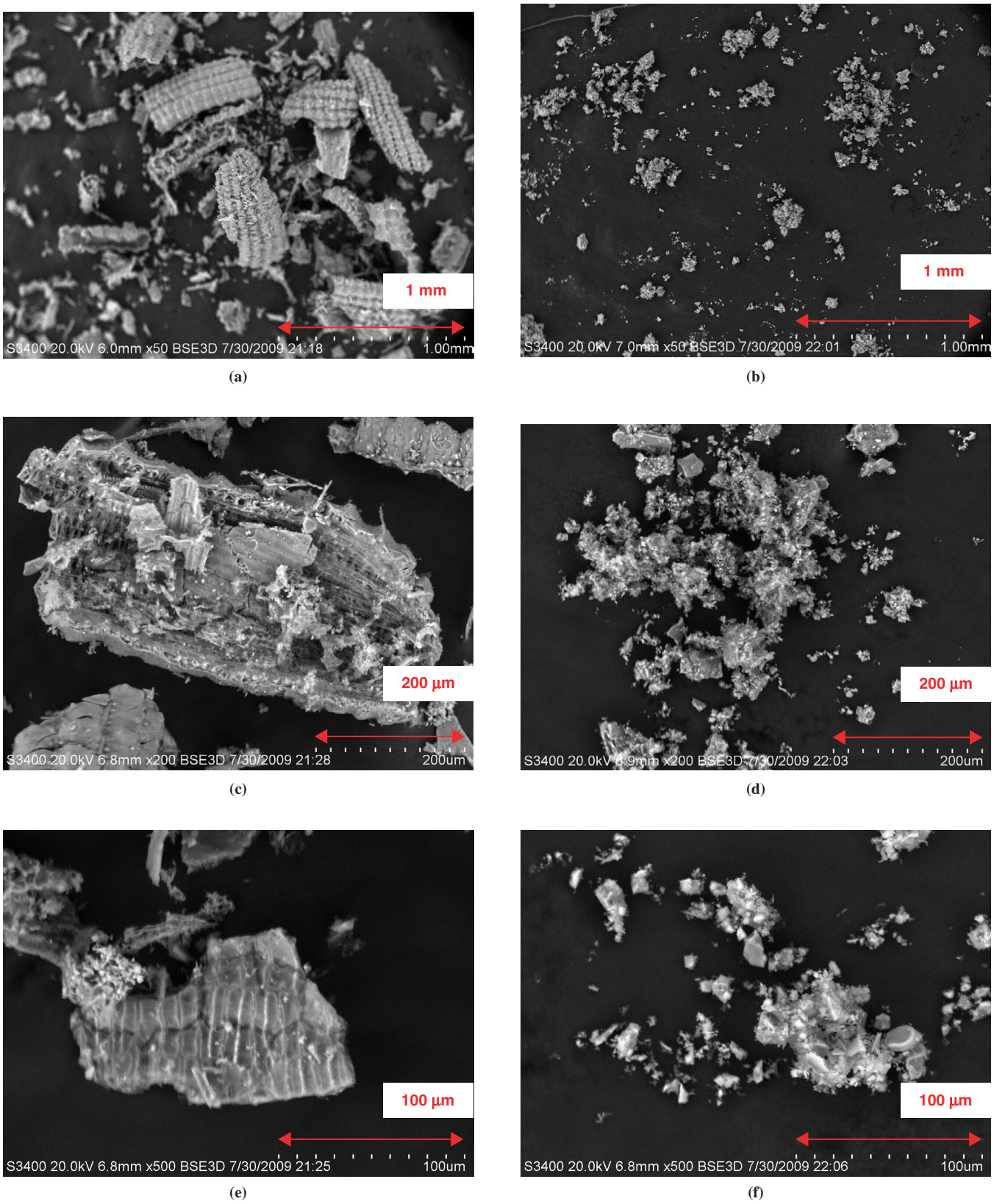


FIGURE 2 SEM photographs of RHA samples: 50× magnification of (*a*) URHA and (*b*) GRHA, 200× magnification of (*c*) URHA and (*d*) GRHA, and 500× magnification of (*e*) URHA and (*f*) GRHA.

