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Comparison of two processes for treating rice husk ash for use in high performance concrete

Andres Salas [a,*], Silvio Delvasto [a], Ruby Mejía de Gutierez [a], David Lange [b]

[a] Escuela de Ingeniería de Materiales, Grupo de Investigación de Materiales Compuestos, Universidad del Valle, Colombia
[b] Department of Civil and Environmental Engineering, University of Illinois at Urbana-Champaign

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ABSTRACT

Rice husk ashes (RHA) have been used as pozzolanic admixtures for high performance concrete (HPC). This study reports on a chemical treatment before burning that improves the effectiveness of the RHA. The resulting ash (ChRHA) was compared to ash produced by conventional incineration (TRHA). The digestive chemical treatment before burning produced an RHA with properties comparable to silica fume. The ChRHA was highly amorphous, white in color, presented higher specific surface area and exhibited greater pozzolanic activity. The fresh and hardened properties of HPC made with different percentages of these RHAs were compared. The hardened concrete testing included the determination of the modulus of elasticity and the compressive and flexural properties. It was shown that ChRHA and TRHA were effective supplementary cementing materials, although concrete mixes required higher dosages of superplasticizer compared to the control concrete mix.

1. Introduction

Rice husk ash (RHA), produced by the controlled burning of rice husk, has been used as a highly reactive pozzolanic material, leading to a significant improvement on strength and durability of normal concretes [1–8]. Since the end of the 1960s, extensive research has been carried out [1–17] on the preparation, properties and applications of RHA in pastes, mortars and concretes and many papers and patents have been published on this subject. Although high quality RHA has been produced by many researchers under controlled conditions, the use of this material is still limited in many countries due to its sensitivity to burning conditions [17].

Some researchers have found [13–16] that various metal ions in the husk and unburned carbon influence the purity and color of the ash. Controlled burning of the husk after removing these ions with an acid leaching can produce white silica of high purity that is amorphous, reactive, and characterized by high surface area and pore volume. Acid treatment has been found to decrease the degree of crystallization of silica and carbon in rice husks reducing the sensitivity of the pozzolanic activity of the rice husk ash to burning conditions.

The aim of this study is to examine the effect of a partial replacement of Portland cement by a chemically treated rice husk ash (ChRHA) on the mechanical and durability properties of high performance concrete, and to compare the results with concretes made with conventional RHA (TRHA) and silica fume (SF) at the same replacement levels.

2. Experimental program

Rice husk ash samples were produced by two different methods: a conventional type of RHA (TRHA) was obtained by a thermal treatment of the rice husk and the other (ChRHA) was based on a chemical–thermal attack to the rice husk. These were subjected to various tests to analyze the amorphous silica content, color, particle size distribution and surface area. The strength and durability of the cement-pozzolan concretes produced from these samples (TRHA and ChRHA) were compared to those of a reference concrete without mineral admixtures (control) and a concrete with silica fume (SF).

2.1. Materials

2.1.1. Cement

The study used Type V Portland cement, conforming to Colombian standard codes NTC 30 and 31. The physical, chemical and mechanical properties of this cement are given in Table 1.

2.1.2. Pozzolanic materials

The chemical composition and some physical characteristics of all the pozzolanic materials used are given in Table 2.

2.1.2.1. Rice husk ash. The rice husk samples were collected from a rice mill “Arrocera La Esmeralda” located in Valle del Cauca, Southeastern Colombia, in the framework of research project “PUZOSIL” [18].

* Corresponding author. 205 North Mathews Avenue, University of Illinois, Urbana, IL, 61801, USA. Tel.: +1 217 333 6724; fax: +1 217 265 8040.
E-mail addresses: asalas@uiuc.edu (A. Salas), delvasto@univalle.edu.co (S. Delvasto), rudeguti@yahoo.com (R.M. de Gutierez), dlange@uiuc.edu (D. Lange).

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Table 1
Physical, chemical and mechanical properties of Type V Portland cement.

