

Study of Characteristics of Rice Husk and Silica Obtained from Rice Husk

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Abstract—This paper presents the study of the features of the rice husk and to investigate the presence of silica in the rice husk. To obtain the pure silica, the rice husk was firstly washed and went through chemical treatment, prior to combustion. The combustion process was conducted at temperature of 700°C for 2 hours. The rice husk ash obtained was studied and has been named as C3. The characteristics of C3 samples can be acquired by using scanning electron microscopy (SEM), x-ray diffractometer (XRD) and thermogravimetric analysis (TGA). The SEM images showed the material was in agglomeration. The silica content of C3 was observed to be 89.85% using EDS. The thermal properties of the C3 also was performed using TGA as the change with temperature up to 1000°C with heating rate of 5°C/min. As from the result, the energy of organic compound lose at 700°C was 0.0141mg/min and at 1000°C was 0.00217 mg/min.

Index Terms—Rice husk, silica, combustion, structural studies.

I. INTRODUCTION

For decades, the exponential population and social civilization expansions, different in cycle of life and resources that have been used with extended advances of the industrial and technologies have generated to a rapid growth of modernization and metropolitan [1]. One of the environmental sustainability targets is to recycle the agricultural waste products [2]. The potential of rice husk silica to be developed as the membrane filter is quite promising since the statistics shows that the worldwide annual rice husk output is about 80 million tones. From that amount, over 97% of the husk was generated in the developing countries, including Malaysia. According to the statistics compiled by the Malaysian Ministry of Agriculture, 408,000 metric tonnes of rice husk are produced in Malaysia each year [3].

Rice husks are a sheltered material because they are difficult to burn and less likely to allow moisture to propagate mould or fungi. It is also contains abundant floristic fibre, protein and some functional groups such as carboxyl and amidogen [4]. Application for various industries shall conduct research on the usage of rice husks in major rice

producing countries. Research on fully utilize the agriculture waste means to avoid burning the rice husks as wastes are worth like. Rice husk is a very cost-effective raw material to be used and it is very easy to obtain. A few current implementations on the usages of the rice husks are building material, pillow staffing and fertilizer [5]. The utilizing of the rice husk will provide twofold advantage to the environmental management. First, the large volume of rice husk waste could be partly reduced, converted to useful, value added adsorbent. Second, the development of the low-cost adsorbent may overcome the waste waters and air pollution at a reasonable cost [6]. Reported physicochemical characteristic of rice husk is shown in the Table I [7].

TABLE I : THE PHYSICO-CHEMICAL CHARACTERISTIC OF RICE HUSK

Characteristics	Unit	Value
Bulk density	g/ml	0.73
Solid density	g/ml	1.5
Moisture content	%	6.62
Ash content	%	45.97
Particle size	mesh	200-16
Surface area	m ² /g	272.5
Surface acidity	meq/gm	0.1
Surface basicity	meq/gm	0.45

To obtain high silica content, the combustion process according to the specification of amorphous content should be performed. The combustion process was conducted at 700°C for 2 hours. After combustion, the produced rice husk ash may contain 80% -95% of silica mineral, while others are metallic impurities. The metallic impurities such as iron (Fe), manganese (Mn), calcium (Ca), sodium (Na), potassium (K) and magnesium (Mg) could be eliminated through chemical treatment with hydrochloric acid (HCl). The chemical treatment and combustion process were believed to lead the change of the microstructure silica from amorphous to crystalline state [8].

The production of silica from rice husk was easy and requires low cost. The silica content found in rice husk is widely used in electronics, ceramics, plastic, rubber and photoelectric industries. Other than that, silica in rice husk also can act as a heat exchanger agent in composite additives [9].

II. MATERIAL AND METHOD

Firstly, rice husk was washed using tap water to remove dirt and unwanted substances. It was then dried for 48 hours

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under the sun to ensure that the rice husk was completely dried. After that, 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 for 2 hours with heating rate of 5°C/min [10].

The produced rice husk ash was then called as sample C3. Sieved process took place by passed through 63 μm mesh sieve. Lastly, the C3 samples were characterized by using SEM, XRD and TGA to find the phases of silica oxide when firing at 700°C.

III. CHARACTERIZATION

A. Scanning Electron Microscopy (SEM)

The morphology of rice husk was identified by using scanning electron microscopy (SEM). The samples were gold-coated prior to morphological studies to obtain clear images and to avoid charging effect. The compositions 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).

B. X-Ray Diffractometer (XRD)

C3 samples have been studied for their mineralogical characteristics 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.

