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PRODUCTION OF INSULATING CERAMIC FROM ISEYIN CLAY

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Abstract: The effect of adding varied amount of rice husk on thermal and mechanical/refractory properties of Iseyin clay was investigated. The clay obtained from Iseyin in Oyo State was preprocessed to very fine particles and characterized using scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) and x-ray diffractometry analysis (XRD). Rice husk procured from a large rice plantation in Akure was dried. It was then pulverized into powder. A mixture of this dried rice husk powder with the processed clay was made at various proportions of the rice husk, with a little addition of water. Samples of standard dimensions were produced through uni-axial compaction. The samples were dried and then fired at 1000°C. The sintered samples were characterized for various properties using standard ASTM method. The microstructures of the fired samples were characterized with SEM. It was observed that increase in the rice husk leads to increase in the porosity of the sample. This resulted in the reduction in other properties of the samples. It was concluded that service condition where the ceramic will be in contact with molten metal/slag, sample with 0% admixture is optimum. For insulation, sample with 40% admixture is optimum.
Keywords: porous ceramic; sintering temperature; thermal conductivity; insulating brick; clay

INTRODUCTION

Clays are vital raw materials for the industries. They are used in making ceramics, paper making, paint, petroleum industry, catalysis etc. Their area of applications depends upon their structure, composition, and physical attributes [1, 2].

Insulating ceramics are materials with low thermal conductivity due to their high porosity. They are generally used in the conservation of heat in furnaces where they will not be in contact with molten metal [3]. Because of their high porosity they are inherently lightweight refractories that have very low thermal conductivity and heat capacity than other refractories [4]. They are also used in other industrial areas, such as catalyst supports for heterogeneous chemical reactions, filters, membranes and bioceramics [5]. Porosity is normally incorporated by the addition of combustible materials to the ceramic raw material mixture. These combustible materials are burnt off during firing, which leaves a large fraction of pores within the ceramic body. There are different types of pore formers available today such as sawdust, foam polystyrene, fine coke, binders and organic foams, or granular materials such as hollow microspheres and bubble alumina [4, 6].

Many researchers have worked extensively on clay; production of refractory and insulating ceramics from natural clay. Aramide, [1] reported on the effects of firing temperature on the mechanical properties of masonry bricks produced from Ipetumodu clay. He reported that the optimum mechanical properties of the masonry bricks were obtained at 950°C. The same author in 2015 [2] investigated the effects of sintering temperature on the phase development and mechanical properties of ceramics produced from Ifon clay. He observed that the sintered samples were composed mainly of quartz, microcline and anorthite and only the sample sintered at 800°C contains muscovite. He concluded that the sample which was sintered at 800°C held for 1 hour and cooled in the furnace. Moreover, Hassan *et al*., (2014) [7] investigated the effects of saw dust and rice husk on the properties of local refractory clay.

Furthermore Aramide, [5] produced and characterized porous insulating fired brick from Ifon clay. In all the references above only the later investigated the thermal conductivity among the properties examined. The aim of the present work is to evaluate the effects of varying porosity (through varied amount of saw dust admixture) on the properties (including thermal conductivity) of the porous ceramic bricks produced from Ipetumodu clay.

EXPERIMENTAL PROCEDURE

The materials used for this study include; dried saw dust, Ifon clay as mined, water, wooden sieve, beaker, conical flask and thermometer. The equipment used for this study include; a furnace, mounting press, grinding mill, pulverizer, seive shaker and set of sieves, and com- pression strength tester. Dried saw dust from mahogany tree acquired from a saw mill in Akure was sun dried further to remove moisture present. Clay acquired from Ifon, also in Ondo State was soaked in water for three days to dissolve the clay and at the same time to form slurry. The resulting slurry was then sieved to remove dirt and other foreign substances using a sieve. More water was thereafter poured into the clay to form slurry once again. This is then allowed to settle down for seven days. The floating clear liquid was decanted after the seventh day. The settled fine clay was then poured into a P.O.P mould and left undisturbed for three days in other to allow the liquid still present to drain out completely. The resulting clay was then sun dried for two days. This was followed by grinding in a grinding mill to reduce the particle sizes. A pulverizer was

