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Characterization and Utilization of Rice Husk Ash (RHA) from Rice Mill of Bangladesh

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Abstract

The characterization of Rice Husk Ash (RHA) was carried out using conventional chemical analysis and instrumental techniques. Chemical analysis reveals that RHA contains mainly silica along with minor inorganic oxides. Phase analysis by X-ray diffraction (XRD) indicates the presence of quartz, crystobalite and anorthite, while micro structural features obtained from Scanning Electron Microscopy (SEM) shows that RHA particles are highly porous and honeycombed structure. Thermal analysis indicates the presence of surface moisture. A wide range of particles (0.001-0.100 mm) are present in the sample with 59% below the size of 0.05 mm. However after characterization, utilization of RHA as a potential cost effective ingredient in developing a variety of construction materials (e.g. building brick, insulating brick and pozzolana cement) have been examined. The results obtained are very promising.

Keywords : Charcterization, Rice husk ash, XRD, SEM, Construction material.

Introduction

Rice husk (RH) is an agricultural waste. In Bangladesh, about 39.3 million ton of rice is produced annually (Mustafi, 2005) which generate about 9.83 million ton of RH after milling of the paddy. The RH is generally used as fuel in rural areas and a small quantity is used as animal feed. Huge quantity of rice husk ash (RHA) is generated in Bangladesh during per-boiling of rice in rice mills. This ash is treated as a waste material usually dumped at the backyard causing unforeseen environmental hazards. No systematic efforts have yet been made for exploitation of this replenishible major agricultural by-product on commercial basis due to lack of detailed characterization of the RHA produced in Bangladesh.

RHA is predominantly silica together with some minor oxides (Agarwal, 1989, Kumar, 1993). The nature of this silica depends on the burning temperature. Controlled combustion of RH produces reactive silica (Agarwal, 1989, Kumar, 1993, Ibrahim, and Helmy 1981) which is suitable for making pozzolana cement (Kumar, 1993). It is reported (Agarwal, 1989) that silica present in RHA remains amorphous up to 973 K. With further increase in temperature, crystallization of silica occurs. Techniques for burning RH to produce amorphous silica, which is suitable for pozzolana cement production, have been developed (Kumar, 1993,

Ahmed, *et al*.1993). Nehdi *et al.* (2003) have developed a new technique for complete combustion of Egyptian RH to make it suitable for construction industry. A mini incinerator has been designed by Ahmed, *et al*.(1993) in BCSIR laboratory, Dhaka to produce RHA and a comparative study has been carried out in relation to compressive strength and setting time of the pozzolana cement prepared by using this ash and boiler ash.

Characterization of RHA by SiMAS NMR, XRD and FESEM has been investigated by Harridan *et al.* (1997) for synthesis of zeolites. Borthakur *et al*. (1980) have reported thermal and infrared spectra of RHA prepared at different temperatures. SEM images have indicated (Zhang *et al.*, 1996) that the ash is a highly porous and fibrous material with high surface area and honeycombed microstructure. Scanning Force Microscope has been used for the surface characterization of calcined RH and it is reported that the surface roughness of RHA is decreased while the number of voids is increased with the increase of temperature (Vempati *et al.*, 1995).

The primary objective of the present study is to characterize the RHA collected from rice mill to evaluate its suitability as a building material such as pozzolana cement, composite

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cement, building brick, tiles and insulating brick. A number of methods that include chemical analysis, X-ray diffraction, DTA/TGA, particle size analysis, SEM have been used in this study.

Materials and Methods

Materials

RHA was collected from rice milling area of Munshigonj and was finely ground in a pulverizer

Chemical analysis of RHA

Chemical characterization of RHA was carried out using classical methods as well as by instrumental techniques like PFP7 JENWAY (UK) flame photometer and UV-2201 SHIMADZU (Japan) UV spectrophotometer.

