World Academy of Science, Engineering and Technology
International Journal of Chemical and Molecular Engineering
Vol:7, No:1, 2013

Enhanced Quality of Zeolite LSX: Studying Effect of Crystallized Containers

Jitlada Chumee

***Abstract*—**Low silica type X (LSX) Zeolite is one of useful material in many manufacturing due to the advantage properties including high surface area, stability, microporous crystalline aluminosilicates and positive ion in an extra–framework. The LSX was used rice husk silica source which obtained by leaching with hydrochloric acid and calcination at 500 C. To improve the synthesis method, the LSX was crystallizated in Teflon–lined autoclave will expedite deceasing of the amorphous particles. The mixed gel with composition of 5.5 Na2O : 1.65 K2O : Al2O3 : 2.2 SiO2 : 122 H2O was crystallized in different container (Polypropylene bottom and Teflon–lined autoclave). The obtained powder was characterized by X–ray diffraction (XRD), X–ray fluorescence spectrometry, N2 adsorption-desorption analysis BET surface area Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy to justify the quality of zeolite. The results showed the crystallized zeolite in Teflon lined autoclave has 102.8 nm of crystal size, 286 m2/g of surface area and fewer amounts of round amorphous particles when compared with the crystallized zeolite in Polypropylene.

***Keywords*—**LSX Zeolite, rice husk and crystallized container.

Open Science Index, Chemical and Molecular Engineering Vol:7, No:1, 2013 publications.waset.org/5904/pdf

I. Introduction

RICE husk is an agricultural solid waste, rice milling byproduct, produced in large quantities for no value. This significant problem may be overcome by utilizing this waste into useful materials. The ash obtained from rice husk is composed of silica with small impurities of other inorganic metal depending on geographic location and climate [1]–[3].

The utilization of rice husk is extraction of higher purity silica by leaching with hydrochloric acid and calcination at 600°C under a static air or flowing atmosphere.

The rice husk silica can use as a source for preparation of porous materials such as zeolite and mesoporous MCM–41, increasing value [3]–[5].

Zeolite is widely used in manufacturing due to its property such as high surface area, stability, micropore, crystalline aluminosilicates and positive ion in an extra-framework. Therefore, it can apply for catalyst support, gas separation and detergents in manufacturing.

The low silica type X zeolite (LSX) is faujasite (FAU) zeolite, Si/Al ratio in rang of 1–1.5. The LSX can use a silica source from natural clay and rice husk for synthesis in polypropylene bottom [5]–[7]. However, the results show a number of amorphous particles, possibly it had unstable pressure during the synthesis method. Therefore, the synthesis method should be improved.

Jitlada Chumee is with the Department of Chemistry, Faculty of Science and Technology, Suan Sunandha Rajabhat University, Bangkok, 10300, Thailand (e-mail: jitladachumee@yahoo.com).

The aim of this work was to study effect of crystallized container between polypropylene bottom and Teflon lined autoclave.

II. Experiment

A. Materials

The chemical reagents for synthesis zeolite LSX were analytical grade hydrochloric acid (37% HCl, Carlo Erba), sodium aluminate ($NaAlO_2$, Riedel-de Han), sodium hydroxide (NaOH, Carlo Erba) and potassium hydroxide (KOH, Carlo Erba)

B. Synthesis and Characterization of Zeolite LSX

Rice husk silica for synthesis zeolite LSX was extracted by leaching with hydrochloric acid and calcination at 500°C with a modified method from literature [7]. The silica source from rice husk was dissolved in 3.5 M NaOH solution for preparation of sodium silicate solution that was used to synthesize zeolite LSX. The zolite LSX was synthesized by mixing the seed gel and feed gel to obtain initial batch composition of 5.5 Na_2O : 1.65 K_2O : Al_2O_3 : 2.2SiO_2 : 122 H_2O and was poured in different container (Polypropylene bottom and Teflon–lined autoclave). Then, the mixed gel was aged at 70°C for 3 h and crystallized at 100°C for 2 h, filtrated and washed with deionized water until it was pH 10. Finally, the powder product was washed with 0.01 M NaOH solution for hydrolysis protection.

The obtained powder material was characterized by X–ray diffraction (XRD; BRUKER AXS: D8 ADVANCE A25), X–ray fluorescence spectrometry (XRF), Scanning electron microscopy (SEM; Jeol scanning microscope model JSM–5410), N_2 adsorption-desorption analysis BET surface area (Autosorb–1 chrantachrome, BL model) and Fourier transform infrared spectroscopy (FT–IR; BRUKER TENSOR27).

III. Results and Discussion

The rice husk was obtained from Ubonratchathani province, Thailand and analyzed with XRF for determination amount of silica. The results show that it is contained of 87% of silica. After extraction with HCl, the purity silica was increased to 98%. It seems to be suitability for preparation of sodium silicate solution that was used to synthesize zeolite LSX [3], [5].

There are two powder products of zeolite (LSX–A and LSX–B), which was synthesized from rice husk silica. LSX–A and LSX–B refer to the zeolite was crystallized in polypropylene bottle and Teflon–lined autoclave, respectively.

