

Effects of Hydrothermal Duration on Synthesized Zeolite Rice Husk Particles

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Abstract

Zeolite particles were synthesized through in-situ extraction of silica from rice husk ash without seeding method in the absence of organic template by a static hydrothermal condition at various duration 8-24h. The effect of amorphous silica content on hydrothermal duration of synthesis products was evaluated with X-ray diffraction (XRD), and field emission microscopy (FESEM). The present of zeolite Y without seeding method via XRD analysis show a mixture of zeolite A, Y and P. The transformation of amorphous rice husk particle was increased gradually with the increased of hydrothermal reaction. The FESEM images showed cubic-shaped morphology of the powders increased with increase in ageing reaction.

Keywords: Rice husk ash, zeolite Y, hydrothermal.

Introduction

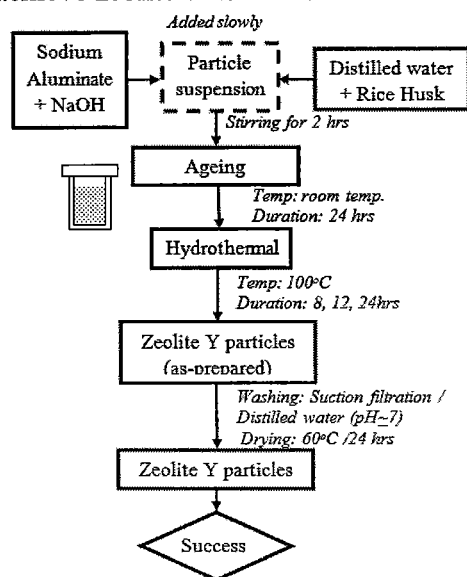
Rice husk (RH) is one of the most abundance agricultural wastes in Malaysia. Exploring value materials from this abundance waste materials i.e zeolite and silica is a potential research that needs to be discovered. Basically, RHA is an active catalyst and good material for catalyst support due to its high surface area properties¹ and can be used as an alternative source for zeolite preparation. Extensive study of zeolite synthesization from RH for separation processes such as sorbent, as well petrochemical processes is widely explored and studied.² Theoretically, the formation of zeolite under hydrothermal condition is very complex process.³ It is strongly influenced by its processing parameters such as agitation time and temperature⁴, ageing time and temperature⁵, crystallization time and temperature⁶. As for example several studies have proved that with increasing

hydrothermal duration and temperature⁷ the formation of zeolite also increased. Thus, in this present work, this hydrothermal technique was addressed in synthesizing zeolite Y powders via static hydrothermal. Omitting the intermediate reaction steps for extraction of silica from RHA is a futher improvement in this present work to provide a simple and easy steps in formation of zeolite phases.

Experimental Method

Preparation of synthesized RH

RH ash, pellets of sodium hydroxide (NaOH), Sodium aluminate (NaAl_2O_3), and distilled water were used as the starting materials in the initial mixture of the synthesis of Na-Y zeolite. This RH source used in this experiments was obtained from Malaysia rice mill (Jelapang Selatan). The process involved burning of RH at 700°C for 5h in atmosphere. All these material was undergone hydrothermal process in order to achieve zeolite Y formation.



Zeolite analysis. The crystal phases of the powder were identified using powder

diffraction technique by Bruker XRD (Model: D8 Advance) with Ni-filtered Cu- K_{α} radiation ($\lambda=0.15418\text{nm}$), operating at 40kV and 40mA. The morphology of the synthesized particles was examined by FESEM (Model: JEOL JSM 7600F) operating with an accelerating voltage of 1kV.

Results & discussion

As shown in fig 1, the formation of zeolite Y show an increase of intensity peak as the hydrothermal duration is increased. The transformation metastable zeolite from amorphous RHA to crystalline phases was clearly observed as increasing the hydrothermal reaction. Microstructure observation also shows that the formation of Zeolite Y to a complete crystalline structure also increases with the increase hydrothermal time. This can be proved with FESEM analysis, the image of cubic shape growth is obviously shown as the hydrothermal time increases. The formation This zeolite Y formation was referred to JCPDS number 38-0240 data by matching the diffractograms pattern.

This figure also demonstrated that at all duration of hydrothermal, the synthesized zeolite Y is in the form of mixture zeolite A, Y and P. This metastable zeolite Y mixture is easily transform to another phases with small changes of the influenced parameter. As can be seen in Fig 1, some of the crystallites were transformed to zeolite P⁸ (with JCPDS number 39-0219) and Zeolite type A (with JCPDS number 40-1646) due to excessive water medium in the mixture compound. According to Turnbull⁹ zeolite A, Y and P have the structure of faujasite where all of these phases are naturally exist as mineral, and are commonly denoted as FAU. The FESEM microstructure of RH zeolite particles obtained at 100°C/24 hr with magnification of 10,000 show that cubic shape particles of NaY was in agglomeration condition.

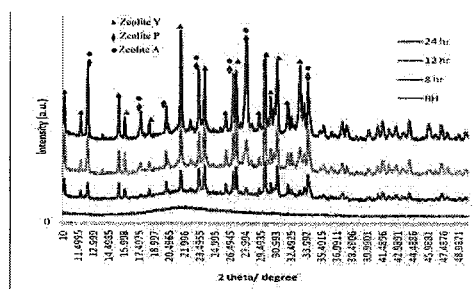


Figure 1 : Zeolite phases formation at different hydrothermal duration.

Conclusion.

In this present study, the zeolite-Y powder was synthesized by static hydrothermal condition by using of rice husk ash as silica source at 100°C, at various hydrothermal duration within 8, 12 and 24 hr. X-ray diffraction peak and microstructure observation revealed that the formation of Zeolite Y was increased as extended the hydrothermal. As proved with FESEM analysis, the image of cubic shape growth is clearly seen with longer hydrothermal time. Preparations of zeolite Y without seeding method also give a mixture of metastable zeolite Y, A and P.

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