International Journal of Engineering

Journal Homepage: www.ije.ir

TECHNICAL NOTE

Synthesis and Characterization of Porcelain Body Developed from Rice Husk Ash

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PAPER INFO

Paper history:
Received 09 August 2017
Received in revised form 04 October 2017
Accepted 12 October 2017

Keywords:
Porcelain Body
Rice Husk
Calcination
Sintering
Silica

ABSTRACT

For a developing country like Bangladesh, waste management is an important issue. Since it is a small over-populated country , strict government regulations regarding landfills is essential. Expansion of ceramic industries is considered to be one of the potential sectors for the economic growth of Bangladesh. However, raw material cost per unit production is the prime concern for industrialists. Utilization of rice husk (RH) for industrial purposes is found to be an effective solution associated with both waste disposal and financial concerns. Since, RH is a rich source of silica, it could substitute quartz used in ceramic industries. Availability of RH in Bangladesh makes it more suitable for manufacturing applications. Hence, in the present work we focused on the synthesis and characterization of porcelain body by incorporating 25% rice husk ash (RHA) as a substitute of quartz and evaluation of structure-property relationship by means of temperature. Three different calcination temperatures (700, 800 and 900^{o}C) were chosen for the conversion of rice husk (RH) to rice husk ash (RHA). True density measurement and phase identification of RHA was conducted to ensure the quality of raw material. Calcination at 900^{o}C for 3 hours provided the preferred quality of RHA. Green samples for the porcelain body were prepared by a homogenous mixture of clay, feldspar, quartz and RHA, followed by densification at 950, 1050 and 1150^{o}C for constant soaking time of 1 hour. The effect of calcination as well as sintering profile on the densification of porcelain body was assessed. Finally physical, mechanical and morphological characterizations were done. The investigation revealed that 1050^{o}C as the optimum sintering temperature for 25% quartz substituted product.

doi: *10.5829/ije.2018.31.01a.04*

NOMENCLATURE

AF	Area factor	n_t	Number of threads
E_t	Total internal energy (Nm)	Q	Conservative vector
F	Parallel fraction of the code	S	Local speedup
RF	Recovery factor	T	Temperature (K)
F_{1inv}	Horizontal inviscid flux vector	u,v	Velocity components (m/s)
G_{1inv}	Vertical inviscid flux vector	**Greek Symbols**	
H_t	Total enthalpy (Nm)	ρ	Density (kg/m^3)
J	Jacobian	ϕ	Limiter function
N	Number of nodes	ξ,η	Curvilinear coordinates

1. INTRODUCTION

Researches on agricultural waste and its utilization have been gaining importance day by day in ceramic community due to some beneficial factors like energy saving, economic profitability and environmental safety. Rice husk (RH) is one of the potential candidates among them owing to its profusion in nature. According to a survey, the global production rate of rice paddy is 600 million tons/annum and as a consequence, the amount of rice husk ash (RHA) is around 21 million tons/ annum [1-3].

This farming byproduct has been employed as an alternative fuel both in the rice manufacturing industries and dwelling sectors for the fulfillment of heat

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requirements since centuries [4]. The combustion process of RH not only creates a residue of 20% RHA but also liberates silica particles, methane and carbon monoxide in air leading to atmospheric pollution and depletion of ozone layer [1, 5, 6]. Normally the generated ash is deposited in open space or abandoned by the side of canal or river which results in severe environmental impact and silicosis ailment [3, 4].

The mineralogical and morphological properties of RHA are intensely dependent on the climate and geographic location of the paddy and also on the combustion process of rice husk specially the firing temperature and soaking time. Amorphous silica seemed to appeare at a range of firing temperature 550-800°C, whereas the tendency of crystalline silica formation increases above this limiting value [7-11].

Serra et al. found the existence of more than 90% amorphous silica having high surface area in RHA. This statement has also been supported by some other researchers [3, 4].

The principal phase observed in RHA is cristobalite which made it eligible as an effective raw material in technological sectors from advanced to traditional level. Several advanced ceramics such as cordierite, solar grade silicon, silicon carbide, silicon nitride, thermal insulator, electronic semiconductor, magnesium-silicate, lithium-aluminum-silicate (LAS), zeolite, cement surfactants, etc. have been effectively produced by the formulation of RHA [12-22].

