ORIENTAL JOURNAL OF CHEMISTRY
An International Open Free Access, Peer Reviewed Research Journal
www.orientjchem.org

Est. 1984

ISSN: 0970-020 X
CODEN: OJCHEG
2016, Vol. 32, No. (4):
Pg. 1923-1928

Preparation and Characterization of Amorphous Silica and Calcium Oxide from Agricultural Wastes

SUPACHAI SOMPECH[1], THANANCHAI DASRI[1] and SUKHONTIP THAOMOLA[2]*

[1]Faculty of Applied Science and Engineering, Nong Khai Campus,
Khon Kaen University, Nong Khai, 43000, Thailand.
[2]Faculty of Science and Arts, Chanthaburi Campus, Burapha University, Chanthaburi, 22170, Thailand.
*Corresponding author E-mail: suky_2547@yahoo.com

http://dx.doi.org/10.13005/ojc/320418

(Received: July 13, 2016; Accepted: August 11, 2016)

ABSTRACT

Rice husk ash and bagasse ash were agricultural wastes that provide an abundance of the silica (SiO_2) source and the chicken eggshells and duck eggshells were important sources for calcium oxide (CaO). Therefore, in this study the rice husk ash and bagasse ash were used as raw materials for synthesis of silica powder, while chicken eggshells and duck eggshells were synthesized for the calcium oxide. The results from the XRD pattern clearly showed the structural formation of amorphous SiO_2 and CaO phase. While the FTIR results indicated that the spectrums which displayed the characteristic peaks of the functional groups presenting in the SiO_2 and CaO powder. However, the SEM images revealed that the particles agglomerated, various sizes and the particle size were found to be in micron level.

Keywords: Rice husk ash, Bagasse ash, Chicken eggshells, Duck eggshells, Agricultural wastes.

INTRODUCTION

Rice husk and bagasse are agricultural waste material abundantly available in Thailand. They are important biomass sources, which used as a fuel in the brick industry and sugar industry, respectively. As a result, a large quantity of rice husk ash and bagasse ash are generated and created a serious dispensation problem. Rice husk ash and bagasse ash are rich in silica (SiO_2) (more than 60% for bagasse ash and above 90% for rice husk ash) and can be used as a raw material for produce the silica powder production[1-5]. Many studies have reported that the rice husk ash and bagasse ash were an excellent sources for synthesis of amorphous silica[6-9] because the fine silica powder is a basic raw material that is widely used in electronic substrates, thermal and electrical insulators, optoelectronic devices and composite fillers, etc.[10].

While the chicken eggshells and duck eggshells are one group of agricultural waste materials which can be found as waste products from restaurants and fresh markets in Thailand, as they are used for cooking. The chemical composition of eggshells is calcium carbonate ($CaCO_3$) typically containing a high level (above 90%)[11, 12]. As the chicken and duck eggshells are composed of calcium carbonate, so being calcined at above 700°C will produce the CaO powder[13, 14], which can be used as a basic adsorbent for carbon dioxide (CO_2)[15] and as a catalyst for the production of biodiesel[13].

Therefore, the aim of this study was to synthesize of amorphous silica and calcium oxide powder from agricultural wastes by using the rice husk ash and bagasse ash used as sources for silica, while the chicken eggshells and duck eggshells were sources for calcium oxide. The synthesized powder were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Brunauer-Emmett-Teller (BET), particle size analyzer and scanning electron microscopy (SEM).

MATERIALS AND METHODS

Synthesis of sample

Rice husk ash and bagasse ash were obtained from a brick factory and sugar industry, respectively and they were rinsed by water several times to remove impurity and then burned at 700°C for 3 h. To evaluate the chemical compositions of the rice husk ash and bagasse ash was analyzed by X-ray fluorescence (XRF), this result was shown in Table 1. Subsequently, twenty grams of rice husk ash and bagasse ash samples were stirred in 80 ml of 2.5 N sodium hydroxide solutions, afterwards heated in a covered 250 ml Erlenmeyer flask for 3 h after that, the solution was filtered and the residue was washed with 40 ml of boiling water. The filtrate was allowed to cool down at room temperature. Then, it was added by 5 N H_2SO_4 with constant stirring until pH 2. Then, the filtrate was added NH_4OH until pH 8.5 left for 3 h and dried at 100°C for 12 h and finally was pulverized by mortar.

