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OPTIMIZED PREPARATION OF RICE HUSK ASH (RHA) AS A SUPPLEMENTARY CEMENTITIOUS MATERIAL

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ABSTRACT: For years, supplementary cementitious materials (SCM) has been extensively used as an integral component in the production of concrete. This has been motivated by sustainability efforts and reduced environmental impact. Correspondingly, this study intended to develop an appropriate method for the use of rice husk ash (RHA), a by-product of rice husk (RH) which exists abundantly in the Philippines, as a partial replacement to ordinary Portland cement (OPC). Different studies have suggested optimal percentage replacement values of RHA, from 10% to 20%, which demonstrated vast improvement in strength of the resulting blended concrete. These optimal values, however, immensely depends on the burning preparation of RHA which adversely affects the amorphous silica components, and therefore the pozzolanic activities of RHA in concrete. In this research, a potential approach to convert RH into optimized and highly reactive RHA by controlled burning and grinding is provided. The effect of RHA in the strength of cement mortar was investigated through the various proportions of 5, 10, 15, and 20% RHA by weight replacement of cement. Some of the findings are: (i) controlled combustion of RH with temperature ranging from 400 to 600 °C and a slow method of cooling for 6 to 8 hours were some of the critical factors needed to produce high silica content, (ii) concrete containing up to 10% RHA replacement is optimal in maximizing the strength of cement mortar, and (iii) the incorporation of RHA in concrete by 15% and 20% replacement by weight indicated a lower workability.

Keywords: Rice husk ash, Cementitious, Incineration, High silica, SCM, Pozzolans

1. INTRODUCTION

Recently, rapid urbanization and the massive leap in industrialization created an ever-increasing demand for concrete. Concrete production has then been castigated as it involves consumption of massive amounts of natural resources and persistently constitutes to several issues on carbon dioxide (CO2) emissions which pose a huge threat on both the society and environment. Waste management is another interrelated issue that is constantly being addressed by environmentalists and many researchers. It was established that one of the solutions to lessen the adverse effects of CO2 emissions brought about by the production of concrete was to build sustainable and environmentally sound cities. Most recent studies [1,2,3] leaned towards green technology and sustainability distinctly acknowledge utilization of SCMs in concrete.

In evaluating SCMs, chemical analysis using any appropriate methods as standardized by ASTM C114, C311, and D4326 should be carried out [4]. One notable characteristic of SCMs is its ability to improve the mechanical properties of concrete, due to its highly reactive silica content, which relatively makes it a good pozzolanic material [5]. Commonly and successfully established known SCMs in the concrete industry include silica fume, fly ash, and blast furnace slag. These SCMs indeed essentially contribute to the durability and strength properties of concrete and generally help reduce CO2 emissions.

Countless significant researches have been carried out and various explorations were done in the search for a material that can pass as a cementitious material. A popular candidate is RHA (rice husk ash).

RH (rice husk) is one of the most common agricultural wastes in the Philippines which serves as an outer covering of the rice grain during its growth. According to [6], once the RH is detached from the grain during the milling process, it usually ends up being dumped in an open space making it worthless thus inducing deterioration to land and environmental problems [7]. On the other hand, RH has its compelling usage; it has been broadly employed as a fuel for boiler feed to produce electricity for the power generation of the rice mill. With this, a voluminous amount of RHA is being produced.

RHA is a natural carbon by-product obtained from RH that is converted to ash during ignition process [6]. The potential of RHA as SCM has very well been regarded since it contains a high content of silica.

Several types of research have been made concerning the factors that affect the mechanical strength of concrete incorporated with RHA such as time of incineration, temperature, the time required for cooling process and fineness of substance [8]. The nanoscale analysis is being performed to verify the physical and chemical properties of RHA through different test methods namely SEM, XRD, XRF-EDX [6,8,9]. Based on the analysis stated in the study of [7], results showed that RHA possesses amorphous SiO_2 when gathered from a low temperature of 600°C [9]. Many studies validated that generating RHA by controlled burning of RH between 550°C and 600°C temperature converts silica content into an amorphous phase [8,9,10], and partially substituting cement with 10% RHA by weight tends to improve the compressive strength of Portland cement concrete [9].