C. Thermogravimetric Analysis (TGA)

The thermogravimetric analysis was performed on a Linseis Thermobalance simultaneous thermal analysis (STA). Sample of 15.1 mg was heated at heating rates of 5°C/min from 20°C to 1000°C.

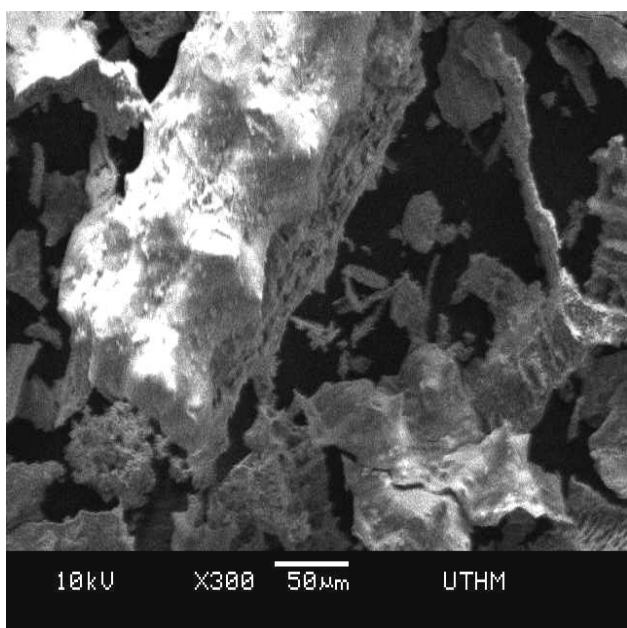


Fig. 1. (a) The micrographs of C3 under the particle distribution indicated a large scale from 0.030 to 50 μm .

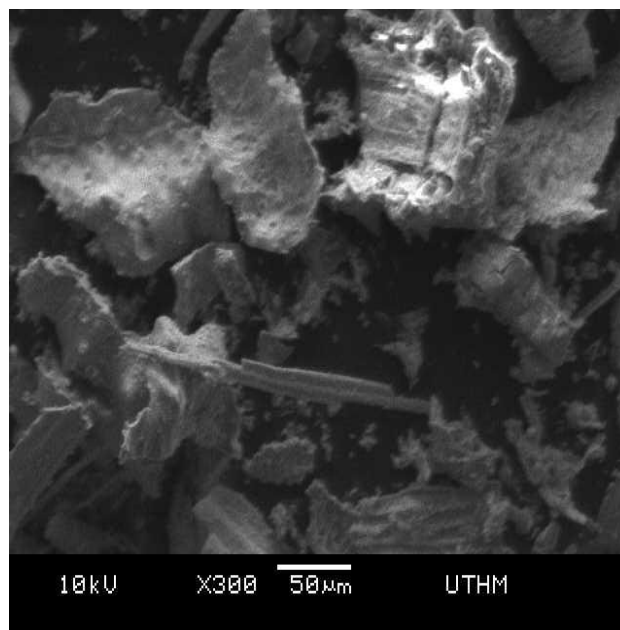


Fig. 1 (b) The micrographs of C3 under the particle distribution indicated a large scale from 0.030 to 50 μm .

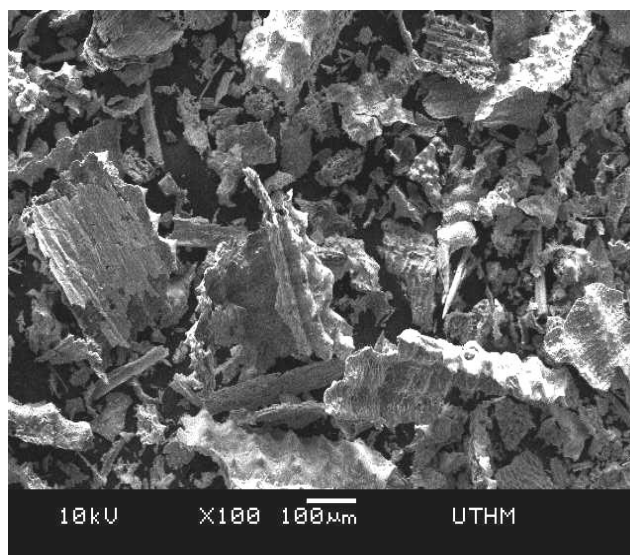


Fig. 1. (c) The micrographs of C3 under the particle distribution indicated a large scale from 0.030 to 100 μm .