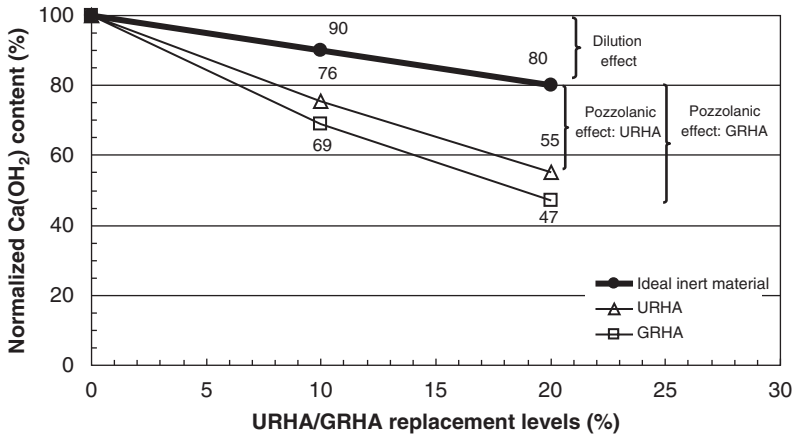


FIGURE 3 TGA results for 10% and 20% RHA replacement level: $Ca(OH_2)$ = calcium hydroxide.

Flow measurement was done with the same batch each time a superplasticizer was added. The initial mixing of the materials was done with Hobart mortar mixer according to ASTM C331 with certain modifications. Remixing of the mixtures was carried out for 2 to 3 min to achieve homogeneity. The time taken for individual flow measurement is not greater than 4 min. Delayed flow measurements may lead to slump loss in mixes. Thus, all the flow measurements were taken within 20 min from the time water was added to cement.

Strength Properties

The compression and flexure tests were carried out according to ASTM C109 and ASTM C348, respectively, to understand the strength properties of these mortars. Split tensile strength tests were conducted on mortar cylinders following ASTM C496 procedure. Standard-sized cubes (50 × 50 × 50 mm); cylinders (50 × 100 mm); and beams (40 × 40 × 160 mm) were cast and tested after 28 days in a universal testing machine, according to standard procedures.

Durability Properties

Water Absorption and Effective Porosity Water absorption of mortars containing RHA was determined according to ASTM C642 after 28 days of moist air curing. The specimens were kept inside an oven at a temperature of 105C° ± 2°C for 2 days, removed, and allowed to cool in dry air; the initial mass was measured. The specimens were then immersed in water, and the mass was measured at 30-min intervals for the first 3 h and then a final measurement after 24 h. Water absorption refers to the percentage increase in mass of specimens at 24 h of immersion in water. On the basis of results from this study, an effective porosity was determined, as follows:

$$\text{effective porosity } (\%) = \frac{(B - A)}{V} \times 100$$

where

A = mass of oven-dried sample in air,
B = saturated mass of surface dry sample in air after immersion at time t (h), and
V = bulk volume of sample.

Rapid Chloride Ion Penetration Test Ability of RHA to resist penetration of harmful chloride ions was determined using this test according to the ASTM C1202 test method. However, in this study, mortar specimens were used instead of concrete specimens. In this investigation, the effect of URHA and SF was evaluated at 10% and 20% replacement levels. GRHA was not evaluated in this study. The test was conducted after curing the specimens for 28 days.