Several investigations have shown the successful utilization of RHA in concrete blend, coating formulations i.e. glazes, ceramic stains, adhesives, refractories and whitewares [23-32].

In construction management, RHA is used as admixtures [33]. Due to the strong pozzalonic action and highly permeable nature, use of RHA as partial replacement of cement in concrete blend is increasing tremendously [34]. Safabaksh et al. [35] have showed the effectiveness of RHA in developing the mechanical properties of pervious concrete. The yellow ceramic pigment has been developed by Federica et al. via solid state route using RHA as silica precursor [7]. Light weight RHA composed bricks have also been successfully manufactured and characterized by Tonnayopas et al. [36]. S.E. Mousvi [37] has also represented the useful implementation of RHA in green bricks.

Furthermore, quartz has been commendably substituted by RHA in porcelain products by Prasad et al. [38]. They observed the physical and mechanical characteristics of quartz substituted porcelain goods were as good as former products.

Kula et al. stated the similar effects of RHA addition in whitewares. Meanwhile, Andreola et al. [29] have reported the enhanced bending strength and hardness by the application of RHA as SiO_2 precursor in glass-ceramic products. The opportunity of utilizing RHA for the fabrication of LAS and cordierite has been evaluated by prasad et al. [38].

Highly porous alumina ceramics having improved mechanical properties has also been created by the incorporation of RHA as a pore-creating agent [39]. It was used efficiently in reducing atmosphere for vitrified tiles as cited by Vishal et al. [3].

The present work focuses on the fabrication of porcelain body by utilizing RHA and examines the influence of calcination and sintering temperature on the properties of the finished product.

2. PROCEDURE

Traditional synthesis technique was followed for the preparation of RHA. The employed rice husk was collected from the rice boiler and washed thoroughly with distilled water to eliminate dirt and fine particles. After the pre-cleaning treatment, RH was dried at 80°C for 12 hours followed by calcination in an electric kiln, for a dwelling time of 3 hours at three different temperatures-700, 800 and 900°C. Samples were abbreviated later as S_1, S_2 and S_3 for the calcination temperature of 700, 800 and 900°C respectively. Next, the calcined product referred as RHA was taken for true density measurement using ultimate gas pycnometer (Ultrapyc 1200e, Germany). The phase identification was conducted with the access of X-ray diffractometer (Bruker D8 Advance, Germany). The parameter was varied from 20-60° having a step size of 0.02° with Cu K_α radiation (wavelength λ=1.5418 Å).

Ball clay, china clay, feldspar, quartz and RHA were used as raw materials for the formulation of porcelain body. Enhanced properties of porcelain products were reported by the addition of 20-30 % RHA in several studies [40, 41]. Hence, 25% RHA was adopted as a substitute of quartz in this work. The weighted materials were ball milled for 24 hours in acetone media for adequate particle sizing and homogeneous mixing. After milling, the drying of the slurry was conducted for 48 hours at 100°C. Both pellet (Dia=32 mm, Thickness=6 mm) and rectangular (Length=30 mm, Width=12 mm, Thickness=8 mm) samples were prepared by manual hydraulic press (Pellet press-PP 25, Germany) using Carboxy Methyl Cellulose (CMC) as a binder at a pressure of 5 ton. Rectangular shape samples were used for bending and compressive strength testing. Electric kiln was engaged for the sintering of samples at temperatures 950, 1050 and 1150°C with a constant soaking period of 2 hours.

Finally, density measurement and water absorptivity calculation was done for the matured samples. For analyzing the morphology, scanning electron microscope (ZEISS EVO-18, Germany) was used.

Before imaging, the samples were coated via gold-palladium (Au-Pd) coater. Mechanical properties like modulus of rupture (MOR) and impact strength was also evaluated by Payne, UK three point bending tester and impact tester respectively. Finally, compressive strength measurement was conducted by Universal Testing Machine (UH64200).

