NANO HIGHLIGHT
Microwave Synthesis of Nanostructured Catalysts (1)

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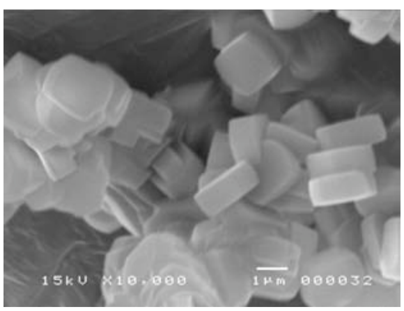
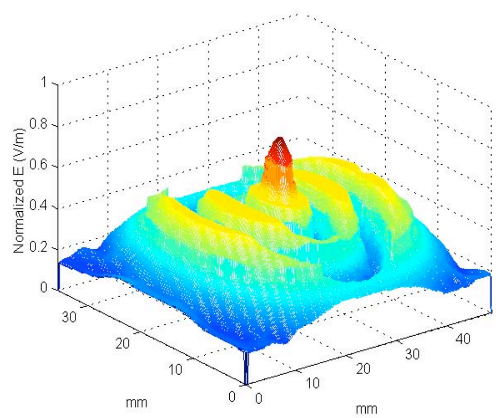
Microwave energy is proving to provide an efficient new approach in the synthesis of new materials. In many cases, solid molecular sieves are synthesized by microwaves in minutes compared to days for conventional approaches to synthesis. The nanoporous materials are employed as catalysts and in separations based on molecular dimensions. Microwave synthesis obviously saves both time and energy. The reasons for this enhancement are not understood; so, we are employing a variety of in situ techniques and are developing the theory to understand the engineering and microwave chemistry of this enhancement.

As an example, Silicalite, a zeolite with pores of dimension slightly larger than benzene (ca. 0.6 nm), can be synthesized in less than an hour in the presence of microwave energy, which controls the synthesis average solution temperature at 175ºC. Conventionally the synthesis would take a day. We discovered that the size and shape of the synthesis vessel could make the rate of synthesis vary by up to an order of magnitude (minutes to over an hour)(2).



Within 15 minutes, 45% of the starting materials are converted to a uniform distribution of low-defect zeolite crystals within a reactor 33mm in diameter. This is shown in the micrograph. As time progresses, the size increases while the number of the particles decrease. Nucleation of the initial particles is rapid as is subsequent growth, both processes are enhanced by microwave exposure. A smaller reactor, 11mm in diameter, shows only 1% yield in 15 minutes.

We simulated the distribution of microwave energy within the two reactors and find a significant variation of energy as a function of radial position in the larger, more-productive reactor. This is s shown in the figure on the right. We further find that different zeolites exhibit vastly different rates of microwave enhancement, certainly due to differences in their synthesis mechanisms and kinetics. Our ongoing studies include in situ X-ray (WAXS, and SAXS) as well as vibrational spectroscopies (Infrared and Raman) to understand the unique enhancement due to microwave chemistry and engineering.



References
[1] For further information about this project link to **< http://www.ecs.umass.edu/che/micro-synth/ >** or email **<wconner@ecs.umass.edu>**
[2] J. Physical Chemistry B, 108,37, 13913-20 (9/16/2004) Cover Article