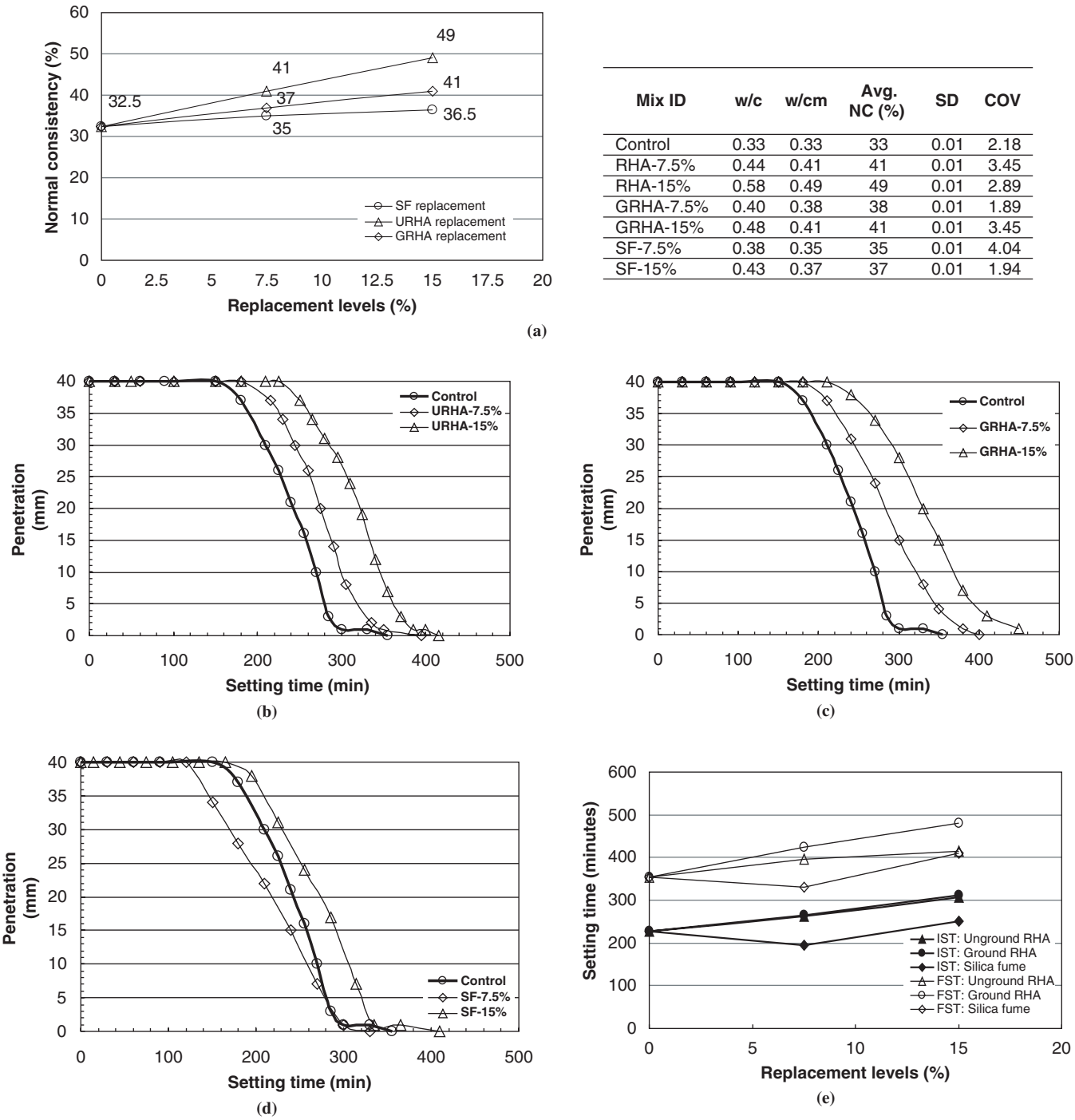
DISCUSSION OF RESULTS

Normal Consistency

The normal consistency test results are shown in Figure 4*a*. From this figure, it is clear that as the URHA replacement level increases the water required to produce a cementitious paste of normal consistency also increases. For instance, at a 15% replacement level, the water demand for normal consistency is 48% higher compared with that of control mixture. This may be partly due to the cellular microstructure of RHA (see Figure 2) and its water absorption characteristics, by which it holds the water within itself as soon as water is added to the dry mixtures. Also compared with that of URHA, the water demand for normal consistency of GRHA pastes is lower. This is because as the particle size of URHA is lowered through grinding, the cellular nature and therefore the water-holding ability of RHA grains is reduced (see Figure 2). The findings from this investigation suggest that grinding of URHA to a lower particle size might significantly improve the workability of concrete mixtures. Also, the water-absorption characteristics of concrete might be similarly improved with the use of GRHA. When compared with URHA and GRHA, the water demand for normal consistency of pastes containing SF is much lower. This may be due to the lower specific surface area of SF compared with that of RHA. Also, SF is not made of particles with a very high internal surface area.

Setting Time

Figure 4*b* shows the initial and final setting time behavior of URHA, GRHA, and SF pastes, respectively, prepared at normal consistency, and results are compared with those of the control mixture. Figure 4*b* shows the influence of these pozzolans on setting time at different cement replacement levels. The initial and final setting times for the control mixture were found to be 228 and 355 min, respectively.