IV. RESULT AND DISCUSSION

A. Scanning Electron Microscopy (SEM) Analysis of Rice Husk

Fig. 1 show the micrographs of a C3 sample. It was observed that the particle was in the existence of amorphous silica oxide and firing rice husk at 700°C was not influence yet to the level of purely crystalline silica oxide. According to Q. Feng et al., the firing temperature definitely has to be more than 700°C to obtain of silica in crystalline state [11].

According to the SEM micrograph of sample C3, Fig. 1 (a) and (b) show the particle distribution indicated a large scale from 0.030 to 50 μm . Average particle size was found to be at 45 μm . Fig. 1 (c) shows the particle distribution of large scale from 0.030 to 100 μm . The average particle size was 80 μm . All figures show a spherical morphology with an irregular geometry in a form of agglomerates in micron scale.

Furthermore, the C3 sample was collated with the original rice husk sample. The original rice husk sample was not fired

and not been soaked out with chemical. The sample was an originally used to see any physically differentiation with C3 sample. From the Fig. 2 (a), (b) and (c), the micrographs showed that at large scale from 0.030 μm to 10 μm and 0.030 μm to 100 μm . It was observed that the sample was highly at amorphous silica state and the structure was rough and rutted surface. According to N. A. A. Ismail et al., the crystalline structure was not formed around 700 $^{\circ}\text{C}$ to 900 $^{\circ}\text{C}$. The formation was existed after firing the rice husk at 1000 $^{\circ}\text{C}$ and above until it reach the phase of breaking point to quartz phase [12].

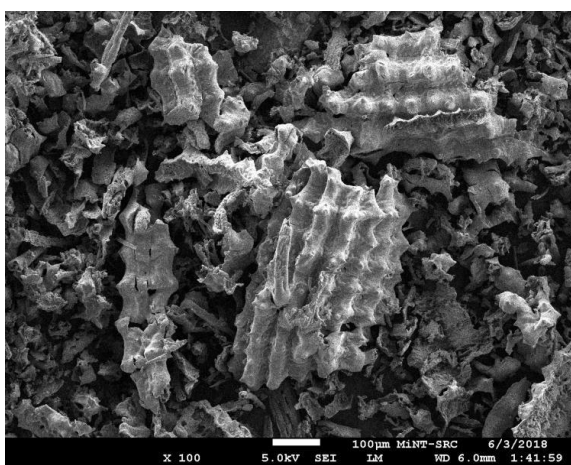


Fig. 2. (a) The micrographs of original rice husk under the particle distribution indicated a large scale from 0.030 to 100 μm .

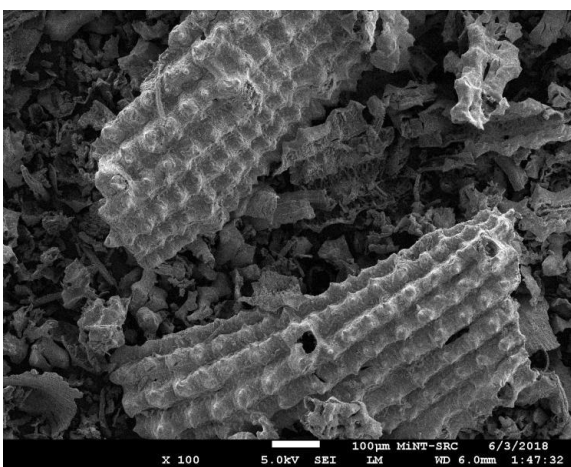


Fig. 2 (b) The micrographs of original rice husk under the particle distribution indicated a large scale from 0.030 to 100 μm .

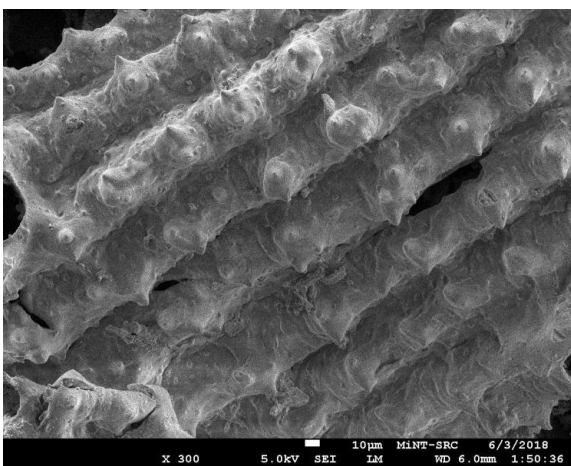


Fig. 2. (c) The micrographs of original rice husk under the particle distribution indicated a large scale from 0.030 to 10 μm .