then used to reduce the sizes of the clay particles further into still finer particles. A final sieving of the pulverized sample was then carried out. A sieve analysis of the clay and saw dust used in this project is as stated: Clay; <850 µm, Saw dust; <1700 µm. A mixture of clay and saw dust was made with the saw dust in various proportions of 0%, 5%, 10%, 15%, 20%, 30% and 40%. Each mixture was made thoroughly with a little addition of water to induce some plasticity and homogeneity of both the clay and saw dusts. The resulting mixtures (for each proportions of saw dust), were then compacted in a mounting press to obtain cylindrical shaped samples. These samples were then placed in the furnace and fired at 1000°C (held at the temperature for 1 hour) such that the saw dust burns off leaving some ash and pores. Series of tests were then performed on the fired samples. These tests include; cold crushing strength, bulk density, porosity, thermal shock resistance, permeability and thermal conductivity.

Apparent Porosity

Test samples from each clay/saw dust blend (for varying proportions) were dried for 12 hours at 110°C. The dry weight of each fired sample was taken and recorded as D. Each sample was immersed in water for 6 hrs to soak and weighed while been suspended in air. The weight was recorded as W. Finally, the specimen was weighed when immersed in water. This was recorded as S. The apparent porosity was then calculated from the expression:

$$p = \frac{(W-D)}{(W-S)} \times 100 \quad (1)$$

Cold Compression Strength

Cold compression strength test is to determine the compression strength to failure of each sample, an indication of its probable performance under load. The shaped samples of clay blends with saw dust were dried in an oven at a temperature of 110°C, allowed to cool and then placed between two plates of the compression strength tester. This was followed by the application of a uniform load to it. The load at which a crack appears on the sample was noted and the cold compression strength (CCS) is calculated from the equation:

$$CCS = \frac{\text{Load to Fracture}}{\text{Surface area of sample}} \quad (2)$$

Thermal Shock Resistance

Each sample of the clay/saw dust blend was placed in an electrically heated furnace to attain the test temperature of 1000°C for over 3 hours. Each sample was then with- drawn from the furnace and held for 10 minutes. The procedure was repeated until an appearance of a crack was visible. The number of cycles necessary to cause a crack was recorded for each of the samples and taken as a measure of its thermal shock resistance.

Bulk Density

The test specimens were dried at 110°C for 12 hours to ensure total water loss. Their dry weights were measured and recorded. They were allowed to cool and then immersed in a beaker of water. Bubbles were observed as the pores in the specimens were filled with water. Their soaked weights were measured and recorded. They were then suspended in a beaker one after the other using a sling and their respective suspended weights were measured and recorded. Bulk densities of the samples were calculated using the formula:

$$\text{Bulk density} = \frac{D}{(W-S)} \times 100 \quad (3)$$

where: D = Weight of dried specimen, S = Weight of dried specimen suspended in water, and W = Weight of soaked specimen suspended in air

Thermal Conductivity Test (Using Ibrahim's Thermal Conductivity Apparatus; the Steam Method)

Test specimens of area 0.002 m^2 and thickness of 0.01 m were cut from their respective mother bricks. The test specimens were tested one after the other. Each specimen was fixed between two copper discs provided within the equipment. A conical flask containing 50 ml of water was placed directly above and in contact with the specimen. A cork having a thermometer passing through it was used to cork the mouth of the conical flask. The thermometer reads the temperature changes of the water in the flask. The test section was then closed and the initial water temperature was noted. A second thermometer with the aid of a cork was inserted into the steam outlet pipe offset to monitor the steam temperature so as to ensure a constant base temperature of 100°C.