Mix ID	w/c	w/cm	Avg. NC (%)	SD	COV
Control	0.33	0.33	33	0.01	2.18
RHA-7.5%	0.44	0.41	41	0.01	3.45
RHA-15%	0.58	0.49	49	0.01	2.89
GRHA-7.5%	0.40	0.38	38	0.01	1.89
GRHA-15%	0.48	0.41	41	0.01	3.45
SF-7.5%	0.38	0.35	35	0.01	4.04
SF-15%	0.43	0.37	37	0.01	1.94

FIGURE 4 Cementitious paste samples: (*a*) normal consistency; setting time behavior for (*b*) URHA, (*c*) GRHA, and (*d*) SF; and (*e*) setting time versus replacement level (FST = final setting time; IST = initial setting time; NC = normal consistency; w/c = water-to-cement ratio; w/cm = water-to-cementitious-material ratio).

Initial Setting Time

As the URHA replacement level increases, the setting time also increases; a similar trend was seen for GRHA mixtures also. However, the initial setting time of pastes with URHA is slightly lower than for pastes with GRHA. Unlike mixtures with RHA, SF mixtures showed a different behavior. Compared with the control mixture, the initial setting time of SF mixtures decreased as its dosage increased up to a 7.5% replacement level. At higher dosage levels of SF, the initial setting time increased beyond that of the control mixture (251 min).

Final Setting Time

The final setting time behavior was similar to that of initial setting time for all the mixtures. There is also a substantial increase in the final setting time of pastes with GRHA compared with pastes with URHA at both replacement levels.

Workability Analysis

Figure 5 shows the flow characteristics of cementitious mortars at 0.5% and 1.5% superplasticizer levels. It can be seen that flow behavior of all mixtures containing RHA and SF is significantly affected at both 10% and 20% replacement levels, compared with that of the control mixture. When compared with SF mixtures with URHA exhibited lower flow values, while GRHA resulted in equivalent flow at both replacement levels. The reason for lower flow of mixtures with URHA may be due to the larger median grain size of RHA, along with its porous internal structure that is potentially trapping mix water. Also, due to the uneven surface of GRHA grains, physiomechanical interlocking with cement grains may occur, resulting in lower flow values. In addition, one may notice that it is possible to obtain an optimum flow of 110% for all the mixtures within the superplasticizer dosage of 0.5% to 1.5%, except RHA at 20%.

Strength Properties

Figure 6 shows the 28-day compressive, split tensile, and flexural strengths of cementitious mortar mixtures containing URHA, GRHA, and SF at 10% and 20% replacement levels. It is evident from these results that mixtures containing URHA perform better than did the control mixture at all replacement levels. Mixtures with GRHA performed better than mixtures with URHA at all replacement levels. Mixtures with SF showed higher strengths than did mixtures with URHA and GRHA at the 10% replacement level; however, at the 20% replacement level, mixtures with URHA and GRHA performed better than those containing SF. From these results, it seems that the optimum dosage level of URHA and GRHA from a strength perspective may be significantly higher than for SF. With split tensile strength and flexural strength, although all the mixtures with URHA, GRHA, and SF were higher than for the control mixture, no definitive trend could be observed as a function of replacement level. Also, mixtures with URHA, GRHA, and SF exhibited comparable split tensile and flexural strengths at both replacement levels. The coefficient of variation for split tensile strength and flexural strength of all samples was less than 4% and 10%, respectively.

Water Absorption and Effective Porosity

Water absorption and effective porosity are presented in Figure 7*a* and 7*b,* respectively. The effective porosity values were determined at 1 h and 24 h. Figure 7*a* shows that the water absorption values within the first 3 h for all the mortar mixtures were somewhat lower compared with that of the control mixture, whereas at 24 h, the water absorption values for URHA at 10% and URHA at 20% were slightly higher than those for the control mixture. Mixtures with GRHA registered slightly lower absorption values compared with the control mixture at 24 h. However, the water absorption values of SF are significantly lower compared with those of all the other mixtures. The superior performance of silica fume is due to its very fine particle size and its ability to create pore-filling effect, in addition to pozzolonic reactivity. Although URHA and GRHA can affect pozzolanic reaction in mixtures, the unreacted grains with their porous matrix can potentially serve as a medium for the movement of water. Therefore, URHA and GRHA seem to be less effective in controlling absorption behavior than do SF paste specimens.

