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# Heterogeneous Fenton process by natural pyrite for removal of a textile dye from water: Effect of parameters and intermediate identification

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#### ABSTRACT

The ability of natural pyrite particles in the degradation of C.I. Reactive Orange 29 (RO29) solution by heterogeneous Fenton (pyrite/ $H_2O_2$ ) process was studied. X-ray diffraction, scanning electron microscopy and Fourier transform infrared spectroscopy analysis were performed to characterize the pyrite particles. The decolorization efficiency of the RO29 by pyrite/ $H_2O_2$  process was obviously influenced by pyrite particle size, suspension pH, initial concentrations of  $H_2O_2$  and RO29, and pyrite dosage. Decolorization efficiency of 94.4% was resulted at the initial RO29 concentration of 10 mg/L,  $H_2O_2$  concentration of 3 mM, catalyst dosage of 3 g/L, pH of 2 and reaction time of 120 min. Decolorization efficiency through pyrite/ $H_2O_2$  process was not decreased significantly even after five successive cycles because of the low release (0.6 mg/L) of pyrite iron in each decolorization cycle. Plausible degradation pathway of RO29 was proposed by using gas chromatography–mass spectroscopy analysis.

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#### 1. Introduction

There has been a considerable increase in investigation of the mineral materials' ability in the environmental remediation processes especially through the wastewater treatment during the last decades. This is because of their low cost and optimal environmental harmony in comparison with synthetic compounds [1]. Depending on the composition of mineral materials, they have potential to use in wastewater treatment with the mechanism of adsorption, Fenton-like, photocatalytic degradation and so on [1–3]. The natural magnetite and hematite particles are used in the adsorption of humic acid [4], and natural vanadium–titanium magnetite [5] and sulfurized limonite are used in degradation decomposition of organic compounds by nonhomogeneous Fenton reaction [6].

It is so important to mention that Pyrite ( $FeS_2$ ) is one of the most abundant and nontoxic semiconductors found in the earth's crust [7]. This bright yellow to golden compound was synthesized and used in the adsorption of various organic and inorganic chemicals such as organic dyes [7], molybdate [8] and arsenite [9]. Furthermore, the synthesized pyrite has gained an increasing interest to use in removal of organic pollutants through the advanced oxidation processes because of its potential to use as a catalyst in the heterogeneous Fenton reaction [10,11]. However, few studies have been conducted on

\* Corresponding author. Tel.: +98 41 33393165; fax: +98 41 33340191. *E-mail address:* a\_khataee@tabrizu.ac.ir, ar\_khataee@yahoo.com (A. Khataee). the application of the natural pyrite ore for the treatment of polluted water. Through the present work, for the first time, ability and durability of the pyrite ore as a catalyst in degradation of Reactive Orange 29 (RO29) dye from polluted water and textile wastewater were investigated through the heterogeneous Fenton process. The pyrite particle size, pyrite dosage (g/L), initial H<sub>2</sub>O<sub>2</sub> concentration (mg/L), initial dye concentration (mg/L), reaction time (min) and pH were considered as the operational variables to find the treatment condition with proper performance. The second step of experiments was to perform gas chromatography–mass spectroscopy (GC–MS) analyses on the treated RO29 solution to investigate the destructive side reactions occurrence.

#### 2. Materials and methods

#### 2.1. Materials and reagents

Natural pyrite ore was obtained from Morvarid iron mine (Zanjan, Iran). The mono azo dye, C.I. Reactive Orange 29 was obtained from Ciba-Geigy Ltd (Switzerland). The properties of RO29 are shown in Table 1. RO29 is commonly used as a textile dye for wool and silk. The RO29 containing wastewater was obtained from Farsh & Patu Textile Factory, Tabriz, Iran. Chemical oxygen demand (COD), RO29 concentration, conductivity, and pH of the wastewater were 2173 mg/L, 21 mg/L, 2.26 mS/cm and 8.5, respectively. Hydrochloric acid (37%), sodium hydroxide (99%) and hydrogen peroxide (30%) were

