CHAPTER 5

SYNTHESIS OF SiO_2 NANOPOWDER FROM RICE HUSK ASH

Overview - In the present work, we demonstrate a processing technique for SiO_2 nanopowder preparation from rice husk ash using a vibro-milling following by a heat treatment technique. Rice husk ash was milled by vibro-milling for 4 h to obtain nanopowder. In order to eliminate carbonaceous material, the nanopowder was heated at 1000°C for 3 h. Characteristic of the powder was then investigated by XRF, XRD, and SEM techniques. The silica nanopowder with 95% purity was obtained after the heat-treatment. The result indicated that the present method is a beneficial process to produce the silica nanopowders of low cost and high mass productivity.

5.1 Introduction

Silica (SiO_2) is a basic raw material that is widely used in agriculture, medical, electronic, ceramic, and polymer material industries[37-38]. Many authors reported that the silica powder could be produced from rice husk ash (RHA) source with not only has the benefit of producing valuable silica powder but also of reducing disposal and pollution problems[37-38,41-42]. Recently, much attention has paid on the production of silica nanopowders. The silica nanopowders can be prepared from many methods, including vapor-phase reaction, sol-gel and thermal decomposition technique[38-40]. However, most of those methods have high cost and give only small quantities that limited their wider applications. In the present work, a method to produce silica nanopowder with high mass productivity and relatively inexpensive was reported. The characteristic of the silica nanopowder was investigated.

5.2 Experimental procedures

The rice husk grains were washed with distilled water to remove adhering soil and dust. The rice husk samples were fired at 1000°C for 3 h in electrical furnace to form RHA. The resulting product was milled in a ball mill pot for 24 h. The dried powders were reground by vibro-milling method for 4 h. The product was heat treated at 1000°C for 3 h to eliminate carbonaceous material. Quantitative chemical analyze of as-prepared powder was accomplished by X-ray fluorescence (XRF). The phase evolution was investigated by X-ray diffraction technique (XRD). The powders were mounted on stubs, gold-coated in vacuum and viewed under scanning electron microscope (SEM).

5.3 Results and Discussions

The results of phase analysis by XRD of the samples are shown in Fig. 5.1. Both samples of as-received vibro-milled and calcined powder, showed the sharp peak at the position $2\theta = 22$, which indicated the crystalline state of cristobalite[37-38,41-42]. The concurrent of crystalline cristobalite may be due to the high temperature of firing at 1000°C, where as most authors reported that amorphous cristobalite was formed at the low firing temperature (~700°C).

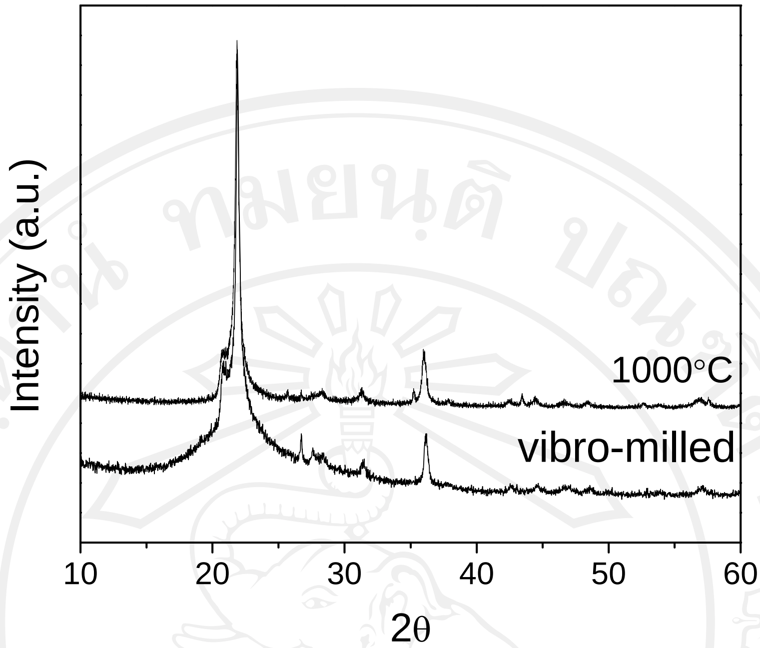
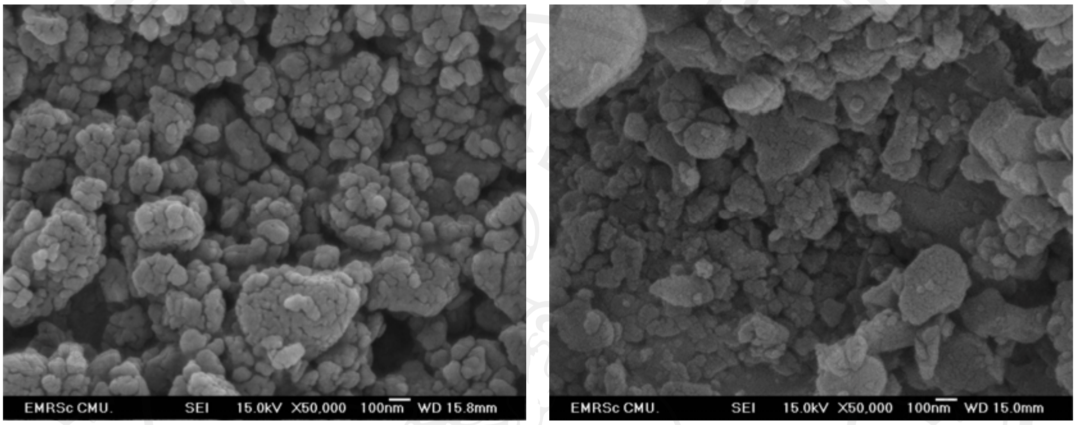


