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Microwave heating synthesis of zeolite W (K, Sr)

A. C. B. de Faria[a*], C. L. Ribeiro[b] and A. M. A. Silva[b]
[a] Institute of Chemistry, University of Brasília, Brasília, Brazil
[b] Department of Mechanical Engineering, University of Brasília, Brasília, Brazil
[*] fariacaca@hotmail.com

The separation of $CO_2/CH_4/N_2$ mixtures is of great importance in several industry applications, such as natural gas purification, coalbed methane development and CO_2 capture [2]. Compared with the traditional aqueous amine solution, adsorption and membrane separation methods have attracted much attention because of their simple equipment, low energy consumption and non-corrosive properties [2]. In recent years, preliminary studies have been carried out on the separation of mixed gases over MER zeolite. The zeolite W is described in the literature as a member of the MER zeolite structure (similar to the natural mineral merlinoite), characterized by interconnected 8-membered ring pores of 3.8 x 3.8 Angstroms [6]. It is thermodynamically stable at temperatures up to 950 °C, with an ideal composition in the gel of Al_2O_3:5 SiO_2:7.5 K_2O:600 H_2O when using only KOH as the potassium source and heated at 150 °C for 10 days [1].

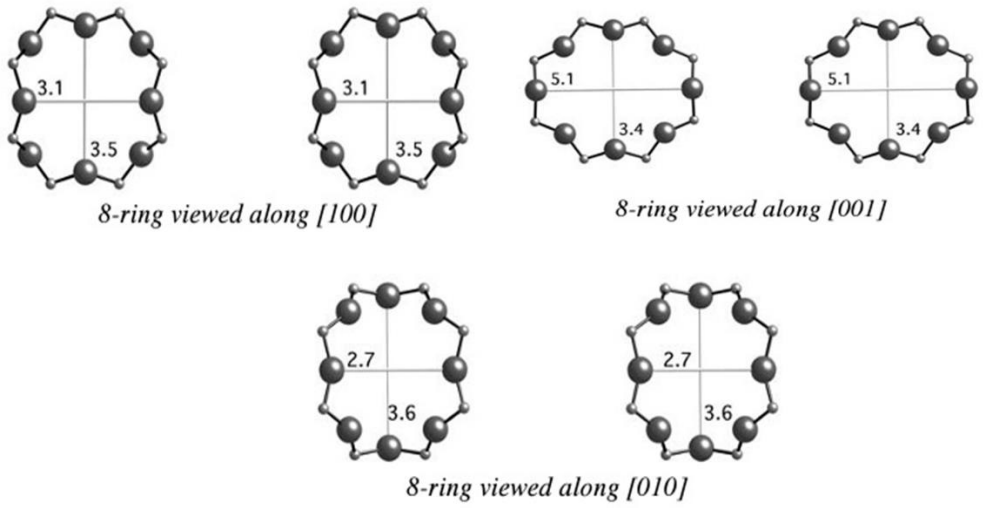

Figure 1. 8-ring window aperture of silicon MER zeolite.

To achieve competitive applications such as inorganic membranes and advanced nanodevices it is essential to provide high-quality zeolite crystals, which means a more defined morphology, high crystallinity, and a homogeneous particle size distribution. Zeolites is generally synthesized under hydrothermal conditions using conventional furnaces [4]. Crystal size and morphology are directly controlled by adjusting the initial gel composition (water content, alkalinity, or oxide ratio) as well as the heating conditions (heating rate and/or temperature) [3,4]. A new approach that has gained prominence in recent years and promotes several improvements in the properties of zeolites is microwave-assisted synthesis. This simple and effective technology can reduce synthesis time, improve dimensional and compositional uniformity, and enhance the dissolution of precursor gel [5]. In this research, MER-type zeolites were produced from microwave heating, with the insertion of the Sr ion and modifications in the molar proportion to produce a material with improved properties for membrane applications, the characterization occurred by XDR spectra to determine the zeolite type.

Zeolite W was synthesized using a microwave system (300 W) with hydrothermal conditions at 150°C for 5,10 and 24 hours in a Teflon pressure vessel. Different alumina source (aluminum isopropoxide and aluminum wire) was used to verify its influence in zeolite structure formation. A gel in the molar ratio of Al_2O_3: 2.3 K_2O: 10 SiO_2: 0.1 $Sr(NO_3)_2$: 160 H_2O was employed for all reactions.