The boiler water outlet valve was closed while 5 litres of water was measured and poured into the boiler. The steam inlet valve, outlet valve, and condensate outlet valve were all closed. With the boiler cover remaining opened, the boiler was switched on. Immediately the water started boiling, the boiler cover was closed, while the steam inlet valve was fully opened with all the remaining valves closed. Timing commenced with the aid of a stopwatch immediately the steam inlet valve was opened. The testing was timed in each case for 10 minutes and final temperature of the water in the beaker was noted at the end of time. Each specimen was tested (experimented) twice and a mean temperature value was obtained. At the end of each experiment, the steam outlet valve was opened to release steam. The water in the boiler was refilled to maintain 5 litres and the experiment was repeated as stated above for other specimens. The value of the thermal conductivity, K for each of the specimen was determined using the formula [8, 9];

$$K = \frac{2.303\frac{MCL}{A}[\log(\frac{\theta_1}{\theta_2})]}{t} \quad (4)$$

where, K = thermal conductivity of the specimen, T_l = temperature of steam (in Kelvin), T_i = Initial temperature of water in conical flask, T_4 = Final temperature of water in conical flask, t = Time (s), A = Specimen area, (m^2), M = mass of water in conical flask (kg), C = specific heat capacity of water in conical flask (J/kgK), L = thickness of specimen (m), $\theta_1 = T_1 - T_i$, $\theta_2 = T_1 - T_4$.

Qualitative and Quantitative XRD

The samples were prepared for XRD analysis using a back loading preparation method [10]. They were analysed using a PANalytical X'Pert Pro powder diffractometer with X'Celerator detector and variable divergence- and receiving slits with Fe

filtered Co-Kα radiation. The phases were identified using X'Pert Highscore plus software. The receiving slit was placed at 0.040°. The counting area was from 5 to 70° on a 2θ scale. The count time was 1.5 s. The temperature-scanned XRD data were obtained using an Anton Paar HTK 16 heating chamber with Pt heating strip. Graphical representations of the qualitative result follow below.

The relative phase amounts (weight %) were estimated using the Rietveld method (Autoquan Program) as reported by Young et al [11]. Amorphous phases, if present were not taken into consideration in the quantification.

Scanning Electron Microscopy

Morphology and microanalysis of raw clay and sintered ceramic composite samples were determined using ultrahigh resolution field emission scanning electron microscope (UHR-FEGSEM) equipped with energy dispersive spectroscopy (EDS). The pulverized clay samples/sintered ceramic composite samples were previously gold coated. The samples were studied using ultra-high resolution field emission scanning electron microscope (UHR-FEGSEM) equipped with energy dispersive spectroscopy (EDS). Particle images were obtained with a secondary electron detector.

RESULTS AND DISCUSSION

Figures 1 to 5 show the effects of varying the amount of rice husk admixtures on various physical and mechanical properties of the ceramic samples. While Figure 6 (a) to (g) show the SEM images of the various sample revealing their relative pores.

Aramide et al. 2014 [12] characterized and discussed the Iseyin clay sample with some other clay samples from other part of the country. They carried out x-ray diffractometry analysis and scanning electron microscopy of the raw clay sample used in this work. From their report it can be seen that the Iseyin clay contains 39.71% kaolinite and 39.55% quartz (which are refractory materials).

Effect rice husk admixture on the bulk density of the ceramic sample

Figure 1 show the effect of varying the amount of rice husk admixture on the bulk density of the ceramic sample. From the figure it is observed that the bulk density of the sample reduced with increase in the rice husk admixture. It is is observed that the bulk density of the sample was 2.42 g/cm^3 when there was no rice husk incorporated into the sample. However, when 5% rice husk was incorporated into the sample, the bulk density reduced to 1.34 g/cm^3. Further increase in the rice husk admixture to 10% leads to further reduction in the bulk density to 1.19 g/cm^3. As the rice husk admixture was increased to 15% the bulk density is observed to reduce further to 1.076 g/cm^3. Furthermore, increasing the rice husk admixture content to 20% leads to the bulk density of the sample being decreased to 1.04 g/cm^3. This is because when the sample is fired at the sintering temperature of 1000°C, all the rice husk is burnt off from the matrix of the ceramic leaving empty pores within the ceramic. This can be observed in the Figure 6 (a) to (g); it can be estimated from the figure that the pore volume in each of the micrographs increased with the least (very little) in Figure 6 (a) and the highest pore volume in Figure 6 (g). The more the rice husk admixture added to the sample, the more the pores that will be left after sintering. This is the reason for the reduction in the bulk density of the sample with increased rice husk admixture [5, 13].

