

Rice Husk Ash Selection as Solid Oxide Fuel Cell Sealing Ceramic Material

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ABSTRACT

Various ceramic glass compositions have been designed and tested as Rigid Bonded Sealing in Solid Oxide Fuel Cell (SOFC) applications. The ion of silica dioxide (SiO_2) synthesized with other oxides as character enhancers. SiO_2 can be obtained from mines or from decomposition of living things, one of which is Rice Husk Ash (RHA) as a thermochemical transformation product. Conversion process conditions e.g. (pyrolysis, gasification or combustion) may affect the physical-chemical characteristics of the final product. RHA generally contains approximately 60% silica (SiO_2), 10-40% carbon and a small portion of other material compositions. In this study, an RHA production process was carried out from Rice Husk (RH) samples from Klaten (R1) and Wonosobo (R2) Central Java. The calcination process was carried out by washing the RH route with Sodium Lauryl Sulfate (SLS) as a surfactant and 0.5 M sulfuric acid (H_2SO_4) for leaching. The drying process was done with an oven for 24 hours at 110°C . The production process ended with burning in a muffle furnace at 650°C for 2 hours. The characterization of RHA from both samples produced amorphous silica (SiO_2) with an average particle size of $R1 = 0.217$ and $R2 = 0.268$ μm . Both samples were consisted of amorphous silica with purities above 97%. The surface area of silica grains produced after washing RH with sulfuric acid are $R1 = 265.73 \text{ m}^2/\text{g}$ $R2 = 294.37 \text{ m}^2/\text{g}$. The average pore diameter of R1 is $\sim 2.04 \text{ nm}$ and R2 is 1.89 nm , indicating that the silica produced by R1 is mesoporous while R2 is microporous. Microstructural visualization through SEM strengthens the information of RHA produced. High purity amorphous silica has a large grain surface area. useful as an absorber or supporting catalyst in chemical synthesis. This supports the synthesis of RHA with other oxides in its production as a seal to the SOFC system.

Key words : Seal, SOFC, RHA, Characterization.

1. INTRODUCTION

Among fuel cell technologies, Solid Oxide Fuel Cell (SOFC) is the most promising and shows great potential in distributed generator applications due to its strong electrolyte properties,

ability to reform gas fuels and high efficiency [1]. SOFC works electrochemically at intermediate temperatures with its basic components of anode, electrolyte, cathode, interconnection and sealant. Each has a special function and is made of ceramic materials that are able to adapt well to extreme operating cycles [2].

Various ceramic glass compositions have been designed and tested as Rigid Bonded Sealing in SOFC applications. The material composition consists of basic composition of silica dioxide (SiO_2) with other oxides to achieve the desired material performance. Ceramic sealing materials are derived from those developed material using Silica Diocide (SiO_2) as a base material. This material is then synthesized with other oxide materials (AlO_3 , BaO , CaO , MgO , TiO_2 and others) as a reinforcement. The material developed must be able to withstand the load cycle temperature and will support the operation for a relatively long time [3-9].

One of the main challenges in implementing solid oxide fuel cells (SOFC) is the development of a suitable sealing material as an air and fuel separator. Several approaches have been used to achieve adherence to the necessary prerequisites, mechanical integrity and stability [10], including rigid seals (no-load applied during operation) and compressive seals (load applied during operation).

Seals are generally used as a gap cover in the cell and chamber (reaction chamber). This seal is usually made of ceramic material which can withstand temperature cycle loads. The CTE must be in accordance with the electrodes material, not easily cracked, and have high resistivity [11]. Rigid seals make effective bonds on the surface and offer advantages over compressive seals because they require a relatively simple method for binding ceramic and metal parts (rather than applying an external sealing load).

