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Short Communication

Nano-sized $AlPO_4$-5 Crystals: Synthesis and characterization

A. Hassanvand
M. Asghari *

Separation Processes Research Group (SPRG), Department of Engineering, University of Kashan, Kashan, Iran

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* Corresponding author:
Morteza Asghari
Separation Processes Research Group (SPRG), Department of Engineering, University of Kashan, Kashan, Iran
Tel +98361 591 2427
Fax +98361 555 9930
Email asghari@kashanu.ac.ir

ABSTRACT

Molecular sieves and zeolites are materials whose crystalline frameworks form nanometer or subnanometer pores. A variety of different crystal structures are known having a range of pore sizes. Because the pore sizes are usually smaller than 2 nm, they are classified as microporous materials. Synthesis of microporous materials is usually conducted by the high temperature treatment (80-200 °C) of aqueous synthesis gels. The process of heating aqueous mixtures to elevated temperatures for crystallization is typically described as hydrothermal synthesis. A hydrothermal treatment has been used to synthesize nano-sized $AlPO_4$-5 (AFI) crystals. The treatment involved three main steps: (1) to prepare a synthesis gel and let it get well mixed, then filling autoclave with the gel and sealing it; (2) crystallization at 180 °C for 6 h; and (3) Calcination at 600 °C for 4 h to separate template materials. The crystals have been structurally characterized using X-ray diffraction (XRD), scanning electron microscope (SEM), energy dispersive X-ray analysis (EDAX). The analyses show an acceptable fitness to the referred pattern. The SEM analysis and Scherer equation revealed that the size of the obtained $AlPO_4$-5 crystals was about 65 nm.

Keywords: *Nano-sized crystal; $AlPO_4$-5; Zeolite; Hydrothermal; Molecular sieve*

INTRODUCTION

Zeolites have crystalline structures with uniform, molecular-sized pores. These inorganic structures have been used extensively as catalysts and adsorbents. More recently, continuous polycrystalline zeolite layers have been deposited on porous supports and used as zeolite membranes [1]. $AlPO_4$-5 has a hexagonal open framework structure with a = 13.827 Å, b =13.827 Å, c =8.580 Å and α =90°, β =90°, γ = 120°.

The structure is composed of alternating alumina and phosphate tetrahedra with the main porosity along the [001] plane as seen in Figure 1. The pore size of $AlPO_4$-5 is 7.3 Å. The space group is P6/mcc (mass centered cubic), and the largest ring size is 12 atoms [2]. The typical unit cell composition is $|(C_{12}H_{28}N)\ (H_2O)x\ (OH)|\ [Al_{12}P_{12}O_{48}]$ [3].

In this study, we prepared nano-sized $AlPO_4$-5 crystals by aging process of gel and hydrothermal as synthesis method.

EXPERIMENTAL

Aluminum triisopropylate ($Al(C_3H_7O)_3$) and Orthophosphoric acid (H_3PO_4) have been used as sources of Al and P, respectively.

$AlPO_4$-5 was hydrothermally synthesized with different initial solutions with composition Al_2O_3 : 1.3 P_2O_5 : 1.6 TEA : 1.3 HF : 425 H_2O : 6 C_3H_7OH and conditions of hydrothermal crystallization were as follows:

The reactants were phosphoric acid (H_3PO_4 85 wt %), triethylamine (TEA, 99.5%) $(C_2H_5)_3N$ and hydrofluoric acid (HF, 40 wt%). First, H_3PO_4 was added in deionized water. Aluminum triisopropylate was given in a beaker containing mixture at 0°C then the mixture was stirred at room temperature for 2 hours to homogenize. TEA was added to the mixture dropwise into the mixture while stirring. In a parallel process, another solution was prepared by solving HF in deionized water. First solution was then added to the latter rapidly and left it to be homogenized by stirring for 2 hours at room temperature. The prepared gel was transferred to a Teflon-lined steel autoclave. Autoclave was placed into a preheated oven, maintaining at 180°C for 6 hours without agitation. After crystallization, the autoclave was cooled to room temperature. The product was obtained by Buchner vacuum filtration funnel, then washed with deionized water to gain pH under 10 and dried at 353 K overnight. The dried product calcined at 600°C for 4 hours (Figure 1).

RESULT AND DISCUSSION

Structural properties of the prepared $AlPO_4$-5 was characterized using XRD and SEM analysis. Figure 2 shows XRD pattern of the powder product, which corresponds to the XRD of standard sample. Using calculations done on the basis of XRD data in Scherrer (Eq. 1) equivalent particles size of the synthesized $AlPO_4$-5 was estimated as 65 nm.

$$D = 0.9\lambda / \beta_{sample} Cos(\theta) \qquad (1)$$

Figure 3 shows SEM images of the nano-sized $AlPO_4$-5. As shown, the images confirmed the nano-size of the $AlPO_4$-5 particles about 65 nm.

