Chemistry and Materials Research
ISSN 2224- 3224 (Print) ISSN 2225- 0956 (Online)
Vol.8 No.7, 2016

Analysis of Rice Husk Ash Nanoparticles with Polyethylene Glycol Surfactants -6000 Using Coprecipitation Method

Eva Marlina Ginting* Karya Sinulingga Mukti Hamzah Harahap
Irma Suryani Siregar Nurdin Bukit
Departement of Physics Universitas Negeri Medan,Indonesia

Abstract
The purpose of this research is the preparation of nano particles of rice husk ash (RHA) and the characterization of the properties of nano-particles with a method that will be prepared coprecipitation.RHA obtained from rice refinery combustion products has white color .This method is performed in the manufacture of nano-particles by means of RHA in ball mill for an hour to get a size of 74 μm and then synthesized with a solution of HCl 2 M ,NaOH 2.5 M and sintesis polyethyelene glycol (PEG) 6000 (1:3;1:4 and 1:5)by methods coprecipitation so it is expected to obtain nanoparticle RHA then be characterized namely by analyzing the composition by means of X-Ray Fluorescent (XRF), morphology analysis by scanning Electron Microscofe (SEM), X-ray structure analysis Difraction (XRD). XRD analysis results obtained by the size of the crystal RHA 52,22 nm, 47,84 nm and 54,54 nm with a crystal system tetragonal ,XRF analysis results obtained dominant SiO_2 content 99,3 % by weight. Morfology results does not occur agglomeration RHA.
Keywords: Rice husk ash,PEG-6000, Coprecipitation

1. INTRODUCTION

Rice husk is a waste of rice being very abundant in Indonesia but its use is still limited in traditional ways. Rice husk has now been developed as a raw material to produce rice husk ash, known in the world as RHA (Rice Husk Ask). RHA is one of the raw materials to produce silica. RHA is one of the raw materials silica containing approximately 90-98% silica after combustion (Thuadaij, N et al, 2008).

Rice husk is a material such as cellulose berligno other biomass but contains high silica. RHA in the form of crystalline silica (quartz) and amorphous concentrated on the outer surface and little surface in (Bakri,et al,2009). Chemical content of rice husk consisting of 50% cellulose, lignin 25-30%, and 15-20% silica (Ismail.M.S,et al, 1996). RHA very high porosity causes RHA can absorb water in large quantities, (Kaboosi.K, 2007).

Silica has been used in many applications,including production of nanomaterials. Tailored materials composed of nanoparticles have potential for application in numerous technological fields (Ana Maria de Sousa ,et al,2009)

The most common value content of silica (SiO_2) in the rice husk ash is 91-96% and if its value is close to or below 90% may be caused by chaff samples that have been contaminated by other substances that lower the silica content (Prasad.CS, et al.,2001 ,Muthadhi .A,et al. 2007). Rice husk ash when burned in a controlled manner at high temperatures (500-600^{0}C) will produce silica ash which can be utilized for a variety of chemical processes. Rice husk when burned to generate about 20% of rice husk ash. The siliceous between 92-95%, with a high degree of porosity and lightweight .

Silica nano now has been applied in various fields including science and industry. RHA has been widely used as a filler. Silica has been used widely as a catalyst, and various kinds of organic-inorganic composite materials (Sun .L et al, 2001). In addition in the form of processed products, silica has also been used directly for the purification of oil, as additives in pharmaceutical products and detergents, as the stationary phase in a column chromatography, as a filler to the polymer thermoplastic polymers, as well as adsorbent (Kalapathy.U,et al, , 2000; Sun L, et al, 2001).

The organic portion of rice husk can be processed further to produce chemicals such as xylose, furfural, xylitol, ethanol, acetic acid, lignosulfonic acid (Chandrasekhar, et al, 2003)

Inorganic substances in rice husks as the minerals in small amounts can be removed by treatment with acid using H_2SO_4, HCl, or HNO (Chakraverty, et al, 1988). According to (Chandrasekhar,et al 2006), hydrochloric acid is a chemical that is highly effective for reducing impurities - impurities contained in the rice husk ash

Has done a lot of research on the manufacture of nano silica from RHA by way of synthesis, among others, (Thuadaij.N. et al, 2008), the particle size of 50 nm is obtained, (Pukird, S, et al, 2009), the size of the particles obtained 40 - 200 nm, so too (Ezzat Rafiee, et al, 2012) the results obtained average 6 nm, with the ball mill for 15 hours obtained the crystal size of 53 nm (Ginting, E.M, et al., 2014).