3. RESULTS & DISCUSSIONS

3. 1 Characterization of Powder True density of RHA as a function of calcination temperature is presented in Figure 1. A consecutive improvement in density with calcination temperature was experienced. The measured true density of powder sample S_1, S_2 and S_3 was found to be 1.9503, 2.1923 and 2.3856 gm/cc correspondingly.

Room Temperature (RT) XRD pattern of the synthesized RHA powder is shown in Figure 2. The chronological formation of cristobalite phase with calcination temperature could easily be observed in the patterns. Major peaks like (111), (001), (122) and (002) confirmed the fact of generating cristobalite having orthorhombic structure as a stable phase.

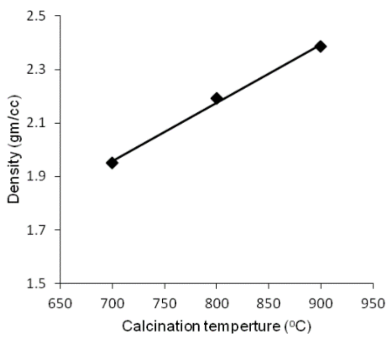

Figure 1. Variation of true density of decarbonated powder with calcination temperature.

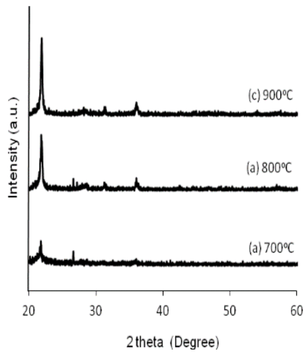

Figure 2. Room temperature XRD pattern of rice husk calcined at (a) 700°C, (b) 800°C and (c) 900°C

It is quite evident from the curves that, calcination temperature greatly influenced the crystallinity of RHA. With increasing temperature, a tendency of improvement in crystallinity was clearly apparent in this observation. In consequence to this, calcination at 900°C showed the utmost crystallinity among the three samples.

3. 2 Density Mesurement The variation of bulk density with sintering temperature of the porcelain products is portrayed in Figure 3. Sintering temperature of 950°C was not adequate for densification which can also be observed in SEM graph. Essentially, densification is a complex process associated with progressive transformation of microstructure by means of diffusion, formation and growth of neck and finally shrinkage and pore elimination [42]. Favorable temperature is essential for proper densification. Sintering at 1050°C turned out to be the optimum firing temperature for densification as evidenced by density data (1.85 gm/cc). With increasing temperature phase composition and densification achieve a saturation point above which grain coarsening might deteriorate property [3, 43].

Excessive grain growth was perceived for the samples sintered at 1150°C which can be attributed to Ostwald ripening. According to this theorem, during the last stage of sintering, small pores are supposed to be eliminated while large pores are exaggerated. Uncontrolled pore growth at this stage could lead to a decrease in density since high gas pressure in the bigger pores tends to inhibit further densification.

3. 3 SEM Analysis Figures 4 (a-c) reveal the SEM micrograph of densified samples of S_3 fired at 950, 1050, 1150°C. The structure for 950°C seemed to be highly porous. Moreover, nonuniform arrangement with both small and large grains was also visible in this case. On the other hand, a uniform pattern with the least amount of pore was confirmed by the SEM micrograph sintered at 1050°C.

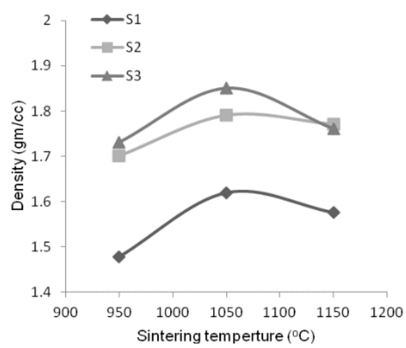

Figure 3. Variation of bulk density with sintering temperature for the samples S_1, S_2 and S_3