Chemical composition (%)		Physical properties		Mineralogical composition (%)	
SiO_2	21.27	Density (kg/m^3)	3,050	C_3S	53.29
Al_2O_3	4.63	Blaine fineness (m^2/kg)	377	C_2S	20.79
Fe_2O_3	3.96	Mechanical strength		C_4AF	12.05
CaO	63.05			C_3A	5.56
MgO	1.56	Compressive strength (MPa)		Free CaO	0.54
Na_2O	0.16	1 day	10.1		
K_2O	0.18	3 days	23.3		
SO_3	1.75	7 days	36.0		
P.F.	2.25	28 days	46.7		

The samples were washed, dried and separated in order to produce two different types of ashes. The first one was produced by burning rice husk in a programmable temperature furnace at a rate of 10 °C/min to 600 °C for 3 h; the other husk samples were separately treated with hydrochloric acid at a concentration of 1 N for 24 h; the supernatant liquid was decanted and the sample was washed thoroughly with water until free from acid (neutral pH). Then, the treated rice husk was dried and burned to ash in a muffle furnace at a rate of 10 °C/min to 600 °C for 3 h. The burning process and heating rate was established in earlier work and reported elsewhere [19].

In order to improve the pozzolanic behavior of these materials, RHAs were ground by ball milling for 90 min using ceramic balls. All the ash samples were characterized for the chemical and physical properties.

Crystallographic structure of the silica in the powders was examined by X-ray diffractometry (XRD). The Silica Activity Index (SAI) defined by Mehta [20] was used in order to evaluate the proportion of amorphous silica in the RHAs. X-ray fluorescence was used for quantitative chemical analysis. The surface area determination was performed by the BET method. The pozzolanicity of these materials was corroborated following the procedures established in ASTM C618 *Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete* and in ASTM C311 *Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete*. The results shown in Table 2 demonstrate that both TRHA and ChRHA meet the minimum performance levels that define pozzolanicity.

Table 2
Chemical and physical properties of pozzolanic materials.

	SF	ChRHA	TRHA
Chemical analysis (wt.%)			
SiO_2	90.00	99.00	90.00
Al_2O_3	0.46	<0.01	0.68
Fe_2O_3	4.57	0.13	0.42
K_2O	1.69	0.06	2.80
CaO	0.49	0.49	1.23
MgO	0.68	<0.07	0.35
Na_2O	0.05	<0.32	<0.32
TiO_2	0.02	0.02	0.04
P_2O_5	0.06	0.11	0.68
MnO	0.16	0.02	0.19
P.F (%)	0.54	0.16	0.3
Ba (ppm)	10	20	10
Sr (ppm)	100	40	40
Zr (ppm)	10	10	10
Physical properties			
Specific gravity (kg/m^3)	2230	2080	2160
Specific surface BET (m^2/Kg)	27,000	274,000	24,000
Average particle size (μm)	0.1	17	19
Color	Dark gray	White	Pink
Silica activity index (%)	–	99.4	89.5
Pozzolanic activity index ASTM C618–ASTM C311	123	125	95

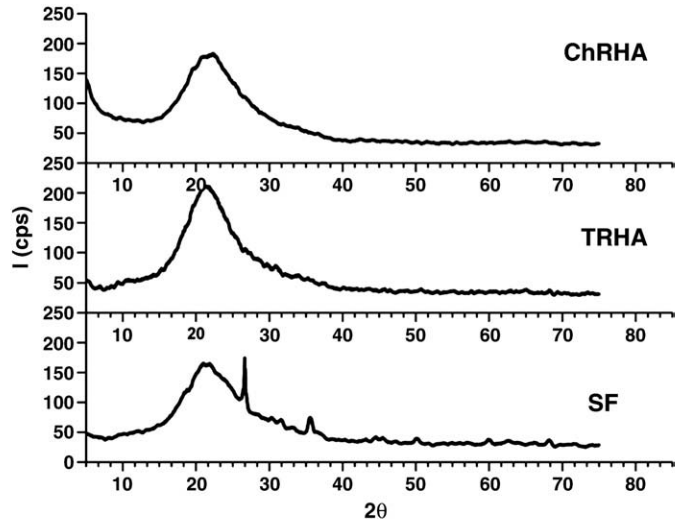

Fig. 1. X-ray spectrum of pozzolanic materials.

