The Investigation of SiO$_2$ structure obtained from the combustion of rice husk

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Abstract. In this work, SiO$_2$ was obtained from the combustion of rice husk. The influence of temperature on its structure and morphology was investigated. At low combustion temperatures, the rice husk ash appeared to be black due to an insufficient temperature for the oxidation process. When the temperature was raised to 1000°C, the color changed to a pale pink. The elemental composition of the rice husk ash was determined by means of x-ray photoelectron spectroscopy. The survey analysis of the XPS spectra revealed the existence of Si, O, C, Ca P, K and N. The element fraction of Si was increased as a function of temperature. However, the N 1s XPS signal can be collected from the rice husk after the combustion at 300°C only. Si L edge XANES spectra were recorded to determine the structure of the SiO$_2$. The results suggested the similarity between the SiO$_2$ structure of the rice husk ash and the standard SiO$_2$. The microstructure of the rice husk ash was studied by SEM. It was clearly seen that the pores were created on the rice husk ash. By increasing the pyrolysis temperature, the cellulosic material was removed and consequently produced channels.

1. Introduction

Rice husk (RH) is a byproduct of the rice milling process. The outer covering of the rice grain is an agricultural waste material in all rice growing countries [1]. RH can be used as a fuel for power plants [2], a raw material in the production of activated carbon [3] and as the feed stock of industrial chemicals based on silica and silicon compounds [4]. RH is composed of hemicellulose, cellulose, and lignin totaling about 75 – 90% [5] with small amounts of Si and other trace elements such as Fe, Mn, Ca, Na, K, and P [6]. Rice husk ash (RHA) can be obtained from the combustion of RH under controlled conditions [7]. On burning, cellulose and lignin are removed, leaving behind silica ash. The controlled temperature and environment of burning yields better quality of rice husk ash in terms of its particle size. RHA consists mainly of silica (SiO$_2$) (>90%) [8]. The content of impurities in RHA is influenced...
by geographic location, fertilizers, and agrochemicals. The structure of silica in RHA depends on the temperature and duration of combustion [9]. The amorphous silica can be produced by burning RH at temperatures lower than 700°C under oxidizing conditions [10]. Silica is a raw material that can be used in many applications such as ceramics, agriculture, and medicine [11-13]. Many authors reported the fabrication of silica from RH to increase the value of agricultural waste and to reduce the disposal and pollution problems [14-19].

In this work, silica was fabricated by combustion under an oxygen atmosphere at various temperatures. The characteristics of the silica were investigated by scanning electron spectroscopy (SEM), x-ray photoelectron spectroscopy (XPS) and x-ray absorption near edge structure (XANES).

2. Experimental
Rice husk ash was prepared by the combustion of rice husk (Surin, Thailand) under an O$_2$ (99.99%) at a flow rate of 50 ml/min and ambient pressure for 1 hour. The combustion temperatures were varied from 300-1000°C at a heating rate of 5°C/min with the interval of 100°C and denoted as RHO300, RHO400, RHO500, RHO600, RHO700, RHO800, RHO900 and RHO1000, respectively. The morphology of the RHA was studied by SEM QUANTA 450, FEI. XPS and XANES were performed to investigate the electronic properties and structural information. XPS (PHI VersaProbe II) was carried out at the SUT-NANOTEC-SLRI Joint Facility, BL5.3: SUT-NANOTEC-SLRI, Synchrotron Light Research Institute. The excitation energy was 1486 eV using an Al Kα source. Si L$_3$-edge XANES was performed at the BL3.2a: PES beamline of the Synchrotron Light Research Institute, Thailand.

3. Results and discussion
The burning of RH under an O$_2$ atmosphere at different temperatures caused the different appearance in the colors of the final ashes (Fig. 1). At 300°C, the color of the RHA was black, suggesting an insufficient temperature for the combustion process. Its elemental compositions were dominated by carbon. The firing of RH at 400-600°C caused changes in the RHA colour from black to white grey. This means that the residual carbon content of RHA obtained in this temperature range was low. The pink color appeared when the temperature was raised to 700°C. At 1000°C, the color of the RHA was pinkish.

![Figure 1](https://example.com/figure1.png)

**Figure 1.** Optical images of rice husk ash obtained from the combustion of rice husk under an O$_2$ atmosphere at 300°C, 400°C, 500°C, 600°C, 700°C, 800°C, 900°C and 1000°C.

The microstructures of the RHO300, RHO500 and RHO1000 were characterized from a scanning electron micrograph (Fig. 2). The morphological structures of all RHAs have a predominantly rough and undulating surface texture. The major parts of all RHAs retained their original shape. The dense membrane structures were observed on both the inside and outside surfaces without the presence of micropores. The cross-section of RHA revealed an interlayer which consisted of a crisscross mesh of
chips between the two surfaces. This inside structure was arranged in a loose honeycombed configuration with a high number of holes. The size of holes on the RHA interlayers were varied ranging from several nanometers to several microns. The presence of these pores correlated to its high surface area when it is ground to powder.

