

FACTORS EFFECTING THE CRYSTALLIZATION OF ZEOLITES SYNTHESIZED BY MICROWAVE HEATING

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Microwave heating has been shown to influence various inorganic chemical reactions. Substantial reductions in the synthesis time for materials have been reported (1). Additionally, product characteristics and quality of materials synthesized by microwave heating are different from those synthesized under conventional heating methods. The mechanism which results in microwave enhancement is not completely understood. Conflicting explanations and experimental results for zeolite synthesis reactions have been reported by various labs. This is primarily due to differences in experimental procedure such as ageing, temperature ramp rate, reaction vessel geometry, power delivery and volume of precursor gel reacted. Often, these parameters are not reported. The importance of such factors in the synthesis of silicalite has been shown in a recent publication by Conner et al (2). Furthermore, they showed that the dielectric properties and microwave field distribution in the synthesis medium are critical. An order of magnitude difference in the yield of silicalite was observed by changing the reactor geometry and giving rise to different simulated field distributions in the liquid. We studied the synthesis of NaY zeolite using the same experimental setup, but found no difference between the products formed in either reactor geometry. The precursors for silicalite and NaY differ. Silicalite is crystallized from a clear silica solution, containing TPAOH as a template material. NaY is crystallized from a solid containing aluminosilicate gel with no structure directing agent. The different response to changes in reactor geometry of NaY and silicalite is a result of different crystallization mechanisms for the materials (3), and different dielectric properties of their precursor gels.

Stirring may effect the nucleation growth of zeolites. Romero et al (4) reported that a rapid stirring rate of low silica X zeolite produced under conventional heating gave an enhancement in reaction rates which were similar to or greater than that observed under microwave heating. Bonaccorsi and Proverbio (5) found that stirring resulted in a higher yield with less secondary product formation in the microwave synthesis of NaA zeolite. NaY zeolite was prepared in both static and stirred conditions under microwave heating. We found no difference in the crystallinity and morphology of the samples produced.

Beta zeolite is an important catalyst due to its stability, acidic sites, hydrophicity and large pore dimensions (6). Beta zeolite samples were rapidly synthesized with microwave heating using tetraethylammonium hydroxide as a structure directing agent. Tetraethylammonium fluoride was used as a fluoride source. The presence of fluoride enhanced the rate of product formation; beta zeolite was produced in less than 8 hours. Work by Kim et al (6) show similar results using ammonium fluoride.

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