



# Nanostructure composite ZnFe<sub>2</sub>O<sub>4</sub>–FeFe<sub>2</sub>O<sub>4</sub>–ZnO immobilized on glass: Photocatalytic activity for degradation of an azo textile dye F3B



Mohammad Hossein Habibi\*, Amir Hossein Habibi

Nanotechnology Laboratory, Department of Chemistry, University of Isfahan, Isfahan 81746-73441, Islamic Republic of Iran

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## ABSTRACT

An efficient and scalable one-pot synthetic method to prepare nanostructure composite of ZnFe<sub>2</sub>O<sub>4</sub>–FeFe<sub>2</sub>O<sub>4</sub>–ZnO (ZfZ) has been investigated. This method is based on thermal decomposition of iron(III) acetate and zinc acetate in monoethanolamine (MEA) as a capping agent. Moreover, thermogravimetric analysis (TG-DTG) was performed to determine the temperature at which the decomposition and oxidation of the chelating agents took place. ZfZ was immobilized on glass using doctor blade method and calcinated at different temperatures. The properties of the ZfZ nanocomposite have been examined by different techniques, such as X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and diffuse reflectance (DRS). FESEM shows that nanocomposite is monocrystallines and a narrow dispersion in size of 48 nm. XRD confirms that the prepared nanocomposite is composed of franklinite, ZnFe<sub>2</sub>O<sub>4</sub> (54%), magnetite, FeFe<sub>2</sub>O<sub>4</sub> (8%) and wurtzite, ZnO (48%). Photocatalytic activity of ZfZ immobilized on glass was carried out by choosing an azo textile dye, Reactive Red 195 (F3B) as a model pollutant under UV irradiation with homemade photocatalytic apparatus and the results indicated that ZfZ exhibited good photocatalytic activity.

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## 1. Introduction

Azo textile dyes are the main chemical class of dyes due to their stability and the range of colors available compared to natural dyes. More than 60% of all textile dyes contains azo dyes which making them the largest group of synthetic colorants released into the environment [1–3]. Nanostructure zinc oxide with a large exciton binding energy and wide band gap has received growing interest in the application of photocatalysis [4–8]. Nanostructure zinc oxides show more advantages than titanium dioxide with lower cost and higher quantum yields [9–12]. Nanoparticles of photocatalysts must be separated and reused after application which may encounter technological problems [13]. The immobilization of nano-size photocatalyst overcomes the difficulties in separation and recycle of photocatalyst [14–18]. There are some reports on immobilization of the nano-size photocatalysts on different supports but the problem is lower efficiency of the immobilized photocatalyst compared with nanoparticles suspensions [19–24]. This can be done by application of nanostructured photocatalyst with more active sites, higher surface-to-volume ratio and less electron–hole recombination is needed [25–27]. However, to our best knowledge, there are no report about the

nanostructure composite ZnFe<sub>2</sub>O<sub>4</sub>–FeFe<sub>2</sub>O<sub>4</sub>–ZnO immobilized on glass and their use as photocatalyst for degradation of an azo textile dye F3B. In continuation of our research in semiconductor metal oxide thin film [6,28–30], in the present work, we report for the first time the preparation and characterization of nanostructure composite ZnFe<sub>2</sub>O<sub>4</sub>–FeFe<sub>2</sub>O<sub>4</sub>–ZnO immobilized on glass. The nanostructure coating was characterized in detail by means of XRD, SEM and DRS. We have applied the coatings for photocatalytic decoloration of a textile dye (F3B) (Fig. 1) as an aquatic environmental pollutant.

## 2. Experimental

### 2.1. Materials

Borosilicate glass was used as a support of the ZnFe<sub>2</sub>O<sub>4</sub>–FeFe<sub>2</sub>O<sub>4</sub>–ZnO (ZfZ) nanocomposite. Zinc acetate dihydrate, Zn(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O, iron(III) acetate, Fe(CH<sub>3</sub>COO)<sub>2</sub>, monoethanolamine and isopropanol were used without any further purification. Double-distilled water was used in all the experiments.