![SEM images of RHA at different temperatures](image)

**Figure 2.** The illustration of SEM images of the selected RHA obtained after the combustion of RH under an O$_2$ atmosphere at 300°C, 500°C, 1000°C, respectively, and the correspondence EDS spectrum.

The elemental distribution of RHA was further studied by EDS analysis. It could be seen that the internal reticular porous structure was a carbon-rich layer while silicon was packed on the outer surface. The EDS analysis also explored the existing trace elements in all RHAs. The trace elements consisted of K, P, and Ca. At low combustion temperatures (RHO300), dispersal among these elements was observed. Their agglomeration occurred when the temperature was increased, whereby the RHO1000
exhibited the greatest agglomeration. Calcium is an essential element required for the structure role in the membrane and cell wall of plants [20]. For RHO300, the remained carbon due to uncompleted combustion of rice husk maintained the high dispersion ability of calcium. However, the lower carbon content was observed on RHO500 and RHO1000. Thus, the remained calcium was favored to agglomerate in RHA at high temperature.

X-ray photoelectron spectroscopy was conducted to examine the chemical composition of carbon compounds in the RHAs (Fig. 3). The survey scan explored the contribution of Si, O, C, K, P and Ca atoms in all samples. For RHO300, the elemental relative ratio of Si:O:C:K:P:Ca was 12.30:44.30:38.15:1.10:0.55:0.62. When the combustion temperature was increased to 500°C, the relative concentration of Si atoms was increased which contrasted with the reduction of carbon. This result suggested the transformation of solid phase carbon to the gas phase carbon dioxide. However, although the temperature was increased to 1000°C, the relative concentration of carbon atoms was not changed significantly. The existing carbon in the RHA confirmed the appearance of a white grey color. As shown in Fig. 3A, the Si 2p XPS spectra of RHA after calcination at all temperatures revealed a similar peak position at 103.0 eV due to the electron emission from SiO\(_2\). The result agreed well with the O 1s XPS spectra that exhibited the photoelectron peak due to the O–Si bond at around 532.5 eV. Among the trace elements, potassium exhibited the highest atomic concentration. The K 2p XPS peaks appeared at 293.2 eV and 296 eV and corresponded to the K\(^+\) in the K\(2O\) compound.

![Figure 3](image.png)

**Figure 3.** The Si 2p (A), K 2p (B) and O 1s XPS spectra rice husk ash after combustion at various temperatures under an O\(_2\) atmosphere.

X-ray absorption spectroscopy was employed to investigate the structure and composition of the RHA samples. XANES spectra were recorded at the energy range of 110-120 eV to examine the photoabsorption characteristics of the Si atom (Fig. 4). Normalized Si L-edge XANES of standard SiO\(_2\) revealed the main absorption peaks at around 105.7 eV, 106.3 eV and 108.1 eV. Si L-edge XANES spectroscopy involves the electronic transition of Si 2p states. The first two well resolved peaks are transitions to the first unoccupied 3s-like states, from the spin-orbit splitting of Si 2p (2p\(_{3/2}\) and 2p\(_{1/2}\)). The peak at 108.1 eV was due to a 2p to 3d transition, which features a broad peak (due to multiple scattering). It has been reported that the splitting and relative peak ratios of the first two peaks relate to the long-range order of Si–O tetrahedral coordination. For the well crystalline phase, the increase in shared oxygen atoms led to better resolved splitting with an increase in the ratio between the intensity of the first and second peaks [20]. The comparison of the XANES spectrum between standard SiO\(_2\) and RHAs showed the similar features for RHA and standard SiO\(_2\). The results suggested the formation of the SiO\(_2\) compound in RHA. However, as described above, the rice husk ash after combustion at 300-
700°C was in a short-range order coordination. A combustion temperature of 1000°C was necessary to transform the SiO from the amorphous to the crystalline phase.

Figure 4. Normalized Si L-edge XANES of standard SiO and rice husk ash after combustion at temperature 300°C, 400°C, 500°C, 600°C, 700°C and 1000°C.

4. Conclusions

Rice husk ash can be prepared by firing under an oxidizing atmosphere. The combustion temperatures of 300-1000°C caused changes in RHA color from black to pinkish. The morphological structure of all RHAs has a predominantly rough and undulating surface texture. After combustion, the major parts of all RHAs retained their original shape. The dense membrane structures were observed on both the inside and outside surfaces without the presence of micropores. The cross-section of RHA revealed an interlayer which consisted of a crisscross mesh of chips between the two surfaces. Using EDS analysis, it could be seen that the internal reticular porous structure was a carbon-rich layer while silicon was packed on the outer surface which agreed well with the XPS result. The rice husk ash after combustion at 300-700°C was in a short-range order coordination. A combustion temperature of 1000°C was necessary to transform the SiO from the amorphous to the crystalline phase.

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6. References

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