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SYNTHESIS OF A SPECIAL TYPE OF ZEOLITE FROM ANALCIME FAMILY WITH TRIETHANOLAMINE AND ITS CHARACTERIZATION WITH XRD, FTIR AND SEM

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Abstract

A type of zeolite from the analcime family was prepared by using triethanolamine, silica gel and sodium aluminate. The average particle size was around 24 μm. FTIR, XRD and SEM results approved the type of zeolite which was formed.

Introduction

During the last two decades, different methods have been used in the synthesis of zeolites. Chemists are still working on the synthesis of new types of them.

The use of organic bases in hydrothermal crystallization was initiated in 1961 [1-5]. The range of materials that can be used as organic materials in the synthesis of high silica is not restricted to positively charged species. For example, instead of quaternary ammonium ions [5], primary and secondary amines, diamines, alcohols, polyamines, salts of organic acids and many other representatives of a wide variety of functional groups have all been shown to be effective [6-10].

In our studies, triethanolamine (TEA) was used as a base for the synthesis of a zeolite type compound and then the prepared zeolite was characterized by FTIR, XRD and SEM.

Experimental Section

Instrumentation

X-ray diffraction spectra of synthesized zeolite was obtained by X-ray diffraction (Philips, PW 1840, with cu tube). The micrograph of zeolite was taken with a scanning electron microscope (NIOC, SEM 360). FT-IR (KBr) spectrum was recorded using a Philips PU-9800 FT-IR spectrophotometer.

Keywords: Analcime; Zeolite; Triethanolamine

Preparation

Analcime type of zeolite with triethanolamine (TEA) was prepared using sodium aluminate, silica gel and TEA. The ratios were chosen as: SiO_2/Al_2O_3=2.7, Na_2O/Al_2O_3= 1.49, TEA/Al_2O_3= 5.5 and H_2O/Al_2O_3= 120.

A solution of sodium aluminate was slowly added to the mixture of silica gel and TEA. The reaction mixture was stirred for five to seven days at ambient temperature. It was then kept hydrothermally in autoclave at 150°C for 48 hours.

After crystallization, filtration and washing with distilled water up to pH 9, a crystalline zeolite was obtained. Chemical analysis showed: SiO_2, 54.72%; Al_2O_3, 20.48%; Na_2O, 13.4%; H_2O, 10.47%; N<0.1; C, 1.2%.

Results and Discussion

The type of our synthetic zeolite was found to be analcime on the basis of XRD (Fig. 1 and Table I) FT-IR spectrum and chemical analysis which showed the

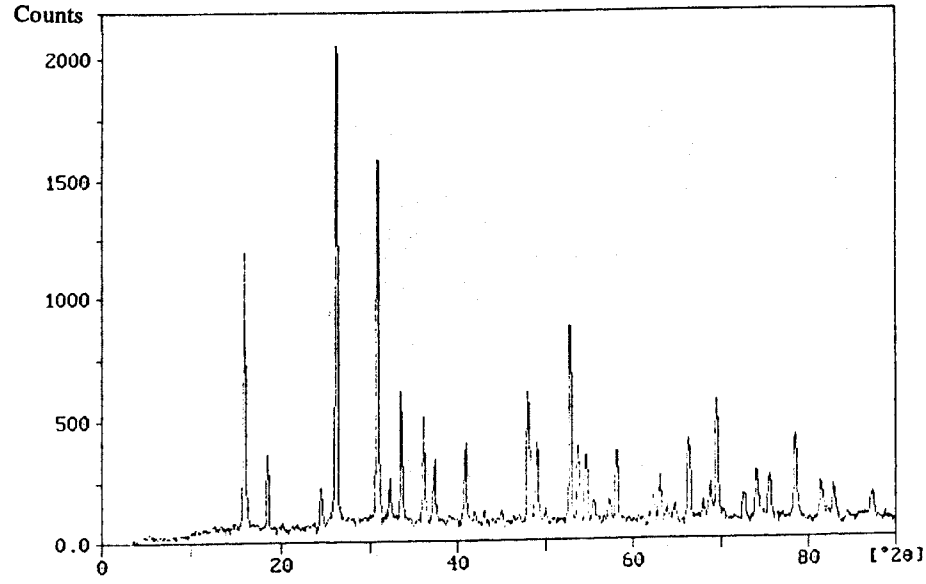

Figure 1. The XRD spectrum of the prepared zeolite (anal cime)