In this study, an attempt to transform locally available RH into high-quality RHA that will exhibit heightened pozzolanic activity to be suitable as a cement replacement is done. To ascertain its quality, chemical composition, physical properties and characterization of the produced RHA will be carried out using X-Ray Fluorescence spectrometry (XRF), X-Ray Diffraction Spectrometry (XRD), and Scanning Electron Microscopy (SEM). Also, further investigation is carried out to determine the optimum level of replacement of cement by the produced optimized and highly reactive RHA through compression test of mortar specimens.

2. THEORETICAL BACKGROUND

2.1 Hydration Of Portland Cement

The hydration of cement is the result of mixing between anhydrous cement with water involving a series of exothermic reactions taking place both simultaneously and successively. The main compounds in cement are as follows: elite ($\mathbf{Ca_3SiO_5}$, $\mathbf{Ca_3S}$), belite ($\mathbf{Ca_3SiO_4}$, $\mathbf{C_2S}$), aluminate ($\mathbf{Ca_3Al_2O_6}$, $\mathbf{C_3A}$), and ferrite ($\mathbf{Ca_4Al_2Fe_2O_{10}}$, $\mathbf{C_4AF}$). A set controlling agent, such as gypsum, is usually added to prevent rapid setting brought about by the high reaction rate of aluminate. However, this paper only examined the chemical reactions involving tri-calcium silicate, $\mathbf{C_3S}$, and di-calcium silicate, $\mathbf{C_2S}$, since only these silicates produce the calcium hydroxide ($\mathbf{Ca(OH)_2}$) needed for further reaction with RHA.

The product of the reaction of cement with water is commonly referred to as Calcium-Silicate-Hydrated (C-S-H) gel which is the primary agent that binds the cement and aggregate particles together in concrete. The approximate hydration reactions of C_2S and C_3S is illustrated using the following chemical equations:

$$2(3CaO \cdot SiO_2) + 6H_2O \rightarrow 3CaO \cdot 2SiO_2 \cdot 3H_2O + 3Ca(OH)_2 \quad (1)$$

$$2(2CaO \cdot SiO_2) + 4H_2O \rightarrow 3CaO \cdot 2SiO_2 \cdot 3H_2O + Ca(OH)_2 \quad (2)$$

2.2 Chemical Contribution Of RHA

High-quality RHA which exhibits high pozzolanic activity can be produced under controlled conditions. From the study of [11], it was found that burning RH at 600°C yielded the highly pozzolanic RHA consisting of amorphous silica. From Eq. (1) and (2), aside from the C-S-H gel produced, the hydration reaction also yields $Ca(OH)_2$. This $Ca(OH)_2$ will further react with the amorphous silica (SiO2) found in high-quality RHA to form a second type of C-S-H gel. In research by [12], a comprehensive study was conducted on the pozzolanic effect of RHA on a cementitious system and provides the following chemical reaction:

$$SiO_2 + 3Ca(OH)_2 + H_2O \rightarrow 2CaO_{1.5}SiO_{3.5} \cdot 2H_2O \quad (3)$$

2.3 Physical Contribution Of RHA

Grinding of RHA is usually done to reduce particle size and improve surface area. Also, it is established that an inverse relationship exists between particle size and pozzolanic activity of RHA. However, this parameter does not significantly change beyond 5.6μm. The study of [13] reported that concrete incorporated with finer RHA resulted in denser concrete mix wherein the finer particles acted as micro fillers to enhance the cement paste pore structure contributing to the overall strength development of the concrete.

3. METHODOLOGY

3.1 Preparation Of RHA

A custom-built muffle furnace, with chimney and an air inlet, was used to burn the RH. The interior dimensions of the furnace are 1m wide, 1m height and 1m deep. The RHs were laid flat, measuring about 2-3cm in thickness, on 28 ceramic hearth plates. The plates were stacked in multiple layers inside the furnace using ceramic tubes as columns. About 12kgs of RH could be loaded in the oven every burning but only about 20% of which are recovered as high-quality RHA.

The muffle furnace utilizes burners (4pcs, 1 in each corner) and is semi-controlled, that is, the fuel is supplied from a propane tank and the main mode of control is through its pressure valve. When in operation, the control of the temperature inside the oven is not absolute but fluctuates within a small manageable range. The temperature is monitored using a Benetech GM700 infrared thermometer through 4 holes (1 on each side and 2 in front) of the furnace, the temperature was strictly limited within the range of 400-600ºC. Since the temperature only ranged below the exact optimal temperature of burning (600°C), the burning duration was prolonged to 10 hours to make up for this temperature deficiency. During burning, fresh air continuously flows into the furnace through the 4 holes, this condition was defined as a free air supply.