Particle size distribution

Particle size was determined by sedimentation and sieve analysis using a density hydrometer and various sizes of sieve respectively. The method was based on Stroke's law (Bowles, 1992).

XRD analysis

The phase composition of the RHA was determined by the XRD analysis of the sample with Philips X-ray Diffiactometer model operating with a CuKα radiation source (Kα=1.5406 A^O). The samples were ground to a fine powder and loaded on a silicon low background sample holder over baseline adhesive. The XRD scans were recorded from 10 - 80° 2θ with 0.20^O step-width and 5.1 s counting time for every step. Phase analysis was performed by comparing the *d* values and intensity ratios of the main fundamental peaks with data available in the data book published by the Joint Committee of Powder Diffraction Standards (1974).

SEM analyses

Morphological examination of the RHA was carried out on a 2600SN Hitachi (Japan) Scanning Electron Microscope (SEM) equipped with Germanium detector and Diamond window. The samples were mounted using double-sided tape.

DTA/TGA analysis

DTA/TGA Labsys ^{TM} from SETARAM was used to evaluate the thermal behaviors of the sample. The heating rate for DTA/TGA analysis was 10°C/min. The temperature difference between an inert (Alumina) and the sample was record-ed and mass loss was calculated from the TG curve.

Preparation of building brick and its characterization:

Building brick was made from RHA - Soil mixtures (RHA: Soil = 70 : 30, 80 : 20 and 90 :10) which was ball milled. The optimum water for making the batch composition in workable condition increases as the quantity of rice husk ash was increased. 2 ' ' cube specimens were prepared by applying a pressure of 60 kg/cm^2. Specimens were released from the mold, dried at 110°C and fired at 1100°C. Water absorption, bulk density (ASTM C 830-00 method) and cold crushing strength (CCS, ASTM C 109 method) were determined.

Linear shrinkage was also determined by preparing rectangular bar (10 x I.5 x 0.6 cm^3) from control (100% soil) and RHA - soil mixtures (100 : 0, 90 : 10, 80 : 20 and 70 : 30). The test sample was mixed with optimum water just to moisten it and placed in a mold and pressed by applying a pressure of 10 ton on the surface and a straight line was drawn on the surface of the test specimen. The sample was dried at 110°C followed by firing at 1000° C in an electric muffle furnace. The difference in the linear change indicates the linear shrinkage.

Insulating brick was prepared by taking RHA (coarse) to soil in the ratio of 70:30 and either CaO (5%) or sodium silicate (5%). The RHA and soil were mixed properly but not ball milled as coarser grain being required for thermal insulation. Except applying 3t pressure, preparation of test specimen follows the same procedure that was used for making building brick. The bulk density (%), % apparent porosity and CCS of the products were determined.

Pozzolanic cement was produced from RHA and lime at different ratios and ball milled for 3 h. The cement was then kept in airtight container. CCS measurements were carried out at a definite interval following ASTM C 109 method. The physico-chemical properties of the soil used were also evaluated (Farooque *et al.*, 2005)

Results and Discussion

Compositional analysis

The composition of the RHA sample has been determined by chemical analyses. The RHA used in this study contains: SiO_2 (91.3%), Al_2O_3 (0.3%), Fe_2O_3 (0.12%), CaO (0.21%), MgO (0.31%), Na_2O (1.52%), K_2O (2.65%), TiO_2 (0.15%), SO_3 (0.24%) and it undergoes 2.7% weight loss on ignition. It is evident that RHA contains mainly silica. A survey of literature (Kumar *et al.,* 1989, Al-Khalaf *et al.*, 1984) reveals that usually 79.5-97.6% silica is found in RHA and 90% of

this silica is present in gel form and the remaining is in the form of metallic silicates or in fine colloidal form. Presence of small quantities of K_2O=2.65% and Na_2O=1.52% (Qijun *et al.*, 1999, Sing *et al.,* 2001) in RHA makes it suitable raw material for producing composite cement. The loss on ignition indicates that small amount of free carbon is retained in RHA due to insufficient asking of the husk.

