



# The Ability of Crystalline and Amorphous Silica from Rice Husk Ash to Perform Quality Hardness for Ceramic Water Filtration Membrane

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**Abstract:** Silica is an inexhaustible resource on earth. It can be found in food-based natural resources. The natural tendency of silica mainly is to contribute to the strength and the hardness of the ceramic material. This paper investigates the hardness formed in ceramic filtration membrane made by amorphous and crystalline silica from rice husk. Four samples from this material were studied based on unwashed and washed material with chemical, namely, crystalline silica treated with chemical (CS1), crystalline silica untreated with chemical (CS2), amorphous silica treated with chemical (AS1) and amorphous silica untreated with chemical (AS2). To acquire the silica phases, the rice husk was experienced to combustion process at 700°C and 1000°C for 2 hours. The properties of the silica were obtained by using Scanning electron microscopy (SEM) and x-ray diffractometer (XRD) while the value of the hardness was tested by using microhardness tester machine. As a result, from SEM both samples of AS1 and AS2 produced an irregular, crinkled, or grainy surface of silica and for sample CS1 and CS2 produced were flushed, unwrinkled and planar surface. For the high hardness value, crystalline silica treated with chemical (CS1) sample was obtained with 171.0.

**Keywords:** Rice husk, Amorphous Silica, Crystalline Silica, XRD, SEM, Hardness

## 1. Introduction

Rice husk can be found on a seed or grain of rice. It is formed from hard materials, including silica and lignin which to protect the seed during the growing process. Compared with other based silica resources like bentonite, sand and diatomaceous earth rice husk has insignificant amounts of impurity that affect performance in applications that requiring high purity [1]. Throughout years of natural evolution of the plant, rice husk had an exclusively developed the nanoporous silica layers. This is why producing highly reactive silica from rice husk is a simple process with various advantages, compared with conventional production methods [2].

The existence of the crystalline and amorphous silica in rice husk is involved with firing at certain temperature. According to Shen et al., [3], amorphous and crystalline silica of rice husk can be obtained at 550°C to 800°C and 900°C to 1300°C. This validation can be supported by [4], which the amorphous silica can be detected when fired the rice husk at 700°C. Aside from that, by chemical treatment also could be help to remove the metallic impurities such as iron (Fe), manganese (Mn), calcium (Ca), sodium (Na), potassium (K) and magnesium (Mg) by treated with hydrochloric acid (HCl). The chemical treatment and firing could lead to change the microstructure of silica from

amorphous to crystalline state [5]. The benefits of the silica in ceramic product will lead to the high abilities of the silica itself in absorption of substances and assemble the good strength and hardness in ceramic product by which fired the rice husk at high temperature generates a high strength [6].

## 2. Material and Method

Initially, rice husk was washed using tap water to remove unwanted substances. Then to make sure the rice husk was utterly dried, it has been placed under the sun for 48. Afterwards, for sample CS1 and AS 1, there undergone for chemical treatment. Firstly, the cleaned rice husk was treated separately with hot hydrochloric acid at 60 °C at concentration of 0.5M for 30 minutes with constant stirring. After the acidic solution was drained off, the rice husk was rinsed with distilled water until it free from acids, filtered and dried in air-oven at 110 °C for 24 hours. Then, the rice husk was incinerated in a furnace at 700 °C (AS1 and CS1) and 1000 °C (AS2 and CS2) for 2 hours. The heating rate used was 5 °C/min.

The produced rice husk ash under non-chemical treatment was called CS 2 and AS2. The process started to take place by heating in a furnace with the same temperature, holding time and heating rate. Later, the sieving process took place by passed through 63 µm mesh sieve. Subsequently, the rice husk ash undergone SEM and XRD to find the phases of silica oxide when firing at 700 °C and 1000 °C [4]. For the ceramic filtration membrane, the process took place by mixing the amorphous and crystalline silica with the binder and shaped it by using compaction process by using pressing machine. To find the hardness value, the sample then undergone hardness testing by using microhardness tester machine.