In Figure 2, the crystallographic patterns of the syntheses conducted in the microwave using $Al(O\text{-}i\text{-}Pr)_3$ and alumina wire as the alumina source, and Ludox® AM-30 as the silica source are exhibited. Figure 2(a) shows patterns for synthesis using alumina wire, and it is possible to observe that the resulting material is predominantly amorphous, with few peaks shown in the 2θ region equal to 10.86, 12.56, 16.58, 17.8, 27.42, and 28.14, corresponding to the (011), (200), (211), (002), (103), and (420) planes of zeolite W, respectively.

Figure 2(b) shows the patterns for synthesis using aluminum isopropoxide as the alumina source and different synthesis times. It can be observed that when using isopropoxide, the obtained product exhibits a profile similar to zeolite W, with some peaks also resembling those of zeolite L [3,4]. However, these peaks are significantly shifted, indicating that the produced material, in addition to presenting itself as a mixture of these zeolites during synthesis, experienced broadening and compression of the unit cells of the zeolite crystals, indicating a lack of purity. This likely occurred due to the synthetic route used in this experiment, such as the use of strontium, which is conventionally not employed in zeolite W synthesis, or possibly non-ideal pressure and temperature conditions for the growth of zeolitic crystals to obtain a pure material.

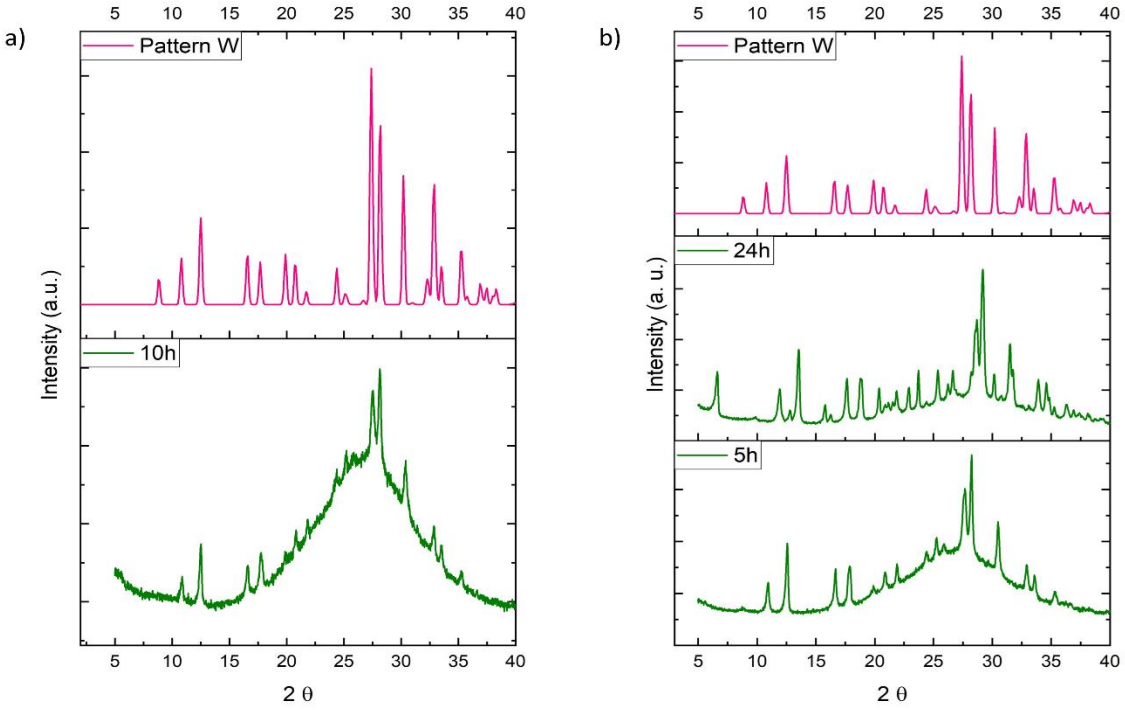

Figure 2. XRD pattern of the microwave syntheses using a) Aluminum wire and b) aluminum isopropoxide with Ludox® AM-30 and different syntheses times.

Therefore, it can be concluded that the synthesized material presents structural characteristics of zeolite W and that the use of microwave heating promoted a reduction in the reaction time of the synthesis, obtaining a material with a time 3x shorter than conventional synthesis. It was also observed that the insertion of Sr into the structure showed promise both for synthesis and for possible application in membranes. Obtaining zeolite W by microwave synthesis is, as far as we know, something not addressed in the literature with conditions similar to those of this work.

References

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