### 2.2. Preparation of ZnFe<sub>2</sub>O<sub>4</sub>–FeFe<sub>2</sub>O<sub>4</sub>–ZnO (ZfZ) nanocomposite

To the solution of isopropyl alcohol, 30 ml as a solvent, monoethanolamine, 0.63 ml as a complexing agent, zinc acetate

\* Corresponding author. Tel.: +98 311 7932707; fax: +98 311 6689732.

E-mail addresses: [habibi@chem.ui.ac.ir](mailto:habibi@chem.ui.ac.ir), [mhhabibi@yahoo.com](mailto:mhhabibi@yahoo.com) (M.H. Habibi).

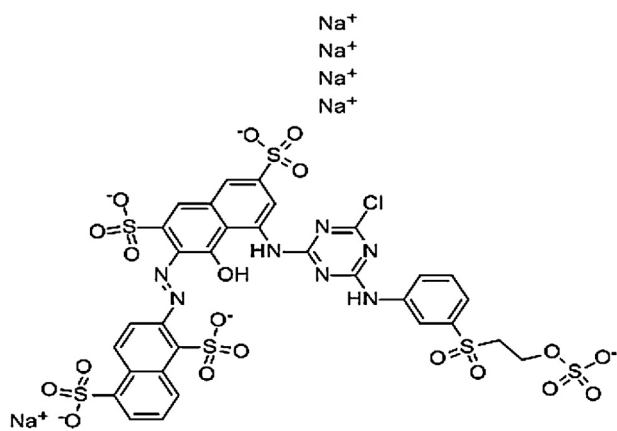


Fig. 1. Chemical structure of Reactive Red 195 (F3B) as a textile dye.

dihydrate,  $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$  (2.304 g), was added while stirring at 60 °C for 1 h to achieve a transparent solution (Sol A). Iron(III) acetate,  $\text{Fe}(\text{CH}_3\text{COO})_3$  (1.826 g) was dissolved in the mixture of isopropyl alcohol, 30 ml and monoethanolamine, 0.63 ml while stirring at 60 °C for 1 h (Sol B). Sol A was added to Sol B with continuous stirring for 10 min at room temperature and aged for 2 days. The solution was heated to 80 °C with formation of a gel. Dried gel was annealed at 700 °C to obtain the  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  (ZFZ) nanocomposite. A schematic diagram of the process is presented in Fig. 2.

### 2.3. Preparation of $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$ paste (ZFZP)

$\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  powders (0.60 g) was grinded in a mortar with 1 mL of acetic acid at 5 min. Double distilled water (1 mL) was added with stirring for 1 min (5 times). Ethanol (1 mL) was added and stirred for 1 min (15 times). Ethanol (2.5 mL) was added and grinded in the mortar for 1 min (6 times). The paste was

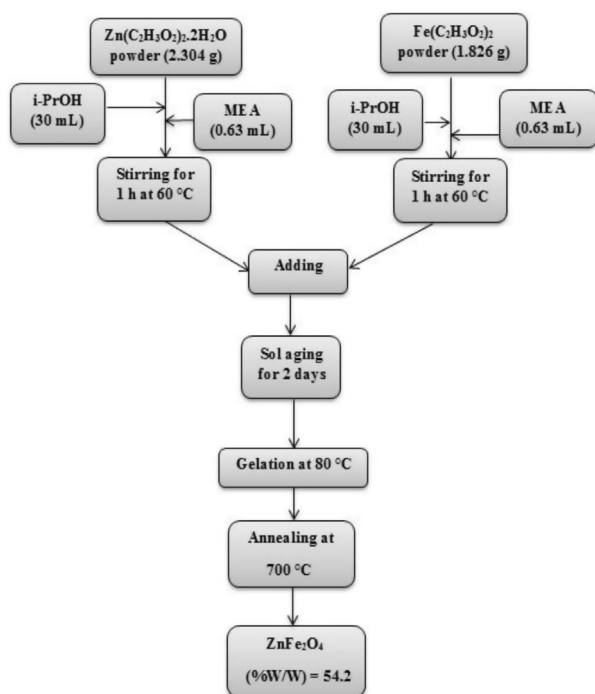


Fig. 2. Flow chart for preparation  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  (ZFZ) nanocomposite paste.