## 3. Characterization

### 3.1 Scanning electron microscopy (SEM)

The structure of rice husk was identified by using scanning electron microscopy (SEM). The samples were coated with gold label to obtain the perceive images and to avoid charging effect. The configuration of the samples were obtained using electron dispersive spectroscopy (EDS) attached to the SEM machine. The process was conducted using FESEM (model JSM 6701F JOEL).

### 3.2 X-ray diffractometer (XRD)

The samples have been studied for their mineralogical characteristics and by using x-ray diffraction (XRD) system. Samples were scanned from 2 hours from 2θ ranging from 5° to 90°. The EVA™ Software was used to record and analyze the structural pattern of the samples.

### 3.3 Hardness analysis

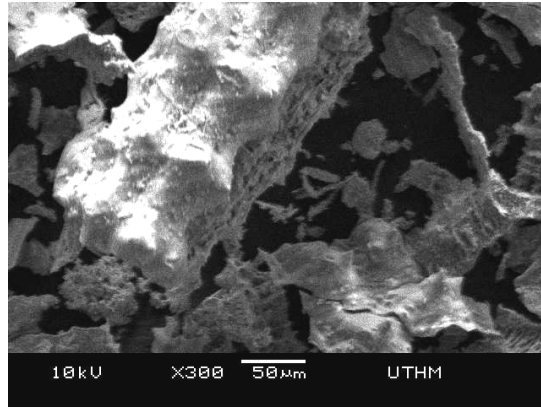
The hardness analysis was performed by using conventional Microhardness Tester HMV SHIMADZU-2 machine. The experiment was conducted about five times to get the average value.

## 4. Result and Discussion

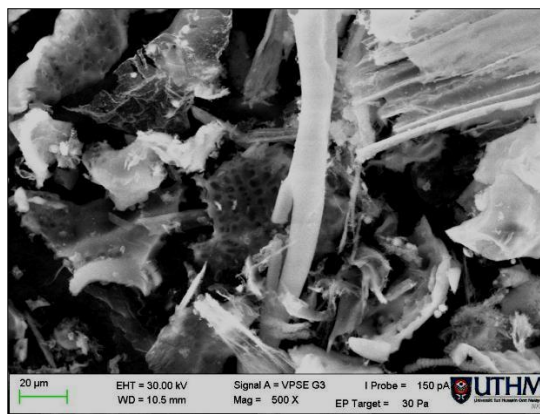
### 4.1 Analysis of Scanning electron microscopy (SEM)

Figure 1 and 2 showed the micrographs of an AS 1 and AS 2 sample. It was observed that particle in the existence of amorphous silica oxide. Nevertheless, the addition of the hydrochloric acid in sample AS1 still formed uneven surface which result in high surface area. Firing rice husk at 700 °C was not influence yet to the level of purely crystalline silica oxide. In this analysis, the particle distribution indicated a large scale from 0.030 to 50 µm. Average particle size was found to be at 45 µm.

According to Q. Feng et al., the firing temperature definitely has to be more than 700 °C to obtain of silica in crystalline state [7]. It proved by formed of an agglomerates and fragment structures. This study is compatible with what has been reported by [8], similar shape for silica based on rice husk ash prepared by fired at temperature below 900 °C. Other than being light, massive, and profoundly permeable, creation of rice husk ash incorporates silica (SiO<sub>2</sub>), carbon (C), potassium oxide (K<sub>2</sub>O), phosphoric oxide (P<sub>2</sub>O<sub>5</sub>), calcium oxide (CaO), and littler measures of magnesium (Mg), iron (Fe), and sodium (Na). Along with impurities including Al<sub>2</sub>O<sub>3</sub>, K<sub>2</sub>O, MgO and CaO are decreased due to the composition of burned rice husk depends upon many factors including the temperature itself, an agricultural practice and geographical location [9].