Silica (SiO_2) is a basic raw material widely used in industrials. Rice Rusk (RH) can be considered as a suitable source of energy and silica in Asian countries. Given that rice production in Indonesia is quite abundant. Resulting rice husk waste is quite large with 15.8 million tons per year. Nowadays, RH has the potential to be developed into silicon-based materials that can be used for the production of

silicon carbide, silica, silicon nitride, silicon tetrachloride, pure silicon, and zeolite [12]. Silica can be obtained in nature such as quartz sand and in living things. RH itself has a large amount of waste. Rice husk through the purification process produces RHA (Rice Husk Ash) containing high-level SiO₂ [12-15]. RHA can be used as a ceramic material for various advanced applications [16]-[20].

The main objective of this research is to develop a sealing material based on RHA as a substitute for pure SiO₂ derived from extracting RH by synthesizing other material as a reinforcement of seal performance. RHA is a product of thermochemical transformation of RH. Conditions in the conversion process (pyrolysis, gasification or combustion) affect physical-chemical characteristics of the final product. RHA generally contains approximately 60% of silica (SiO₂), 10-40% carbon and a small portion of other material compositions. The main components of RH are lignin, cellulose and ash which vary depending on climate, variety and geographical location where rice is planted. White ash obtained from the combustion of this raw material at moderate temperatures contains 87 ± 97% silica in an amorphous form and a number of metal impurities such as iron (Fe), manganese (Mn), calcium (Ca), sodium (Na), potassium (K) and magnesium (Mg) which affects the purity and color of silica and it can be removed by pre-treatment with hydrochloric and sulfuric or nitric acid before combustion [21].

The utilization of rice husk as a source of silica is based on the removal of impurities with low effort and high specific surfaces [14,22-25]. Chen showed that sufficient pure silica can be obtained from RHA by simple acid washing procedures [21]. It has also been shown that with acid leaching, silica with purity of > 99% can be obtained by combusting rice husks at 600°C under normal atmospheric pressure [15],[18],[26]-[28].

The process of combusting RH from various experiences shows the correlation between microstructure and combustion control. The amorphous structure of the SiO₂ produced provides physical information on various combustion treatments on the results [13],[29],[30]. Treatment and combustion process provides an overview of the most inexpensive process options with the most optimal quality and quantity of ceramic material [15]. It is concluded that at 600°C to 1000°C, depending on the combustion time, amorphous silica is formed. However, at higher temperatures, crystalline silica is obtained.

The purpose of this study is to select the Central Java local RHA through characteristic comparisons. The properties revealed include; the determination of the structural properties of the compound, the level of SiO₂ content, grain

surface area, pore diameter, particle size, crystallinity level and morphological structure. This procedure serves as a preliminary determining RHA characteristics prior to combined it with other oxides to form a sealing ceramic material composition.

2. MATERIAL AND METHOD

2.1 RHA Production

IR 64 Rice Husk was obtained from several rice mills in Klaten (R1) and Wonosobo (R2) Central Java. RH was rinsed with Sodium Lauryl Sulfate (SLS) solution as a surfactant with constant stirring of 10 minutes. SLS is also known as Sodium dodecyl sulfate (C₁₂H₂₅SO₄Na), is surfactant anion. This chemical salt is organosulfur anion containing 12-carbon tails bound to sulfate groups, making this chemical substance have ambiphilic properties as a condition of a detergent. SLS is a very powerful type of surfactant used to bind oil and water-soluble impurities.

RHA production can be briefly explained as follows: First, rice husk was rinsed with distilled water to remove surfactants. After that was, the first natural drying step to eliminate moisture. In the next process, RH was rinsed separately and treated with sulfuric acid H₂SO₄ at ~ 60°C with a concentration of 0.5 M for 30 minutes with constant stirring. The use of sulfuric acid was based on the experience of Abu et al. who used various acids in the leaching process. Next, RH was rinsed with distilled water until it was acid-free, then filtered and dried with natural heating. Rice husks that had been dried naturally were then dried further in an oven at 110°C for 24 hours to completely eliminate humidity. The next process was burning the furnace at 650°C for 2 hours to obtain white RHA. Selection of temperatures above 500°C is due to the previous experience of using lower temperatures resulted in observable carbon elements marked with black or brown color.