The ChRHA has an extremely high specific surface of 274,000 m^2/kg by BET method, which is 10 times that for SF and for TRHA. The X-ray diffraction analysis shown in Fig. 1 indicates that the RHAs contain mainly amorphous materials. These results are similar than those obtained by Feng et al. [14] which obtained a specific surface area of 270,000 m^2/kg for a chemically treated rice husk ash with a 95% amorphous silica content. Previous researchers [1,13–16,21], have found that the acid treatment causes a significant increase in the specific surface creating a mesopore internal structure. When rice husk is treated, K^+ ions present in the husk which are responsible for a surface melting of the ash particles are leached out, allowing the creation of mesopores.

Particle size distribution was determined by a particle size analyzer SHIMADZU 3001, and the results are shown in Fig. 2. The average particle size of both ashes, TRHA and ChRHA, are approximately 19 and 17 μm respectively, while that of the SF is 0.1 μm.

2.1.2.2. Silica fume. The SF powder consisted of spherical particles with a high content of amorphous silicon dioxide, which was lower than those of ChRHA and similar to TRHA. XRD pattern in Fig. 1 shows that SF is mainly amorphous with a small quantity of crystalline phases.

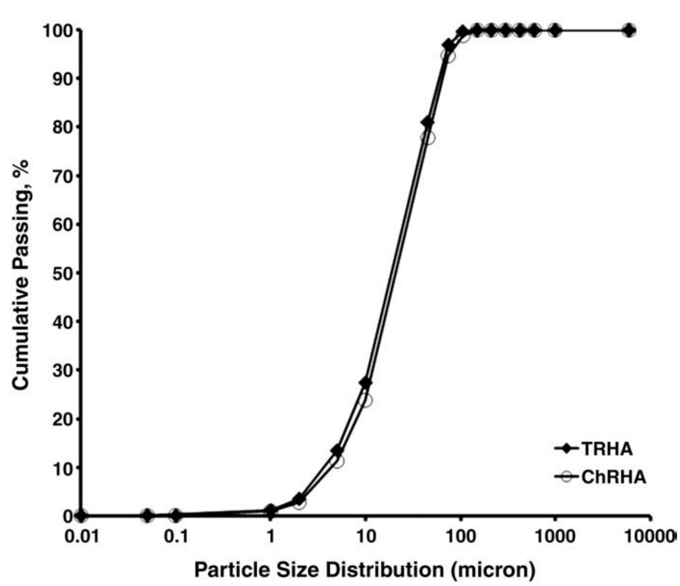

Fig. 2. Particle size distribution of treated and untreated rice husk ash.

Table 3
Grading and physical properties of aggregates.

Sieve size (mm)	Cumulative percentage retained (%)		
	Crushed basalt	Coarse sand	Fine sand
19.05	100	100	100
12.7	63.6	100	100
9.51	31.0	96.6	100
4.76	8.4	81.3	99.9
2.38	5.7	66.2	97.5
1.19	4.1	49.5	90.6
0.595	3.7	27.9	79.8
0.297	3.3	9.5	48.7
0.149	2.3	2.1	4.0
Fineness modulus		3.63	1.78
Volumetric density (g/cm^3)	2.98	2.75	2.71
Absorption, %	1.61	1.64	2.70

2.1.3. Aggregates

Two graded river siliceous sands were used as fine aggregate; the first one, with particle size between 0 and 2.38 mm and with fineness modulus of 1.78, and the second one had particles between 0 and 9.51 mm, with fineness modulus of 3.63. The coarse aggregate was locally available crushed basalt, passing through 19 mm sieve and retained on 4.75 mm. The grading and physical properties of the fine and coarse aggregates are shown in Table 3.

2.1.4. Superplasticizer

A lignosulfate-based superplasticizer, Sikament NS from Sika, Colombia S.A, produced in liquid form was used for the first stage mixes and a naphthalene formaldehyde-based superplasticizer (SP), Viscocrete of Sika, was used as liquid chemical admixture for the second stage mixes.

2.2. Mixture proportions

The objective was to produce high performance concretes using treated and untreated rice husk ash and silica fume. A total of 10 concrete mixes were made. Eight mixes were made to evaluate the role of replacement level of these RHAs on the properties of fresh and hardened concrete. A water/cementitious materials ratio (w/cm) of 0.45 was selected and a control Portland cement concrete mix and the mix incorporating SF were also included for comparison. In the mixes with higher dosages of TRHA, w/cm ratios were insufficient to produce slumps greater than 15 cm; therefore, it was necessary to provide higher dosages of superplasticizer for mixes with 15 and 20% of TRHA, as seen in Table 2. This is because the specific surface of TRHA is higher than cement particles. Hence, a greater amount of SP was required to reach flowability [1,4]. Proportions of concrete mixes are summarized in Table 4.