The purpose of research and writing in this article is to determine the particle size, structure, composition and morphology of RHA taken from the refinery industry paddy combustion products are white, the process of making do with a ball mill process and methods kopresitasi with HCl 2 M and NaOH 2 , 5 M, then synthesized with PEG-6000

2. METHODS

Materials

RHA white color from the combustion of of paddy mill industry, HCl 2 M, NaOH, 2.5 M (Merck KGaA 64271 Darmstadt Germany) Poly Ethylene Glycol- 6000

Tool

Ball mill PM 200, X-ray Diffraction (XRD) Shimadzu 6100 (40 kV, 30 mA) at a rate of 2 ° / min over a range of angles 2θ = 5 ° -80 °. XRF (Torontech Model TT-EDXPRT) and SEM (Zeiss Model), Energy dispersive X-ray Detector - single photon couting / enrgy dipersive X-ray with multi-channel analyze (MCA). Bruker Nano Xflash Detector.

The processing RHA with Coprecipitation Method

The RHA milled using a grinding tool ballmill for 6 hours and then sieved with the size of 74 lm. Nanoparticles are already in the sifter and then synthesized using coprecipitation method. Coprecipitation method is used because it uses simple equipment and can be done in a relatively short time. RHA phase SiO_2 which has weighed as much as 20 g and diluted with 40 ml 2M HCl and generate reactions:

$$SiO_{2\,(s)} + 4HCl_{(l)} \Longrightarrow SiCl_{4\,(s)} + 2H_2O_{(l)}$$

The reaction product is separated through the screening process. Then precipitated dissolved in 30 ml 2.5 M NaOH with the following reaction:

$$SiCl_{4\,(s)} + 4H_2O_{(l)} + 4NaOH_{(l)} \Rightarrow SiO_{2\,(s)} + 4NaOH_{(l)} + 6H_2O_{(l)}$$

The resulting solution is filtered and then washed using distilled water as much as approximately 6 times that remnants acid reaction product can be lost. To obtain nanoparticles SiO_2 powder, precipitate dried in an oven for 4 hours at a temperature of 70^0C after dried and then PEG-6000 in the form of solids are heated and melted at a temperature of 50^0C for ± 15 menit.PEG-6000 that has been melted added to the solution by comparison 1: 3; 1: 4; and 1: 5 and then stirred using a magnetic stirrer at a temperature of 70^0C for 40 menit.Larutan 2.5 M NaOH 30 ml was added to the mixture of PEG-6000 with The RHA solution while stirring using a magnetic stirrer. Furthermore, the solution is separated by the filter paper and washed by using a distilled water and filtered again to separate the The RHA with distilled water, dried in an oven at a temperature of 70^0C for 4 hours.

3. RESULTS AND DISCUSSION

3.1 The Analysis of RHA With XRF (X-Ray Flouresence).

The results RHA analysis using XRF spectrometer Spector Pro Elvax type to determine the Si content contained in RHA and other impurity elements. From the results obtained characterization data on the chemical elements such RHA Table 1

Table 1. The Results of Analysis of RHA with the synthesis of PEG-6000

No	Chemical elements	The content of the element (%) 1:3	The content of the element (%) 1:4	The content of the element (%) 1:5
1.	Si	99,40	99,30	99,29
2.	K	0,48	0,51	0,3
3.	Ca	0,055	0,072	0,27
4.	Mn	0,032	0,038	0,074
5.	Fe	0,012	0,0168	0,024
6.	Zn	0,0061	0,0063	0,016
7.	Rb	0,0039	0,0040	0,0011
8.	Sn	0,0040	0,0039	0,005
9.	Ni	0,0028	0,0028	0,0043
10.	Ag	0,0018	0,0008	0,001

Table 2. The Results Analysis of RHA Without Treatment

Chemical elements	The content of the element (%)
Si	54,5
K	28,5
Fe	4
Ca	4,3
Al	1,4
Mn	2,9
P2	1,96
Rb	1,8
Co	0,4
Zn	1,23

