

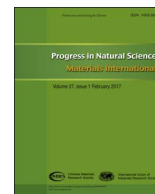
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Original Research

Sulfurized hematite for photo-Fenton catalysis<sup>☆</sup>Yaping Zhang<sup>a</sup>, Kaituo Dong<sup>a,\*</sup>, Zheng Liu<sup>a</sup>, Haolin Wang<sup>a</sup>, Shengxiang Ma<sup>a</sup>, Anyu Zhang<sup>a</sup>, Ming Li<sup>a</sup>, Lianqing Yu<sup>a,\*</sup>, Yan Li<sup>b,\*</sup><sup>a</sup> College of Science, China University of Petroleum, QingDao 266580, China<sup>b</sup> College of Mechanical and Electronic Engineering, China University of Petroleum, QingDao 266580, China

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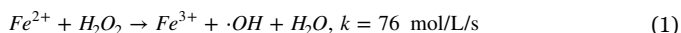
Hematite  
Amorphous sulfur  
Photo Fenton catalysis  
SO<sub>4</sub> radical

## ABSTRACT

A hematite/amorphous sulfur composite was prepared via simple heating hematite and α-sulfur in Teflon-lined autoclave at low temperature. The composite was characterized by X-ray diffraction (XRD), Raman spectrum, Thermal Gravity Analysis (TGA), Transmission Electron Microscopy (TEM) and X-ray photoelectron spectroscopy (XPS). The results revealed that an allotrope sulfur at 5–37% weight percent was found in the composite. After sulfuration, S<sub>n</sub><sup>2-</sup> or S<sub>2</sub><sup>2-</sup> was doped in the lattice of hematite, large amounts of OH and SO<sub>4</sub> were adsorbed on the surface of hematite. Hematite/amorphous sulfur composite had superior photo-Fenton activities than pure hematite. This work also demonstrated that amorphous sulfur also had the activity of photo-Fenton catalysis. OH<sup>•</sup> and SO<sub>4</sub> radicals facilitated dye adsorption and acted as a bridge to link H<sub>2</sub>O<sub>2</sub>. Moreover, SO<sub>4</sub> radicals on hematite served as electron trapping center that can receive photo-induced electron from conduction band of hematite and transfer it to the adsorbed H<sub>2</sub>O<sub>2</sub>, increasing the rate of photo-Fenton reaction eventually.

## 1. Introduction

Dyes are extensively used in textile, paper and printing industries, and their wastewater pollutes river and underground water [1]. Photocatalysis, Fenton and photo-Fenton reaction could degrade the contaminants effectively [2,3]. Hematite (Fe<sub>2</sub>O<sub>3</sub>) could be applied in heterogeneous Fenton-like degradation of dye in the presence of H<sub>2</sub>O<sub>2</sub> with low cost. The vital step of Fenton-like degradation reaction was ·OH generation (Eq. (1)), which has the second highest oxidized potential ([E°(OH/H<sub>2</sub>O)=+2.8 V<sub>NHE</sub>]), is the main active oxidant in Fenton-like system and could mineralize organics to CO<sub>2</sub>, H<sub>2</sub>O and other inorganic substances [4–6].



In the presence of hematite, Fe<sup>2+</sup> originates from reduction of Fe<sup>3+</sup> by photo-induced electron (Eq. (2)).



Taken Eqs. (1) and (2) into consideration, the degradation reactions mainly occur at surface of hematite or interface with dyes. According to the Eq. (1), it involves adsorption of H<sub>2</sub>O<sub>2</sub> on surface of hematite and electron transfer from hematite to H<sub>2</sub>O<sub>2</sub>. No matter whether the photo-generated electron comes from excited dye or hematite, formation of ·OH (Eq. (1)) is the key for increasing rate of photo-Fenton reaction. So both enhance-

ment adsorption of H<sub>2</sub>O<sub>2</sub> or dye on hematite and decrease impedance of electron transfer from Fe<sup>2+</sup> to H<sub>2</sub>O<sub>2</sub> could increase reaction rate of Eq. (1).

