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XAFS Study of Mesoporous Iron Oxyhydroxide Materials

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Introduction

High health risks from arsenic-contaminated drinking waters are forcing to reduce its maximum contaminant level (10 ppb). Affinity of Fe is known for As but the removal of trace level of contaminant is difficult with bulk iron materials. To cope with new regulations, mesoporous iron materials with high surface area were chosen in this study. The framework is relatively unstable as compared to Si and Al mesoporous materials. The local structure at iron site was studied by Fe K-edge XAFS.

Experimental Section

Mesoporous iron oxyhydroxide was prepared by quick oxidation of Fe^{2+} in the presence of surfactant i.e. sodium dodecyl sulfate (as-prepared sample). The surfactant was removed by calcination in air. Another series was prepared by extraction of the surfactant with ethanol for 12 h (ethanol-washed) and then subsequent calcination of the sample in air. Fe K-edge XAFS spectra were measured in transmission mode at beamline 10B.

Results and Discussion

The mesoporous nature of produced iron oxyhydroxide was characterized by XRD patterns. With the increase of calcination temperature, the characteristic *d*(001) peak intensity gradually decreased and became 40% for the 423K-calcined sample. The Fourier transforms of as-prepared (S_{BET} = 2.8 m^2/g) and calcined samples are shown in Figure 1. The magnitude of Fe•••Fe peak of as-prepared and the sample calcined at 423 or 473 K remained essentially the same. For the 473K-calcined sample the *d*(001) peak disappeared suggesting highly disordered structure. But no growth of Fe•••Fe peak for the as-prepared and calcined samples demonstrated that (part of) template still remained in the pore.

In case of ethanol-washed and calcined samples. The ethanol-washed (S_{BET} = 10 m^2/g) and 423K-calcined sample (S_{BET} = 120 m^2/g; S_{BET} = 170 m^2/g when evacuated at 423K) have the same magnitude of Fe•••Fe peak as in Figure 1 and indicated the retention of structure (Figure 2) and was also supported by the XRD results. However, once calcined at 473K, the Fe•••Fe peak became almost double (Figure 2). This demonstrates that ethanol effectively removed the template and due to this elution the framework became less stable and easy iron aggregation occurred as compared to the sample without ethanol washing calcined at same temperature (Figure 1).

Base on these EXAFS observations supported with XRD and BET results, the material can be optimized for the arsenic removal from 100 – 1000 ppb aqueous solutions.

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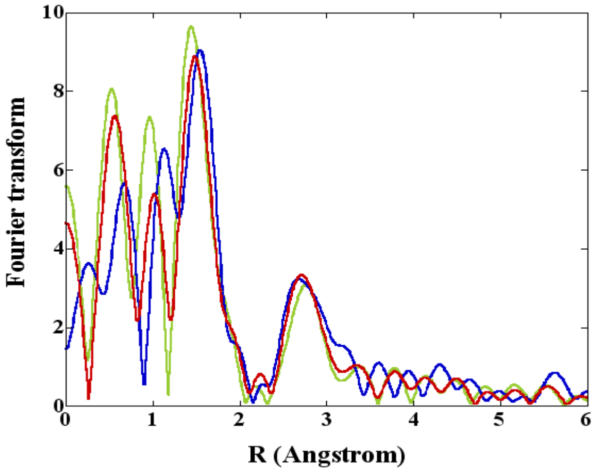

Figure 1. k^3-weighted Fourier transforms of Fe K-edge EXAFS for as-prepared mesoporous iron material (blue line), 423K, 2h calcined in air (green line) and 473K, 2h calcined in air (red line).

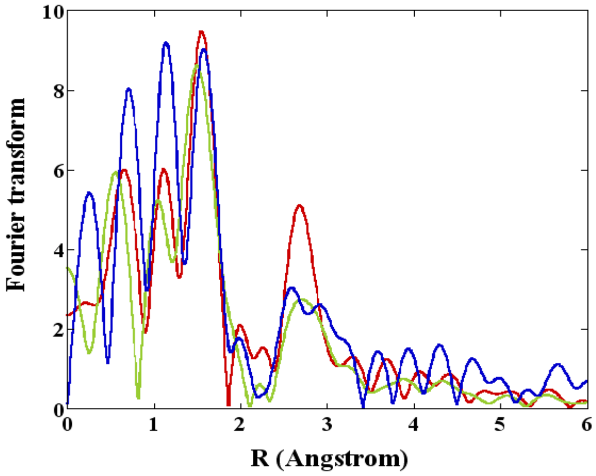

Figure 2. k^3-weighted Fourier transforms of Fe K-edge EXAFS for ethanol-washed mesoporous iron material (blue line), 423K, 2h calcined in air (green line) and 473K, 2h calcined in air (red line).