From the analysis by XRF for RHA were taken from the burning of rice husk of the refinery industry gained silica element content of approximately 54.5% as shown in Table 2. After peparsi process with a solution of HCl and NaOH with coprecipitation method with sisntesis PEG 6000 to a ratio of 1: 3; 1: 4; 1: 5 telihat an increase silicon content constituent into (99.40; 99.30; 99.2 9% can be seen in Table 1 as well as research results (Thuadaij, Net, al., 2012) Approximately 90-98% silica after combustion. it is appropriate according to (Chandrasekhar et al, 2006), hydrochloric acid is a chemical that is highly effective for reducing impurities - impurities contained in the RHA, the same is true of research results (Chakraverty, et al, 1988) and (Sun L, et al, 2001)

3.2 Characterization of XRD (X-Ray Diffraction)

XRD characterization performed to obtain diffraction pattern, crystalline structure and particle size of the nanoparticles RHA. The results X-ray diffraction pattern of phase RHA with PEG-6000 are shown in Figure 1 to 4.

In Figure 1 can be seen the highest peaks on 2θ : 20,530°; 21,729°; 23,282°; 27,392°; 30,076°; 36,100°. The maximum peak located on angle 2θ = 21,729° by distance (d) 4,0867 Å. The results X-ray diffraction pattern of the RHA with PEG-6000 (1: 3) have a cristobalite phase (SiO_2) with the lattice parameter $a = b \neq c$ with velue $a = b = 4{,}9930$ Å $c = 7{,}0050$ Å tetragonal crystal system and has a density of 2,28500 g/cm^3

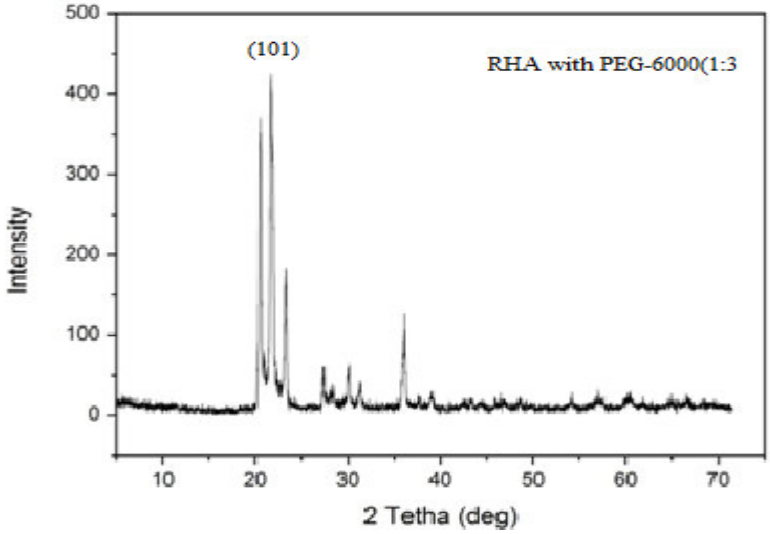

Figure 1 The RHA diffraction pattern with PEG-6000 (1: 3)

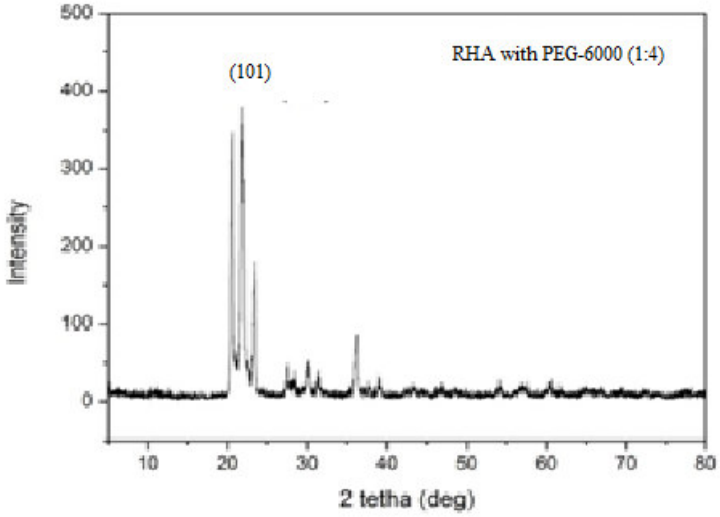

Figure 2 The RHA Diffraction pattern with PEG-6000 (1: 4)

In Figure 2 can be seen the highest peaks on 2θ : 20,593°; 21,799°; 23,352°; 27,443°; 30,153°; 31,327°; 36,100° 39,064°. The maximum peak located on angle 2θ = 21,799° by distance (d) 4,0737 Å. The results X-ray diffraction pattern of the RHA with PEG-6000 (1:4) have a cristobalite phase (SiO_2) with the lattice parameter $a = b \neq c$ with velue $a = b = 4{,}9790$ Å $c = 6{,}9500$ Å tetragonal crystal system and has a density of 2,31400 g/cm^3.

