Zeolite X Synthesis with Different Sources

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***Abstract*—**Zeolite X was synthesis by using different aluminum (alumina, sodium aluminate) and silica source (sodium silicate, sodium metasilicate pentahydrate), sodium hydroxide and distilled water. Sodium silicate solution and a sodium aluminate solution were prepared separately. Before the preparation, the amount of raw materials which used in resulting hydrogel with appropriate mole ratio was calculated. Aging time was fixed at 2 hour. Hydrothermal treatment temperature was kept at 100 °C for 4 hour. The product was characterized by XRD, SEM and FT-IR techniques. The crystal structure of the product was determined as zeolite X by XRD. The morphology of SEM image for zeolite X is octahedron shape and FT-IR bands are in accordance with the other characterization results.

***Keywords*—**Crystallization, Faujasite, ydrothermal, Zeolite X

I. Introduction

ZEOLITES are crystalline microporous aluminosilicates consisting of tetrahedral units producing open framework structures (Fig. 1); which generates a system of pores and cavities having molecular dimensions [1].

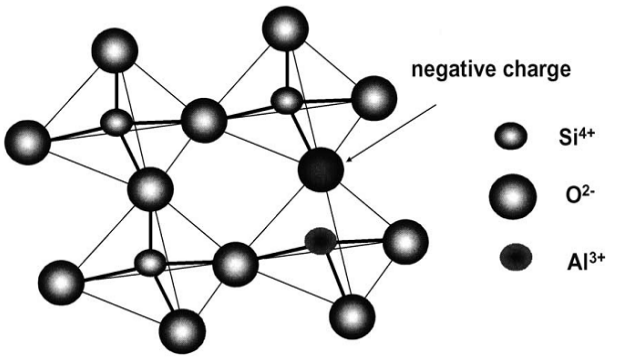

Fig. 1. Idealized structure of zeolite framework of tetrahedral $[SiO_4]_4$ with a Si/Al substitution ($[AlO_4]_5$) yielding a negative charge [2]

Naturals zeolites are mined in many parts of the world, but most of zeolites used at industrial level, are synthesized in alkaline mediums with different sources of silica and aluminum components [3]. Zeolites find wide applications in different areas as waste treatment [4], gas purification [5], [6] construction industry [7], etc.

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There are 46 known zeolite minerals in nature and over 150 synthetic zeolites known in the literature and the number of synthetic zeolites is increasing day by day. The history of man-made zeolites can be traced back to the claimed laboratory preparation of apophyllitte by Wöhler in 1848. However, zeolite technology was initiated during the late 1940s on a large scale, essentially by the groups of Barrer and Milton. They developed hydrothermal synthesis using reactive alkali-metal aluminosilicate gels at low temperatures (~100°C) and pressure (autogenous). By 1958, under the leadership of Milton, the Linde Division of Union Carbide had successfully synthesized nearly all the commercially important zeolites such as Faujasite-type zeolites (zeolite X) (Fig. 2) [8]. In 1962, only 10 years after, these synthetic Faujasite-type zeolites were for the first time used as catalysts and had induced a deep revolution in petroleum refinery and petrochemical industry by the strong improvement of the efficiency of the existing processes [9].

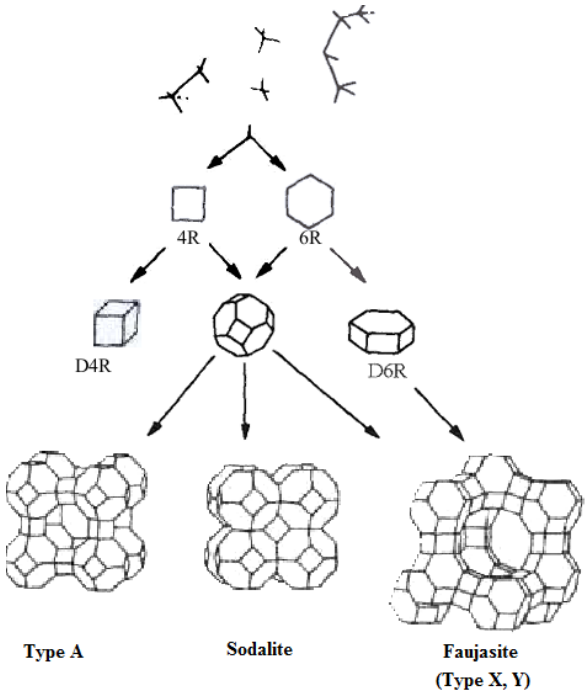


Fig. 2. Three different zeolites [8]

Zeolite X is a very attractive material for technological and environmental applications. Wide micropores make it useful for purification and separation of gases and organic components; high exchange capacity allows for adsorption of heavy cations and radionuclides (10).

II. Materials and Methods

A. Materials

The zeolite synthesis gels used in this study were prepared from sodium aluminate ($NaAlO_2$), alumina, sodium silicate (Na_2SiO_3), sodium metasilicate pentahydrate ($Na_2SiO_3.5H_2O$), NaOH and deionised water. Commercial zeolite X was provided from Ceca Specialty Chemicals Inc., France.