After the burning process, RHA is cooled to room temperature by leaving the furnace door ajar. This step usually takes 6-8 hours before RHA can be safely recovered from the furnace.

Grinding of RHA was done using a Los Angeles machine. About 2.4kgs of RHA is placed into the LA machine for each batch; 12 pieces of 2” steel balls were used for grinding. The machine had a rate of 36rpm and was operated for 2hrs in each grinding. To avoid unwanted absorption of moisture, the RHA is stored in an airtight container every after burning and grinding. The effect of grinding on the fineness of RHA was investigated through determining its specific surface area (SSA) and mean particle size. These were obtained by conducting Blaine permeability test (ASTM C204-17) and particle size analysis.

Table 1 Mix Proportion of Mortar Specimens

Material (g)	0%	5%	10%	15%	20%
	0 RHA	5 RHA	10 RHA	15 RHA	20 RHA
Cement	500	475	450	425	400
RHA	0	25	50	75	100
Sand	1375	1375	1375	1375	1375
Water	242	242	242	242	242

3.2 Physical And Chemical Analysis Of OPC And RHA

The chemical composition of OPC and RHA used in the present investigation were determined using an Olympus Delta Professional Handheld XRF. Mineralogical analysis of RHA was done by X-ray diffraction analysis using Shimadzu Maxima X 7000. A JEOL 5300 scanning electron microscope (SEM) with the aid of a Gold Coater was used to obtain electron micrographs of the RHA sample and powdered RHA blended mortars.

3.3 Mix Proportion And Compressive Strength Of Cement Mortars With RHA

Ordinary Portland Cement type I was used for this study in accordance with [14]. Ottawa sand passing through 1.18mm sieve was used as a fine aggregate [15].

RHA blended cement mortars were prepared by replacing OPC with varying amounts of RHA (5%, 10%, 15%, and 20% by weight of cement). The cement mixes were designated as 05RHA-20RHA, and 0RHA for the control mix. The mix proportions were presented in Table 1.

The mortars were mixed using a planetary 4.8 liter-capacity mixer. Each RHA proportion was produced in batches of 6 mortars and was mixed for a total of 2.5 min with a total of 1.75min rest in between. Preparation and mixing of materials were done in the following sequence: (i) water in mixer bowl (ii) cement poured into bowl and then mixed (30s slow speed) (iii) and slowly added while mixing (30s slow speed) (iv) further mixing (30s medium speed) (v) resting cement mix (15s scraping + 90s) and (vi) final mixing (60s medium speed) [16]. Flow tests were done following each mixing, after which another 15s of mixing were done before casting cement mix into a mortar mold [17]. The compressive strength of RHA blended cement mortars cubes of 5.08cm size was determined after 7 and 28 days of moisture curing, as prescribed by ASTM C 109: Standard test method for compressive strength of cement mortars hydraulics [18].

Table 2 Chemical Properties of OPC and RHA

Oxide Composition (%)	OPC	RHA
Silicon dioxide (SiO_2)	19.6	93.47
Aluminum oxide (Al_2O_3)	5.20	0.92
Ferric oxide (Fe_2O_3)	3.20	1.08
Calcium oxide (CaO)	64.8	0.87
Magnesium oxide (MgO)	1.40	3.18
Sodium oxide (Na_2O)	0.40	-
Titanium oxide (TiO_2)	-	0.04

4. DATA, RESULTS AND ANALYSIS

4.1 Effects Of Burning Conditions And Grinding

Obtaining high-quality RHA largely depends on burning techniques which include (but are not limited to) the duration, and temperature of burning, type of incinerator, method of combustion and others. The process of layering the RH on each slab plate level inside the furnace notably showed an effect on the color characteristic of RHA. As observed, laying the RH thinly flat (less than 2 inches thick) on ceramic hearth plates (Fig. 1a) resulted to a faint pink colored RHA after the incineration process (Fig. 2a). At the same duration, RH placed on the slabs in a triangular prism manner (Fig. 1b), which was done in an attempt to maximize the area of the slabs and mass produce RHA,

(a) (b)

Fig. 1 Layering of RH on slab plates in two manners: (a) thinly flat (b) triangular prism

(a) (b)