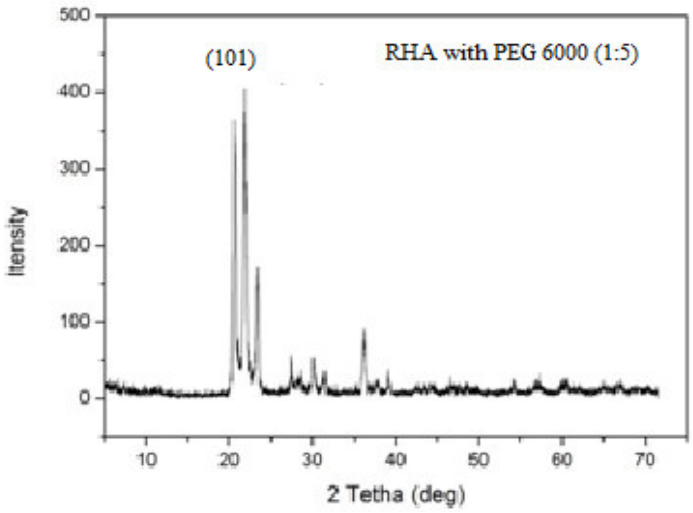

Figure 2 The RHA diffraction pattern with PEG-6000 (1: 4)

In Figure 2 can be seen the highest peaks on 2θ : 20,633°; 21,807°; 23,363°; 27,459°; 30,138°; 31,424°; 36,181° 39,101°. The maximum peak located on angle 2θ = 21,807° by distance (d) 4,0724 Å. The results X-ray diffraction pattern of the RHA with (1:5) have a cristobalite phase (SiO_2) with the lattice parameter $a = b \neq c$ with velue $a = b = 4{,}9719$ Å $c = 6{,}9223$ Å tetragonal crystal system and has a density of 2,3300 g/cm^3 From the results of XRD diffraction pattern RAH with PEG-6000 (1:3, 1:4, 1:5) have a cristobalite phase (SiO_2) with d_{hkl} (101) , according to research (Ginting, E.M ,et al 2015).

To determine the particle size of each sample can be determined using the equation Debay Scherrer.

$$D = \frac{k\lambda}{\beta \cos\theta}$$

where:
k = 0,91,
λ = wavelength Cu K_α 1,54060 Å,
β = FWHM (full width a half maximum).

Table. 3. Particle Size RAH synthesized PEG-6000

RHA with PEG-6000	Cristal size (nm)
(1:3)	52,22
(1:4)	47,84
(1:5)	54,54

Table 3 is based on rice husk ash without PEG-6000 has a particle size of 50.77 nm, rice husk ash with PEG-6000 (1: 3, 1: 4, 1: 5) each have a size of 52.22 nm, 47, 84 nm and 54.54 nm. Interest PEG-6000 was to make no agglomeration of the particles. Proven on the addition of PEG-6000 in a ratio of 1: 4 has a smaller size than the RHA without using PEG.

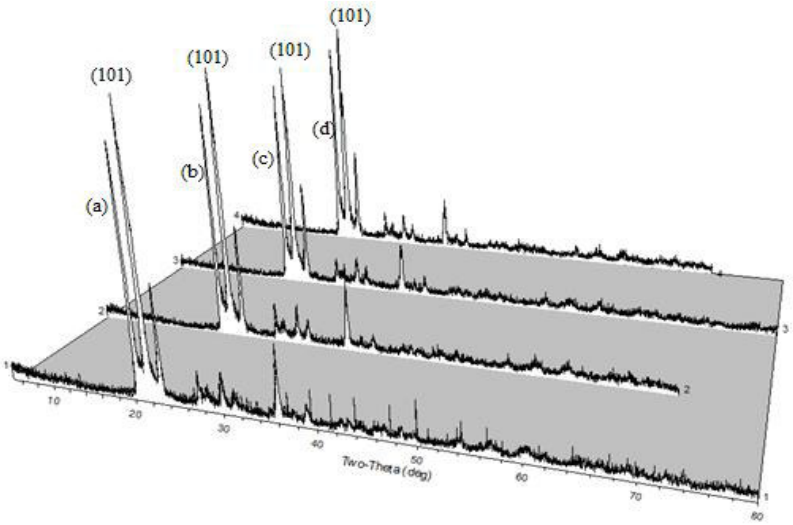

Figure 4 The RHA Diffraction Pattern (a) without PEG (b)1: 3; (c)1:4 ; (d)1,5)