Particle size distribution

Particle size analysis of RHA (Fig. 1) shows that ~8% , ~41% , ~8% , and ~10% of RHA particles are in the range of 0.1 - 0.05 mm, 0.05 - 0.01 mm, 0.01 - 0.005 mm, 0.005 - 0.001 mm respectively. According to ASTM C 593 - 66 T standard the particle size of RHA when wet sieved should retain 20% on 0.045 mm sieve for composite cement/pozzolana cement production. The present result indicates that 59% of RHA particles are below 0.05mm. Therefore, it is suggested that further grinding is required to obtain the required particle size, of RHA for the production of composite/pozzolana cement by utilizing RHA from rice mill.
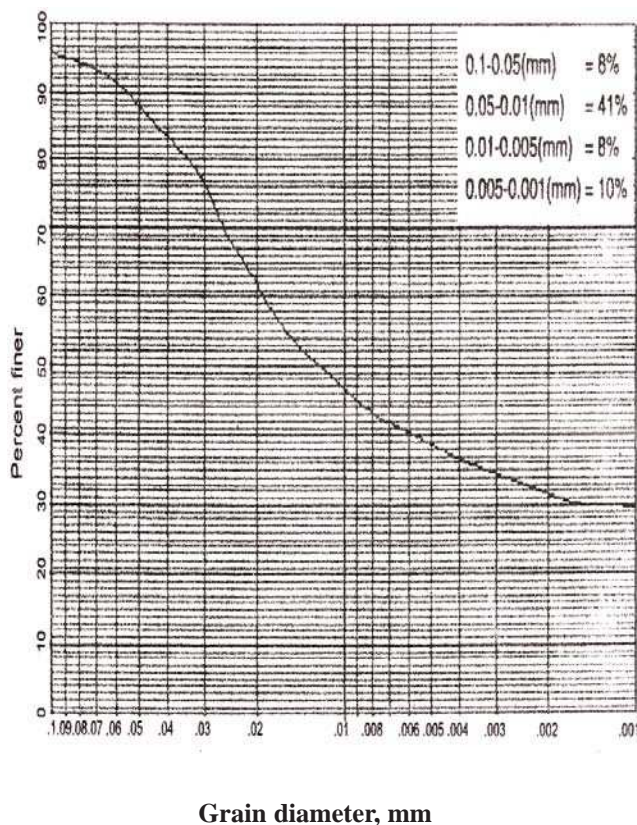

Grain diameter, mm

Fig.1 : Particle size distribution of RHA

Phase analysis:

The XRD pattern of RHA (Fig. 2) indicates the presence of quartz (22.85^O, 26.63^O and 42.47^O 2θ peaks) and crystobalite (21.91^O, 35.99^O and 69.50^O 2θ peaks). Anorthite
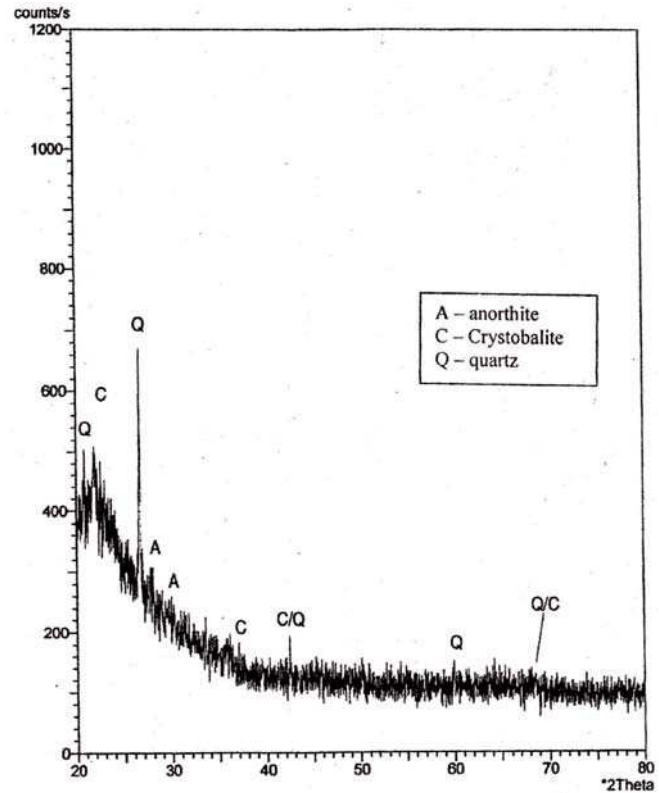