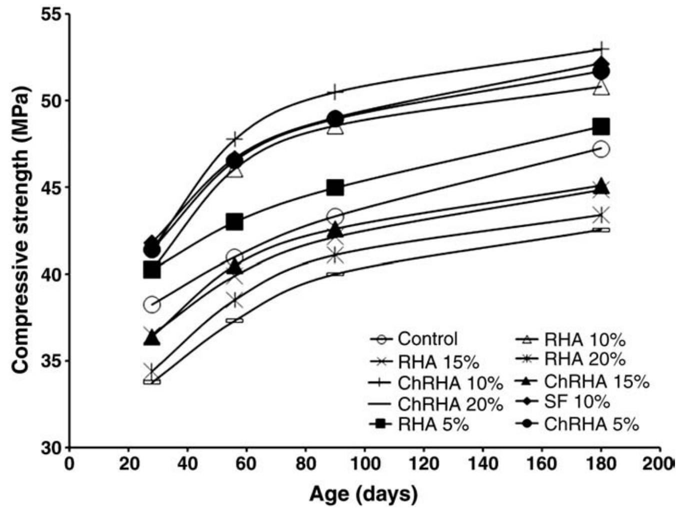

Fig. 3. Development of compressive strength of mixes with different percentages of TRHA, ChRHA, and SF as cement replacement.

2.3. Mixing and casting

The concrete mixes were prepared using a tilting drum mixer. 102 × 203-mm cylinders were cast from each mix for compressive strength and for durability tests; 150 × 150 × 500 mm beams were cast for the determination of flexural strength.

The molds were oiled, cast in three layers and compacted on a vibrating table. After casting, all specimens were covered with a plastic sheet and stored in the laboratory at 20 ± 3 °C for 24 h. The specimens were then demolded, marked and placed under water until the time of testing.

2.4. Testing

Compressive strength was determined on cylinders at 28, 56, 90 and 180 curing days. The modulus of elasticity and flexural strength were determined at 56 days. All the tests were carried out following the ASTM standards.

The resistance to chloride ion penetration was measured according to ASTM C1202 (Rapid Determination of the Chloride Permeability of Concrete) at 56 days of curing using a portion of the cylinders.

3. Tests results and discussion

3.1. Effect of percentage of RHA as cement replacement

3.1.1. Fresh concrete

Table 4 shows the results of the slump test for the fresh concrete mixes. According to these results, the use of these pozzolanic

Table 4
Mix proportions of concrete.

Mix	Pozzolanic material kg/m^3			Cement kg/m^3	Water l/m^3	SP %	w/cm	Aggregates kg/m^3			Slump mm
	HS	TRHA	ChRHA					Crush basalt	Coarse sand	Fine sand	
Control	0	0	0	440	198	0.4	0.45	660	838	285	178
TRHA 5%	0	22	0	418	198	0.8	0.45	655	832	283	127
TRHA 10%	0	44	0	396	198	1.7	0.45	651	827	281	152
TRHA 15%	0	66	0	374	198	1.9	0.45	646	820	279	178
TRHA 20%	0	88	0	352	207	4.3	0.47	645	819	279	254
ChRHA 5%	0	0	22	418	198	1.2	0.45	653	830	282	229
ChRHA 10%	0	0	44	396	198	1.9	0.45	647	822	280	178
ChRHA 15%	0	0	66	374	198	2.0	0.45	645	819	279	127
ChRHA 20%	0	0	88	352	198	5.0	0.45	637	810	276	254
SF 10%	22	0	0	396	198	0.9	0.45	653	829	282	165

Table 5
Characteristics of fresh concrete.

Characteristics	Control	RHA	ChRHA	SF
Superplasticizer, %	0	0.35	0.38	0.20
Slump, mm	191	127	102	102
Density, kg/m^3	2425	2400	2403	2405
Workability	Good	Good	Good	Good

Table 6
Elastic modulus and flexural strength of concrete mixes at 56 days.