The XRD result is shown in Fig 1. Both of LSX–A and LSX–B zeolite have characteristic peaks comparable with the

Open Science Index, Chemical and Molecular Engineering Vol:7, No:1, 2013 publications.waset.org/5904/pdf

standard NaX. The degree that has a highest intensity was selected for calculation crystallite size from Scherrer equation.

The LSX–B showed similar crystallite size compared with standard NaX (102.89 nm) as show in Table I [7], [8]. The other hand LSX–A showed smaller crystallite size than LSX–B. It is possibly, the stable pressure during crystallized zeolite in Teflon-lined autoclave was helped to grow the crystallite size better than polypropylene bottle.

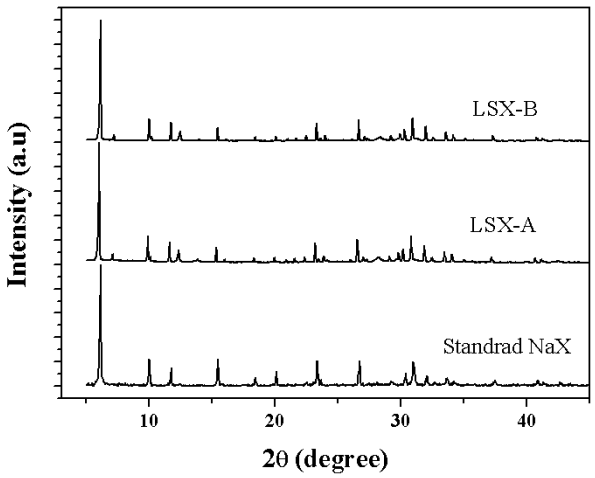


Fig. 1 XRD spectrum of zeolite LSX–A, LSX–B and standard NaX

In addition, both of LSX–A and LSX–B zeolite have similar composition with Si/Al ratio around 1.3 as show in Table II though the LSX–B has higher surface area than the LSX–A, it seem clear the container used for synthesis effect to the quality of zeolite [7], [8].

TABLE I
CRYSTALLITE SIZE AND SURFACE AREA OF ZEOLITE LSX-A AND LSX-B

Zeolite	Crystallite size (nm)	Surface area (m^2/g)
LSX-A	99.26	239
LSX-B	102.88	286

TABLE II
COMPOSITION OF ZEOLITE LSX-A AND LSX-B

Composition	LSX-A (%)	LSX-B (%)
O	48.49	53.69
Na	9.53	7.29
Al	16.73	15.11
Si	22.67	21.76
K	2.58	2.15

Fig. 2 shows SEM images of LSX–A and LSX–B at different magnification. There are two types of particle shape consisted of round amorphous and multi-faceted spherulite crystals. The both of them have mixing particle shape. However, the particle size of LSX–B was larger than LSX–A, corresponding with XRD results [7].