From Figure 7*b,* it is seen that the effective porosity value at 1 h of immersion for the control mixture is higher compared with those of all the other mixtures. Compared with the control mixture, the reductions in the 1-h effective porosity of mixtures with URHA, GRHA, and SF were found to be 15.5%, 0%, and 58.9%, respectively, at the 10% replacement level. At the 20% replacement level, the corresponding reductions in the 1-h effective porosity for URHA, GRHA, and SF were 22.6%, 17.25%, and 49.8%, respectively. At both replacement levels, SF mixtures showed the most reduction in effective porosity. The 24-h effective porosity values for GRHA and SF mixtures follow same trend as do the corresponding 1-h effective porosity values. However, for mixtures with RHA, the 24-h effective porosity was greater than that of the control mixture. The URHA mixtures showed an increased value at both replacement levels—probably due to the porous microstructure of RHA confirming the water-absorption values.

Rapid Chloride Ion Permeability Test

The rapid chloride ion permeability test results from mortar mixtures are shown in Figure 8. It is evident that the control mixture

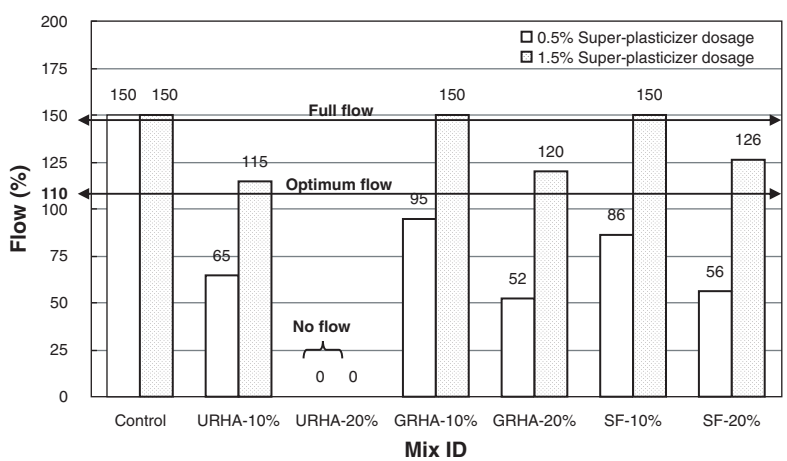


FIGURE 5 Comparison of flow values for URHA, GRHA, and SF mixes at different replacement levels.