Mix	*E* (GPa)	MOR (MPa)
CTRL (control)	29.1	3.7
TRHA	30.2	4.5
SF	31.8	5.1
ChRHA	32.1	5.4

materials increases the water demand; especially mixes with 15% and 20% TRHA and ChRHA were relatively stiff and it was necessary to adjust the dosage level of superplasticizer to maintain similar workability, these mixes showed tendencies to segregation and bleeding affecting the strength of hardened mixes. Fresh concretes with replacement of cement by SF required lower amounts of superplasticizer as compared to the TRHA concretes. This could be due to the high specific surface of the ashes that causes a water retaining effect in the mesopore structure [1,4]. According to Bui [1], this situation could be resolved partially breaking the pore structure with additional grinding of the ash. In addition to this, the lignosulfate-based superplasticizer was not a high range water reducer.

3.1.2. Properties of hardened concrete

Fig. 3 shows the compressive strengths development of all the concretes made in this study. The plotted points represent the mean of five tests, all falling within 7% of the mean value. The compressive strength of concretes with 15% and 20% of TRHA and ChRHA were lower than that of the control Portland cement concrete. It can be caused by the problems of bleeding and cohesiveness in the fresh mixes.

The compressive strength of the concrete added with ChRHA at percentages of 5% and 10% is significantly higher when compared to ordinary Portland cement hardened mix (100% Portland cement), and is similar to the behavior of the silica fume added concrete. These results show that ChRHA has a similar reactivity than silica fume [22] despite its larger particle size. This high reactivity can be explained by its extremely high porosity, which is represented by the high specific surface, and by its high content of pure amorphous silica.

Zhang et al [4] studied the effect of different percentages of TRHA as replacement of Portland cement in concrete mixes. These percentages included 5, 8, 10 and 15% with w/cm ratio of 0.40. According to Zhang et al, 10% is an optimum value because the workability decreased with increasing quantity of TRHA and it is necessary to increase the dosage of superplasticizer.

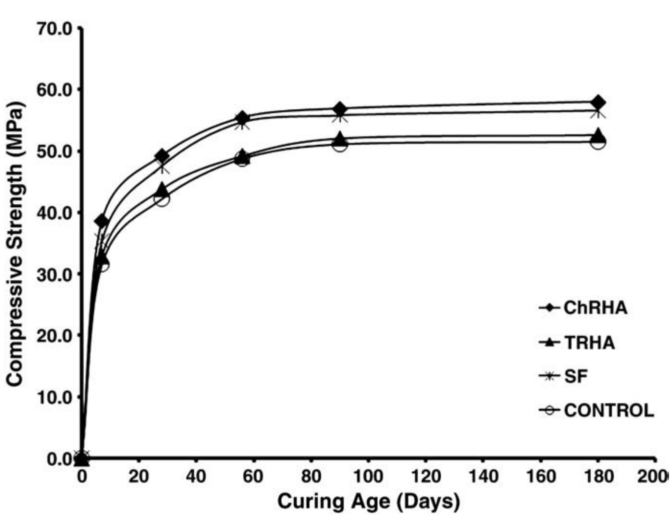

Fig. 4. Compressive strength of concrete mixes.

Following Zhang et al, the current research used 10% of TRHA ashes as an optimum percentage of Portland cement replacement, mainly due to technical and economical reasons. Workability problems and high dosages of superplasticizer were encountered with replacement levels higher than 10%. Thus, the 10% replacement level was established as a standard for the second stage of the current research that compared fresh, mechanical and durability properties of high performance concretes.

3.2. Effect of the optimum percentage of pozzolanic materials replacement on the properties of concrete mixes

3.2.1. Fresh properties

Fresh properties of all mixes are shown in Table 5. Mixes with 10% of TRHA and ChRHA cement replacement required more superplasticizer to reach the same level of workability when compared with control and with 10% SF concretes. The apparent density of each of the mixes with pozzolan is about 1% minor than the control mix.

3.2.2. Mechanical properties

Mechanical properties as compressive strength were evaluated at 7, 28, 56, 90 and 180 days of curing, and durability properties as absorption and chloride permeability were evaluated at 56 days.

Fig. 4 shows the results of the compressive strengths of the concrete materials. As shown in Fig. 4, the compressive strength of ChRHA concrete is comparable to SF concrete and somewhat higher than the control and TRHA materials. The datapoints shown in Fig. 4 represent the average of five tests, and the five individual values fell within 5% of the mean value.