In Fig. 3, the composition of the sample was obtained using (EDS) that has been attached with SEM. It shows that the silica was higher compare to other components such as zinc (Zn), potassium (K), carbon (C) and Oxygen (O).

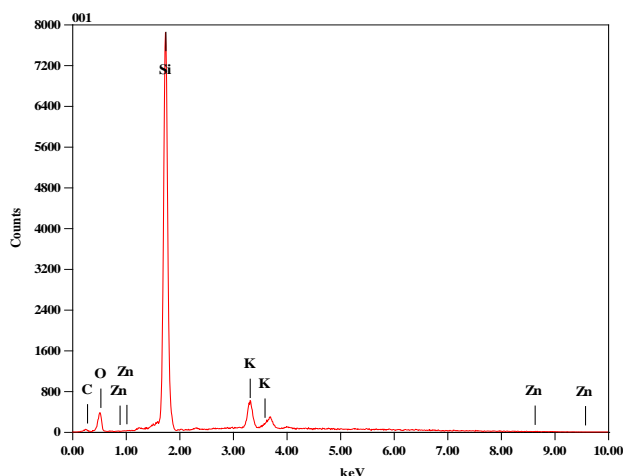


Fig. 3. EDS Graph of sample C3.

The results in Table I were also obtained using (EDS). It shows that the percentage of silica oxide contents was 89.85% in sample C3. It indicates that silica values increased with temperature. According to the previous researchers, the increase in combustion temperature and soaking time of acid treatment may increase the purity of silica [13]. Therefore, sample C3 needs to be fired more than 700 $^{\circ}\text{C}$ or increase the soaking time of acid up to 24 hours to achieve higher silica content.

Table III showed the result from the previous researcher in 2006 [14]. The silica oxide contains was 81.09%. The total Al_2O_3 , CaO , and K_2O were observed to be 0.19%, 0.32%, and 1.64% respectively.

Moreover, in Table IV showed the result from the previous researcher in 2010 [15]. The silica oxide contained about 88.32%. The total for the other components were stated of 0.46% (Al_2O_3), 0.67% (CaO), 0.67% (Fe_2O_3), 2.91% (K_2O), 0.44% (MgO) and 0.12% (Na_2O).

TABLE II: THE COMPOUND CONTENTS IN SAMPLE C3

Element	(keV)	Mass (%)	Compound	Mass (%)
C	0.277	0.61	C	0.61
O		49.47		
Si	1.739	42.00	SiO_2	89.85
K	3.312	7.91	K_2O	9.53
Zn	1.012	0.01	ZnO	0.01
Total		100.00		100.00

TABLE III: THE COMPOUND CONTENTS FROM (PRASETYOKO, 2006)

Compound	Mass (%)
Al_2O_3	0.19
CaO	0.32
Fe_2O_3	0
K_2O	1.64
MgO	0
Na_2O	0
SiO_2	81.09
etc	16.76

TABLE IV: THE COMPOUND CONTENTS FROM (HABEEB & MAHMUD, 2010)

Compound	Mass (%)
Al ₂ O ₃	0.46
CaO	0.67
Fe ₂ O ₃	0.67
K ₂ O	2.91
MgO	0.44
Na ₂ O	0.12
SiO ₂	88.32
etc	6.4

However, the result of potassium as K₂O was investigated by both previous researchers and showed the difference in the range 0% to 2.91%. This is due to the difference in climate, regions and geographical areas in which the rice was planted [16].

B. X-Ray Diffractometer (XRD) Analysis

Fig. 4 (a) shows the pattern performed by XRD for sample C3. At temperature of 700°C, the crystalline structure of silica oxide was not formed. This is indicated by the appearance of single diffuse broad peak at about $2\theta = 20.79^\circ$. On the other hand, this figure proved that sample C3 was completely amorphous structures. This is happened when hinders eutectic reaction with silica by removed the alkali metals. Thus, the optimization of combustion temperature of rice husk is necessary to hinder the crystallization of silica [17].

Fig. 4 (b) shows the pattern performed by XRD for original rice husk sample. Since this sample was not burned at any temperature and was not mixed with any chemical, result showed that the sample was amorphous state. This indicated by the appearance of single diffuse broad peak at about $2\theta = 24.00$, with no other impurities detected.