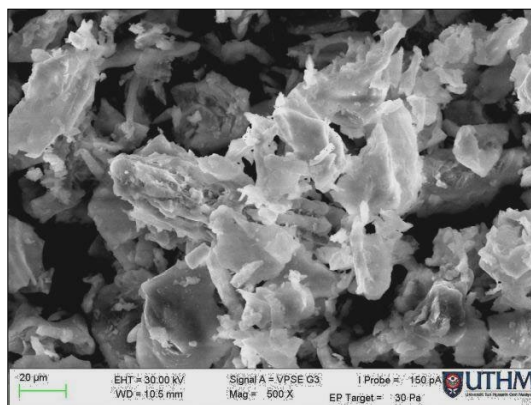


**Fig. 1 - The micrographs of AS1 under the particle distribution.**



**Fig. 2 - The micrographs of AS2 under the particle distribution.**

Figure 3 and 4 showed the micrographs of an CS 1 and CS 2 sample. These two samples were observed to be purely crystalline silica oxide. It proved by formed of an unwrinkled and smooth surface. The particle distribution indicated a large scale from 0.030 to 100  $\mu\text{m}$ . Average particle size was found to be at 80  $\mu\text{m}$ . These micrographs consisted of particles in nano shape with average of less than 200  $\mu\text{m}$  [9]. This is clarified by understanding that rice husk which contains high silica, can respond with high temperature and treatment agent. Muhammad (2011), relate that the disappearance of impurities elements makes progressively surface area and new pore structure. Y. Guo et al (2002) detailed that the surface area depends on the ash content. Along these lines, the lesser the ash content, the more surface area exists.



**Fig. 3 - The micrographs of CS1 under the particle distribution**

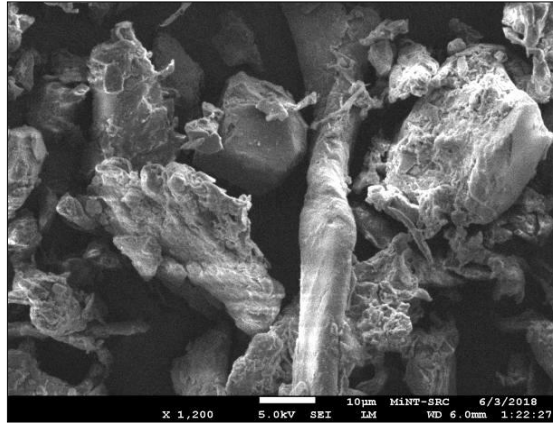


Fig. 4 - The micrographs of CS2 under the particle distribution.

#### 4.2 Analysis of X-ray diffractometer (XRD)

Figure 5 and 6 presented the diffractograms of the samples analysed. Previous studies had conducted an experiment using different time and temperature conditions to induce the existence of amorphous and crystalline silica [13]. In Figure AS 1 showed a single diffuse broad peak at about  $2\theta = 20.79^\circ$  while the AS2 showed the single diffuse broad peak at about  $2\theta = 22^\circ$ . Both of the figures showed the structure of amorphous silica phase.

The EVA™ Software showed that the silica contained in AS1 was 89.85% while AS2 was about 81.90%. These two results showed that the chemical treatment definitely helps to extract the hemicellulose fibre. This formed composition was depends on temperature in furnace. Therefore, the compositions range has been reported [14,15]. The controlled temperature was transforming the husk into amorphous biomass silica ash. The furnace is outfitted with a controller for air (oxygen) to enter and assist a complete burning process in order to produce low carbon content of white amorphous biomass silica ash. Perforated trays in furnace rotated to enhance the mixing process, heat distribution and optimizing the burning efficiency [16].

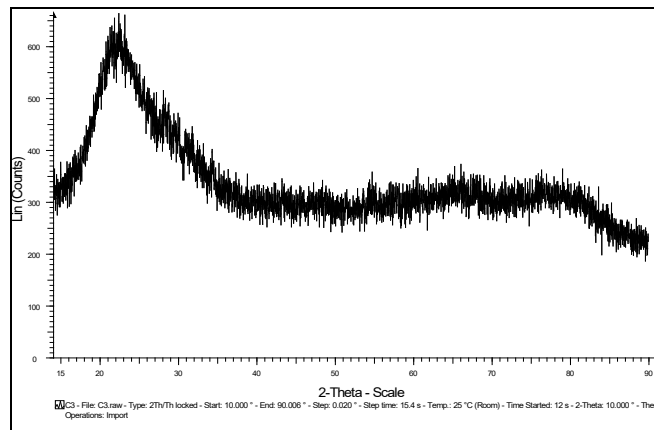


Fig. 5 – XRD patterns of sample AS1 at 700°C.

