



Application of BiFeO₃-based on nickel foam composites with a highly efficient catalytic activity and easily recyclable in Fenton-like process under microwave irradiation



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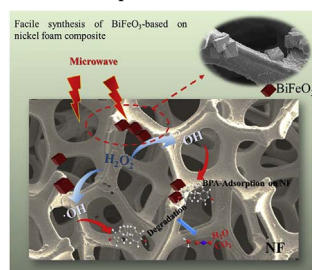
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HIGHLIGHTS

- BFO/NF was prepared via a one-step microwave-assisted hydrothermal method.
- BFO/NF exhibited a superior catalytic activity in MW-Fenton-like process.
- NF has a synergistic effect with BFO and MW on •OH generation.
- BFO/NF facilitates the industrialization application in wastewater treatment.

GRAPHICAL ABSTRACT

Schematic representation of BPA degradation using BFO/NF in MW-Fenton-like process.



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ABSTRACT

In this study, BiFeO₃ (BFO) powders decorated on nickel foam (NF) with a high catalytic activity are prepared via a one-step microwave-assisted hydrothermal method. The factors that influence the degradation of bisphenol A (BPA) with BFO/NFs as catalysts are optimized to improve the catalytic activity in a microwave-enhanced Fenton-like process. BFO/NF exhibit a superior catalytic activity with a high BPA removal ratio (98.4%) and TOC removal ratio (69.5%) within 5 min. Results indicate that NF significantly affect the improvement of the catalytic activity of BFO because it served as a source of hydroxyl radicals (•OH) during degradation. The amount of •OH generated by BFO/NF is approximately 1.65-fold higher than that by pure BFO. After six reaction cycles, the stability and reusability of •OH remain high. These findings provide new insights into the synthesis of composites on heterogeneous catalysts with high efficiency and easy recyclability for water treatment applications.

1. Introduction

BiFeO₃ (BFO), which possesses a highly crystalline perovskite structure, exhibits multiferroic properties with electrical and magnetic parameters in the same phase [1]. With its unique properties, BFO powders have been widely used in many applications, such as multifunctional materials [2], magnetoelectric sensors [3], electronic

memory devices [4], intelligent sensors [5]. BFO powders are usually used as catalysts in photo or photo-Fenton process, few are acted as a Fenton-like catalyst in wastewater treatment [6]. However, its catalytic capability is limited and the targeted pollutant always is dye (such as Huo et al. [7] used BFO as a Fenton-like catalyst to treat methylene blue, An et al. [8] also prepared BFO to degrade methyl violet, Rhodamine B) which should be improved to promote degradation of

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organic contaminants, such as bisphenol A (BPA). BPA is one of the most common Environmental Endocrine Disruptors (EEDs), which can cause bioaccumulation and reproductive toxicity which are very harmful to human health and ecological environment.

In addition, BFO powders are difficult to recycle and separate due to the weak magnetic properties, and these characteristic hampers the utilization of pulverous BFO catalyst powders in wastewater treatment. Thus, BFO nanoparticles will have positive influences in the research of efficient recycling and convenient separation on the application. Several methods, including preparation and loading of magnetic materials on suitable carriers [9], have been developed to prepare immobilized powder-type catalysts and thus overcome defects. Highly porous materials, such as ceramic foam [10], porous alumina porous silica [11], zeolite [12], and activated carbon, are typically chosen as catalyst supports [13]. Nickel foam (NF) as a catalyst support has been attracted considerable attentions due to the excellent electric, heat conductivity and a large active surface area to boost the redox reactions. Some researchers have reported that the composite foams used as an electrode to decorate with (e.g. graphene and TiO₂ [14], CuO [15], carbon nanotubes [16]) for non-aqueous redox flow batteries which could enhance in light harvesting and photoelectric conversion. Nevertheless, few results have been reported the catalytic capability of composite foams in the MW-Fenton-like process. Thus, we prepared BFO powders decorated on NF with a high catalytic activity via a one-step microwave-assisted hydrothermal method for exploitation in widespread environmental applications.

In our previous studies, microwave heating has been investigated in detail for degradation reaction, disinfection for water reuse, adsorbent regeneration or synthesis process [17–22]. We found that microwave (MW)-assisted hydrothermal method is an environmentally friendly technology and it can be automatically controlled. On one hand, MW heating can shorten catalyst preparation time and enhance catalytic activity. On the other hand, MW influences the improvement of molecular thermal motion and molecular collision by eliciting thermal effects [23,24]. Moreover, MW is efficient in the selective heating of MW-absorbing phases and components in materials without energy wastage, thereby increasing the chemical reaction rate [25,26]. BFO was used as a catalyst, which is a MW-absorbing material because of its large dielectric constant [27–29]. The synergistic effect of MW and MW-absorbing materials on the shortening of synthesis time has also been described [30,31].

