

Nanominerals which belong to iron(III) (oxy)hydroxides, represented by ferrihydrite ($\text{Fe}_5\text{HO}_8 \cdot 4\text{H}_2\text{O}$), ferroxhyte ($\delta\text{-FeOOH}$) and akaganéite ($\beta\text{-FeOOH}$), occur as poorly crystalline aggregates of extremely fine particles. Despite the fact that these they are not very stable, they are ubiquitous in various near-surface as well as hydrothermal systems on Earth. The knowledge on the parameters controlling their stability is to understanding the role of iron oxides in geologic processes. Therefore, it is important to investigate and explain the formation/transformation pathways of these minerals.

Natural occurring iron(III) (oxy)hydroxides always contain numerous impurities, particularly Si, which strongly affects their stability. Therefore, the aim of this project is determination of the effect of Si content on thermal stability and mechanisms of thermal transformation of Si-doped ferrihydrite, ferroxhyte and akaganéite. To date, the characteristics of formation and transformation processes of these nanominerals has been limited by many analytical limitations. For the first time complementary use of *ex situ* laboratory instrumental methods combined with synchrotron based *in situ* X-ray scattering and Pair Distribution Function (PDF) analysis is proposed herein to overcome analytical difficulties. More complete characterization of these phases will improve our understanding of global biogeochemical cycling of iron including hydrothermal environments. Also, Si-doped synthetic ferrihydrite, ferroxhyte and akaganéite might in future serve as precursors for manufacturing of attractive and useful magnetic nanomaterials broadly applied in modern technologies.

The research proposed herein involves synthesis and characterization of pure and Si-doped phases followed by thermal transformation experiments accompanied with *ex situ* and *in situ* analysis of the products and by-products. The comparison of the results will allow for determination of Si content effect on thermal stability and mechanisms of formation and transformation of phases in question. To date, literature data is sparse or partial and many of published contributions on doped iron(III) (oxy)hydroxides are controversial or require more systematic approach. It is hypothesized, among others, that Si-doped iron (oxy)hydroxides are more stable, their thermal transformation proceeds through formation distinct intermittent iron(III) oxides depending on Si content, and that application of synchrotron based *in situ* high resolution X-ray scattering as well as PDF analysis will allow solving the structural transformation mechanisms of these phases upon heating. This was never done before with the use of these phases but this analytical technique proved successful with respect to other minerals. The author visited Advanced Photon Source in Chicago on September 2015 and discussed the conditions of the experiment with synchrotron beam scientists.

The objectives of proposed research will be addressed through laboratory experiments and instrumental analysis of synthesized pure and Si-ferrihydrite, Si-ferroxhytes and Si-akaganéites series having different Si/Fe molar ratios between 0.00 (pure phase) and 1.50 (doped phases). To test the above hypotheses, the following research is proposed:

1. Synthesis of pure phases and doped varieties from aqueous solutions followed by chemical and mineralogical characteristics of precipitated solids. This will include powder X-ray diffraction, electron microscopy and infrared spectroscopy for identification as well as “wet” chemical analysis and X-ray fluorescence analysis of solids for elemental composition.
2. Determination of thermal stability (and identification of the products) as a function of Si content with the use of differential thermal analysis and powder X-ray diffraction analysis.
3. Determination of temperatures of all the steps of structural transformations and identification of intermittent phases will be accomplished with the use of *in situ* synchrotron based powder X-ray scattering using reaction cell/furnace for the analysis in real-time upon heating, followed by determination of the effect of Si content on structure of Si-ferrihydrite, Si-ferroxhyte and Si-akaganéite based on Pair Distribution Function (PDF) analysis.

There is a very high novelty in the experimental approach proposed herein combining classical with state-of-the art synchrotron based techniques. The expected results and the development of this analytical approach are universal and in future they will be applied to other systems. PDF analysis is a powerful tool for elucidating the structures of natural and synthetic nanoparticulate materials. This technique is seldom used in Earth science disciplines. There is a need for broader implementation of this modern approach in mineralogical sciences which will contribute to the development of the whole discipline. The results of this basic research are potentially interesting for both, Earth scientists and the industry. In future, the outcomes will be used for development of various applications and technologies including nanotechnology of Fe compounds.