

Microwave Reactor Engineering: Zeolite Synthesis

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Introduction

Studies of the use of microwaves in chemistry have increased exponentially in the last score of years resulting in over two thousand publications in the last year. Reaction rates are often increased by over an order of magnitude while selectivities can also be enhanced. It is recognized as at the forefront of Green Chemistry¹. Continuous processes might even replace batch processes². Microwave enhanced syntheses reactions span from ceramics to pharmaceuticals to polymers to zeolites³⁻⁸. The reasons for microwave enhancement are not understood and results from different laboratories are not reproducible.

This paper will discuss and give examples of the microwave reactor engineering of zeolite catalyst syntheses. We will demonstrate how reactor geometry, temperature profile, power delivery and distribution influence the synthesis kinetics. Simulation of the distribution of electromagnetic energy within the microwave delivery systems (oven, open or tuned waveguides) for different reactor geometries helps to explain many of the differences^{9,10}. Molecular theory and kinetic theory and simulation in the presence of microwave exposure are recently beginning to unravel the reasons for microwave effects in interfacial reactions such as syntheses, sorption and catalysis¹¹⁻¹³. Both experiment and theory demonstrate that microwave exposure creates systems with multiple local temperatures at steady state.

Results & Discussion

The enhancement of synthesis reactions under microwave heating is dependant on many complex factors. We investigated the importance of reaction engineering parameters relevant to microwave synthesis. Of interest to this investigation were the reaction vessel size, volume of precursor reacted, microwave power delivery and microwave cavity design. The synthesis of Silicalite, NaY zeolite and Beta zeolite were carried out under a number of varying conditions to determine the influence of these parameters on the nucleation rate, the crystallization rate and the particle size and morphology. The rates of NaY and beta zeolite nucleation and crystallization were more rapid in the multi-mode MARS®-5 oven compared to the mono-modal Discover®. The faster synthesis rate in the MARS®-5 may be the result of the multi-mode microwave electric field distribution. Slower rates of NaY and beta

zeolite formation observed in the Discover® and the waveguide may be the result of an even microwave electric field distribution. Changes in reaction vessel size and precursor volume during the microwave synthesis of beta and NaY zeolite were found to influence the rate of zeolite formation. These results indicate that reactor geometry needs to be considered in the design of systems used for microwave synthesis.

Comparative synthesis reactions were carried out with conventional heating, and microwave heating was shown to be faster and more selective in most cases; however, the reasons for enhanced syntheses differed depending on the specific syntheses. The reasons for these various forms of enhancement are based on in situ X-ray and Raman spectroscopies, and microwave energy distribution simulations that will be presented and discussed.

Significance

Microwave enhancements of chemical reactions in a broad range of chemistries are experimental facts. The unique phenomena are due to the chemistries and distribution of microwave energies in time and space as well as their effects on the molecules and their reaction intermediates. The crucial component in understanding and taking advantage of microwave enhancement in chemistry/catalysis is microwave reactor engineering, a multidisciplinary field in its infancy.

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