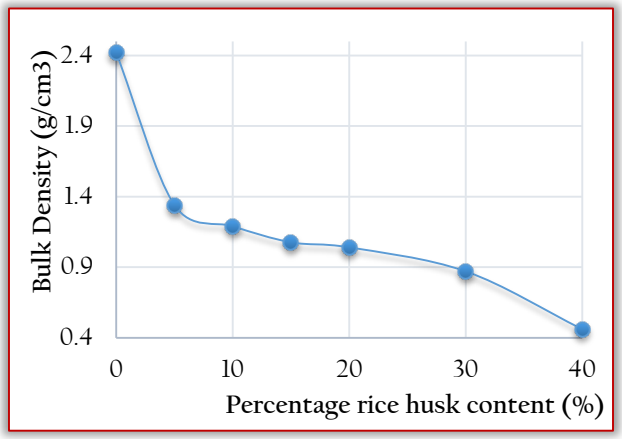


Figure 1. Effects of percentage rice husk content on the bulk density of the samples

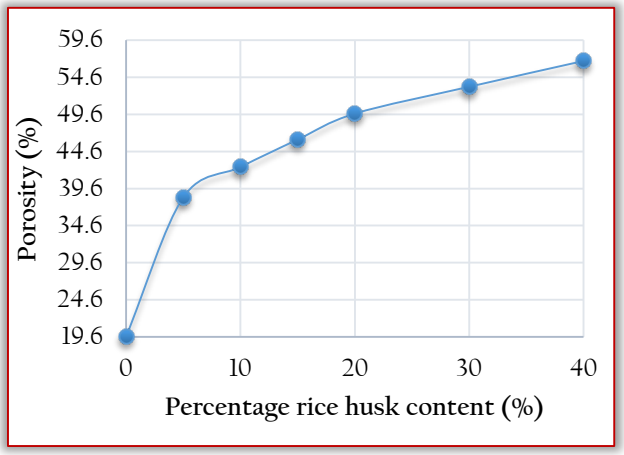


Figure 2. Effects of percentage rice husk content on the porosity of the samples

Effect rice husk admixture on the porosity of the ceramic sample

Figure 2 shows the effects of rice husk admixture on the porosity of the sintered ceramic samples. From the figure, it is observed that the porosity of the ceramic samples increases with increase in the rice husk admixture. The porosity of the sample was 19.65% when it rice husk admixture was 0%; it increased to 38.4% as the rice husk admixture increased to 5%. Moreover, it is observed that the porosity of the sample increased further to 42.5% as the rice husk content was increased to 10%. Further increase in the rice husk admixture to 15% is observed to lead to increase in the porosity of the sample to 46.2%. A porosity of 49.7% is observed for the sample with 20% rice husk admixture, further increase in the rice husk admixture content to 30% is observed to result in the porosity being increased to 53.32%. Lastly, the porosity of the sample is observed to increase further to 56.8% with increase in the rice husk admixture to 40%. This is because

when the samples are sintered at the 1000°C, the combustible rice husk content got burnt off thereby leaving empty pores within the ceramic matrix. As earlier explained, the more the rice husk content, the more the empty pores that would be left after sintering [13, 14, 15].