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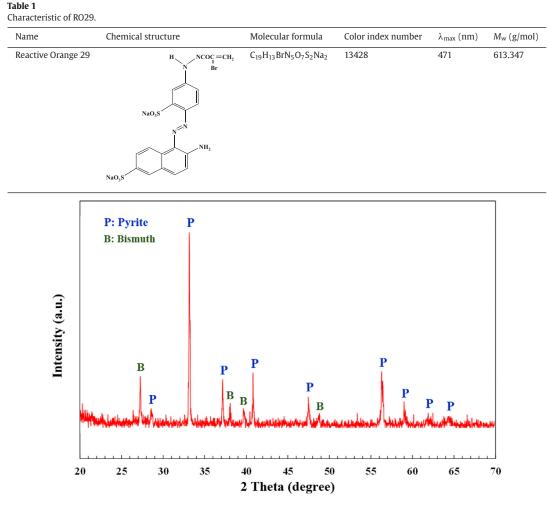


Fig. 1. The XRD pattern of the natural pyrite.

obtained from Merck (Germany). Ethanol was obtained from Jahan Alcohol Teb Co. (Arak, Iran). Distilled water was used throughout the experiments.

#### 2.2. Pyrite ore characterization

Powder X-ray diffraction (XRD) was done by the PANalytical X'Pert PRO apparatus, (Germany) with monochromatic Cu K $\alpha$  X-radiation 0.15406 nm, the accelerating voltage of 40 kV and the emission current of 40 mA. The surface morphology of the material was studied by scanning electron microscopy (SEM) model Mira3 FEG-SEM (Tescan, Czech). The SEM analysis was executed after gold plating of the particles. Fourier transform infrared (FT-IR) spectrum was recorded by Tensor 27, Bruker spectrometer (Germany). The spectra were recorded at the wavenumber ranging from 400 to 4000 cm<sup>-1</sup> on the pellet obtained by mixing the samples in KBr.

#### 2.3. Heterogeneous Fenton process

Batch decolorization studies were carried out to investigate the ability of pyrite/ $H_2O_2$  process in decolorization of RO29 solution. The decolorization reaction was started by adding the desired amount of  $H_2O_2$  solution and pyrite in 250 mL of dye solution under the magnetic stirring. All the experiments were carried out at room temperature. pH of the suspension was determined with a Metrohm pH meter (Switzerland) and adjusted by using HCl and NaOH solutions during the decolorization process. The experiments were carried out

varying pyrite particle size, pyrite dosage (g/L), initial RO29 concentration, initial H<sub>2</sub>O<sub>2</sub> concentration, retention time, and pH. To determine the variation of RO29 concentration during the treatment process at pre-selected time intervals, 5 mL of samples were withdrawn from the flask and taken into the tubes containing 1 mL ethanol solution to quench the reaction by increasing the pH to 8.5–9.0 and analyzed spectrophotometrically at 471 nm by using a UV spectrophotometer (WPA, S2000, England). It was observed that there was no significant change in UV-vis spectrum of RO29 within pH of 2-9. The decolorization efficiency (%) for each sample was equal to  $(C_0 - C_t)/C_0$  where  $C_0$  is the initial concentration of RO29 and  $C_t$ is the RO29 concentration at selected times t (min) in the solution. The released concentration of iron ions to the solution was measured by using novaa 400, Analytikjena Atomic Absorption Spectrometry (AAS) apparatus (Germany). GC-MS analysis was performed to determine the intermediate products formed during the RO29 degradation process. Preparation of sample for GC-MS analysis has been discussed in our previous work [12] and it should be mentioned that silvlated products, which could be detected more conveniently, were prepared by adding N,O-bis-(trimethylsilyl)acetamide after extraction. The solutions 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 temperature of 50 °C (hold 4 min); increase up

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