B. Zeolite X synthesis

Prepared sodium silicate solution was introduced into the sodium aluminate solution and the resulting mixture was homogenized at room temperature for 2 hours. The reaction mixture was charged into the autoclave for hydrothermal reaction (100 °C, 4 h).

3 different experiments involved different sodium sources and aluminum sources, were investigated that were given in

TABLE I
SILICA AND ALUMINUM SOURCES FOR ZEOLITE X SYNTHESIS

	Ex.1	Ex.2	Ex.3
Silicasource	$Na_2SiO_3.5H_2O$	$Na_2SiO_3.5H_2O$	Na_2SiO_3
Aluminum source	Al_2O_3	$NaAlO_2$	$NaAlO_2$

The mechanism is illustrated schematically in Fig. 3. The elements (Si, Al) which will make up the microporous framework are imported in an oxide form. These oxidic and usually amorphous precursors contain Si-O and Al-O bonds. During the hydrothermal reaction in the presence of a ‘‘mineralising’’ agent (most commonly an alkali metal hydroxide), the crystalline zeolite product containing Si-O-Al linkages is created [11].

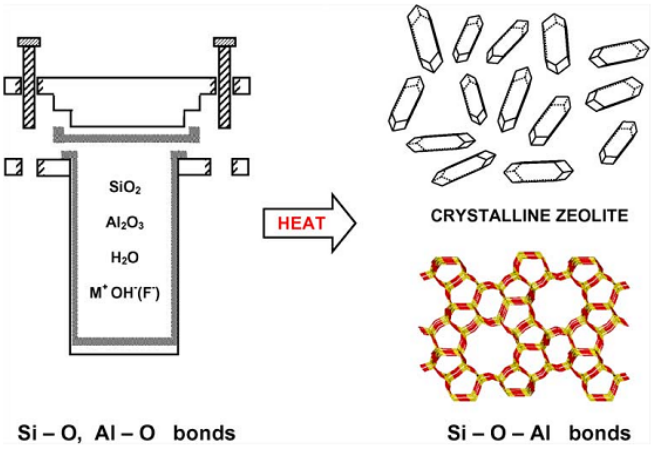


Fig. 3 Hydrothermal zeolite synthesis [11]

After completing crystallization, the resultant precipitate was separated from the mother liquor by filtration. The crystallized zeolite was then washed with deionized water until a pH 10 and dried at 105 °C for 12 hours.

C. Characterization of the product

The particle morphology was observed by Camscan Apollo 300 scanning electron microscopy (SEM).

The crystalline phases were identified by XRD (Philips PanAnalitical, X’pert Pro). Operating conditions involved the use of CuKα radiation at 45 kV and 40 mA. The samples were scanned from 3-50° (2θ).

Infrared spectroscopic analysis of different materials was carried out to study their structural features. Infrared spectroscopic analysis of the prepared zeolite samples was performed with a Perkin Elmer (Spectrum One) spectrometer and the samples were prepared as KBr pellets for wave number range of 400-4000 cm^{-1}.

III. Results and Discussion

Zeolite X was prepared different sodium and aluminum sources (Table 1). Fig. 4 shows the XRD pattern of synthesized zeolites and commercial zeolite X. It is evident from the XRD patterns that Ex. 1 and Ex.2 are comparable to the commercial zeolite X. But Ex. 3 is has an amorphous phase. So it is not crystallized as zeolite X. The “d” spacings for the synthesized zeolite compare well with the reported values.

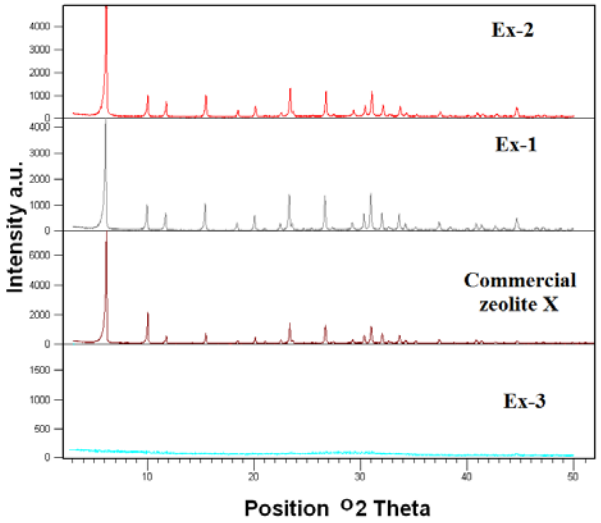
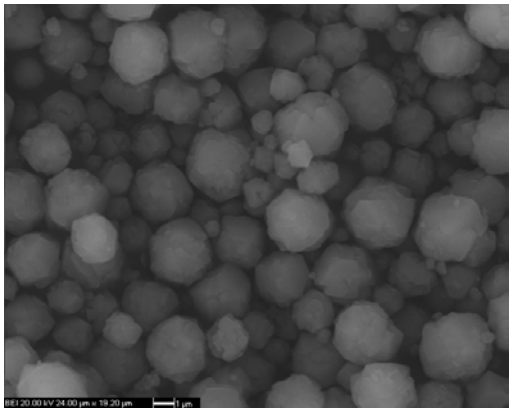
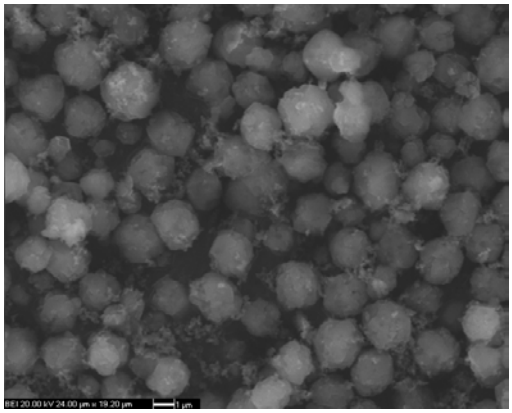