Effect rice husk admixture on the thermal conductivity of the ceramic sample

Figure 3 shows the effect of the rice husk admixture on the thermal conductivity of the ceramic samples. From the figure, it is observed that the thermal conductivity of the sintered ceramic samples decreases with increase in the rice husk admixture. It could be observed that the thermal conductivity of the sample was 0.24 W/m/K when the rice husk admixture was 0%; it then reduced to 0.19 W/m/K as the rice husk admixture was increased to 5%.

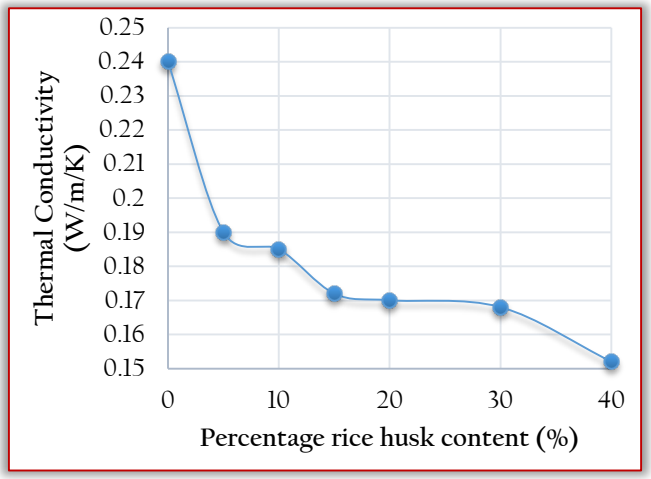


Figure 3. Effects of percentage rice husk content on the thermal conductivity of the samples

Moreover, further increased in the rice husk admixture to 10% is observed to lead to further reduction in the thermal conductivity of the sample to 0.185 W/m/K. When the rice husk admixture was 15% the thermal conductivity is observed to be 0.172 W/m/K, as the rice husk content increased to 20% the thermal conductivity is observed to reduce to 0.17 W/m/K. Moreover, increase in the rice husk admixture to 30% is observed to lead to further reduction in the thermal conductivity of the samples to 0.168 W/m/K. This is majorly because the porosity of the samples increases with the increase in the rice husk admixture.

Increased porosity means increased percentage pores within the ceramic matrix which insulates the flow of heat through conduction from the hotter part to the cold part [5, 13, 15].

Effect rice husk admixture on the thermal shock resistance of the ceramic sample

Figure 4 shows the effect of the percentage rice husk admixture on the thermal shock resistance of the ceramic samples. From the figure it is clearly observed that the thermal shock resistance of the samples reduces with increase in the rice husk admixture. The thermal shock resistance of the sample with 0% rice husk admixture was observed to be 26 cycles. Addition of 5% rice husk admixture lead to the reduction of the thermal shock resistance to 18 cycles. It was also observed that the thermal shock resistance of the sample reduced to 14 cycles as the rice husk admixture was increased to 10%. Furthermore, increase in the rice husk admixture to 15% lead to further reduction in the thermal shock resistance of the sample being reduced to 11 cycles.

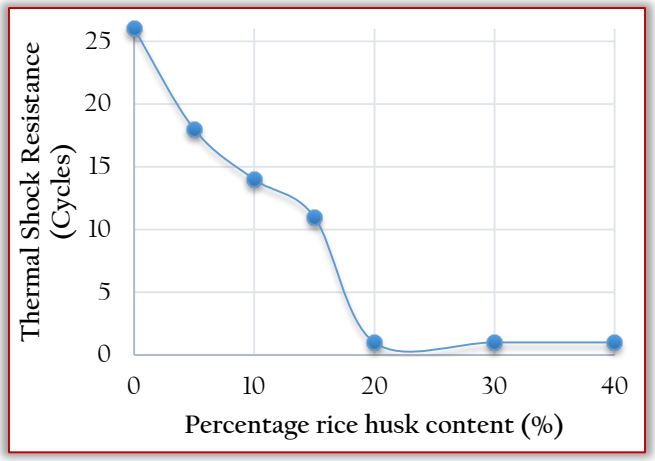


Figure 4. Effects of percentage rice husk content on the thermal shock resistance of the samples

Beyond this point, further increase in the rice husk admixture of the sample lead to the sample possessing the lowest value of thermal shock resistance of 1 cycle. The explanation that could be given to this is that the shapes of the pores have great influence on the mechanical properties of the ceramic. A pointed end pore will aggravate any form of stress within the ceramic body. That is it will have a multiplying effect on the stress (thermal or mechanical), thereby making the ceramic to fail through propagation of intrinsic cracks at much lower stress than it should have failed [16, 17].