2.2 RHA Characteristics

RH combustion producing RHA-R1 and RHA-R2 were analyzed using various analytical tools to help characterization process. The functional groups in the sample were determined using the FTIR Thermo Nicolet Avatar 360. Particle size was measured using the Particle Size Analyzer (PSA) using the Static Light Scattering method. The spectrum was recorded with 32 scans at 4 cm⁻¹ resolution in the range 4000-400 cm⁻¹. X-ray diffraction (XRD) patterns were obtained using X-ray diffraction (Bruker D2 Phaset 2nd Gen) with CuKα which was operated at 40 kV and 40 mA and 2 Theta between 5° to 50°. About 0.05g of the sample was mixed in 10g of distilled water after mixed strongly and sonicated for 30 minutes. The silica and metal impurity content in the sample was estimated with Wavelength Dispersive X-ray Fluorescence (WDXRF) - Rigaku Supermini200. Surface area and pore volume of the sample

were measured using Brunauer-Emmett-Teller (BET) Surface Area Analysis and / or Barrett-Joyner-Halenda (BJH) Pore Size and Pore Volume Analysis (Equipment / Method: Gas Sorption / Physisorption by Quantachrome Instruments).

BET analysis provides an evaluation of specific surface area of the material by utilizing multilayer nitrogen adsorption measured as a relative pressure function using a fully automatic analyzer. This technique involves evaluating the external area and pore area to determine the total specific surface area in m^2/g , producing important information in studying the effects of surface porosity and particle size in many applications. BJH analysis can also be used to determine pore area and specific pore volume using adsorption and desorption techniques. This technique characterizes the pore size distribution independent from external area due to the particle size of the sample.

Scanning Electron Microscope (SEM) is a type of electron microscope capable of producing high-resolution images of a sample surface. This microscope is used to study the surface structure of objects, which are generally magnified between 1,000-40,000 times. Through analysis using SEM, it is expected to be able to observe the morphological characteristics of the particles produced.

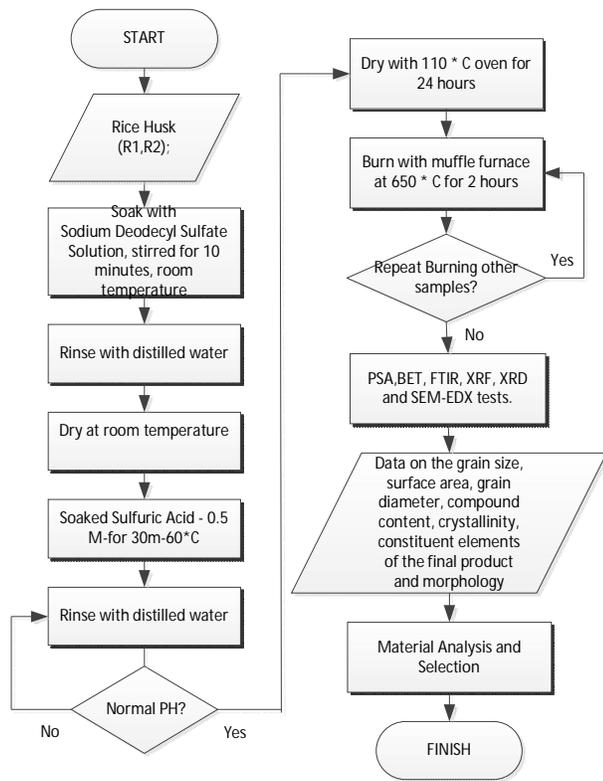


Figure 1: Production process flow and characterization

Table 1: Typical group uptake functions on RHA-R1

Wave Number (cm ⁻¹)	Group	Compound
3478	NH	Amina Primer
2363	C=C	Alkuna
1635	C=C	Alkena
1098	C-O-C	Eter
802	-(CH ₂) _n	Other
465	-(CH ₂) _n	Other

Further reading on ceramics, its wear characteristics and the surface treatment can be found in [31], [32] and [33].