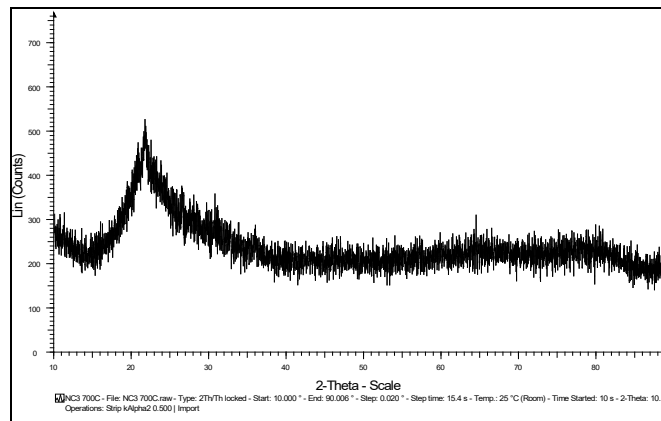
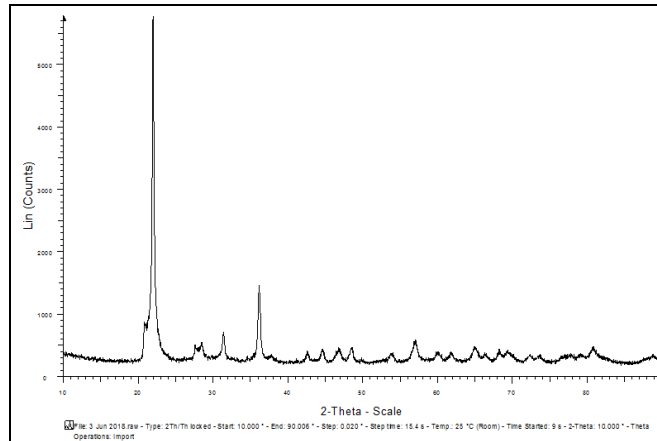


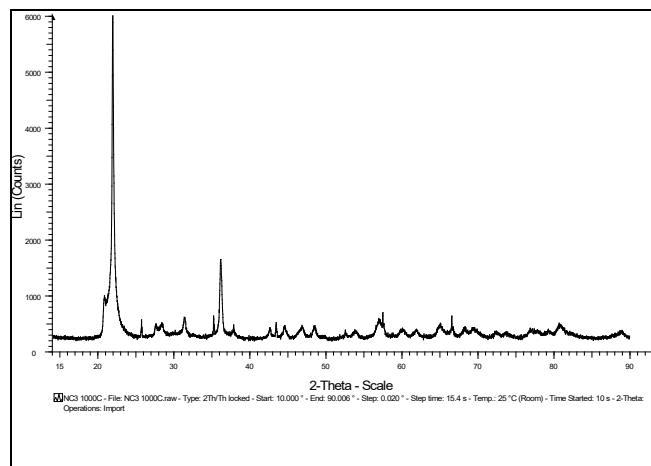
Fig. 6 - XRD patterns of sample AS2 at 700°C.

Figure 7 and 8 showed the pattern of the highly the crystalline structure of silica oxide was formed. This indicated by the appearance of a few diffuse broad peaks at Figure 7 at about  $2\theta = 22.5^\circ$ . Other than that, several diffuse peaks at Figure 8 also formed with the highest peak resulted at about  $2\theta = 23^\circ$ . The EVA™ Software showed that the silica contained in CS 1 was 98.70% while CS2 was about 95.70%. These result proved that by using chemical treatment helps to lift up the silica contained.

