Ultrasonics Sonochemistry xxx (2015) xxx–xxx

Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

journal homepage: www.elsevier.com/locate/ultson

Sonocatalytic decolorization of textile wastewater using synthesized γ-FeOOH nanoparticles

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ARTICLE INFO

Article history:
Received 30 January 2015
Received in revised form 17 April 2015
Accepted 18 April 2015
Available online xxxx

Keywords:
γ-FeOOH nanoparticles
Nanocatalyst
Sonocatalysis
Sono-Fenton process
Textile wastewater

ABSTRACT

γ-FeOOH nanoparticles were synthesized and applied for decolorization of a textile wastewater containing reactive orange 29 (RO29) by ultrasonic/γ-FeOOH/H_2O_2 process. X-ray diffraction, transmittance electron microscope, scanning electron microscope, and nitrogen adsorption/desorption analysis approved synthesis of γ-FeOOH nanoparticles with the average width of 60–70 nm. The catalyst dosage, H_2O_2 concentration, pH, and ultrasonic power had significant effect on catalytic performance of γ-FeOOH nanoparticles. γ-FeOOH nanoparticles can be effectively used as catalyst in successive US/γ-FeOOH/H_2O_2 processes without significant activity loss. Gas chromatography–mass spectrometry analysis was used to verify the main intermediates produced through the RO29 degradation. Mineralization of the textile wastewater during treatment process was determined by chemical oxygen demand analysis.

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1. Introduction

Hazardous colorful wastewater realized from industries particularly textile industry are one group of the major causes of environmental pollutions in water resources [1,2]. The release of synthetic dyes into the aqueous environment causes serious problems because of theirs strong color and low biodegradability [3,4]. The removal of these pollutants from contaminated aqueous solutions is of great importance in environmental processes [5]. Thus, there is currently considerable interest in developing effective processes for degradation of these types of organic pollutants.

Fenton process as of the advanced oxidation processes (AOPs) is regarded as a promising technology for wastewater treatment at room temperature and pressure, particularly for degradation of nonbiodegradable and toxic components to H_2O and CO_2 [6,7]. It is based on production of various reactive species, often hydroxyl radical (·OH). The ·OH is generated through the decomposition of H_2O_2 in the presence of Fe^{2+} as catalyst (Eq. (1)) [8]. The generated reactive species are able to degrade and mineralize various organic pollutants [9].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {\cdot}OH + OH^- \quad (1)$$

By the way, poor recycling property as well as separation of homogeneous catalyst from the treated wastewater restrict the utilization of Fenton process [10]. For the first problem a great effort has been spent to enhance reproduction of the Fe^{2+} as a most effective form of iron in Fenton process [11]. The application of ultrasonic vibration is one of the promising methods could promote Fenton reactions [10]. In sono-Fenton process, the ultrasonic vibration could promote Fenton reactions by cavitation (Eqs. (2) and (3)) [12]. The cavitation is defined as formation of hot spots with extremely high local temperature and pressures by formation, growth, and sudden collapse of bubbles in liquids [13].

$$Fe^{3+} + H_2O_2 \xrightarrow{\text{Ultrasonic waves}} Fe^{2+} + HO_2^{\cdot} + H^+ \quad (2)$$

$$Fe^{3+} + H_2O \xrightarrow{\text{Ultrasonic waves}} Fe^{2+} + {\cdot}OH + H^+ \quad (3)$$

To solve the second problem, heterogeneous Fenton catalysts such as iron oxides are used [14]. In this compounds, heterogeneous iron catalyzes production of ·OH from oxidation of hydrogen peroxide while most of the catalyst remains in the solid phase and can be reused [15].

Most of the studies in application of heterogeneous iron sources in sono-Fenton process were focused on Fe_2O_3 and Fe_3O_4 particles [16–19]. But in this work, γ-FeOOH nanoparticles was synthesized and used as heterogeneous catalyst in sono-Fenton process for treatment of textile wastewater contains reactive orange 29 (RO29) as an azo dyestuff. To evaluate ability of this catalyst in sono-Fenton process the effect of γ-FeOOH dosage, H_2O_2

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http://dx.doi.org/10.1016/j.ultsonch.2015.04.023
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Please cite this article in press as: M. Sheydaei, A. Khataee, Sonocatalytic decolorization of textile wastewater using synthesized γ-FeOOH nanoparticles, Ultrason. Sonochem. (2015), http://dx.doi.org/10.1016/j.ultsonch.2015.04.023

concentration, ultrasonic power and initial solution pH was investigated. Oxidative ability of H_2O_2 in decolorization of the wastewater was compared with that of other oxidative agents. Degradation of RO29 molecules and mineralization of the textile wastewater were followed by Gas chromatography–mass spectrometry (GC–MS) and chemical oxygen demand (COD) analyses.

2. Experimental procedure

2.1. Materials

The wastewater sample containing RO29 was obtained from Farsh & Patu textile factory in Tabriz, Iran. RO29 concentration, pH, COD and conductivity of the wastewater were 21 mg/L, 8.5, 2173 mg/L and 2.26 mS/cm, respectively. RO29 (Ciba-Geigy Co., Switzerland), Iron (II) sulfate heptahydrate (99.5%, AppliChem), butyl amine (98%, Merck), potassium bromate (99%, Fluka), potassium peroxydisulfate (98%, Fluka), potassium periodate (99.8%, Fluka), and hydrogen peroxide (Merck) were used in this work. All other materials used in the experiments were analytic reagents and used without further purification.