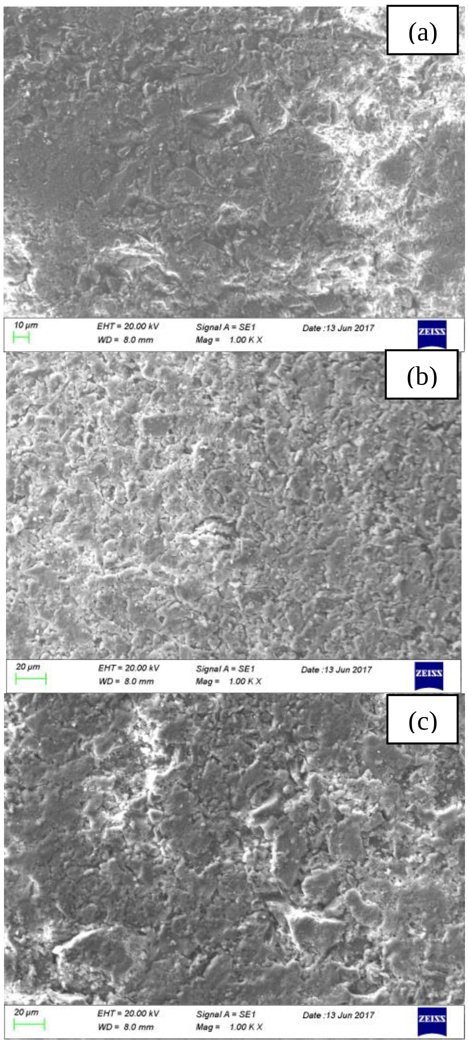

Figure 4. Microstructural change of samples S_2 fired at (a) 950°C, (b) 1050°C and (c) 1150°C

Occurrence of excessive grain growth viewed for the samples sintered at 1150°C indicates the utilization of overfiring temperature. It comes with no surprise, since excessive grain growth adversely affects the pore transportation mechanism by creating pore entrapment inside the structure. In consequence to this phenomenon, less dense body is produced.

The microstructure of samples S_1, S_2 and S_3 sintered at 1050°C is shown in Figure 5 (a-c) respectively. A more consistent microstructure with least porosity was observed for the sample S_3. It signifies that, the quality of RHA strongly influenced the manifestation of microstructure. As a matter of fact, complete removal of organic component and greater crystallinity of the RHA calcined at 900°C as established by X-ray diffraction gave highest density. Furthermore, from Figure 5 (b), it can be seen that the appearance of image for S_2 is in between the sample S_1 and S_3 which is in harmony with the density data.

