MICROWAVE IRRADIATION TECHNIQUE FOR SYNTHESIS OF ZEOLITE A

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Abstract

Synthesization of Zeolite A from colloidal silica was performed by means of microwave irradiation technique under different conditions and was compared with hydrothermal technique. The molar composition at 1 Al_2O_3: 1.96 SiO_2: 3.165 Na_2O: 128 H_2O. X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM) were used to characterize zeolite A. XRD results for all samples demonstrated a typical diffraction peak of zeolite A. SEM images show almost no-different in crystal size of zeolite A synthesized by microwave irradiation and hydrothermal technique. Thus, we concluded that crystallization of zeolite A has occurred rapidly by microwave irradiation. Microwave technique is a time and energy saving due to crystallization time and higher heating rate as compared to hydrothermal technique.

Keywords: Zeolite A, Microwave irradiation technique, hydrothermal technique.

1.0 Introduction

Applications of microwave energy in the synthesis of inorganic materials have been exploited since the mid-1980s (Mingos, 1993). Nowadays numerous papers and patents have been published on the argument and in several conventional synthesis routes; successful use of microwaves has been proved (Mingos, 1994). Microwave irradiation is more efficient for transferring thermal energy to a volume of material than conventional thermal processing which transfers heat through the surfaces of the material by convection, conduction and radiation. The oscillating electromagnetic field, which is generated by microwaves, interacts with the dielectric properties of materials leading to rotation of molecular dipoles and subsequent energy dissipation as heat from internal resistance to that rotation (Kingston, et al., 1997).

In zeolite synthesis, the occurrence of hydrothermal conditions can be considered particularly favorable to the use of microwave energy because water, present as solvent in the reaction mixture, is very receptive to commonly used microwave frequencies. A number of publications have recently appeared on microwave zeolite synthesis to reduce

crystallization time by rapid heating of the reaction mixture. According to various authors (Rao, et al., 1999), this reduction of time was a consequence of two main effects ascribed to microwaves: a relatively fast dissolution of the gel and a rapid homogeneous heating of the synthesis mixture that lead to a more abundant nucleation. Preparation of zeolite NaA, zeolite Y and ZSM-5 has been performed with reduction in crystallization time from several hours to few minutes by combining pressure with microwave heating in suitable autoclaves (Ocelli and Robinson, 1992). In the first reported attempt of a microwave synthesis of zeolite A (Chu, et al, 1988), crystallization of zeolite was obtained in 12 min but the product was contaminated with hydroxysodalite (HS), even if the mixture was aged for 2 h at room temperature before microwave heating. Slangen, et al. (1997) was able to obtain pure zeolite A in 5 min crystallization, but after 20 h of ageing at room temperature.

In the present work, zeolite A has been synthesized from colloidal silica under microwave technique and compared with hydrothermal technique. To the best of our knowledge, zeolite A was first time synthesized from colloidal silica by using microwave technique and has never been reported before.

2.0 Experimental procedures

2.1 Equipments

Microwave equipment used in the experimental work consisted of a household type microwave oven with a working frequency of 2.45 GHz *(Panasonic NN-MX26WF)* and an output power that can be varied from 180, 360, 550W to a maximum of 800 W. The microwave oven was modified by the introduction of a thermocouple inside the cavity, in order to monitor reaction temperature. Atmospheric pressure was chosen because atmospheric hydrothermal synthesis is the common method for manufacturing NaA zeolite on a commercial scale. Teflon vessel was used as a reaction vessel and located into microwave.

2.2 Experimental

Synthesis mixtures had the following molar ratio: 1 Al_2O_3: 1.96 SiO_2: 3.165 Na_2O: 128 H_2O. Sodium aluminate, NaOH and distilled water were mixed together and the solution was boiled until it was clear. Once the solution had cleared up, the colloidal silica and NaOH was slowly added under stirring and the mixture stirred for one hour at 95°C. The prepared solution with the substrate was transferred into Teflon vessel and placed into microwave to get radiation for 10min. The substrate was filtered until pH <9 in order to eliminate any impurities and excessiveness of NaOH and let dried for 25min at 110°C. Synthesis of zeolite A by hydrothermal technique was done with the same procedure as mentioned before. The prepared solution with the substrate were placed in the oven for treatment at 110°C for 4h and let dried overnight. The products were analyzed by X-Ray diffraction (XRD) and Scanning Electron Microscopy (SEM) to determine the identity, crystal size distribution and morphology of zeolite A.