Effect rice husk admixture on the cold crushing strength of the ceramic sample

Figure 5 shows the effect of rice husk additive on the cold crushing strength of the ceramic sample. From the figure, it can be observed that the cold crushing strength of the samples decreases with increase in the rice husk admixture. It is observed that the cold crushing strength of the sample with 0% rice husk additive is 14.5 N/mm^2, it declined slightly to 13.7 N/mm^2 when the rice husk admixture was increased to 5%. Moreover, the cold crushing strength is observed to decline sharply to 9.7 N/mm^2 when 10% rice husk was incorporated into the sample. Furthermore, additional increase in the amount of rice husk additive into the sample (to 15%) leads to further reduction in the cold crushing strength of the sample (5.7 N/mm^2).

The cold crushing strength of the sample is observed to reduce to 4.9 N/mm^2 as the rice husk admixture is increased further to 20%. It was earlier explained that when the samples were fired at the sintering temperature, the rice husk content, being combustible, is burnt off, leaving voids/pore within the ceramic matrix.

The empty voids in the ceramic matrix makes the samples to contain less matter per unit surface area; this implies that increased rice husk admixture means less matter would be available within the sample to bear the applied load. In other

word, less matter per unit surface area means that the samples would (more porous) lighter with increased rice husk admixture. Porous bricks are lighter, hence they cannot carry heavy load [18].

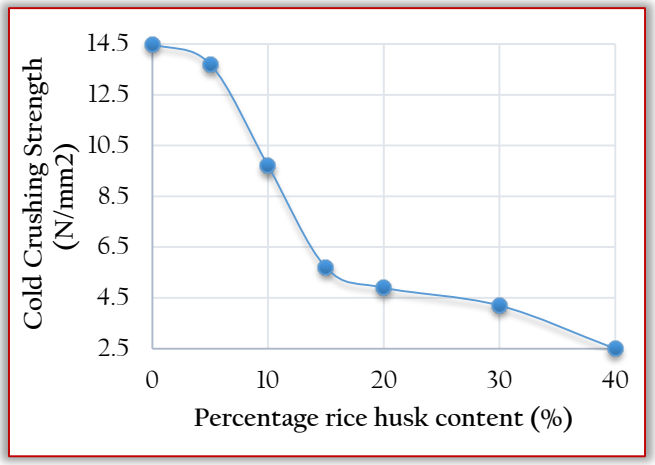
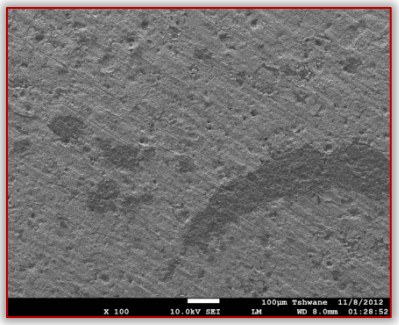


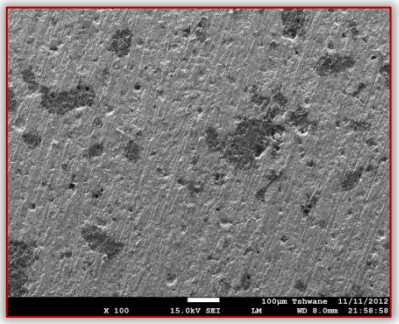
Figure 5. Effects of percentage rice husk content on the cold crushing strength of the samples

From Figure 6, the scanning electron microscope of the various samples is shown.