3. RESULT AND DISCUSSION

3.1 Fourier Transform Infrared (FTIR) Analysis

The FTIR spectra of silica immersed in sulfuric acid after combustion at 650°C are shown in Figure 2. The main absorption peak at 1098 cm⁻¹, 465 cm⁻¹, 3478 cm⁻¹, 802 cm⁻¹, 1635 cm⁻¹, 2363 cm⁻¹ and R2 = 1098 cm⁻¹, 464 cm⁻¹, 3465 cm⁻¹, 802 cm⁻¹, 1638 cm⁻¹ in Figure 3 as an attribute for O-Si-O stretching and bending vibrations.

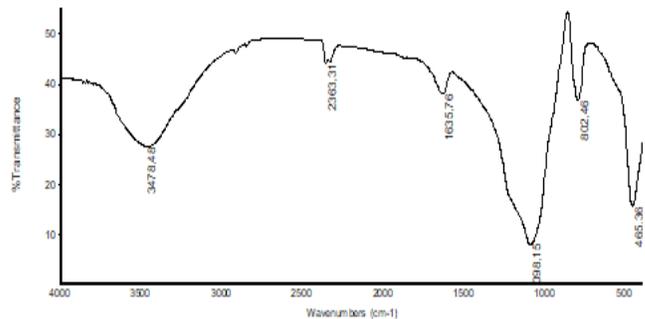


Figure 2: FTIR analysis for sample R1

Table 2: Typical group uptake functions on RHA-R2

Wave Number (cm ⁻¹)	Group	Compound
3465	NH	Amina Primer
1638	C=C	Alkena
1098	C-O-C	Eter
802	-(CH ₂) _n	Other
464	-(CH ₂) _n	Other

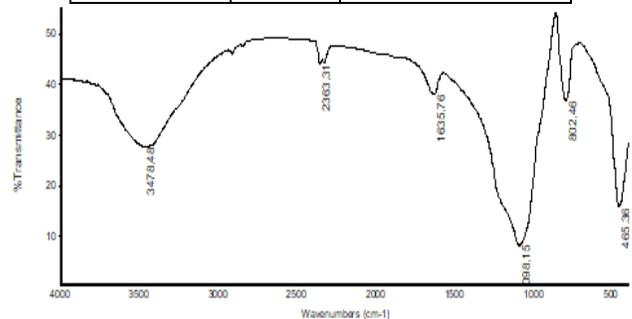


Figure 3: FTIR analysis for sample R2

Table 3: Particle size

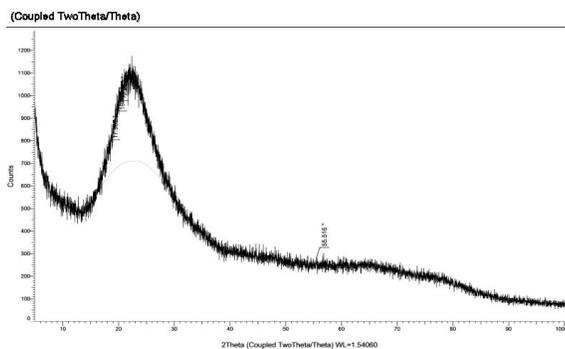
(Static Light Scattering) using PSA	R1	R2
	µm	
S1	265.93	216.3
S2	274.7	214.59
S3	263.38	221.52
Average Particle Size	268.00	217.47