Simultaneously, the eggshells waste received from restaurant were rinsed with water several times to remove additional residues or any unwanted substance from the surface and then heated at 100°C for 12 h to dry by oven. Completely dried eggshells was pulverized by using a hammer mill, after that crushed with a juice blender and then passed through a sieve. Next the chemical compositions of eggshells were analyzed by X-ray fluorescence (XRF) shown in Table 2. The dried eggshells were calcined in a muffle furnace under static air conditions at 900°C for 3 h to transform the calcium carbonate to CaO powder.

Characterization

The powders obtained were characterized by various techniques. The average particle size distribution was determined by using particle size analyzer (Mastersizer S, Malvern) and examination the functional groups studied by Fourier transforms infrared spectrometer (Perkin Elmer spectrometer). X-ray diffractometer (Phillips X' pert X-ray diffractometer) was used to identify phase of the powder sample. The specific surface area of the powder sample was measured using the BET nitrogen adsorption method (Quantachrome), and morphology of sample observed by scanning electron microscope (LEO/1450).

RESULTS AND DISCUSSION

The chemical composition of the raw materials for this study is shown in Table 1 and Table 2. In the Table 1, it showed that the chemical composition of rice husk ash and bagasse ash samples, after burning out at 700°C for 3 h, presented the highest amount of SiO_2; while, the major component of both eggshells displayed calcium $CaCO_3$ (Table 2).

The X-ray diffraction pattern of SiO_2 powder obtained from rice husk ash and bagasse ash is shown in Fig. 1. The results showed the strong broad peak at around 22°(2θ) of both sample after calcining ash at 700°C for 3 h. This results confirmed that this peak is nature of silica, which supposed to be the characteristic of amorphous SiO_2. For the results from FT- infrared (FTIR) spectrum of SiO_2 powder is presented in Fig. 2. It clearly showed a slightly broad band since 2,800 to 3,700 cm^{-1} which assigned to present the OH stretching frequency of silanol groups and adsorbed water. A strong intense band at 1,100 cm^{-1} is associated to the siloxane Si-O-Si vibration of the molecules. Amorphous silica exhibits a relatively strong peak around at 800 cm^{-1}.

For the X-ray diffraction pattern of the CaO powder obtained from chicken eggshells and duck eggshells are shown in Figs. 3 and 4, respectively. The results revealed that the thermal process of

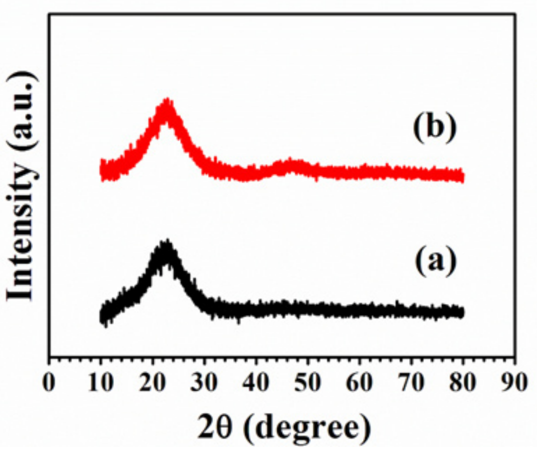

Fig. 1: XRD patterns of SiO_2 powder after prepared from (a) rice husk ash and (b) bagasse ash

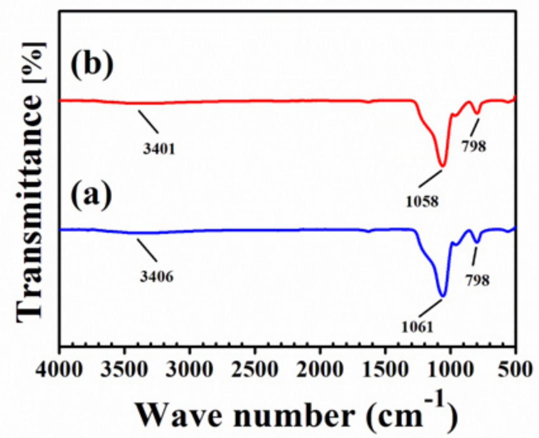

Fig. 2: FTIR spectrum of SiO_2 powder after prepared from (a) rice husk ash and (b) bagasse ash