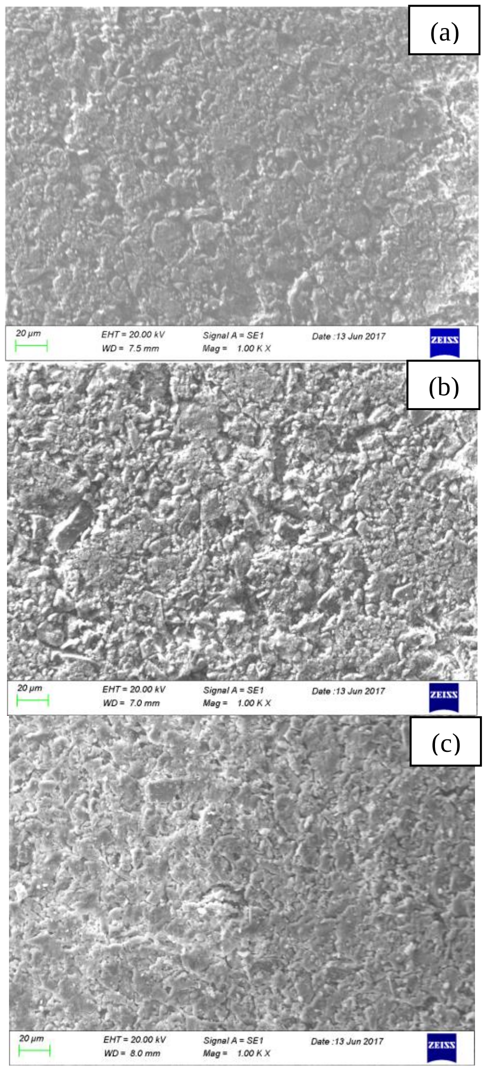

Figure 5. Micrograph of samples S_1, S_2 and S_3 sintered at 1050°C

3. 4 Water Absorption Test Figure 6 shows the water absorption behavior of the samples S_1, S_2 and S_3 sintered at different temperatures. The influence of both calcination and sintering temperature was prominent on water absorptivity as represented in the curve. Among the three type of samples, S_3 exerted lowest absorption (~12%) due to the fact of enhanced density caused by a suitable calcination sequence. As it is well known that, a lesser amount of water absorption is observed for a well sintered sample, firing at 1050°C resulted lowest percent of water absorption for all the samples (S_1, S_2 and S_3).

3. 5 MOR Test The results of bending strength measurement are represented in Figure 7. It was determined according to ASTM 133/97.The values of MOR showed an increasing trend with sintering temperature upto 1050°C. Above this temperature the situation was different.

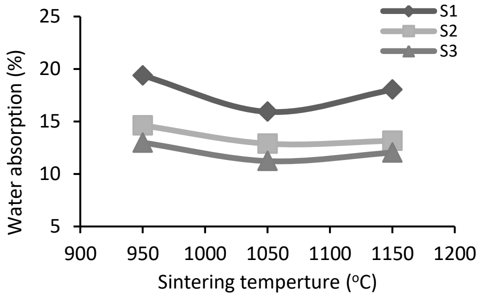

Figure 6. Water absorption vs sintering temperature for all samples.

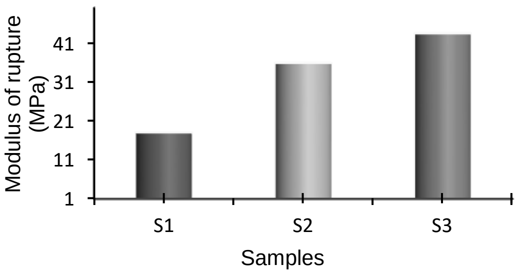

Figure 7. Change in modulus of rupture of porcelain body S_1, S_2 and S_3 fired at 1050°C.

The flexural strength of the sintered body decreased noticeably at high temperature i.e. 1150°C.

Fundamentally, increase in strength of a solid material is extensively affected by its microstructure i.e. porosity, precipitates, grain size etc. Higher the porosity lower should be the strength. Again, the strength of material is closely related to its grain size. Hence, closely packed uniform grains provide better bending property. This statement is in good agreement with the experimental data.

3. 6 Compression Test The graphical representation of compressive strength vs sintering temperature is shown in Figure 8. The values of compressive strength increased with temperature and then declined after attaining maxima at 1050°C. The strengthening mechanism of porcelain body could be demonstrated by the perception mullite hypothesis [44]. According to the theorem, existence of secondary mullite phase in structure should enhance the compressive strength due to its acicular shape along with small dia.

3. 7 Impact Strength Test Figure 9 shows the relationship between impact strength and soaking temperature. Impact resistance of a material is the measure of resistivity on shock loading. Its values are highly sensitive to various factors like microstructural features (grain size), rate of loading, temperature and existence of notches.

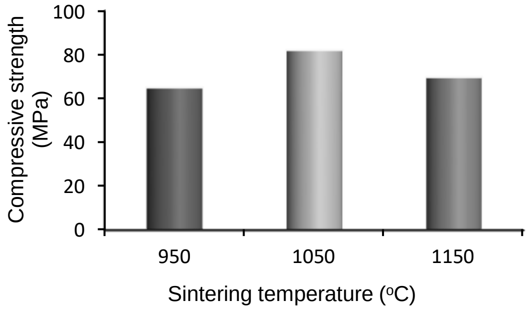

Figure 8. Variation of compressive strength with temperature of porcelain body

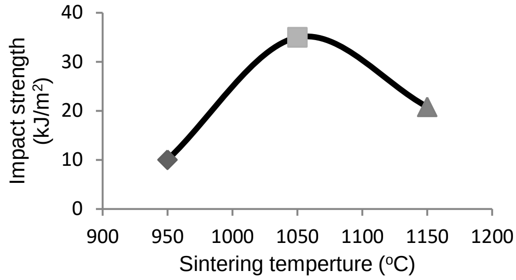

Figure 9. Variation of impact strength with soaking temperature

Coarse grained structures have adverse effect on impact strength [45]. The concept was completely coherent with the experimental data as the finer grained sample obtained at 1050°C showed a better result than 1150°C. The values of impact strength at different temperatures were 10.05 kJ/m^2, 35 kJ/m^2 and 20.85 kJ/m^2 respectively.