3.2 Fourier Transform Infrared (FTIR) Analysis

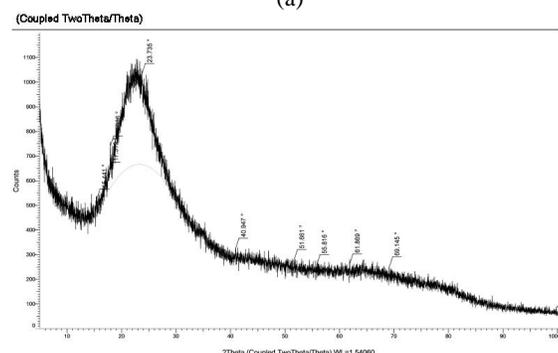
The particle size of silica at various combustion temperatures is given in Table 3. It was measured by PSA using the Static Light Scattering method with results varying from 214 µm to 275 µm.

3.3 Characterization of Crystallinity

The XRD pattern of insoluble silica at combustion temperatures is shown in Figure 3 and Figure 4, respectively. The XRD pattern of the two silica materials shows the same pattern as leached sulfuric acid. A wide-spread peak with maximum intensity at $2\theta = 22^\circ$ was observed, indicating the amorphous nature of silica. Silica released by acid showed a truly amorphous structure after combustion below 900°C, after which there was an increase in crystallinity [15]. This is due to the removal of alkali metals during acid leaching which inhibits the eutectic reaction with silica. Therefore, optimization of the combustion temperature of rice husk is needed to inhibit the crystallization of silica.



(a)



(b)

Figure 4: (a) Spectrum of X-Ray RHA-R1, (b) Spectrum of X-Ray RHA-R2

3.4 Analysis of XRF

XRF was used in identifying the chemical composition and purity of silica produced from rice husks. The silica production process at R1 reached 97.27% which was higher than R2 which was 97.03%. It needs to be emphasized here that the route chosen in getting SiO₂ was quite effective in removing metal impurities in rice husks. Table 4 shows that silica (SiO₂) is the main component and also contains a low amount of metal impurity.

Some Al₂O₃, CaO compound was identified as oxides reinforcing SOFC sealing ceramic characters, so the presence of these oxides can be considered not as impurities.

Table 4: Chemical Composition

Content	SiO ₂	Al ₂ O ₃	P ₂ O ₅	SO ₃	K ₂ O
mass%					
R1	97.27	-	0.68	0.14	0.48
R2	97.03	0.08	1.20	0.10	0.84
Content	CaO	MnO	Fe ₂ O ₃	RuO ₂	Ag ₂ O
mass%					
R1	0.68	0.15	0.23	0.11	0.26
R2	0.56	0.09	0.08	-	-

3.5 Analysis of BET-BJH

Ideally, a porous material should have a pore size distribution that depends on a particular application. In addition, it must exhibit high chemical properties, thermal, hydrothermal and mechanical stability, with high surface area and large pore size. This material must have a specific particle size which is around 1-100 nm. The porous material is initially defined according to its adsorption properties. Based on this, porous material is distinguished by its porous size. Mesoporous material is a material that has an average diameter of its axis between 2-50 nm which refers to the IUPAC classification. According to IUPAC, a porous material is divided into three classifications, namely micropores having shaft diameters of less than 2 nm, mesopores having shaft diameters between 2-50 nm, and macropores having shaft diameters of more than 50 nm.

Table 5: Result of BET-BJH Test

Surface Area Results	unit	R1	R2
Multipoint BET	m ² /g	265.74	294.38
BJH adsorption	m ² /g	128.09	123.72
BJH desorption	m ² /g	164.49	159.63
Pore Volume Results			
BJH adsorption cumulative	cc/g	0.22	0.21

micropore volume			
BJH desorption cumulative micropore volume	cc/g	0.24	0.23
Total Pore Volume	cc/g	0.27	0.28
Pore Size Results			
BJH adsorption pore radius	nm	1.64	1.64
BJH desorption pore radius	nm	1.85	1.85
Average Pore Size	nm	2.04	1.89