Fig. 2 Distinguished color of resulting RHA (a) faint pink (b) blackish

produced RHA which appeared blackish in color (Fig. 2b). According to [1], RHA exhibited a dark color due to its high carbon content resulting from partial combustion while a gray or pinkish-white colored RHA indicated lower carbon content which is due to complete combustion. Since complete combustion implied better activity of the produced ash [19], the latter color appearance is highly preferred. Furthermore, it is reckoned that attaining such color of RHA is vital as it substantially ascertains the degree to which RHA can be suitable for use in concrete. produced RHA which appeared blackish in color (Fig. 2b). According to [1], RHA exhibited a dark color due to its high carbon content resulting from partial combustion while a gray or pinkish-white colored RHA indicated lower carbon content which is due to complete combustion. Since complete combustion implied better activity of the produced ash [19], the latter color appearance is highly preferred. Furthermore, it is reckoned that attaining such color of RHA is vital as it substantially ascertains the degree to which RHA can be suitable for use in concrete.

Experimentally, it was found that incinerating RH kept at a semi-controlled temperature ranging from 400°C-600°C yielded a predominantly optimized pinkish-white RHA by-product. This indicated that RH was burned to ash completely. Since the burning process involved large amounts of RHA, the slow cooling method was done which, in turn, affected the chemical compositions of RHA.

Blaine permeability test was executed to determine the specific surface area as a result of

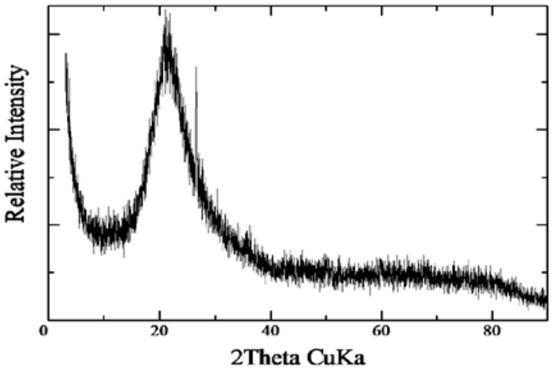


Fig. 3 The XRD Analysis of RHA

grinding. SSA for RHA ground for 2 hours was 365m2/kg, while that of RHA ground for only 1 hour was 286.3m2/kg. Percent passing through sieve no. 325 for the former is 84.78% while the latter got 70.85%. It was apparent from these tests that an increase of 1 hour in grinding time improved SSA and percent passing of RHA.

4.2 Properties Of RHA

The microstructure of RHA and ground RHA were observed through a scanning electron microscope as shown in Fig. 4 (a-d). As these figures showed, grinding RHA have broken down its cellular structure; its microporous and multilayered particles have become fine and uniformly sized particles. The significantly smaller RHA particles are anticipated to improve its pozzolanic activity.

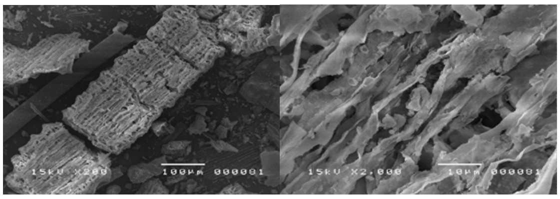
XRD analysis was performed to identify the mineralogical phase (amorphous or crystalline) of the RHA produced. Figure 3 displayed the XRD pattern of the produced RHA. The broadband on 2θ around 22° represented the RHA as mainly amorphous in form. Table 2 presented the chemical analysis of both OPC and the produced RHA using XRF analysis. This particular RHA consisted of 93.47% silica. The aforementioned results confirmed that the burning process was carried out properly and that the RHA produced can be used as an SCM due to its potential high pozzolanic activity.

4.3 Fresh Concrete Properties

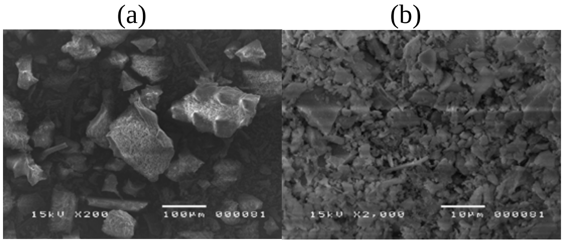
Workability is a property of fresh concrete mixture used to assess the ease of mixing, placing, compacting and finishing of concrete. Flow tests were performed on each proportion of RHA blended cement mortars using a flow table to quantitatively evaluate the effects of RHA on the property of workability.