In the present study, BFO/NF catalysts were prepared via a one-step hydrothermal method followed by calcination. After optimizing the influencing factors for BFO/NF preparation, we investigated the catalytic activity in detail by using the removal rate and TOC removal rate of BPA as evaluation indicators in the MW-Fenton-like process. The mechanism of the modified catalytic activity was discussed comprehensively by analyzing the structural changes and effects of free radicals.

2. Experimental methods

2.1. Preparation of BFO/NF materials

To synthesize BFO, we prepared a mixture (1.0 mM) of Bi(NO₃)₃·5H₂O and Fe(NO₃)₃·9H₂O with a molar ratio of 1:1 in deionized (DI) water. The mixture was stirred for 15.0 min while maintained in a water bath at room temperature. NaOH (1.0 M) solution was slowly added to the mixture to produce a brown precipitate with stirring for 15 min. After the BFO precursor solution was prepared, it was added with NF (1.0 g), mixed, and stored in a Teflon container. The mixture was transferred into a MW oven (MDS-6G, Shanghai Sineo Microwave Chemistry Technology Co., Ltd., China) and treated at a MW power of 300.0 W with a frequency of 2450.0 MHz at 200.0 °C for 40.0 min [29]. A composite was generated, collected, and repeatedly washed with DI water and ethanol thrice. The prepared BFO/NF was dried in a vacuum

drying oven at 70.0 °C for 2.0 h. The final product was labeled as BFO/NF and used as a catalyst.

The supported catalyst was systematically optimized with respect to catalyst dosage. The loading number of active components was calculated by using the following equation:

$$L (\%) = (Wt - Wt_0) / Wt \times 100\% \quad (1)$$

where L is the loading amount of active components. Wt_0 and Wt are the weights of NF (net) and BFO/NF (g), respectively. All the loading amounts of active components exceeded 50%.

2.2. Characterization

The samples were characterized with using a scanning electron microscope spectrometer (JEOL JSM-6700F), an X-ray diffraction analyzer (XRD, Rigaku, RXIII), an X-ray photoelectron spectrometer (XPS) system (K-Alpha, Thermofisher Scientific Company, US), and a 6000 Thermal Electron inductively coupled plasma mass spectrometry.

2.3. Catalytic degradation of BPA

To reach the adsorption–desorption equilibrium of the concerned compounds, we dispersed BFO/NF (2.0 g L⁻¹) into 100.0 mL of BPA (30.0 mg L⁻¹) solution at pH 5.0 and agitated it with a beaker for 30.0 min. Aqueous samples were taken every 3.0 min to determine the BPA concentration in the solution. After reaching adsorption equilibrium, H₂O₂ (24 mg/L) was added into the aqueous BPA solution. Subsequently, the solution was irradiated for 5.0 min under 200 W in an MW oven (2450 MHz, Cooplex-E, Shanghai, China) [29]. During the treatment, the samples (at a given time interval of 1 min) were filtered through a 0.22 μm polyether sulfone filter (JinTeng Laboratory Equipment Co., Ltd.), obtained, and measured immediately by using a UV-vis spectrophotometer (L5S, Shanghai instrumental analysis instrument Co., Ltd., China). All of the experiments were conducted in triplicate to confirm their reproducibility.

$$R (\%) = (C_0 - C) / C_0 \times 100\% \quad (2)$$

where R represents the removal ratio of BPA. C_0 and C represent the initial and instantaneous concentrations of BPA (mg L⁻¹), respectively.

The amounts of •OH generated were detected using a molecular fluorescence spectrometer (RF-5301PC, Island Ferry International Trading Co., Ltd., Japan). During this reaction, terephthalic acid was used as a capture agent (30.0 mg/L) to replace BPA. The process was the same as that for the MW-Fenton-like one. •OH was determined with a photoluminescence technique via our reported method [18]. Methanol (MeOH) and tert-butanol (TBA) obtained from Aladdin were used as radical scavengers to detect active groups.

2.4. Regeneration of BFO/NF

The used composite was collected, washed thrice with DI water and ethanol, dried in a vacuum drying oven at 70.0 °C for 2.0 h, and calcined at 450 °C for 3.0 h under N₂ atmosphere.

3. Results and discussion

3.1. Characterization of the catalyst of BFO/NF composites

The morphological characteristics of naked NF, BFO/NF composite, and BFO are presented in Fig. 1. Bare NF exhibits a uniform and smooth three-dimensional network structure with a large specific surface area (Fig. 1a). Fig. 1c shows that BFO has a cubic structure with a dimension between 8.0 and 12.0 μm. The holes of the NF framework coexist with many cubic particles, indicating that the BFO particles are closely deposited on the surface of NF (Fig. 1e). When the BFO particles are

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