Surface sulfur-doping and sulfate radical modification of hematite is an effective route that could accelerate reaction of Eq. (1) [7]. As reported in previous works, surface modification of sulfur species was achieved along with crystallization of hematite via bulk doping using sulfur source such as inorganic matter (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> et al.) or organic matter (thiourea et al.) [3,4,7]. For example, Jinlong Zhang and Jianguo Bao et al. used FeSO<sub>4</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> as iron and sulfur source, sodium hydroxide and oxalate as precipitant, to synthesize sulfur doped and sulfur species modified hematite [4,7]. In our opinion, surface modification on pre-crystallized and non-doped hematite may be another effective method that could also improve reaction of Eq. (1). In comparison with surface modification of sulfur species along with crystallization of hematite, this synthesis route could simplify the reaction system of every step and avoid impurity generation.

Some sulfur-containing matters such as Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, thiourea, sulfuric acid et al. had been adopted as sulfur source to involve doping reaction. Among these, α-sulfur (S<sub>8</sub>) powder had advantages of non-toxic, low cost, free of organic solvent, more environmental friendliness and low activity temperature et al.

In this paper, a facile method was adopted to modify hematite via mixing α-sulfur and hematite directly at low temperature. The results indicated that not only sulfur-containing radicals adsorbing on the

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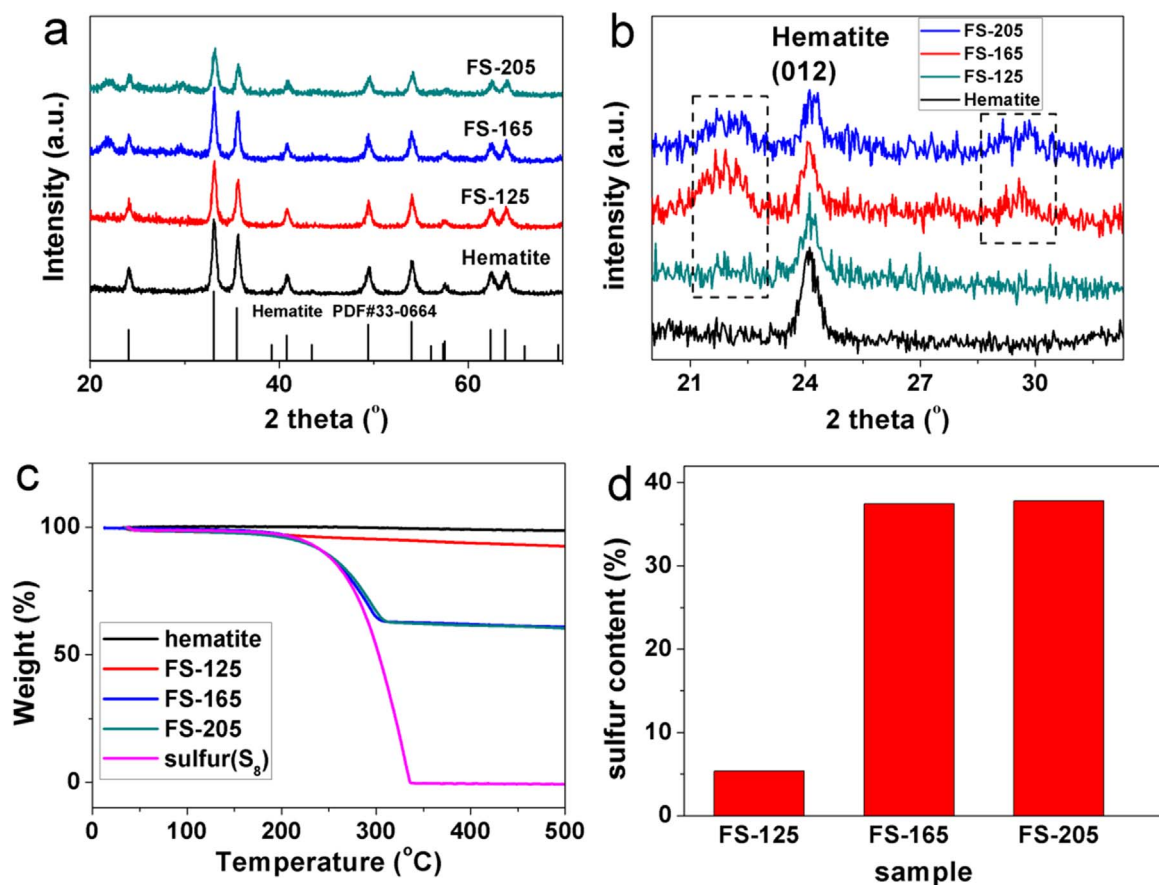