4. CONCLUSIONS

This work assured the immense possibility of utilizing RHA as silica precursor in porcelain product. Following conclusions can be drawn from this work:

1. White colored, highly active and crystalline silica particles were generated from RH through the calcination process. Calcination at 900°C produced a better quality ash for further processing.
2. Application of RHA in porcelain body triggered the physical as well as mechanical properties substantially.
3. 1050°C temperature was established as the threshold sintering temperature as evidenced by the microstructures.
4. The maximum values of flexural, compressive and impact strengths of the porcelain bodies were found to be ~44 MPa, ~82 MPa and ~35 kJ/m^2, respectively.

5. ACKNOWLEDGEMENT

The authors wish to thank Department of Glass & Ceramic Engineering, Rajshahi University of Engineering & Technology (RUET), Rjshahi-6204, Bangladesh.

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Paper history:
Received 09 August 2017
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Silica

چکیده

برای یک کشور در حال توسعه مانند بنگلادش، مدیریت زباله یک مسئله‌ی مهم است. از آن‌جا که این کشور دارای جمعیت بیش از حد است، رعایت مقررات دقیق دولت در مورد دفن زباله امری ضروری است. گسترش صنایع سرامیک به عنوان یکی از بخش‌های بالقوه رشد اقتصادی بنگلادش محسوب می‌شود. با این حال، هزینه‌ی مواد خام به ازای تولید واحد محصول نگرانی اصلی صنعت‌گران است. استفاده از پوسته‌ی برنج (RH) برای اهداف صنعتی، یک راه حل موثر در ارتباط با دفع زباله و نگرانی‌های مالی است. از آن‌جا که RH منبع غنی سیلیسیم است، می‌توان از آن برای جایگزینی کوارتز مورد نیاز صنایع سرامیک استفاده کرد. در دسترس بودن RH در بنگلادش آن را برای تولید قطعات سرامیکی مناسب می‌کند. از این رو، در این مقاله، با ترکیب ۲۵ درصد خاکستر پوسته‌ی برنج (RHA) به عنوان یک جایگزین کوارتز و ارزیابی رابطه‌ی بین ساختار و خواص آن با تغییر دما برای تولید بدنه‌ی چینی تحقیق کردیم. برای تبدیل پوسته‌ی برنج (RH) به خاکستر پوسته‌ی برنج (RHA)، سه دمای کالسینه کردن (۷۰۰، ۸۰۰ و ۹۰۰ درجه‌ی سانتیگراد) انتخاب شدند. اندازه‌گیری چگالی واقعی و تعیین فاز RHA برای اطمینان از کیفیت مواد خام انجام شد. کالسینه کردن در دمای ۹۰۰ سانتی‌گراد به مدت ۳ ساعت کیفیت مطلوب RHA را به دست می‌دهد. نمونه‌های خام برای بدن چینی توسط مخلوط همگن از خاک رس، فلدسپات، کوارتز و RHA تهیه شده و پس از آن با نگاه داشتن در ۹۵۰، ۱۰۵۰ و ۱۱۵۰ درجه‌ی سانتیگراد برای مدت زمان ۱ ساعت، تهیه شد. تاثیر کلسینه کردن و پروفیل پخت و بر چگالی بدنه‌ی چینی ارزیابی شد. در نهایت، مشخصات فیزیکی، مکانیکی و مورفولوژیکی انجام شد. این تحقیق نشان داد که دمای ۱۰۵۰ سانتی‌گراد دمای مطلوب برای شرایط جایگزینی ۲۵ درصد کوارتز است.

***doi**: 10.5829/ije.2018.31.01a.04*