Table 3 Flow Test Results

% of RHA	Flow (mm)
0RHA	175.30
5RHA	161.20
10RHA	141.33
15RHA	133.33
20RHA	-



(a) (b)



(c) (d)

Fig. 4 SEM Photographs (a) RHA (x200) (b) RHA (x2000) (c) ground RHA (x200) (d) ground RHA (x2000)

The result of the flow table test indicated that the incorporation of RHA in concrete led to decrease flow, and is inversely related to RHA content as shown in Table 3. As listed in the table, 20RHA mix was so vicious and did not flow at all. The large surface area of RHA particles attracted more water molecules to its surface, causing the absorption of water by the RHA particles and consequently reducing the flow value of a mortar mix. Hence, the amount of water intended for the hydration of cement and fluidity of the mixture is decreased so it is imperative that incorporation of RHA increases the water demand of concrete.

4.4 Compressive Strength

Figure 5 exhibited the development of compressive strength of mortars with varying amounts of RHA. Because of the hydration process that took place during the curing of mortars, the result showed that the compressive strength values at 28 days were higher compared to 7 days except for mortar with 20% RHA. As shown in Fig. 5, the compressive strength of the concrete comprising 10% RHA at the age of 7 and 28 days were 33.972 MPa and 52.768 MPa, respectively. The results clearly showed that 10% RHA has the highest compressive strengths compared to other mix proportions including the control OPC mix for 7 and 28 days with only 32.808 MPa and 40.228 MPa, respectively. Thus, the replacement of 10% is considered to be the optimal proportion of RHA in concrete to maximize strength. This can be due to the high reactivity of the pozzolanic material burned under controlled conditions consistent with previous studies [6, 8, 9, 10]. The resulting high silica content of RHA validated the report by [6] in

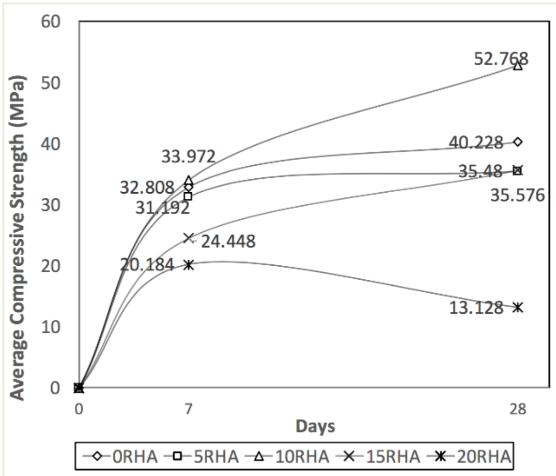


Fig. 5 Comparison of compressive strengths obtained for 7 and 28 days of RHA blended cement concrete mortars.

which RHA can be considered as SCM in concrete.

Moreover, a substantial decrease in the strength of concrete was observed for concrete mortars with 15% and 20% RHA. The decline in strength indicated that there is too much silica content in RHA blended concrete specimens wherein the produced C-S-H during hydration could not react anymore with the amount of silica available in a chemical reaction. Thus, an increase in the replacement level above the optimized value would less likely enhance the properties of concrete.

5. CONCLUSIONS

Recently, more aggressive actions are being initiated to address environmental issues on concrete production. Use of SCMs has been seen to help on this regard. As shown in this study, RHA was used as a partial replacement of cement. The effects of RHA preparation and burning techniques on enhancing its composition and pozzolanic properties were discussed. Some of the substantial conclusions drawn are as follows:

1. Critical factors to be considered in attaining an optimized and highly reactive RHA included correct and proper burning conditions,

controlled incineration temperature, a method of cooling, grinding, and the type of incinerator or furnace used.
2. High-quality RHA was produced by burning RH at a semi-controlled temperature of 400°C-600°C and by slowly cooling the resulting RHA for 6-8 hours.
3. A pinkish-white RHA indicated complete combustion while a blackish colored one resulted from partial combustion. The former has better reactivity during hydration.
4. As a result of subjecting RH under the specified burning temperature, the silica content obtained for RHA is 93.47% which made it highly siliceous and qualified as SCM.
5. Incorporation of RHA in mortars by 15 to 20% drastically affects workability.
6. 10% is the optimal replacement level in maximizing the strength of cement mortars as it significantly enhances the compressive strength as compared to OPC and other mix proportions.

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