In a study by [17], to incinerate the rice husk, the tested has been run in various temperatures and the material previously leached ones. However, the authors underlined and highlighted that the sharpeners of the peak increases with combustion temperature, starts around  $900^\circ\text{C}$  and the removal alkaline species during acid leaching is an obstacle to the eutectic reaction of silica.



**Fig. 7 - XRD patterns of sample CS1 at 1000°C.**



**Fig. 8 - XRD patterns of sample CS2 at 1000°C.**

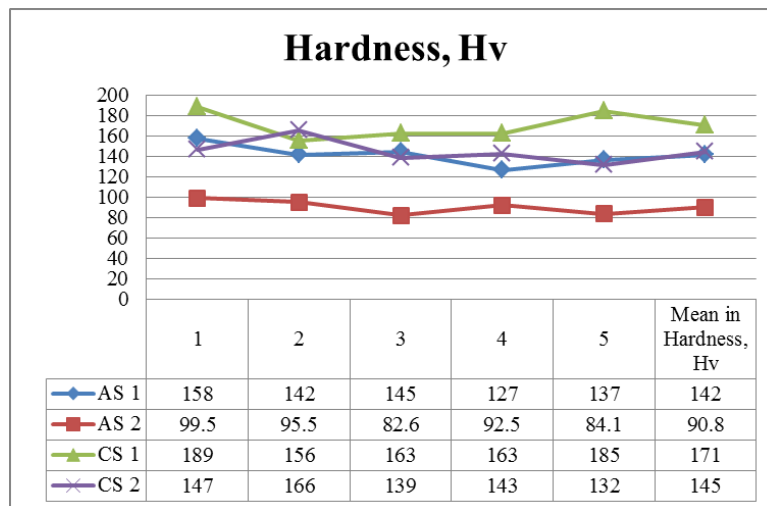
### 4.3 Analysis of hardness of ceramic filtration membrane

Table 1 showed the result of hardness test. It has been run for five times to get an average result. Sample CS 2 showed the higher resistance of hardness compared to other. The value of the hardness was about  $171.0 \mu\text{m}$ . The higher the value of the result, the harder the sample. Figure 7 manifest the result from Table 1 in the form of graph.

This can be supported by the consolidation of aggregated systems has been largely considered in the framework of solid-liquid separations, which involved filtration, compression, drying or centrifugation. The efficiency of solid-liquid processes depends on the way the sample built up, how the network, strong bonds of material and binder respond to applied pressure. At low to moderate applied pressures, the organization of the sample changes very little while for the highly compressible sample leads to the weak bond connects to the particles [18]. The decreasing of the hardness of ceramic membrane was due to an excessive of high specific surface area and high-water absorption capacity of the material. This phenomenon was also happened in cement in view of the unreasonable volume of voids created in the sample [19]. Thus, by using rice husk ash made the material especially in ceramic and concrete with relatively high strength and good resistance [20].

**Table 1 - Result of hardness value.**

Sample	1	2	3	4	5	Hv
AS 1 (700°C)	158.0	142.0	145.0	127.0	137.0	142.0
AS 2 (700°C)	99.5	95.5	82.6	92.5	84.1	90.8
CS 1 (1000°C)	189.0	156.0	163.0	163.0	185.0	171.0
CS 2 (1000°C)	147.0	166.0	139.0	143.0	132.0	145.0



**Fig. 7 - Graph of the hardness value.**

## 5. Conclusion

Rice husk ash can produce different phase of silica. By fired the rice husk at 700°C, an amorphous silica phase was formed while fired the rice husk at 1000°C was leads to the form of crystalline silica. Chemical treatment increased the percentage of silica, prior to combustion. Bonds between the material and binder definitely effect the harden of the sample. The higher the result, the higher the hardness of the sample. The objective was to investigate the hardness formed in ceramic filtration membrane made by amorphous and crystalline silica from rice husk was achieved by which CS2 sample showed the high quality of hardness, the result was about 171.0 μm.

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