The tests results for flexural strength (modulus of rupture, MOR) and modulus of elasticity of the concretes are given in Table 6. These data indicate that flexural strength and modulus of elasticity of the control and TRHA mixes are comparable, while the same properties are comparable for SF and ChRHA added mixes.

3.2.2.1. Water absorption test. The ingress of various ions from the environment and its movement through building materials are responsible for the deterioration of structures. By this reason, the control of the permeability of concrete plays an important role in providing resistance to aggressive environments.

The water absorption was evaluated by ASTM C642. The results are showed in Table 7. In general, the results of the absorption test showed that concrete with the ChRHA and SF exhibited lower permeability than the control and the TRHA concrete, similar to the ranking of the four material compressive strengths. The total absorption and porosity values, lower than 3 and 10% respectively, are useful parameters to

Table 7
Density, absorption and voids in hardened concrete.

Mix	Abs. after immersion (%)	Apparent density	Vol. permeable pores (%)
CTRL (control)	4.63	2.70	11.22
SF	3.77	2.64	10.15
TRHA	4.26	2.64	10.68
ChRHA	3.61	2.65	9.82

Table 8
Chloride Ion permeability of concrete mixtures.

Concrete	Transferred charge		Resistivity
	(Coulombs)		ρ (kΩ cm)
CTRL (control)	3529	Moderate	6.25
TRHA	1413	Low	14.9
SF	970	Very low	22.0
ChRHA	960	Very low	22.2

describe compaction and durability. These results indicate that the concrete with ChRHA is a relatively durable material.

3.2.2.2. Chloride permeability. The resistance to the penetration of chloride ions was measured following the ASTM C1202 "Standard Test Method for Electrical Indication of Concrete's ability to resist Chloride Penetration" as the charge passed through the concrete under the application of an external electrical field (60 V) during a period of 6 h. The test was carried out on cylindrical specimens of 50 mm thick after 56 days of curing. This test, called rapid chloride permeability test (RCPT), is essentially a measurement of electrical conductivity which depends on both the pore structure and the chemistry of pore solution. The results are shown in Table 8.

The incorporation of ChRHA in concrete enhances its durability properties by reducing the concrete permeability. The results for ChRHA concrete are comparable to the results for the SF concrete, a very effective pozzolan with finer particles than many cementitious materials that makes the pore structure of concrete denser [23]. Because of this, the incorporation of ChRHA into concrete mixtures plays its major role in properties related to permeability and durability properties.

The refinement of the pore structure was confirmed using the mercury intrusion porosimetry (MIP) technique with cement pastes having the same percentage of pozzolanic materials and the same water/cementitious material ratio used for the concrete mixes [24]. The average diameter of pores at 56 day old was 0.041 µm for control paste, 0.023 µm for TRHA paste, 0.021 µm for ChRHA paste, and 0.021 µm for SF paste. These results showed that the pore structure of paste with ChRHA and TRHA were found to be finer than that of control paste and similar to SF paste.

As shown in Table 2, the average particle size of the rice husk ashes was much higher than that of SF and similar to Portland cement. The finer particles of SF have a physical or filler effect in pore structure of the cementitious paste microstructure and a pozzolanic reaction [25]. Because of its size the RHA particles may not generate the denser packing (i.e. filler effect) as SF but its contribution to refinement of the cementitious paste microstructure is attributed mainly to its pozzolanic effect.

This reduction in the average pore diameter of cement paste caused by the incorporation of rice husk ash in the mix will effectively reduce the pore sizes, reduce permeability, and reduce diffusivity of chloride ions in concrete.

3.2.2.3. X-ray analysis of the hydration of rice husk with OPC. An X ray diffraction analysis was developed in order to study the pozzolanic behavior of the mineral additives used in this research and to identify the hydration products formed during the hydration of ordinary Portland cement (OPC). The additives were mixed with OPC in 10% replacement ratio to produce cement pastes with the same water/cementitious material ratio than that of the concrete mixes. The X-ray diffraction (XRD) patterns of the hydrated samples were recorded with a Rigaku DMAX-B X ray diffractometer using Cu-K α radiation.