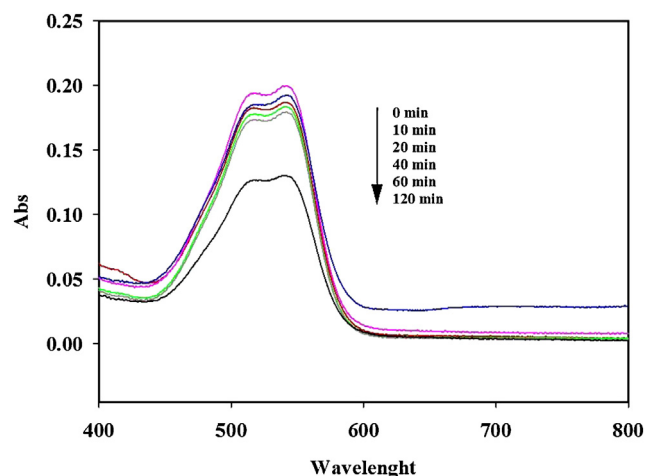


Fig. 3. Absorption spectra of F3B taken at different photocatalytic degradation times using  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  (ZFZ) nanocomposite coated on glass as photocatalysts.

transferred to a 300 mL beaker with addition of 100 mL ethanol. The sample was sonicated for 1 min. Terpineol (20 mg) was added and the paste solution was sonicated. A solution of ethyl cellulose in ethanol was sonicated the solvent was evaporated in a rotary evaporator. It was found that  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$ /ethyl cellulose/terpineol paste is helpful to increase the powder loading and make it possible to produce  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  films with higher surface uniformity and particle density after sintering process.

### 2.4. Coating of $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$ paste on glass by doctor blade (ZFZPC)

The paste was coated on a glass slide by doctor blade method and the thin film annealed at 550 °C for 2 h. Microscope slides were carefully cleaned by ethanol and dried in an oven.  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  paste immobilized on glass substrates were fabricated using the doctor blade method [31,32]. The area of the  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  coated glass was 11.25 cm<sup>2</sup>, length 7.5 cm and width 1.5 cm. The ZFZPC coated glass was air dried, annealed at 550 °C and cooled [33,34].

### 2.5. Characterization

The thermoanalytical measurements (TG-DTG) study for the thermal decomposition of precursors were carried out with a using a Mettler TA4000 system from 20 to 700 °C at a heating rate of 5 °C min<sup>-1</sup>.  $\text{ZnFe}_2\text{O}_4\text{-FeFe}_2\text{O}_4\text{-ZnO}$  paste on glass by doctor blade was characterized by XRD analysis using X-ray diffractometer (D8 Advance, BRUKER) in the diffraction angle range  $2\theta = 20\text{--}60^\circ$ , using  $\text{Cu K}\alpha$  radiation. The crystallite size  $D$  of the sample was estimated using the Scherer's equation,  $(0.9\lambda)/(\beta \cos \theta)$ , by measuring the line broadening of main intensity peak, where  $\lambda$  is the wavelength of  $\text{Cu K}\alpha$  radiation,  $\beta$  is the full width at half-maximum, and  $\theta$  is the bragg's angle. Field emission scanning electron microscopy (FE-SEM, Hitachi, model S-4160) was used to observe the surface morphology of the  $\text{ZnFe}_2\text{O}_4$  photocatalyst. Diffuse reflectance spectra (DRS) were collected with a V-670, JASCO spectrophotometer and transformed to the absorption spectra according to the Tach relationship. FT-IR absorption spectra of selected samples were obtained using KBr disks on a FT-IR 6300. A UV-Spectrophotometer (Varian Cary 500 Scan) was used to measure F3B azo textile dye concentrations at various time intervals during the reactions at wavelength 563 nm (Fig. 3).

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