Fig. 5.1 XRD of RHA after 4 h vibro milling and after heat treatments at 1000°C.

Fig. 5.2(a) shows the SEM micrographs of the RHA samples after 4 h vibro milling time. The RHA nanoparticles have unifrom morphology with diameter smaller than 100 nm. However, agglomeration of nanoparticles was occurred. The agglomeration may be due to van der Waals attraction as suggested by ref.31. Morphology of the nanopowder after heated at 1000°C is shown in Fig. 5.2(b). The coarsening of crystallites on heating was illustrated that the particles which approach each other closer due to the increased van der Waals force or presence of liquid phase within the granules. It can be seen from Fig. 5.2(b) that although the individual particles were loosely aggregated into spherulites, the grain size of SiO_2 powder remains smaller than 100 nm. Concentrations of trace elements in the SiO_2 nanopowder are listed in Table 5.1. After heat treatments at 1000°C, the nanopowder presented the high silica of >95% purity. The trace elements in the SiO_2 nanopowder were alumina, iron, calcium, sodium, potassium, manganese and phosphorus. Compared with the previous work[37-38,41-42], we obtained the higher purity of SiO_2 nanopowder.



(a) (b)

Fig. 5.2 SEM micrographs of the RHA sample: (a) after 4 h vibro milling time and (b) after heat treatments at 1000°C.

Fig. 5.3 shows macroscopies of the samples. Color change was depended on the result of combustion process and compositions in the RHA. They have been classified into high carbon char, low carbon gray ash, and carbon- free pink and white ash[41]. It was observed that the as-received RHA samples showed black with some gray particles. Color was changed to gray after 4 h vibro-milling. In addition, the white color characteristic of SiO_2 nanopowder was found after heat treatment, indicating that there are no carbonaceous materials left in the powder.

Table. 5.1. Chemical composition and the trace elements of SiO_2 nanopowder after heat treated at 1000°C for 3 h.

Compositions	% of trace elements (expressed as oxide)
SiO_2	95.50
Al_2O_3	0.30
Fe_2O_3	0.20
CaO	0.80
Na_2O_3	0.25
K_2O	1.70
MnO	0.20
P_2O_3	0.64
Loss on fire	0.20

5.4 Conclusions

Silica nanopowder was synthesized via a vibro-milling followed by a heat-treatment method. We obtained the higher purity of SiO_2 nanopowder (>95% purity), compared with the previous works. The present method is found to be a beneficial process to produce the silica nanopowders of low cost and high mass productivity.

Fig. 5.3 (a) rice husk ash (b) After 4 h vibro-milling time and (c) After burning out at 1000°C for 3 h.