Fig. 5 shows the compounds observed by XRD in the hydrated pastes at 28 and 90 days. The ChRHA samples showed a similar XRD pattern with smaller intensities of characteristic peaks of portlandite when compared with control pastes, TRHA and with SF pastes. This suggests a considerable amount of pozzolanic activity in the OPC/ChRHA blends at all ages of hydration.

In the case of control OPC, a smaller amount of CH (see Fig. 5) is present at 90 days of hydration, compared to that at 28 days. It is possible that a part of CH became carbonated. In the case of OPC ChRHA cement, still has the smaller amount of CH at 90 days of hydration when compared with control, TRHA and SF pastes. This phenomenon reveals the reaction between ChRHA and portlandite. Therefore, in comparison to the pastes and concrete without ChRHA addition, there will be more C–S–H gel and less portlandite in the concrete with RHA addition. This will contribute to improvement in the strength of the concrete and its resistance to acid attack, carbonation, and penetration.

The results showed that although the pozzolanic reaction is in progress, the total amount of CH available for a pozzolanic reaction has not been consumed. The presence of belite and alite peaks of relatively higher intensity indicate an incomplete hydration of the both phases even at 90 days of hydration.

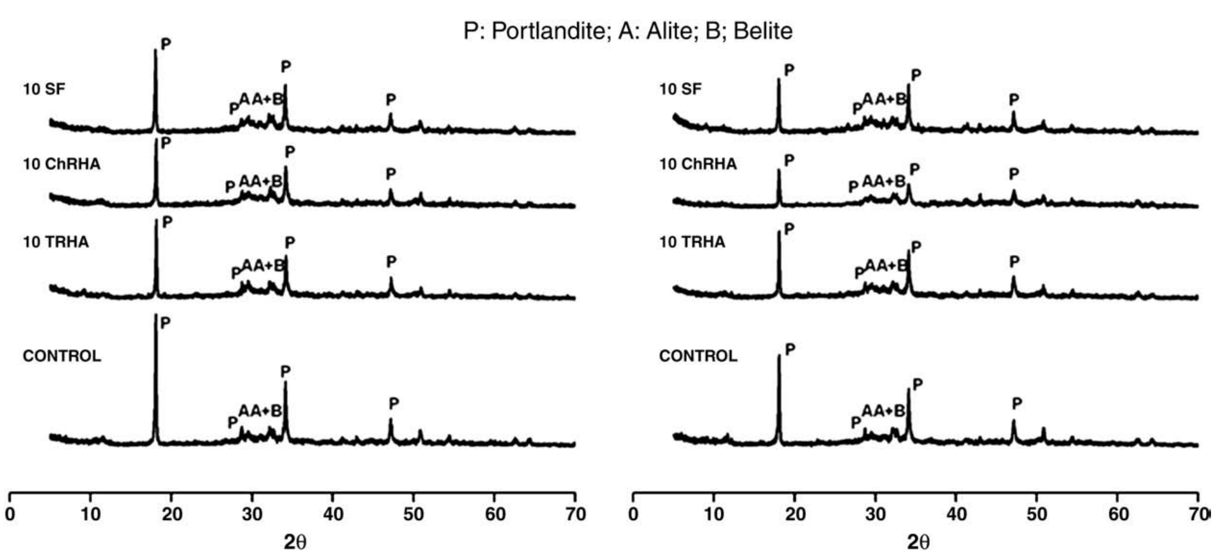

Fig. 5. X-ray diffractograms of cement pastes at 28 days (left) and 90 days (right). Control paste, Portland cement paste; 10 TRHA, paste with 10% conventional RHA; 10 ChRHA, paste with 10% chemically treated rice husk ash and 10 SF, paste with 10% silica fume.

4. Conclusions

Based on the experimental results obtained in this study, the following conclusions may be drawn:

1. Because of the high content of amorphous SiO_2 in ChRHA with great reactivity, a significant increase in the compressive strength of concretes was observed.
2. Compressive and flexural strengths of ChRHA concrete are comparable to a SF concrete made with the same replacement level, and these strengths are higher than the control and TRHA mixtures.
3. Incorporation of ChRHA in concrete enhances durability properties by refining its pore structure. These results are similar to those of SF concrete.
4. It is possible to produce high performance concretes using ChRHA as supplementary cementing material.

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