3.0 Results and Discussion

3.1 Effect of Preheating

Fig.1 shows the X-Ray diffraction pattern of the substrate synthesized from different radiation time under microwave irradiation and different in synthesized temperature. It can be seen that after 10min, the typical diffraction peaks of zeolite A crystals appeared, suggesting the formation of zeolite A crystals on the surface of the substrate. These results imply with samples synthesized at 95°C with 10min radiation in microwave ($A_{95°C/10}$) and room temperature with 10min radiation in microwave ($A_{RT/10}$). In contrast, the reaction at room temperature with 5min radiation in microwave ($A_{RT/5}$) indicates that no peak corresponding to zeolite A.

Fig 2 displays the SEM image of zeolite A synthesized under microwave irradiation under different temperature. It can be seen that after 10min, there was no big variation of crystal size indicate to both samples, with zeolite A crystal size between 2-5μm for sample $A_{95°/10}$ and 2-3μm for sample $A_{RT/10}$. This suggests that during formation of crystal in microwave technique, the ellipsoidal morphology of zeolite A may be due to the collision of nuclei at the genesis of crystal formation (eeeeeee).

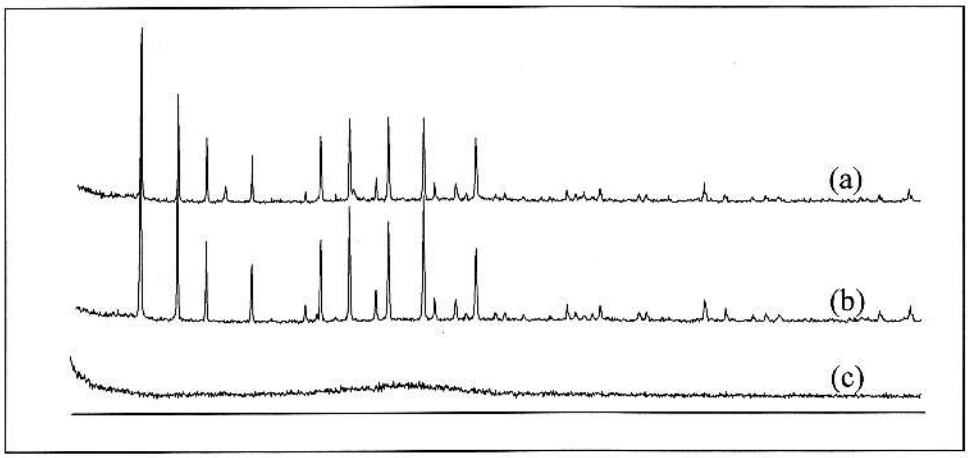
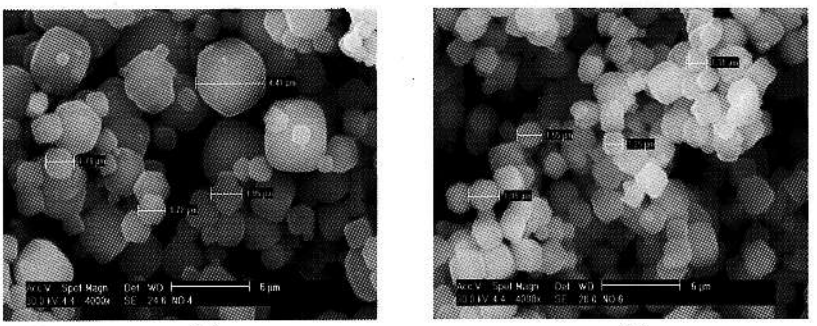


Fig. 1: XRD patterns of zeolite A synthesized with a) $A_{95°C/10}$; b) $A_{RT/10}$; c) $A_{RT/5}$



(a) (b)

Fig. 2: SEM analysis of zeolite A synthesized with a) $A_{95°C/10}$; b) $A_{RT/10}$; c) $A_{RT/5}$

3.2 Effect of Synthesis Method

Fig. 3 shows the X-Ray diffraction pattern of the zeolite A synthesized under microwave irradiation ($A_{95°/10}$) and hydrothermal technique ($A_{95°C/4}$). The intensity of diffraction peak of both samples was similar and consist the typical peak for zeolite A. Thus, it proved that microwave irradiation technique had advantage compared to hydrothermal technique where the reaction occurred in a shorter time to produce zeolite A.