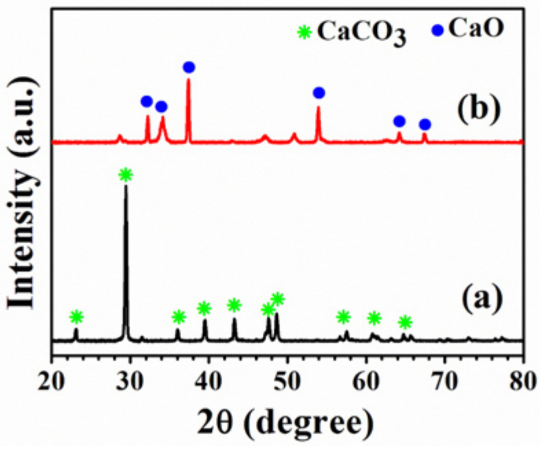

Fig. 3: XRD pattern of chicken eggshells (a) before calcined and (b) after calcined at 900°C

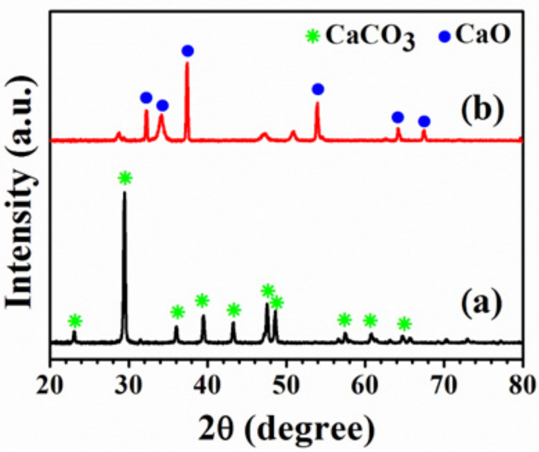

Fig. 4: XRD pattern of duck eggshells (a) before calcined and (b) after calcined at 900°C

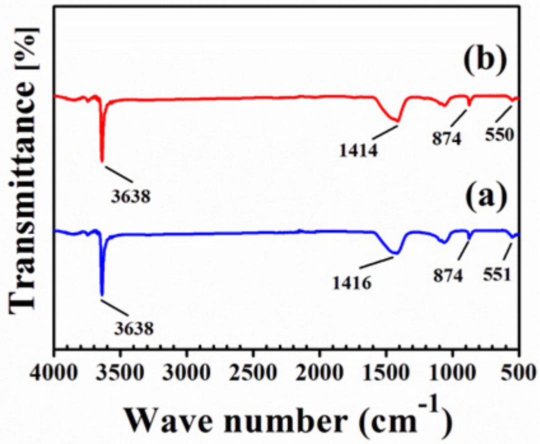

Fig. 5: FTIR spectrum of CaO powder after prepared from (a) chicken eggshells and (b) duck eggshells

$CaCO_3$ phase is completely decarbonized and turned to be CaO phase after both chicken and duck eggshells were calcined at 900°C. The shape of the sharp and strong diffraction peaks indicated that the samples are well crystallized and corresponded to the standard values of file JCPDS No. 37-1497.

Fig. 5 showed the FT-infrared (FTIR) spectrum of the CaO powder after prepared from chicken eggshells and duck eggshells, respectively. In Fig. 5a and 5b presented the strong band at 3638 cm^{-1} can be attributed to the (OH) stretching vibration. The broad band around 1400 cm^{-1} indicated the symmetric stretching vibration of carbonate. Small band at 874 cm^{-1} demonstrated the presence of carbonate species. The tiny band at 550 cm^{-1} identified vibration of the Ca-O bond[16].

While, the average particle size and specific surface area of the SiO_2 and CaO powder is shown in Table 3. The results displayed the average particle size of the amorphous SiO_2 powder obtained from rice husk ash and bagasse ash is not significantly different, the particle sizes are 46.74 μm and 47.16 μm, respectively. In addition, the specific surface area of amorphous SiO_2 powder was also similar as well. However, the average particle size of the CaO powder obtained from chicken and duck eggshells after being calcined of $CaCO_3$ phase are 57.13 μm and 23.91 μm, respectively. And the specific surface area of CaO powder are 13.20 m^2/g and 16.42 m^2/g, respectively.