Table I. The XRD data of the prepared zeolite (analcime)

Peak No.	$d_{exp.}$	Rel.int %
1	5.5004	57.3
2	4.772	15.3
3	3.6222	8.54
4	3.4003	100.0
5	2.8969	76.5
6	2.7717	9.96
7	2.6649	27.76
8	2.4839	22.78
9	2.4087	13.88
10	2.2087	27.04
11	1.8836	27.40
12	1.8549	17.081
13	1.7323	40.57
14	1.7053	16.014
15	1.6788	14.24
16	1.6559	4.63
17	1.6082	4.91
18	1.5883	14.95
19	1.4894	5.34
20	1.4738	9.96
21	1.4079	17.44
22	1.3655	8.19
23	1.3524	24.91
24	1.2995	5.69
25	1.2799	10.68
26	1.2581	9.96
27	1.2177	17.79
28	1.1803	8.19
29	1.1631	7.47
30	1.1159	5.69

empirical formula: $Na_2O.\ Al_2O_3.\ 4\ SiO_2.\ 2H_2O$.

A systematic investigation of the framework structures of many synthetic zeolites has been carried out in the 200-1300 cm^{-1} region [11]. According to Table II, a strong vibration mode at 1027.3 cm^{-1} is due to the internal tetrahedron vibration (T-0 stretching) and a peak at 441.4 cm^{-1} is assigned to its bending. Two bands in the region 766.76 and 737.3 cm^{-1} are related to the presence of double (D4R) rings in the framework structures or as external linkages due to symmetric stretching of tetrahedral atoms. It should be mentioned that the pattern in the region 800-200 cm^{-1} agrees exactly with the analcime-type zeolite [11].

The particle size of the prepared zeolite was 20.8 to 28.3 μm (average size ~24.0 μm), which on the basis of scanning electron microscopy photograph (Fig. 2), is the biggest synthetic zeolite of analcime which has been prepared to our knowledge [12, 13]. Alkanolamines have been used recently for the preparation of ZSM-5 producing particles with a size of 0.3 μm [14].

Table II. Vibrational spectra of the prepared zeolite (analcime) Obsd freq. cm^{-1} and rel intense

V	%T
1027.316	20.158
766.775	57.069
737.312	57.961
625.351	68.074
441.415	35.222

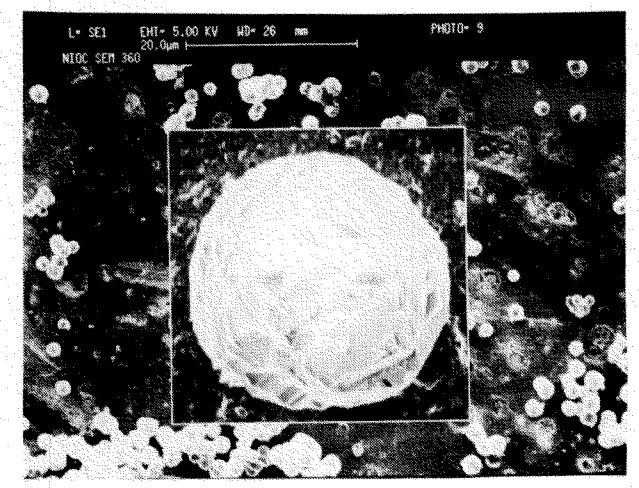

Figure 2. Scanning electron micrographs of analcime sample produced by using TEN

Thermal decomposition patterns from 25-750°C gave two distinct weight losses: 25-300°C and 500-550°C. The first loss was due to the desorption of water (~10.5%), the second loss was from the decomposition of TEA (less than 2%). The essential role of TEA is to gather the reacting species for the formation of zeolite.

It was also found that amorphous aluminosilicate compound could be obtained in the absence of TEA or using the other amines under these conditions.

Acknowledgements

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