Fig. 4 illustrates the scanning electron microscope of zeolite A samples synthesized by both microwave and hydrothermal. SEM images show almost no-different in crystal size of zeolite A synthesized by microwave irradiation and hydrothermal technique. Thus, the crystallization of zeolite A has occurred rapidly by microwave irradiation .

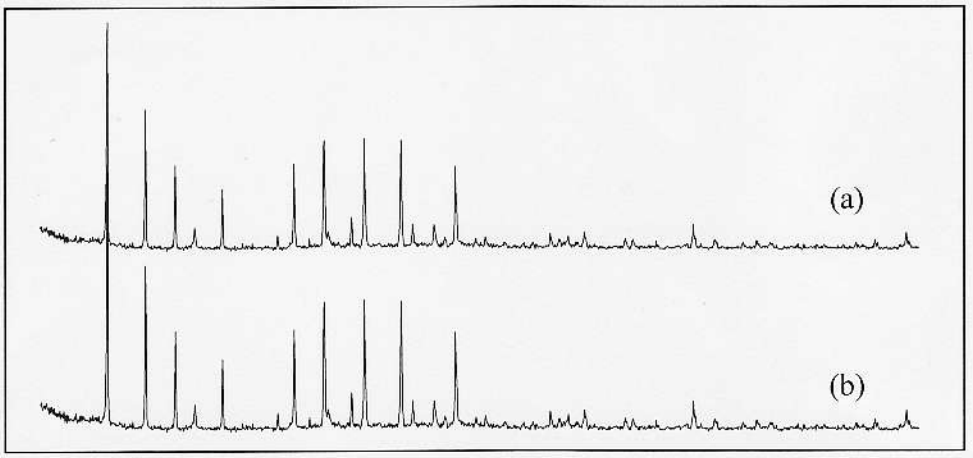
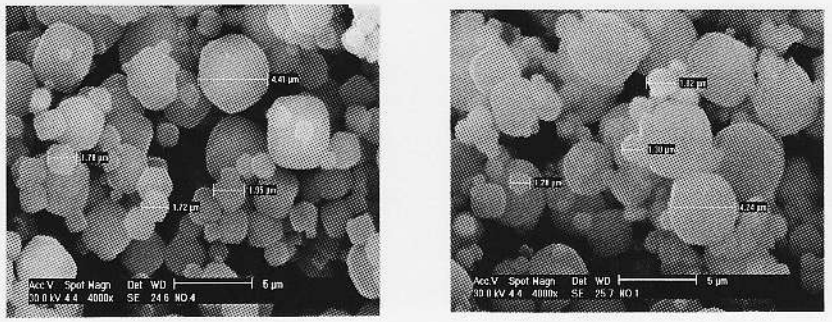


Fig. 3: XRD patterns of zeolite A synthesized with a) $A_{95°C/10}$; b) $A_{95°C/4}$



(a) (b)

Fig. 4: SEM analysis of zeolite A synthesized with a) $A_{95°C/10}$; b) $A_{RT/10}$

4.0 Conclusion

The capability of microwave irradiation to speed up zeolite crystallization has been already demonstrated by numerous authors. Zeolite A from colloidal silica has been synthesized for the first time by microwave irridation. X-ray diffraction pattern shows the similar diffraction peak for each sample; $A_{95°/10}$, $A_{RT/10}$ and $A_{110°C/4}$.

SEM images did not show any big variation in crystal size of each sample. This proved that microwave technique is time and energy saving due to the crystallization time and higher heating rate compared to hydrothermal technique. As a conclusion, microwave irradiation has a potential to use in synthesized zeolite A from colloidal silica.

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