The morphology of the amorphous SiO_2 powder obtained from rice husk ash and bagasse ash is shown in Fig. 6. The SEM images indicated that the particles of powder are agglomerated, various sizes, irregular morphology, and particles size more than 1 μm were recognized. In addition, the average particle size of SiO_2 powder obtained from rice husk ash is in the range of 1-7 μm (Fig. 6a), while the average particle size of SiO_2 powder obtained from bagasse ash is in the range of 1-10 μm (Fig. 6b).

Fig. 7 showed the SEM images of the CaO powder after calcining of $CaCO_3$ from chicken eggshells and duck eggshells. In Fig. 7a can be seen that the average particle size and shape of CaO particles obtained from chicken eggshells

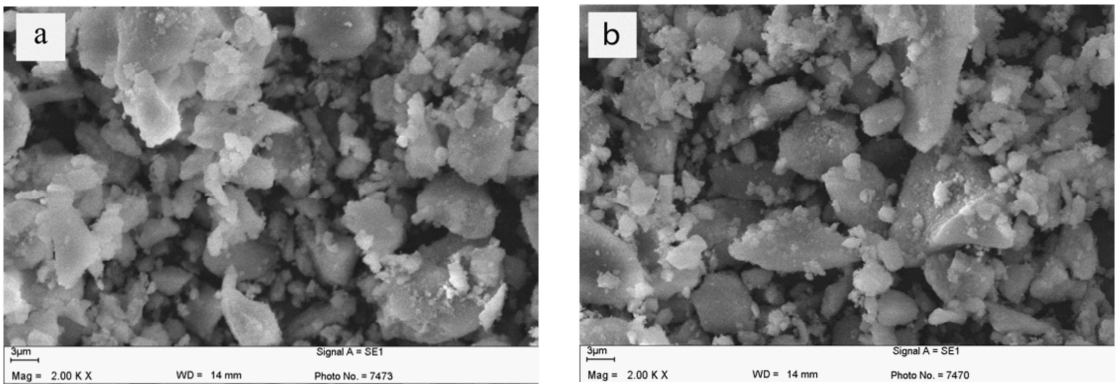

Fig. 6: SEM micrographs of amorphous SiO_2 powder after prepared from (a) rice husk ash and (b) bagasse ash

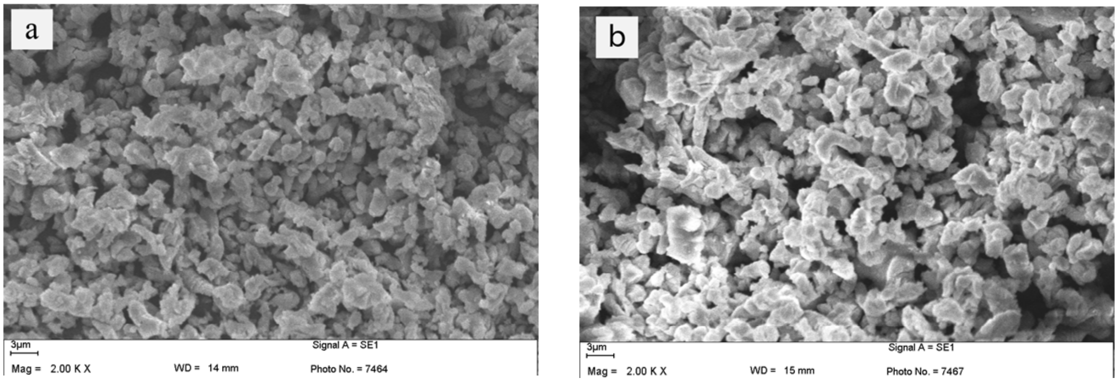

Fig. 7: SEM micrographs of CaO powder after prepared from (a) chicken eggshells and (b) duck eggshells

Table 1: Chemical composition of rice husk ash and bagasse ash

Samples	Inorganic component (wt.%)										
	SiO_2	Al_2O_3	MgO	Fe_2O_3	CaO	P_2O_5	Mn_2O_3	SO_3	K_2O	TiO_2	Total
Rice husk ash	93.57	0.80	0.66	0.59	0.90	1.02	0.28	0.10	2.01	0.08	100
Bagasse ash	80.46	1.24	4.07	1.14	4.43	5.24	0.25	0.69	2.36	0.15	100

