

## Structure of Nanosized Iron Particles Intercalated in Montmorillonite

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### Introduction

The design of metal clusters stabilized on metal oxides and the application to the elimination of NO<sub>x</sub> have been studied utilizing metal K-edge XAFS in this Proposal. As major results of this topic were already described in *Activity Report 2000B*, p.22 and *2001B*, p.18, the preliminary XAFS study for the design of iron (Fe) clusters stabilized between the montmorillonite layers is described in this report.

Adsorption methods based on Fe oxides and oxyhydroxides are known for the arsenic (As) removal in environmental water. The improvement of Fe site dispersion and morphologic stability are required to remove trace amounts of As in water repeatedly. Nanosized particles of Fe species were introduced and stabilized between the montmorillonite layers to satisfy these requisites for practical use.

### Experimental Section

0.43 M of Fe nitrate and double molar amount of 0.75 M of sodium hydroxide were mixed and left in dark at 290 K for 24h. The reaction mixture was slowly dropped to 0.5 wt% of Na montmorillonite (Kunipia F, Na<sub>1.5</sub>Ca<sub>0.096</sub>Al<sub>5.1</sub>Mg<sub>1.0</sub>Fe<sub>0.33</sub>Si<sub>12</sub>O<sub>27.6</sub>(OH)<sub>6.4</sub>) suspended in distilled water. The mixture was stirred at 290 K for 72h, followed by centrifugation and decantation. Finally, it was washed by several times and dried at 313 K (Fe 15.3 wt%). Fe K-edge XAFS spectra were measured at 13 K in transmission mode using beamline 10B. In the curve

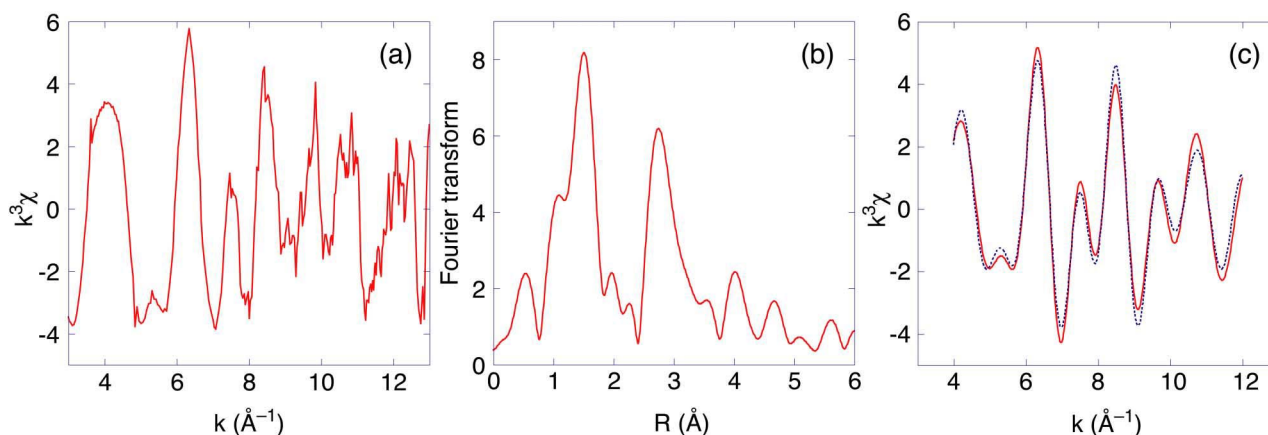
fit analysis, empirical amplitude and phase shift parameters of Fe-O and Fe•••Fe bonds were extracted from the EXAFS of α-Fe<sub>2</sub>O<sub>3</sub>.

### Results and Discussion

Fe K-edge EXAFS spectrum for the Fe-montmorillonite is shown in Figure 1. In the Fourier transform (b), Two dominant peaks appeared at 1.5 and 2.8Å (phase shift uncorrected). The peak at 1.5Å was fit well with one wave of Fe-O. Obtained distance was 2.063Å with the coordination number (*N*) of 5.8. The fit to the peak at 2.8Å with one wave of Fe•••Fe was not satisfactory and two waves fit of Fe•••Fe dramatically improved the *R<sub>f</sub>* value. Obtained distance was 3.259Å with the *N* of 2.4 and 3.753Å with the *N* of 0.5.

Obtained values were compared to reported crystallographic data for Fe oxides and oxyhydroxides. Fit values were only comparable to the γ-FeO(OH) (Lepidocrocite) crystal structure, where Fe-O, shorter Fe•••Fe, and longer Fe•••Fe distance is 2.034Å (*N* = 6), 3.066Å (*N* = 6), and 3.870Å (*N* = 2). The structure consists of double chain of [FeO<sub>6</sub>] octahedral linkage. Based on the obtained *N* values, four [FeO<sub>6</sub>] units were connected in the direction of montmorillonite layers. The nanosized FeO(OH) particle height was consistent with the increase of interlayer spacing starting from 12.2 (no Fe) to 15.7Å (Fe 15.3 wt%) based on X-ray diffraction measurements.

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**Figure 1.** Fe K-edge EXAFS spectrum measured at 13 K for Fe-montmorillonite (Fe 15.3 %). (a)  $k^3$ -weighted EXAFS oscillation ( $\chi$  function), (b) its associated Fourier transform, and (c) curve fit analysis to the inversely Fourier transformed  $k^3\chi$  function. (—) Observed and (••••) calculated.