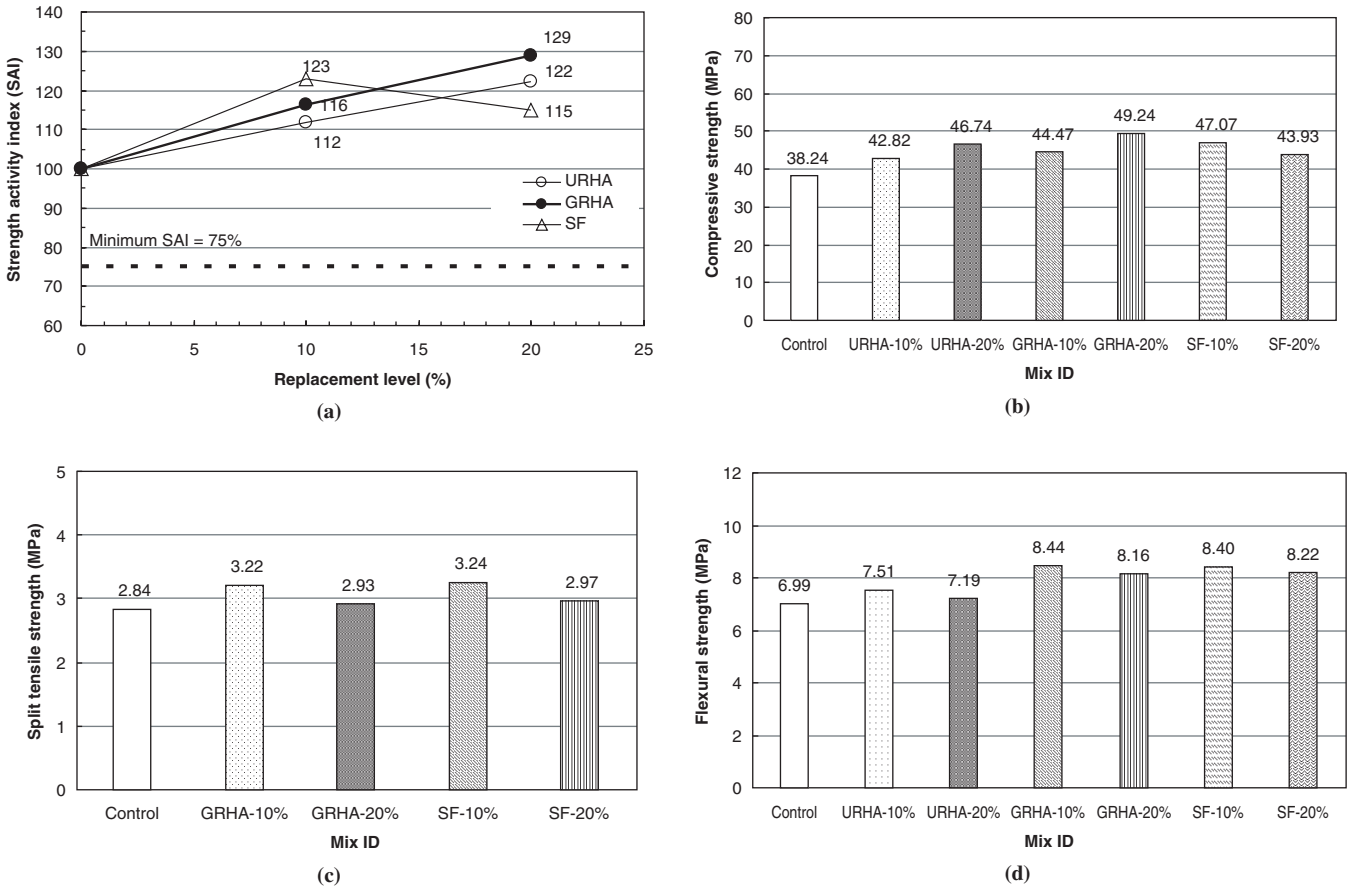


FIGURE 6 Strength properties of cementitious mortars after 28 days: (*a*) 28-day strength activity index versus replacement level, (*b*) compressive strengths, (*c*) split tensile strength, and (*d*) flexural strength.

registered a chloride ion permeability value of 5,117 coulombs (>4,000), indicating the permeability to be in the high range according to ASTM C1202 assessment criteria. With the 10% URHA substitution, the chloride ion permeability value was reduced to <2,000 coulombs (low range); with the 20% substitution level, the permeability value was reduced to below 1,000 coulombs (very low range). SF mixtures also showed similar reductions in chloride ion permeability at 10% and 20% replacement levels. However, at the 20% replacement level, URHA mixtures exhibited somewhat lower chloride ion permeability values than did SF mixtures.

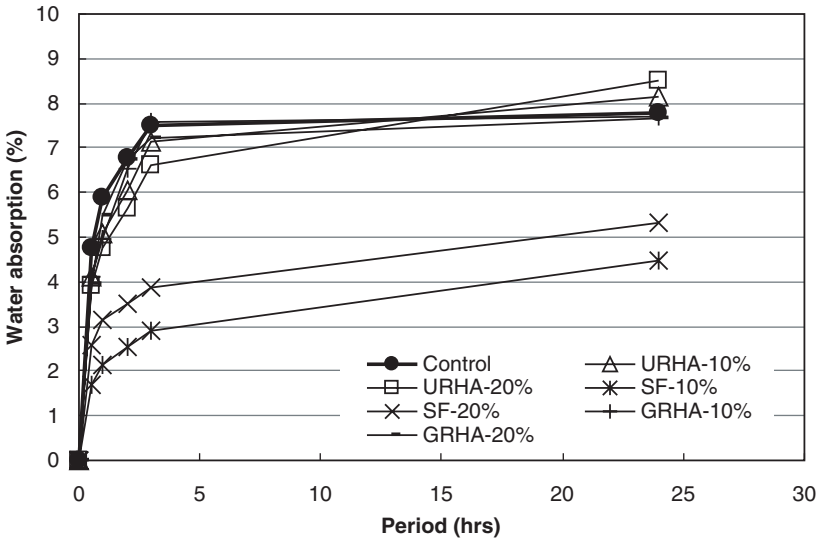
CONCLUSIONS

From the investigations conducted on paste and mortar specimens, to understand the fundamental behavior of URHA, GRHA, and SF, the following conclusions can be drawn:

