Preparation and Characterization of Rice Husk based Alumina and its Applications

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Abstract: Rice husk is an abundantly available waste material in all rice producing countries. In certain regions, it is sometimes used as a fuel for parboiling paddy in the rice mills. The partially burnt rice husk in turn contributes to more environmental pollution. There have been efforts not only to overcome this but also to find value addition to these wastes using them as secondary source of materials. The organic materials in rice husk consist of celluloses (55–60 wt.%, including cellulose and hemicellulose) and lignin (22 wt.%). Approximately one-fifth of the ash is obtained on burning rice husk in air. The ash contains >90% silica by mass with minor amounts of metallic elements. Because the ash is obtained as a fine powder, it does not require further grinding [8], making it the most economical source of nanoscale silica. Other agro-product used is jute fiber which is used for synthesis of alumina fiber. The incorporation of porosity within a tailored structure gives porous ceramics many intrinsic properties such as high Permeability, high surface area, and good insulating characteristics. Porous ceramics have found a wide variety of applications including as filters, membranes, sensors, catalyst carriers, piezoelectric ceramics, biomedical and construction materials. In the present work we have Prepared sample, a composition consisting of 80% Alumina powder, 10% Rice husk and 10% of sucrose solution was taken as binder and we have prepared 4 different samples 1 with pure Alumina and 3 with the above compositions but with different Size of Rice husk particle(microns) 75,320, and 425 respectively. XRD, SEM, relative density and flexural strength of all samples have been studied.

1. Introduction

The microstructure such as porosity and pore size distribution are very important factors for many potential applications of porous ceramics. For instance, the increase in porosity of porous ceramic caused an increase in the permeability and the ideal combination of pore size and porosity could optimize the relationship of permeability and mechanical strength.

The microstructure includes most grains, secondary phases, grain boundaries, pores, micro-cracks and hardness microindentions. Most bulk mechanical, optical, thermal, electrical and magnetic properties are significantly affected by the microstructure. The fabrication method and process conditions are generally indicated by the microstructure. The root cause of many ceramic failures is evident in the microstructure. Ceramography is part of the broader field of materialography, which includes all the microscopic techniques of material analysis, such as metallography, petrography and plastography. Ceramography is usually reserved for high-performance ceramics for industrial applications, such as 85–99.9% alumina (Al2O3), zirconia(ZrO_2), silicon carbide(SiC), siliconnitride (Si₃N₄), and ceramic-matrix composite. It is seldom used on white ware ceramics such as sanitary ware, wall tiles and dishware. The great majority of alumina is consumed for the production of aluminum, usually by the Hall process.

Porous ceramics with different porous morphology and size distribution can be fabricated by different routes, such as Burning out a polymeric sponge impregnated with a ceramic slurry, Solid-state sintering, sol–gel process, Foaming Process, Gel-casting process.

The effect of porosity on variables such as the elastic modulus and thermal conductivity can be readily quantified [1]. However its effect on the fracture energy is not well understood with many different types of behavior apparently

being observed. It is often suggested that the fracture energy should change with porosity in the same way as the elastic modulus on the basis that a material's surface energy is related to its elastic modulus. Whilst this may be correct for a homogeneous material, there is no reason to believe that the surface energy of the alumina in the porous structure should be modified by the presence of the pores. Rather one might expect that introducing porosity reduces the amount of material that can break and hence the energy that is dissipated. The fracture energy of a porous body, Rp, would therefore be expected to decrease linearly with the area fraction of pores on the crack plane, A, according to (1) where Rd is the fracture energy of the dense material. Two types of porous structure may be considered. In the first, the body is made up of discrete pores surrounded by a matrix. This is the structure that might be made by adding particles of a fugitive phase to the powder compact and then removing them by heating. In that case, provided the crack cuts through the structure in some random plane then the area fraction of pores in the crack plane, A, is equal to the volume fraction of pores in the body, P. Porous materials are more usually made by sintering a powder compact so that it is not fully dense. Here fracture occurs by failure of the necks between the particles that are developing during sintering.

The situation is more complex than before, but there are two limits. The first is where the body contains no pores, so that the fracture energy is equal to that of the dense material. The second is the un-sintered powder compact, where the porosity can be obtained from the relative density of the powder compact and where the fracture energy is approximately (but not equal) to zero. Calculations suggest that the variation between these two points should be nonlinear so that the variation in fracture energy is given by (2) where Pg is the volume fraction of pores in the un-sintered powder compact. The value of the exponent is normally unknown, although there is data obtained from partially sintered materials that are consistent with a value for n of 1.

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However it has been shown recently that the change of fracture energy with porosity reported above is due predominantly to changes in the grain size of samples sintered at different temperatures (and hence associated also with different porosities). Once these effects are removed, the fracture energy appears to vary in a similar way to that observed in structures consisting of isolated pores in a matrix. That is the fracture energy remains approximately constant as the volume fraction of pores is increased up to a volume fraction of 0.2, after which the fracture energy falls more rapidly.

