CHAPTER 6

Conclusions

Reviewing the objectives of this project, which were:

- To produce magnetic nanostructured iron oxide particles by precipitation from within the confines of a zeolite matrix, and
- To investigate the properties of the prepared samples, specifically its elemental and phase composition, crystal structure, particle morphology, response to an applied magnetic field, and to analyze its pore size and surface area,

it can be concluded that iron oxide nanoparticles in nanometer dimensions have been successfully prepared within the matrices of Na-Y zeolite via an ion exchange procedure followed by a precipitation process. EDS results showed that Fe was successfully introduced into the zeolite system, replacing Na ions that were inherent in the zeolite. XRD analyses revealed the presence of a mixture of iron oxide phases, with maghemite as well as hematite peaks identified in the spectra of precipitated samples.

Various characterization methods including BET calculations, application of Scherrer's equation on XRD data and observation under electron microscopes have revealed the particles to be within 7-14 nm in diameter. The zeolite-iron oxide system treated with the highest concentration of NaOH exhibited a cluster-like morphology under SEM observation while little agglomeration was observed on the equiaxed samples under TEM. These are visibly different to acicular iron oxide particles precipitated without the constrained space of a zeolite matrix. The magnetic sizes, which are frequently smaller than the physical size, for all the samples were between 4-8 nm in diameter.

Magnetization results indicate that most of the precipitated samples were superparamagnetic in nature, with the absence of hysteresis, and a very tiny remanent

magnetization (less than 1.1 emu/g) upon removal of applied field. The highest magnetization recorded was 10.2 emu/g at the maximum applied field of 10 kOe. The natural ferrimagnetic behavior of maghemite was diminished due to the ultrafine size of the particles, which was below the critical diameter of 10 nm for superparamagnetic maghemite and 8 nm for superparamagnetic hematite.

Very high concentrations of NaOH (5.0 M to 12.5 M) were also found to distort and ultimately, to dissolve the zeolite matrix. Thus, the iron oxide nanoparticles could be retrieved from the confines of the zeolites by using an extremely high concentration of NaOH.

Possible future studies that can be pursued from the results of this work include the application of the zeolite-iron oxide system in bioseparation processes as well as ferrofluids.