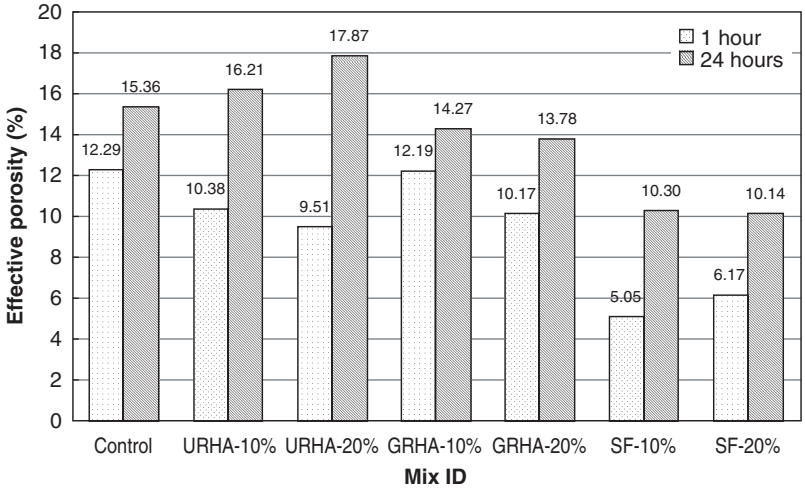
- URHA proved to be an effective pozzolanic material due to its high amorphous silica content and specific surface area.
- Mixtures with URHA performed better than did the control mixture in all the tests, except for water absorption and workability. Therefore, when URHA is considered for use as an SCM, care should be taken to set limit on maximum replacement levels. On the basis of findings from this study, a replacement level of 10% by mass of cement can be considered optimum from all considerations.
- GRHA proved to be more beneficial than URHA with regard to pozzolanic reactivity, flow behavior, and strength considerations. Hence, GRHA can be considered a more competent pozzolan than RHA, and similar in performance to that of SF. When GRHA is used as an SCM, higher replacement levels than for URHA (i.e., up to 20%) can be considered for deriving maximum benefits from this material.
- The strength and durability performance of mixtures with URHA is slightly lower compared with those of mixtures with SF. However, mixtures with GRHA performed in ways comparable with those of mixtures with SF in all the tests conducted.

FUTURE STUDIES AND RECOMMENDATIONS

Findings from this study indicate that URHA produced using the rotary furnace method has shown significant promise as a carbon-neutral and sustainable SCM. However, additional studies involving concrete mixtures are recommended to validate the findings from this study. In additional other studies such as impact of URHA and GRHA on ASR mitigation, sulfate-attack, shrinkage and freeze–thaw resistance of concrete are needed. Furthermore, optimization studies on fineness of RHA are necessary to realize the maximum potential of RHA as an SCM. Ongoing efforts at Clemson University are addressing these additional research needs.



(a)



(b)

FIGURE 7 Water absorption and effective porosity values after 28 days: (*a*) water absorption test results and (*b*) effective porosity test results.

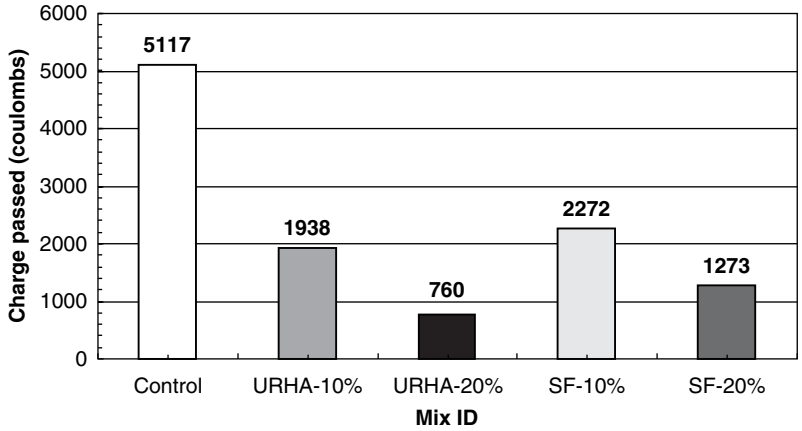


FIGURE 8 Rapid chloride permeability test results of cementitious mortars after 28 days.

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