2.2. Synthesis of γ-FeOOH

γ-FeOOH nanoparticles were synthesized using the following procedure. First, 0.9 g of the Iron(II) sulfate was dissolved in 300 mL double distilled water at 35 °C. Then, constantly a CO_2-free air stream was bubbled into the Iron(II) sulfate solution, and 3) butyl amine solution (2 M) was dropping to the iron solution to adjust its pH at 5.5–6.0. During the synthesis, color of the solution was changed to dark greenish blue and finally to orange. The obtained orange suspension was left to reach the room temperature and filtered. Finally, the obtained γ-FeOOH nanoparticles were washed with ethanol and double distilled water and dried in an air oven at 70 °C for 12 h.

2.3. Characterization of synthesized γ-FeOOH

To determine the structure and purity of the γ-FeOOH nanoparticles, the X-ray diffraction (XRD) analysis has been performed by an X-ray diffractometer (Siemens D-5000, Germany) with CuKa radiation (40 kV, 30 mA, and 0.15418 nm). The diameter and morphology of the γ-FeOOH nanoparticles were observed by scanning electron microscopy (SEM) model LEO 1430VP (England) and transmission electron microscopy (TEM) model Philips CM120 (The Netherlands). Nitrogen sorption analyses were obtained with a Micrometrics, Gimini series (Japan) Sorptometer using standard continuous procedures at 77.15 K on prepared γ-FeOOH samples. The surface area was calculated according to the Brunauer–Emmett–Teller (BET) model over a relative pressure range of 0.05–0.90.

2.4. Wastewater decolorization

Wastewater decolorization experiments were carried out in a 250 mL erlenmeyer placed in ultrasonic irradiation apparatus (EP S3, 40 kHz, 300 W, Sonica, Italy) for 90 min avoiding light. The distance between the bottom of the reactor and the ultrasonic irradiation source was fixed at 1.0 cm. In general, 100 mL total volume wastewater and desired dosage of the γ-FeOOH nanoparticles were used in all the experiments. Then the initial concentration of H_2O_2 and pH values of wastewater solution were adjusted to the desired values and left uncontrolled during the experiments. During the degradation, the 3 mL of sample was taken out with pipette at every 15 min. Then the suspended catalytic particles were completely separated from the treated wastewater solution with centrifugal separator. Furthermore, the concentration of organic dyes was measured by UV–vis spectrophotometer. The COD was measured using the standard open reflux method [20]. Furthermore, the released concentration of total iron ions from catalyst to the solution was measured using Atomic Absorption Spectrometry (AAS) apparatus (Novaa 400, Analytik-Jena, Germany).

2.5. Analysis of degradation products

In order to evaluate the intermediates of RO29 degradation through the treatment of the textile wastewater, the GC–MS analysis was performed. To emit the effect of wastewater matrix on identification of RO29 degradation products, a synthetic wastewater was prepared by dissolving the dye in distilled water. 100 mL of this solution was treated by ultrasonic/γ-FeOOH/H_2O_2 process for 10 min. The resulted solution was saturated with Na_2SO_4 and the organic components were extracted with 30 mL of diethyl ether three times. The collected organic solution was held to evaporate the organic solvent. The remaining solid was dissolved in 100 μL of N,O-bis-(trimetylsilyl)acetamide under heating at 60 °C and stirring for 10 min. The obtained products were analyzed by GC–MS apparatus (Agilent 6890 gas chromatography and 5973 mass spectrometer, Palo Alto, Canada) with the following instrumental conditions: inlet temperature: 250 °C; transfer line temperature: 250 °C, detector temperature: 300 °C and energy of electron: 70 eV. The temperature program was as follows: initial 50 °C (hold 4 min) to 300 °C at rising rate of 8 °C/min and hold time of 4 min at this temperature.

3. Results and discussion

3.1. Characterization of γ-FeOOH nanoparticles

XRD diffractogram of γ-FeOOH nanoparticles is shown in Fig. 1. Nine observed diffraction peaks at $2\theta = 27.0$, 36.2, 38.1, 43.3, 47.0, 49.4, 52.8, 60.6, and 65.08 are assigned to γ-FeOOH [21,22]. No other phase was observed in the synthesized nanoparticles, which indicates the purity of the sample.

TEM image of γ-FeOOH nanoparticle in Fig. 2 confirms nanoscale dimension of the synthesized particle with the width of about 70 nm.

Fig. 3a shows SEM image of γ-FeOOH nanoparticles. Both SEM and TEM figures confirm synthesize of needle-like γ-FeOOH particles. The average width of γ-FeOOH particles was determined from SEM images using the image analysis software (Manual Microstructure Distance Measurement Software, Nahamin Pardazan Asia, Iran). The obtained results shown in Fig. 3b suggest that most of γ-FeOOH particles widths are in nano-range of 60–70 nm. BET surface area was measured to be 68.1 m^2/g. This relatively high BET surface area is beneficial for γ-FeOOH performance in Fenton reactions due to the presence of considerable amount of iron ions on its surface.

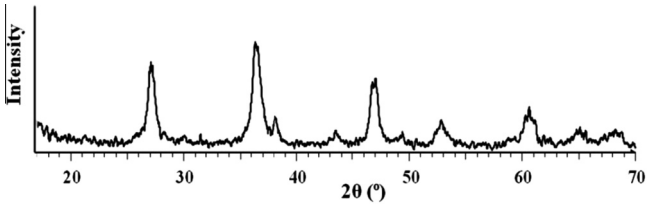

Fig. 1. XRD pattern of synthesized γ-FeOOH.

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