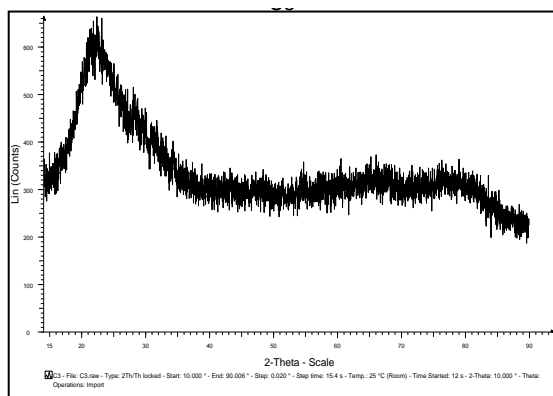


Fig. 4.(a) XRD patterns of sample C3 at 700°C.

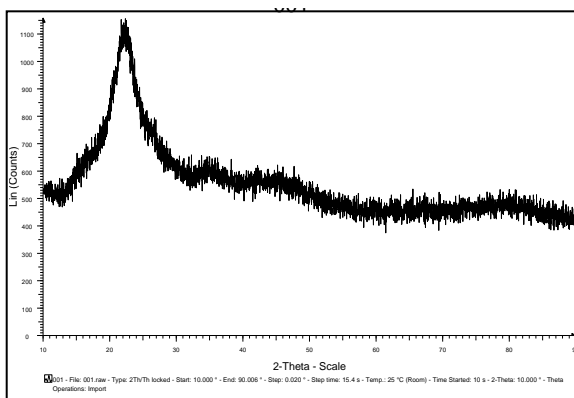


Fig. 4(b) XRD patterns of an original rice husk sample.

TABLE V: WEIGHT LOSS IN SAMPLE C3

Temperature (°C)	Weight loss (mg/°C)
50	-0.25
100	-0.95
150	-1.29
200	-1.58
250	-1.87
300	-2.17
350	-2.50
400	-2.87
450	-3.27
500	-3.75
550	-4.26
600	-4.82
650	-5.45
700	-6.10
750	-6.85
800	-7.64
850	-8.45
900	-9.32
950	-10.23
1000	-11.27

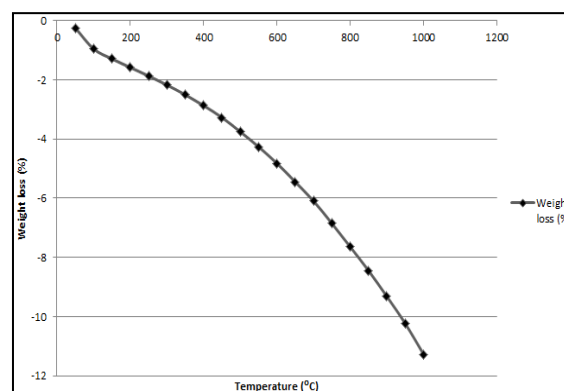


Fig. 5. Thermogravimetry analysis (TGA) curves of sample C3.

A. Thermogravimetric Analysis (TGA)

TGA was used to determine the changes in the mass of a material that occur in response to the programmed temperature changes. The changes in the mass can be caused by a variety processes such as decomposition, degradation, sublimation, vaporization, adsorption, desorption, oxidation, and reduction [18].

It can be seen from Table 5 that in the first stage, the initial weight loss occurs at the range of 50°C to 150°C with weight loss of -0.25 mg/°C to -1.29 mg/°C effects from water loss and other strained substances. The second stage showed a huge weight loss of about -10.23 mg/°C to -11.27 mg/°C which happened at 950 °C to 1000 °C. Both results showed an amorphous state.

This process happened because of thermal decomposition of hemicellulose and cellulose; a major organic component in the rice husk. Previous study said that mainly at 150 °C to 360 °C which is the least stable component of rice husk and cellulose decomposed between 275 °C to 350 °C [12].

Fig. 5 shows the graph of weight loss in terms of percentage against the temperature starting from 50°C to 1000°C. From the observation, higher temperature leads to high amount of percentage of weight loss in sample C3.

V. CONCLUSION

Silica was obtained from the rice husk sample through acid treatment and combustion at 700 °C for 2 hours. It was shown

that by burned the rice husk at the temperature of 700 °C, the resulted silica was in an amorphous state. The average particle size of the rice husk silica was in micron. With high specific surface area, agglomeration was easily formed. Through this study, it was found that high amount of silica in crystalline state could be produced by increasing the combustion temperature and soaking time of acid during treatment, prior to combustion.

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