

In Situ X-Ray Study of Hydrothermally Prepared Titanates and Silicotitanates

Abraham Clearfield, Akhilesh Tripathi, Dmitri Medvedev,
*Department of Chemistry, Texas A&M University, College Station, TX,
USA. E-mail: clearfield@mail.chem.tamu.edu*

In a typical ex-situ hydrothermal or solvothermal reaction the investigator knows what is input and what the end result is but has no experimental evidence of what transpires in between. In situ methods, whether using X-ray, NMR, IR or other techniques aims to follow the reaction through this in-between stage. For crystal growth studies, in-situ X-ray diffraction is eminently satisfactory. We will describe our crystal growth studies of titanium silicate phases and the ion exchange mechanism of Cs⁺ ion uptake in their tunnel structures.

One of the most vexing problems facing the nuclear industry and countries with nuclear weapons is the safe disposal of the generated nuclear waste. Huge quantities of nuclear waste arising from weapons manufacture are stored at the Hanford and Savannah River sites. The general method of remediation involves the removal of Cs-137, Sr-90 and actinides from a huge quantity of salts, principally NaNO₃, organics and complexing agents. It has been found that a sodium silicotitanate is able to remove Cs⁺ selectively from the waste and certain sodium titanates remove Sr²⁺ and actinides. These compounds have been prepared by ex-situ hydrothermal methods. We have studied the in situ growth of these materials at the National Synchrotron Light Source, Brookhaven National Laboratory.* In addition we will describe the mechanism of ion exchange in the titanosilicate as observed by in situ methods and how the combination of these techniques coupled with an intimate knowledge of the structure of the solids is helping to solve the remediation process. In general, the in situ method allows the investigator to follow the nucleation and crystal growth or phase transformations occurring in hydrothermal reactions, and as a result of ion exchange reactions.

* In collaboration with Aaron J. Celestian, Department of Geosciences and John B. Parise and Jonathan Hanson, Department of Chemistry, SUNY Stony Brook, NY, USA.

[1] Medvedev D.M., Tripathi A., Clearfield A., Celestian A.J., Parise J.B., Hanson J., *Chem. Mater.*, 2004, **16**, 3659.

Keywords: hydrothermal synthesis, in situ reactions, in-situ powder diffraction