3.3 Analysis SEM (*Scanning Electron Microscopy*)

The characterization results are shown in Figure 5, the morphology of the sample RHA without PEG-6000 shows that the surface structure is more organized and form small particles shaped oval, and still there is agglomeration of particles In RHA by synthesis using PEG 6000, RHA mostly distributed homogeneously and there is no agglomeration each other in accordance with the function of PEG to prevent agglomeration of particles in order to obtain a uniform size. In general results the sample morphology patterns RHA with PEG-6000 1: 3, 1: 4 and 1: 5 is the same

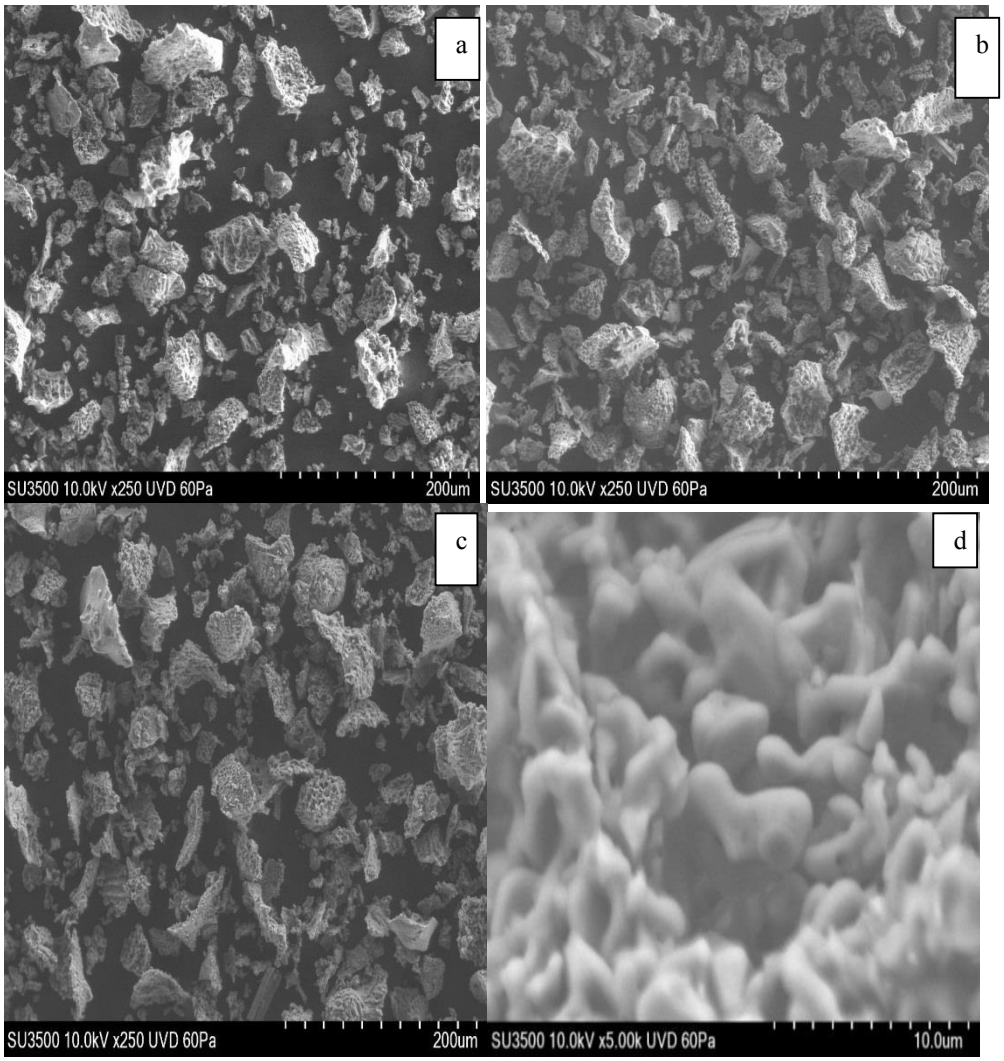

Figure 5. Morphology RHA with PEG-6000 (a),1:3 ,(b). 1:4 (c), 1:5) , (d) without PEG

ACKNOWLEGEMENTS

Researchers would like to thank the Ministry of Higher Education Research and Technology on the research of funding in research grants fundamental 2016 No:022A/UN33.8/KU/2016 and labaratorium Physics, State Unimed and LIPI Jakarta on the facilities provided.