Being fairly chemically inert, relatively non-toxic, and white, alumina is favored filler for plastics. Alumina is a common ingredient in sunscreen. Alumina catalyses a variety of reactions that is useful industrially. In its largest scale application, alumina is the catalyst in the Claus process for converting hydrogen sulfide waste gases into elemental sulfur in refineries. It is also useful for dehydration of alcohols to alkenes. Aluminum oxide is used for its hardness and strength. It is widely used as a coarse or fine abrasive, including as a much less expensive substitute for industrial diamond. Many types of sandpaper use aluminum oxide crystals. In addition, its low heat retention and low specific heat make it widely used in grinding operations, particularly cutoff tools. In chemistry laboratories, alumina is a medium for chromatography, available in basic (pH 9.5), acidic (pH 4.5 when in water) and neutral formulations. In chemistry laboratories, alumina is a medium for chromatography, available in basic (pH 9.5), acidic (pH 4.5 when in water) and neutral formulations. Insulation for high temperature furnaces is often manufactured from aluminum oxide.

2. Experimental

Refining and grinding of rice husk has been done as per following flow chart shown in Figure 1.



Figure 1: Flow chart of Refining and grinding of rice husk

The figure above shows the schematic process for extraction of silica from rice husk. Rice husk (RH) was collected from a rice mill in Varanasi, India. The RH was separated from rice grain by air blowing and washed with tap water for several times till all the impurity was completely removed. The rice husk was then dried at 110oC for 8 h. The dried husk was grinded and sieved to different sizes in microns. For the preparation of sample, a composition consisting of 80% Alumina powder, 10% Rice husk and 10% of sucrose solution was taken as binder. First of all, Alumina powder was taken and mixed with appropriate amount of Rice husk with desired particle size. After proper mixing of raw material, 40% wt% sucrose solution was poured and mixed vigorously. Thereafter the mix was pressed under pressure of 8 tones. The composition of specimen are as follows:

Table I: Composition of all Prepare	d Samples
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Sample no.	Alumina powder (% by weight)	Rice husk (% by weight)	Binder (% by weight)	Size of Rice husk particle (microns)	
1.	90	0	10		
2.	80	10	10	75	
3.	80	10	10	325	
4.	80	10	10	420	

For the purpose of sample preparation, alumina was taken and appropriate amount of rice husk with particular particle size was mixed. After 10 minutes of mixing the binder was added and mixed. The mix was then pressed into pellets under the pressure of 8 tones. This green pellet was then calcined at 800 degrees Celsius. However, proper care was taken in this heating schedule so as to avoid the cracking of samples. During calcination there is decomposition of rice husk and also sucrose. At early stage, first physically bonded water at around 100-110 degree Celsius and then chemically bonded water is removed. So, the heating rate is kept so that removal of the gases thus formed due to decomposition moves slowly, avoiding any crack in sample. The heating rate was kept as low as 1 degree Celsius per minute. After reaching of temperature 800 degree Celsius, a holding period of 2 hours was given to sample so that there is complete removal of the gaseous material. Once the samples were calcined, it was sintered at 1450 degree Celsius. Thus the samples were now ready for testing for various properties.

3. Results and Discussion

We have performed the XRD of prepared samples and The XRD pattern of sample no.1 is in congruence with the fact that it is purely Alumina. So almost all peaks except one shows the presence of alumina. The presence of minute amount of silica may due to unhygienic condition of handling. In sample no. 2 XRD pattern show the dominant presence of Alumina. It also shows that silica is present in the sample. Along with them the sample also contains minute amount MgO. The presence of silica and MgO can be justified in the light of fact that rice husk contains above 90% of silica and about 0.5 % MgO. The peaks in sample no. 3and sample no. 4show the dominant presence of Alumina and also shows that silica and minute amount MgO is present in the sample. The presence of silica and MgO can be justified in the light of fact that rice husk contains above 90% of silica and about 0.5 % MgO.

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Figure 2: XRD data of a) Sample no. 1, b) Sample no. 2, c) Sample no. 3, d) Sample no. 4

Figure: 3 shows the SEM images of all four samples and it is clear from figure a) that the pure alumina sample does not have surface pores. It is also depict able from figure b) sample 2, figure c) sample 3 and figure d) sample 4 that the micro pores are increasing with the particle size of rice husk.



Figure 3: SEM images of a) Sample no. 1, b) Sample no. 2, c) Sample no. 3, d) Sample no. 4

Relative density of sample shows decreasing pattern and it is clear from figure:4. It is because pure alumina has almost negligible amount of pore. And the rest three sample, though having equal rice husk in their making have different pore structure which may be the cause of slight density variation among them. Figure:5 contains the data related to flexural strength and flexural strength of reference sample is as high as 226 MPa and it has significantly reduced to around 80MPa on addition of rice husk. The reason behind this phenomenon can be attributed toporosity of the sample thus produced due to rice husk.

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Figure 5: Flexural strength of all four Samples

4. Conclusion

Thus from above discussion it may be concluded that through addition of rice husk we can generate the porosity within the alumina specimen. These pore structure also varies with variation in the size of rice husk particle. The pore size seems to be increasing with increasing particle size of rice husk. However, more effort is required to analyze these variation i.e. porosity, microstructure of pore and its distribution. We also need to find the ways to increase the strength of material thus prepared.

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