Fig. 4. XRD pattern of synthesis zeolite X and commercial product

The crystal structure of the synthesized zeolite was determined to be octahedral crystals (Fig. 5). But it is seen in the Fig. 5 that Ex-3 has an amorphous structure.



a



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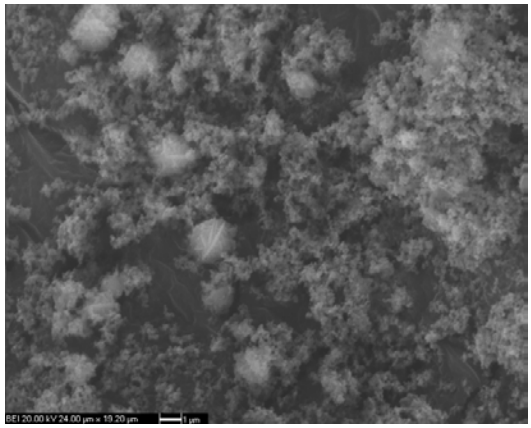

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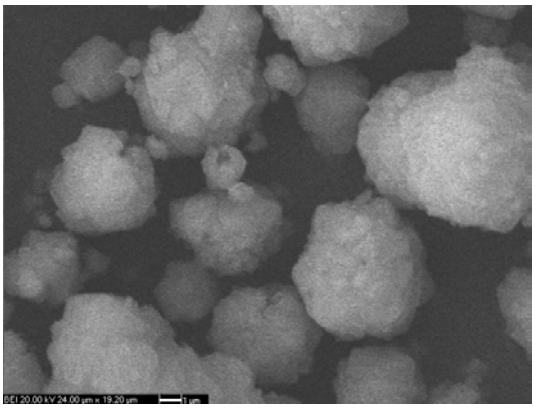

d

Fig. 5 Scanning electron micrographs of (a) Ex-1, (b) Ex-2, (c) Ex-3, (d) Commercial zeolite X

In general, each zeolite has a characteristic infrared pattern. However, some common features are observed, which include the asymmetric and symmetric stretch, double ring vibrations, T–O bending modes, and possibly pore opening modes.

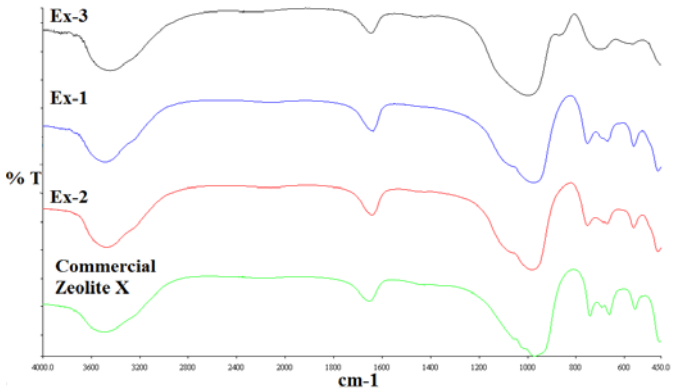

Fig. 6. FT-IR spectra of synthesis zeolites and commercial zeolite X

The IR spectral data for Na–X type zeolites from the literature that of the synthesized zeolite are presented in Table 2. The results

TABLE II
INFRARED SPECTRAL DATA FOR ZEOLITE X

Parameter	Wave number (cm^{-1})			
	Zeolite X	Ex-1	Ex-2	Ex-3
Double ring	560	563.45	563.58	564.34
Asymmetric stretching	1060, 971, 746	979.17, 753.89	982.21, 754.13	995.92 700.02
Symmetric stretching	668 690	671.15	674.19	-
T-O bending	458	463.52	464.01	-

IV. CONCLUSION

Zeolite X was obtained with different aluminum and silica source with octahedral crystal structure, confirmed by XRD patterns and scanning electron micrographs, and FT–IR studies. The results indicate that raw material sources are effected the structure of the product.

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