Figure 6 (a) shows the SEM image of the sample with 0% rice husk admixture, (b) shows the SEM image of the sample with 5% rice husk admixture, (c) shows the SEM image of the sample with 10% rice husk admixture, (d) shows the SEM image of the sample with 15% rice husk admixture, (e) shows the SEM image of the sample with 20% rice husk admixture, (f) shows the SEM image of the sample with 30% rice husk admixture and (g) shows the SEM image of the sample with 40% rice husk admixture. The figure shows relative pores on each of the samples.



(a)



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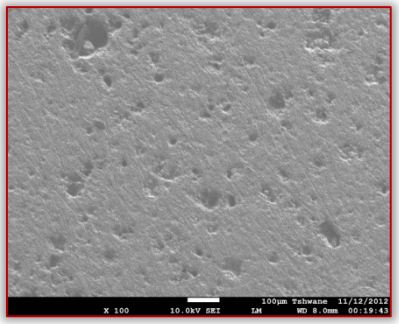

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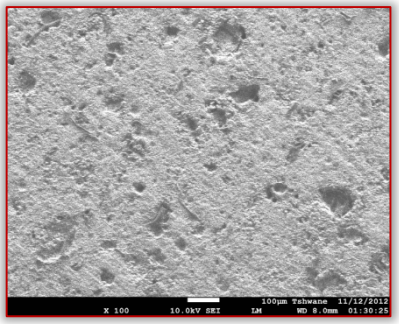

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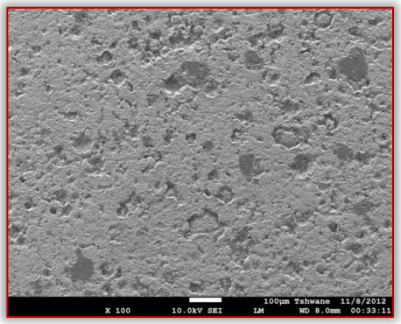

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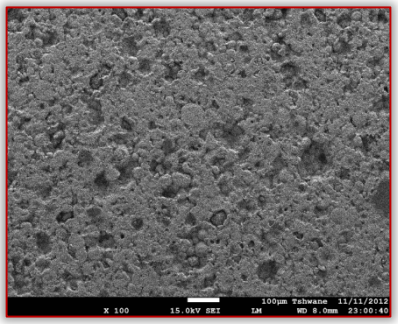

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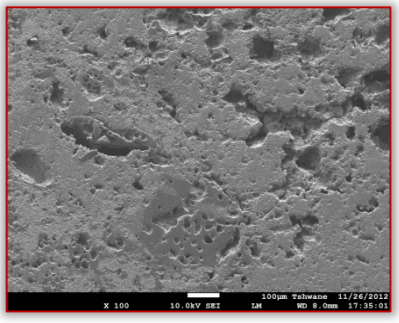

(g)

Figure 6. Scanning electron microscope images of the various sintered ceramic samples: (a) 0% rice husk, (b) 5% rice husk, (c) 10% rice husk, (d) 15% rice husk, (e) 20% rice husk, (f) 30% rice husk, (g) 40% rice husk

CONCLUSION

Increase in the rice husk admixture in the sample leads to increase in the porosity of the sample. This also resulted in the reduction in the bulk density, cold crushing strength, thermal shock resistance and thermal conductivity of the sintered ceramic samples. The choice of optimum parameter for application of the sintered ceramic sample depends on the service condition the sample will meet in application. For a service condition where the ceramic will be in contact with molten metal or slag, sample with 0% rice husk admixture would be optimum. But for efficient insulation in a heat treatment furnace sample with 40% rice husk admixture would be optimum.

ACKNOWLEDGEMENT

The financial assistance of the National Research Foundation (NRF) towards this research is hereby acknowledged. Opinions expressed and conclusions arrived at, are those of the author and are not necessarily to be attributed to the NRF

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ISSN: 2067-3809