Surface area, pore volume and pore diameter of silica are given in Table 3. Surface Area reached R1 265.74 m² / g and R2 294.38 m² / g. The granular surface area and pore volume of sample R2 is greater than R1. This shows that the source of rice husk has variations, one of which is the origin. The high porous structure was obtained as confirmation by Pore R1 volume 0.27 cc / g and R2 0.28 cc / g. The average pore diameter of R1 is ~ 2.04 nm and R2 is 1.89 nm, indicating that the silica produced by R1 is mesoporous and R2 is micropore. It should be noted that hydrochloric acid or sulfuric acid has significantly increased surface area and pore volume, and reduced pore diameter.

3.6 Characterization of RHA : SEM-EDX Silica

SEM-EDX analysis using Scanning Electron Microscopy (SEM) was used by the Phenom Pro X brand, displaying morphological images as shown in Figure 5 and Figure 6.

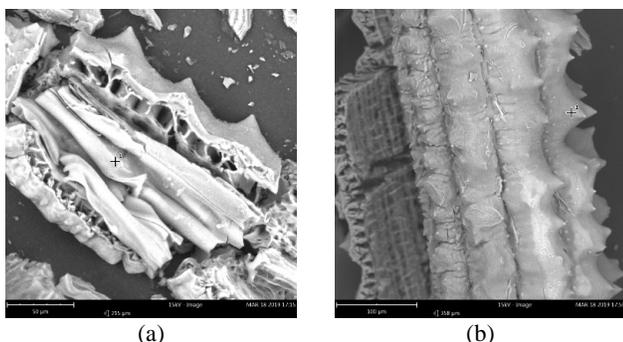


Figure 5: (a) The Morphology and Size of Silica at R1 and (b) R2 on the Field of View 215 µm, Mode: 15kV - Image, Detector: BSD Full

Table 6: The Composition of Elements and R1 Oxide through EDX

Element Symbol	Atomic Conc.	Weight Conc.	Oxide Symbol	Stoich. wt Conc.
Si	35.01	51.68	Si	73.06
O	34.80	29.26		
C	30.19	19.06	C	26.94

SEM results from the sample were taken with a size of 1 micron in 1 micron which equals 1000 nano and from the results of specimens that used 28,000 cycles speed results in nano size i.e. 215 µm. So the percentage of elements Si 35.01% and O as much as 24.80% which formed SiO₂ oxide compounds as much as 73.06%.

Table 7: The Composition of Elements and Oxide R2 through EDX

Element Symbol	Atomic Conc.	Weight Conc.	Oxide Symbol	Stoich. wt Conc.
O	75.22	67.74		
Si	16.79	26.55	Si	82.28
C	5.20	3.51	C	10.89
N	2.79	2.20	N	6.83

From the SEM images, the samples were taken with a size of 1 micron where 1 micron equals to 1000 nano, and from the results of specimens using 140,000 cycles speed obtained nano size result i.e. 358 µm. So, the percentage of Si elements 16.79% and O as much as 75.22% which formed SiO₂ oxide compounds as much as 82.28%

5. CONCLUSION

Based on the analysis of the husk ash sample by calcination route using sulfuric acid leaching with controlled combustion at 650°C for 2 hours, the SiO₂ content at R1 reached 97.27% and R2 obtained 97.03% with a number of distributed impurities. The characterization of rice husk ash from both samples produced amorphous silica SiO₂ and the average particle size was in the range of R1 = 0.217 and R2 = 0.268 µm. The surface area of silica grains produced after washing rice husk with sulfuric acid R1 = 265.73 m² / g R2 =, 294.37 m²/g. The average pore diameter of R1 is ~ 2.04 nm and R2 is 1.89 nm, indicating that the silica produced by R1 is mesoporous and R2 is micropore. Amorphous silica with high purity has a large granular surface area, useful as an absorber or supporting catalyst in fine chemical synthesis, especially oxide character booster in its preparation as a seal on the SOFC system.

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