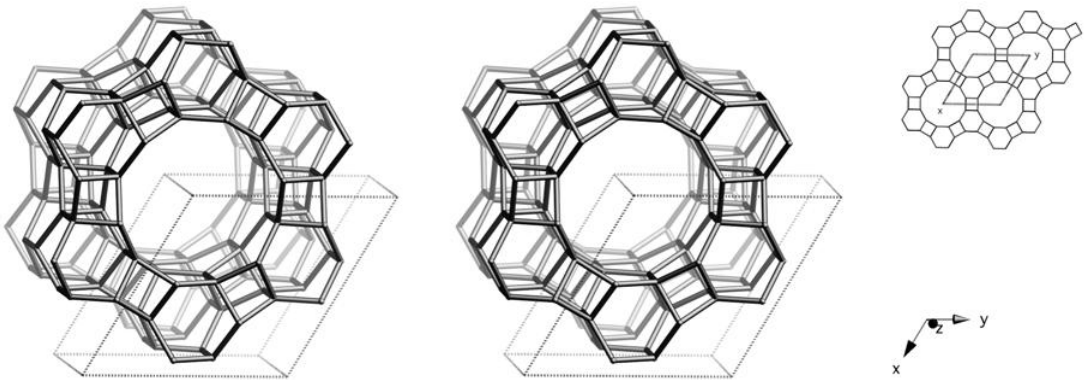

Fig.1. Framework viewed along [001](upper right: projection down [001]).

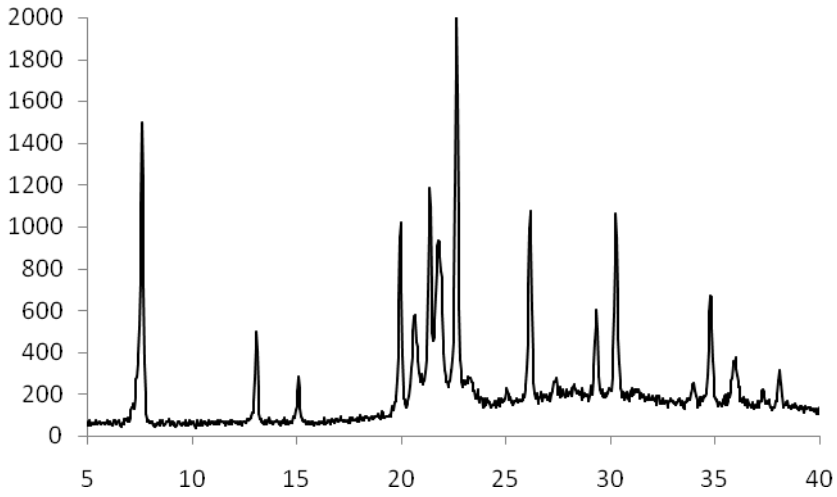

Fig.2. XRD pattern of the prepared $AlPO_4$-5 crystals

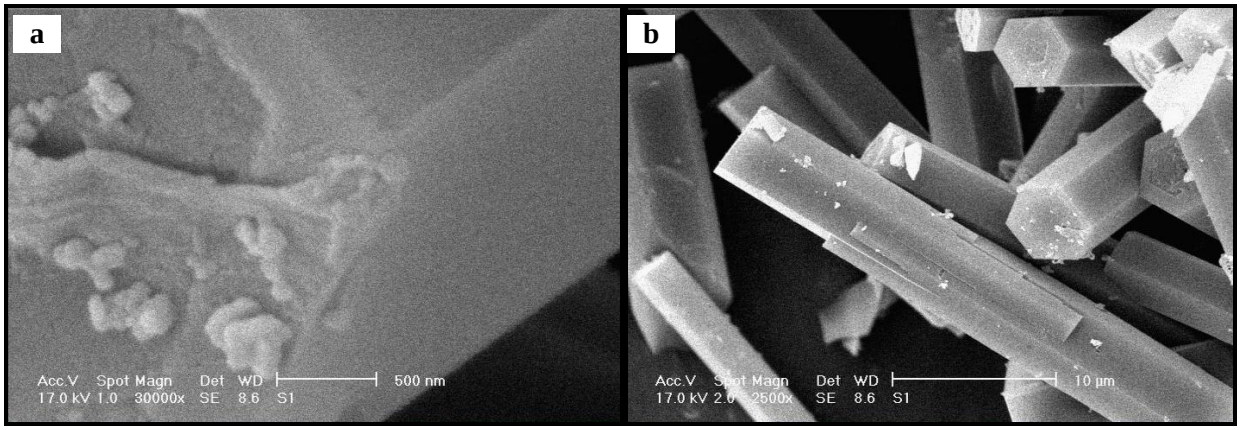

Fig.3. SEM image of the prepared $AlPO_4$-5 crystals; (a) 30000 X and (b) 2500 X

CONCLUSION

Synthesis and characterization of $AlPO_4$-5 powder were investigated. Crystals as small as 65 nm, were obtained from the crystallization of a precursor gel. A short-time hydrothermal treatment was used in this study. XRD pattern of the obtained powder showed a high-crystallinity $AlPO_4$-5 phase. The SEM images and Scherrer's equation revealed the nano-structure of the $AlPO_4$-5 powder about 65 nm.

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