Fig.2 : X-ray diffraction of RHA

phase is also found (27.91^O and 29.42^O 2θ peaks). It has been reported (Agarwal, 1989) that at low temperature (873 - 973 K) silica in RHA is amorphous, and crystallization occurs when temperature goes above 973 K (Agarwal, 1989). The presence of crystobalite phase indicates that RHA is produced above 973 K. At high temperature, quartz is transformed to tridymite (Metha *et al.*, 1976) which is favored in presence of impurities in RHA (Metha and Pith 1974).

Morphological analysis

A typical SEM image of RHA is presented in Fig. 3. The SEM photomicrograph reveals the siliceous nature of the ash, which is also confirmed by the presence of quartz in the XRD. Close examination of the SEM photomicrograph also suggests that RHA is highly porous which is in agreement with others (Zhang and Malhotra 1996). The porous nature

of RHA and its honeycombed structure is responsible for its high specific surface and makes it suitable for making insulating brick.

Fig. 3 : Microstructure of RHA

Thermal analysis

The thermal analysis curve (DTA) of RHA is presented in Fig.4. The endotherm starting from room temperature to around 423 K is accompanied by weight change in TG curve. The endothermic peak corresponding to around 373 K can be attributed to elimination of surface moisture. The endotherm is followed by a broad and diffused exotherm in the temperature region 623 - 873 K which has also been observed by Borthakur *et al.,* (1980). No other change such as structural transformation or formation of new compounds has been
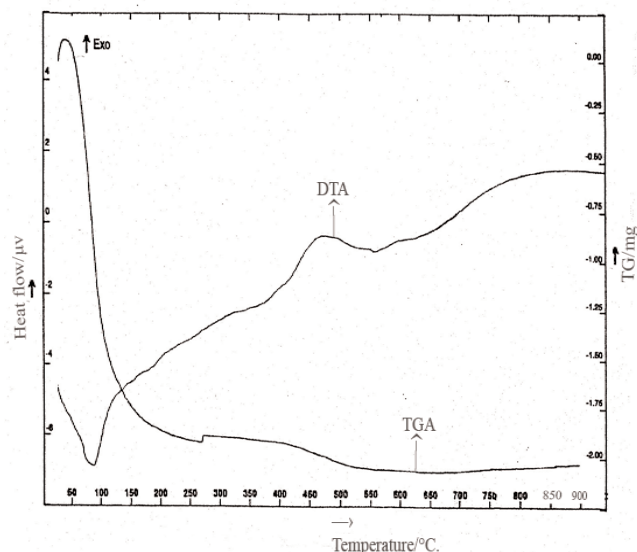

Fig. 4 : DTA/TGA Curves of RHA

observed with the increase in temperature. The shifting of baseline of thermogram at higher temperature is possibly due to change of surface properties of silica present in RHA (Borthakur *et al.*,1980).

The properties of brick specimens produced from a mixture of rice husk ash and red soil (Mirpur) in different proportion are shown in Table (I). As the proportion of RHA increases a gradual decrease in strength and bulk density whereas, an increase in water absorption are observed. The main reason may be due to the presence of unburnt carbon, which makes the specimen porous on firing at high temperature. Another cause may be the increase in optimum water content when quantity of rice husk ash increases. This is in good agreement with the study carried out by Hajela and Gupta (1997).