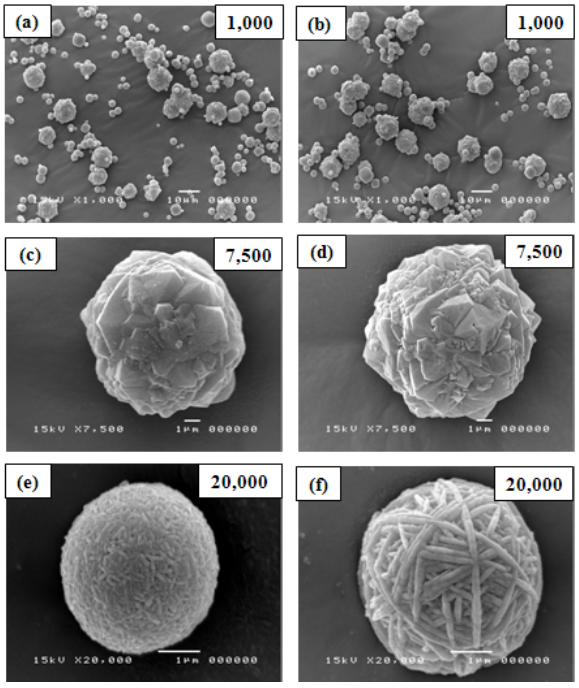


Fig. 2 SEM images of zeolite LSX–A and LSX–B at different magnification

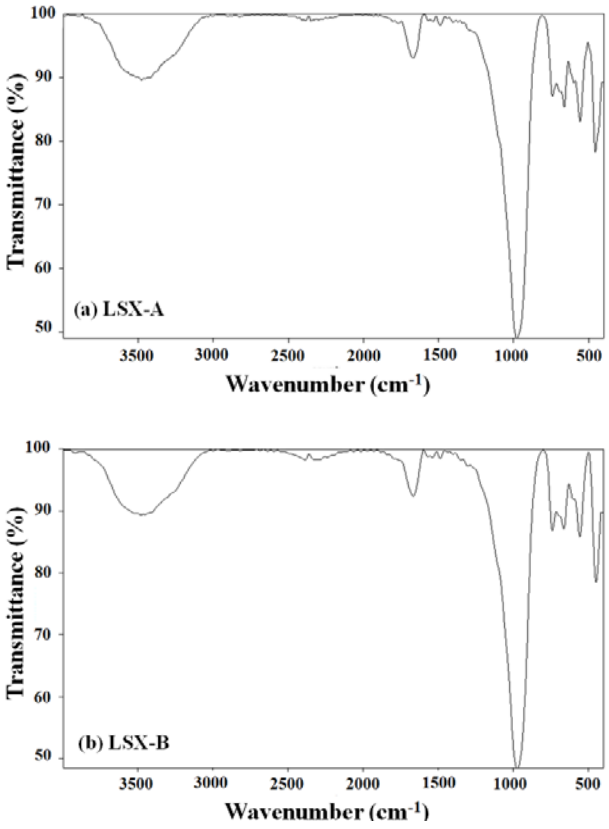


Fig. 3 The FTIR spectra of zeolite LSX–A and LSX–B

The FT–IR spectra of zeolite LSX–A and LSX–B are showed in Fig 3. The presence of transmission bands at 3,478 cm^{-1} and 1630 cm^{-1} were the vibration of –OH stretching and $–OH_2$ bending of adsorbed water molecule. The strong peak at 950 cm^{-1} was corresponded to Si–O stretching or Al–O stretching in the tetrahedral framework of zeoilte.

Moreover, the bands appear lower than 700 cm^{-1} referred to finger print region which was the vibration of symmetric stretching double ring and Si–O or Al–O bending in tetrahedral framework that are characteristic of FAU zeolite [7]–[10].

IV. Conclusion

The type of crystallized container has effect to the surface area and the particle size of zeolite LSX. Moreover, the enhancement of zeolite LSX quality was obtained in Teflon-lined autoclave.

Acknowledgment

This work was financially supported by Research and Development of Suan Sunandha Rajabhat University, Bangkok, Thailand.

References

[1] S. Chiarakorn, T. Areerob and N. Grisdanurak "Influence of functional silanes on hydrophobicity of MCM–41 synthesized from rice husk" *Sci. Technol. Adv. Mater.*, vol. 8, Jan. 2007 pp. 110–115

[2] M. M. Mohamed, I. F. Zidan and M. Thabet. "Synthesis of ZSM-5 zeolite from rice husk ash: Characterization and implications for photocatalytic degradation catalysts" *Micropor. Mesopor. Mat.*, vol. 108, Feb 2008, pp.193–203.

[3] K. Kordatos, S. Gavela, A. Ntziouni, N. K. Pistiolas, A. Kyritsi and V. Kasselouri-Rigopoulou "Synthesis of highly siliceous ZSM-5 zeolite using silica from rice husk ash" *Micropor. Mesopor. Mat.*, vol. 115, Oct 2008, pp.189–196.

[4] M. M. Rahman, N. Hasnida and N. Wan "Preparation of Zeolite Y Using Local Raw Material Rice Husk as a Silica Source" *J. Sci. Res.*, vol. 2, Dec. 2009, pp. 285–291.

[5] J. Wittayakun, P. Khemthong and S. Prayoonpokrach, "Synthesis and characterization of zeolite Y from rice husk silica" *Korean J. Chem. Eng.*, vol. 25, Nov. 2008, pp. 861–864.

[6] S. Li, A. V. Tuan, J. L. Falconer and R. D. Noble. "X-type zeolite membranes: preparation, characterization, and pervaporation performance" *Micropor. Mesopor. Mat.*, vol. 53, Jun. 2002, pp. 59–70.

[7] P. Khemthong, S. Prayoonpokarach and J. Wittayakun, "Synthesis and characterization of zeolite LSX from rice husk" *Suranaree J. Sci. Technol.*, vol 14, Aug. 2007, pp. 367–379.

[8] D. Chen, X. Hu, L. Shi, Q. Cui, H. Wang and H. Yao "Synthesis and characterization of zeolite X from lithium slag" *Appl. Clay. Sci.*, vol. 59–60, May. 2012, pp. 148–151.

[9] C. S. Cundy and P. A. Cox. "The hydrothermal synthesis of zeolites: Precursors, intermediates and reaction mechanism" *Micropor. Mesopor. Mat*. vol. 82, Aug 2005, pp. 1–78.

[10] S. Sang, Z. Liu, P. Tian, Z. Liu, L. Qu and Y. Zhang, "Synthesis of small crystals zeolite NaY" *Mater. Lett*. vol. 60, Nov. 2006, pp.1131–1133.

Jitlada Chumee was born on March 30, 1981 in Ubonratchathani province, Thailand. Chumee received her Doctoral's Degree in Chemistry from Suranaree University of Technology, Thailand in 2008. After graduation, Chumee studied postdoctoral research at Thammasat University for 1 year.

Research areas are heterogeneous catalysis of supported transition metal (monometallic and bimetallic) and synthesis and utilization of porous material as adsorbent and catalyst support.

At the moment, she works in position of lecturer at Department of Chemistry Faculty of Science and Technology, Suan Sunandha Rajabhat University, Bangkok, Thailand.

Open Science Index, Chemical and Molecular Engineering Vol:7, No:1, 2013 publications.waset.org/5904/pdf