Table 2: Chemical composition of eggshells

Samples	Inorganic component (wt.%)										
	$CaCO_3$	Al_2O_3	MgO	Fe_2O_3	SiO_2	P_2O_5	Mn_2O_3	SO_3	K_2O	TiO_2	Total
Chicken egg	99.66	-	-	-	-	-	-	0.34	-	-	100
Duck egg	99.19	-	-	-	-	-	-	0.82	-	-	100

Table 3: Average particle size and specific surface area of prepared samples

Samples	Average particle size [D 4,3] (μm)	Specific surface area (m^2/g)
SiO_2 from rice husk ash	46.74	271.21
SiO_2 from bagasse ash	47.16	233.85
CaO from chicken eggshells	57.13	13.20
CaO from duck eggshells	23.91	16.42

was similar to the CaO particles obtained from duck eggshells as shown in Fig. 7b. The characteristic of CaO particles of both chicken eggshells and duck eggshells are regular micro morphology of rod like, the average particles with size ranging from 2 μm to 4 μm of width are observed.

CONCLUSION

Amorphous SiO_2 is successfully synthesized from the rice husk ash and bagasse ash that used as raw materials. While the CaO powder can be synthesized by using $CaCO_3$ from chicken eggshells and duck eggshells that used as raw materials, and then burning at 900°C for 3 h. The XRD results clearly exhibited the structural formation of SiO_2 and CaO phase. The FTIR spectrums demonstrated that the characteristic peaks of the important functional groups present in the powder samples of SiO_2 and CaO. The SEM images illustrated that the particles are agglomerated, various sizes, and the particle size is found to be in the micro regime. This study revealed that the agricultural waste material of rice husk ash, bagasse ash, chicken eggshells, and duck eggshells acts as an alternative source for the production of SiO_2 and CaO powder.

ACKNOWLEDGMENTS

This work was financial supported by the young researcher development project of Khon Kaen University.

REFERENCES

1. Thuadaij, N.; Nuntiya, A. *Chiang Mai J. Sci*. **2008**, *35*, 206-211
2. Yalçin, N.; Sevinç, V. *Ceram. Int*. **2001**, *27*, 219-224
3. Thuadaij, N.; Nuntiya, A. *CMU J. Nat. Sci*. **2008**, *7*, 59-65
4. Hariharan, V.; Sivakumar, G. *Int. J. Chem. Tech. Res*. **2013**, *5*, 1263-1266
5. Nazriati, N.; Setyawan, H.; Affandi, S.; Yuwana, M.; Winardi, S. *J. Non-Cryst. Solids*, **2014**, *400*, 6-11
6. Real, C.; Alcala, M. D.; Criado, J. M. *J. Am. Ceram. Soc*. **1996**, *79*, 2012-2016
7. Nandi, K. C.; Biswas, A. K.; Acharya, H. N. *Mater. Lett*. **1991**, *12*, 171-174
8. Faria, K. C. P.; Gurgel, R. F.; Holanda, J. N. F. *J. Environ. Manage*. **2012**, *101*, 7-12
9. Aigbodin, V. S.; Hassan, S. B.; Ause, T.; Nyior, G. B. *J. Mineral Mater. Charac. Eng*. **2010**, *9*, 67-77
10. Liou, T.-H. *Mater. Sci. Eng. A*, **2004**, *364*, 313-323
11. Ok, Y. S.; Lee, S. S.; Jeon, W.-T.; Oh, S.-E.; Usman, A. R. A.; Moon, D. H. *Environ. Geochem. Health*, **2011**, *33*, 31-39
12. Cho, Y. B.; Seo, G. *Bioresour. Technol*. **2010**, *101*, 8515-8519
13. Wei, Z.; Xu, C.; Li, B. *Bioresour. Technol.* **2009**, *100*, 2883-2885
14. Niju, S.; Meera, K. M.; Begum, S.; Anantharaman, N. *J. Saudi. Chem. Soc.* **2014**, *18*, 702-706
15. Ives, M.; Mundy, R. C.; Fennell, P. S.; Davidson, J. F.; Dennis, J. S.; Hayhurst, A. N. *Energ. Fuels*, **2008**, *22*, 3852-3857
16. Mirghiasi, Z.; Bakhtiari, F.; Darezereshki, E.; Esmaeilzadeh, E. *J. Ind. Eng. Chem*. **2014**, *20*, 113-117.