4.CONCLUSION

The XRD analysis results obtained with nano-particles of rice husk ash (RHA) using PEG 6000 in a composition ratio of 1: 3.1: 4 and 1: 5 obtained crystal size of 52.22 nm, 47.84 nm and 54.54 nm with SiO_2 tetragonal crystal system, with d_{hkl} (101) and the average Si content of 99.3% by weight

.

REFERENCES

Ana Maria de Sousa, Leila Visconte, Claudia Mansur and Cristina Furtado, (2009), Silica Sol Obtained From Rice Husk Ash Chemistry & Chemical Technology 3, 4, 321-326.

Chakraverty, A., Mishra, P., and Banerjee, D. (1988) 'Investigation of Combustion of Raw and Acid-Leached Rice Husk for Production of Pure Amorphous White Silica', Journal of Materials Science, 23, . 21-24.

Chandrasekhar, S., Pramada, P.N., and Majeed, J. (2006) 'Effect of calcination temperature and heating rate on the optical properties and reactivity of rice husk ash', Journal of Materials Science, 41, . 7926-7933.

Chandrasekhar,S., Satyanarayana. K. G., Pramada, P.N., and Raghavan,P. (2003) 'Review Processing, Properties and Applications of Reactive Silica from Rice Husk— An Overview', Journal of Materials Science.. 38,. 3159 – 3168.

Bakri dan Baharuddin. 2009. Absorpsi Air Komposit Beton Sekam Padi dengan Penambahan Pozzolan Abu Sekam Padi dan Kapur pada Matriks Beton. Jurnal Perennial, 6(2) : 70-78

Ezzat Rafiee, Shabnam Shahebrahimi, Mostafa Feyzi and Mahdi Shaterzadeh ,(2012) , Optimization of synthesis and characterization of nanosilica produced from rice husk (a common waste material . International Nano Letters , 2 :29

Ginting, E. M., Wirjosentono, B., Bukit, N., dan Agusnar, H., (2014), Preparation and Characterization of Rice Husk Ash as Filler Material in to Nanoparticles on Hdpe Thermoplastic Composites, Chemistry and Materials Research, 6 ,7 ,14-24.

Ginting, E. M., Bukit, N., Siregar, M.A. (2015), Preparation And Characterization Of Natural Zeolite And Rice Husk Ash As Filler Material HDPE Thermoplastic , Chemistry and Materials Research 7 .2, 20-27..

Kalapathy, U., Proctor, A., dan Shultz, J., (2000), A simple method for production of pure silica from rice hull ash,Bioresource Technology 73 257-262

Kaboosi, K. 2007. The Feasibility of Rice Husk Application as an Envelope Material in Subsurface Drainage System. Islamic Azad University, Science and Research Branch. Tehran, Iran.

Muthadhi, A., Anitha, R. and Kothandaraman, S. (2007). Rice Husk Ash – Properties and Its Uses: A Review. IE(I) Journal–CV, 88: 50 – 56.

Pukird, S., Chamninok, P., dan Samran, S., (2009), Synthesis and Characterization of SiO_2 Nanowires Prepared from Rice Husk Ash, Journal of Metal, Materials and Minerals, . 19 . 2 . 33-37

Prasad C.S., Maiti K,N., Venugopal R., (2001), "Effect of rice husk ash in whiteware compositions", Ceramic International, 27, 629-635.

Sun, L., Gong, K. (2001) 'Silicon-based Materials from Rice Husks and Their Applications' Ind. Eng. Chem. Res., 40,. 5861-5877.

Thuadaij, N., dan Nuntiya, A., (2008), Preparation of Nanosilica Powder from Rice Husk Ash by Precipitation Method, Chiang Mai J. Sci. 2008; 35(1) : 206-211

Thuadaij, N., dan Nuntiya,A.,(2012), Preparation and Characterization of Faujasite using Fly Ash and Amorphous Silica from Rice Husk Ash, Procedia Engineering 32 , 1026-1032