

Characterization of As(III)/As(V)-Lepidocrocite Complex Using Different Analytical Techniques

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INTRODUCTION.

Arsenic contamination of groundwater in deltaic aquifers has been reported from many parts of the world. Reductive dissolution of iron oxides and oxyhydroxides resulting to release of naturally adsorbed arsenic has been the most popularly advocated theory on arsenic mobilization (McArthur et al., 2000, Nickson et al., 1998). Deltaic aquifers are particularly known to comprise aquifer soils associated with iron oxy-hydroxides and iron oxide coated sands.

Though aquifer soil may contain a number of iron oxides and oxyhydroxides, attention so far has been mainly focused on goethite and ferrihydrite with regards to arsenic adsorption and desorption. Very few studies have been conducted on arsenic adsorption and desorption by lepidocrocite (γ -FeOOH), one of the stable species of iron oxyhydroxide. Transformation of lepidocrocite into goethite or haematite has not been observed under ambient conditions (Schwertmann and Cornell, 1996). Lepidocrocite is typically formed in soils and persists on a pedogenic time scale. In redoximorphic environments it is often found associated with goethite. Adsorption of arsenic by green rust, goethite and lepidocrocite is two or more order of magnitude greater than clay and feldspar minerals (Lin and Puls, 2003.)

An attempt has been made in this communication to understand the nature of As(III) and As(V) adsorbed on the lepidocrocite surface using scanning electron microscopy

MATERIALS AND METHODS.

Materials

Reagents used were of analytical grade. ASTM Grade 1 water was used for making the solutions. As (III) and As(V) solutions were prepared using NaAsO_2 and $\text{NaH}_2\text{AsO}_4 \cdot 7\text{H}_2\text{O}$ respectively.

Synthesis of lepidocrocite

Lepidocrocite was synthesized following the method of Cornell and Schwertmann (1996) using $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and NaOH .

Preparation of As (III) and As(V) complex with lepidocrocite

20 ml of 10,000 mg/L As (III)/As (V) solution was thoroughly mixed with 1 g of the synthesized lepidocrocite. The lepidocrocite-As(III)/As(V) slurry was dried at ambient temperature and washed with distilled water before undertaking further characterization.

Instrumental

Siemens D-500 X-ray diffractometer was used for recording the XRDs. AFM images were captured with Seiko SPA 400 atomic force microscope. Specific surface area and pore volumes were measured using Micromeritics ASAP 2020 BET surface area analyzer. Surface morphology of lepidocrocite-As(III)/As(V) complex was studied using S3400N Hitachi SEM.

RESULTS AND DISCUSSION.

Characterization

Synthesized lepidocrocite phase was confirmed through X-ray diffraction by matching with standard JCPDS values. BET surface area was found to be 124.7 m^2/g .

Figure 1 shows the atomic force micrograph of the synthesized lepidocrocite sample. The crystals have lath-like or tabular morphology. Elongation in the c-direction and (010) face of the crystal is clearly visible.

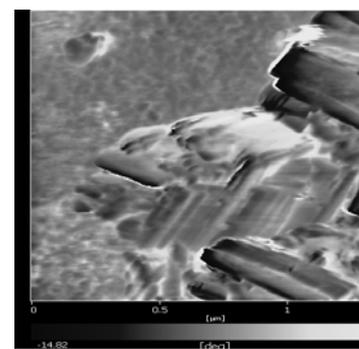


fig 1. Atomic force micrograph of lepidocrocite

SEM study of As (III) and As (V) doped lepidocrocite

Figure 2a shows the scanning electron microscopic images of the synthesized lepidocrocite, As(III) doped lepidocrocite (Fig. 2b, 2c), and As(V) doped lepidocrocite (Fig. 2d) respectively.

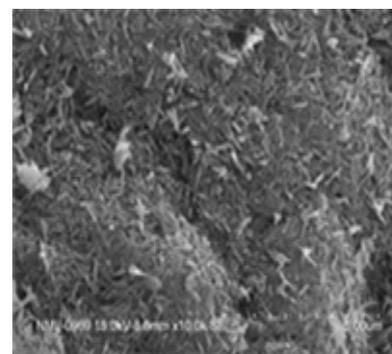


fig 2a. SEM image of lepidocrocite.

The synthesized lepidocrocite was elongated in nature and typical flowery

aggregates were observed. The particles were randomly oriented and preferential directional growth was clearly seen.

A distinct change in morphology was seen in the lepidocrocite doped with As(III) and As(V). Fig 2b and 2c show the morphologies shown by As(III) doped lepidocrocite.



fig 2b. SEM image of As(III) doped lepidocrocite

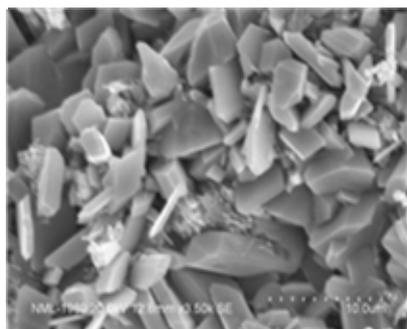


fig 2c. SEM image of As(III) doped lepidocrocite

One long acicular needled (Fig. 2b) and platy lath like tabular particles (Fig. 2c) were seen. The needles varied in length from 5-7 μm in length. Fine encrustations were seen between the lath shaped particles.

A completely different morphology was observed in As(V) adsorbed lepidocrocite (Fig. 2d).

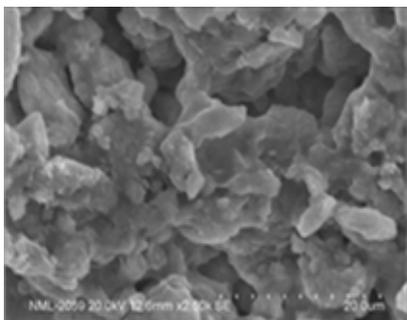


fig 2d. SEM image of arsenate adsorbed lepidocrocite

Agglomeration of particles was seen with the development of voids and spaces. The fused particles do not show any distinct crystalline structure as evidenced in the As(III) doped lepidocrocite.

Elemental analyses from SEM-EDAX showed that As (III) varied from 60.8-74.5 wt.% at different points in fig 2b. For As (V) corresponding range was 10-41 wt.%. As(III) correlated well with O in As(III) adsorbed lepidocrocite while no such correlation was observed in the case of As(V).

CONCLUSIONS.

Lepidocrocite was synthesized in the laboratory and characterized using XRD, AFM and SEM. Lepidocrocite phase was confirmed through XRD. AFM image showed well developed lath like tabular morphology and SEM showed elongated flower like aggregates. As(III)/As(V) doped lepidocrocite develop completely different morphology. EDAX show greater wt % of As (III) on the lepidocrocite surface than As (V). As-O correlation at different points is better on As(III)- lepidocrocite surface than As(V).

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