Fig. 1. (a) XRD patterns of sample hematite, FS-125, FS-165, FS-205; (b) XRD pattern at the range of 20–33°; (c) TGA curves of samples; (d) bar graph of sulfur content in composites XRD patterns of the four samples.

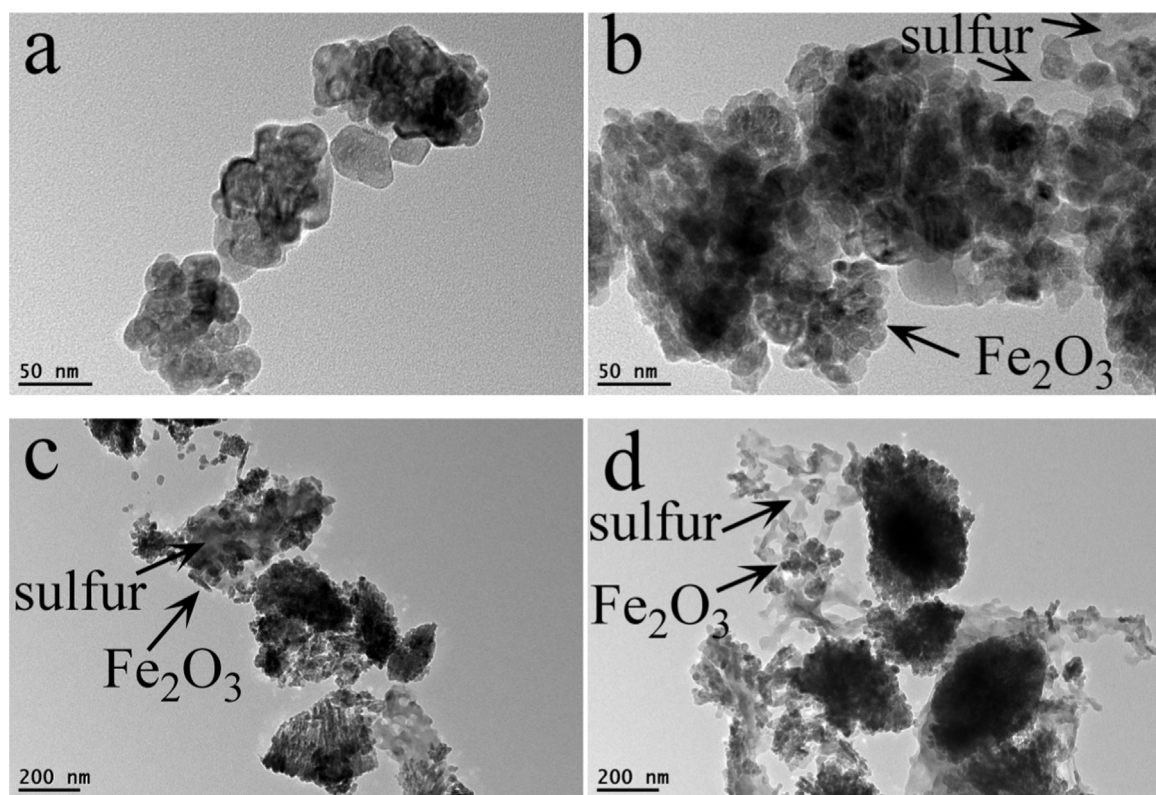


Fig. 2. TEM images of (a) Fe<sub>2</sub>O<sub>3</sub>, (b) FS-125, (c) FS-165, (d) FS-205.

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