Table I. Physical property of RHA - Soil brick.

Soil, (%)	RHA, (%)	CCS, Psi	Water absorption,	Bulk density,	Linear shrinkage
Control	0	2600	15.0	1.78	9.8
90	10	2020	15.45	1.74	8.3
80	20	1850	20.45	1.70	7.6
70	30	1700	22.15	1.53	7.4

Rahman (1988) has observed increase in compressive strength with increase in % RHA. In fact, increase or decrease in compressive strength as well as other properties depend on the type of soil, type of kiln and also manufacturing process. As Bangladesh Standard Specification for common clay brick requires compressive strength of 2000 psi for Grade B and Grade C brick, respectively and water absorption from 12-16% for B and C Grade brick, therefore 10% RHA can safely be used for the production of building brick for wall construction. It is also observed that percent linear shrinkage of RHA - soil mixture (Table I) decreases as the quantity of RHA increases. According to Hazela and Gupta (1997) and Sabrah *et al.* (1989) the linear shrinkage decreases with the increase of RHA used for brick making. This is very advantageous for the production of tile as shrinkage can deform the article during firing. Controlling of shrinkage on firing is an important criterion in manufacturing tile. The burnt specimens for brick and tile show brick red color, which is the primary requirement of the consumer.

Chemical analysis of RHA shows that it contains some unburnt carbon, which is required for thermal insulation. The presence of oxides also favours the formation of vitreous phases during sintering of the insulating brick. The effect of additive (CaO or sodium silicate) on the properties of insulating brick is shown in Table II. It shows that thermal insu

Table II. **Physical property of insulating brick (RHA: Clay = 70 : 30).**

Additive	Apparent porosity (%)	Bulk density (gm/cc)	CCs psi
CaO	0.68	0.73	350
Sodium Silicate	0.61	0.85	680

lating specimens have low bulk density, as a consequence of high porosity.

Change in % of apparent porosity, bulk density and CCS have been observed for the two types of additive. CaO bonded insulating brick achieve lower compressive strength compared to sodium silicate bonded brick as also observed by Kapur (1980). Consequently %AP is more and BD is less for CaO bonded brick than for the sodium silicate bonded brick. This has also been observed by Kapur (1980). The presence of unburnt carbon, oxide as well as coarse particles makes the RHA potentially suitable to be used as a raw material for thermal insulating brick.

Ash-Lime Composite Cements

The CCS of RHA - Lime Composite Cement has been measured at different Ash-Lime ratios for 90 days. The primary objective of this experiment is to determine the optimum ratio of Ash-Lime that can impart maximum strength.

Table III indicates that RHA-Lime ratio of 2:1 achieves better strength compared to RHA - Lime ratios of 1:1 and 1:2. However, various Ash-Lime ratios have been reported (James *et al.*, 1992) as optimum for developing maximum strength and widely varying strength. Recent study of Nair

Table III. **Compressive strength of RHA - Lime composite cement.**

Ageing	Compressive strength, psi		
	RHA: Lime=2:1	RHA: Lime=1:1	RHA: Lime=1:2
7	550	525	350
28	800	650	600
90	1000	800	750

et al., (2006) indicates that unburnt carbon can adversely affect the strength.

Conclusions

The results of characterization of RHA confirm that silica is the major oxide present in RHA and the two most important phases are quartz and crystobalite. About 59% of RHA particles are below 0.05% mm size which implies that RHA particles need further grinding to make it suitable for pozzolanic cement. The particles are porous and irregular that makes it a suitable material for producing insulating brick. The thermogram and weight loss curve of RHA particles show the presence of surface moisture.

The addition of 10% RHA produces brick, which conform to Bangladesh Standard Grade C, which can be used in wall construction. Using a simple technology it is possible to manufacture low cost thermal insulating brick from RHA for dryers, ovens, furnaces. Clay roofing tiles can also be produced from RHA.

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