



## Facile synthesis of a BiFeO<sub>3</sub>/nitrogen-doped graphene nanocomposite system with enhanced photocatalytic activity

Maryam Kiani<sup>a,\*</sup>, Syed Rizwan<sup>a</sup>, Syed Irfan<sup>b</sup>

<sup>a</sup> Department of Physics, School of Natural Sciences, National University of Science & Technology (NUST), Islamabad, 44000, Pakistan

<sup>b</sup> School of Material Science and Engineering, Tsinghua University, China



### ARTICLE INFO

#### Keywords:

Nitrogen-doped graphene  
Photocatalytic activity  
Sol-gel synthesis  
Spinel BiFeO<sub>3</sub>

### ABSTRACT

In this study, BiFeO<sub>3</sub> (BFO) nanoparticles were synthesized via the sol-gel route and BFO/nitrogen-doped graphene (NG) nanocomposites (BFO/NG, NG: 1%, 2%, 3%, 4%, 5%, and 6%) were prepared by dispersing the BFO nanoparticles and NG in ethanol solution, before thermal drying. The structural and morphological properties of the pure BFO nanoparticles, NG, and BFO/NG nanocomposites were studied by X-ray diffraction, scanning electron microscopy (SEM), and transmission electron microscopy. The average crystallite size of the BFO nanoparticles was 57 nm. The SEM observations demonstrated that the BFO nanoparticles were well anchored on the NG. The photocatalytic activities of the as-synthesized samples were calculated based on the degradation of methyl orange under simulated sunlight irradiation. The BFO/NG nanocomposites exhibited enhanced photocatalytic activity compared with the pure BFO nanoparticles. The enhanced photocatalytic performance was attributed to the efficient transfer of photogenerated electrons from the BFO nanoparticles to NG, thereby leading to the increased availability of h<sup>+</sup> for the photocatalytic reaction. Furthermore, hydroxyl (-OH) radicals were detected using the photoluminescence technique with terephthalic acid as a probe molecule and they were generated on the irradiated pure BFO nanoparticles and BFO/NG nanocomposites.

### 1. Introduction

Numerous oxide semiconductors (SnO<sub>2</sub>, TiO<sub>2</sub>, and ZnO) are recognized as highly effective for photocatalytic degradation because of their exceptional ultraviolet (UV) absorbance, low cost, nontoxicity, and high photocatalytic performance [1,2]. Recently, it has been shown that BiFeO<sub>3</sub> (BFO) is an effective visible light-driven photocatalyst due to its appropriate band gap (~2.2 eV), good chemical stability, and intrinsic electric polarization field [3–5]. However, the reported photocatalytic performance of BFO is very low because of the rapid recombination of electron-hole pairs [6]. Multiferroic materials have been studied extensively because of their potential applications in areas such as satellite communication, audio-video devices, spintronic devices, and digital recording [7,8]. Indeed, BFO has great potential for use in magnetoelectric applications at room temperature. BFO has a rhombohedral distorted structure and it belongs to the R3c group [9]. The magnetic behavior of BFO is attributable to the partly filled 3d orbital electrons of the Fe<sup>3+</sup> ions leading to G-type antiferromagnetism [10]. Bi-O hybridization due to the stereochemical activity of the Bi<sub>6</sub>S<sub>2</sub> lone pair explains the ferroelectric property of BFO [10]. Semiconductor-based photocatalysts are important because of their potential

applications in solar energy conversion and environmental purification [11,12]. In particular, TiO<sub>2</sub> is recognized as very useful photocatalyst for the degradation of various organic compounds [13–16]. Moreover, it is necessary to identify suitable visible light-driven photocatalysts to better utilize solar energy, which mostly comprises visible light. Studies have shown that BFO has a visible light-responsive photocatalytic activity during the degradation of organic contaminants [17]. BFO is a potential visible light-driven photocatalyst with applications in the degradation of organic pollutants and water splitting because of its narrow band-gap (2.2 eV) [18–22]. The activity of a photocatalyst depends on various factors. In particular, the effective splitting of photogenerated electron-hole (e<sup>-</sup>-h<sup>+</sup>) pairs is most important for enhancing the photocatalytic activity. Previously, Hu et al. reported Sm-doped BFO nanostructures with improved visible light photocatalytic activity [23]. It has also been reported that substituting Bi<sup>3+</sup> (1.03 Å) with rare earth ions that have smaller ionic radii, such as Gd<sup>3+</sup> (0.938 Å), can enhance the photocatalytic properties due to the significant structural distortion of the BFO lattice [24].

Graphene possesses exceptional properties including chemical stability, high electrical conductivity, electron mobility, thermal conductivity, and mechanical strength [25–27]. Graphene is currently used

\* Corresponding author.

E-mail address: [maryam.kiani@sns.nust.edu.pk](mailto:maryam.kiani@sns.nust.edu.pk) (M. Kiani).

## Synthesis of BFO



Fig. 1. Method employed for producing BFO nanoparticles via the sol-gel method.

as an ideal support because of its excellent properties and it has been integrated with a large number of efficient nanomaterials to form nanocomposite systems with enhanced performance in the fields of photocatalysts [28–35], fuel cells [36], micro-supercapacitors [37], and field-emission emitters [38]. The synthesis of graphene with photocatalysts is an efficient route for promoting the separation of  $e^-h^+$  pairs [28–35]. In graphene-based photocatalyst nanocomposite systems, graphene acts as an electron acceptor because it can freely capture photogenerated electrons. Therefore, more photogenerated holes are accessible for photocatalytic reactions. Hence, many studies have aimed to combine graphene with BFO-based composite nanomaterials. BFO-graphene composites have frequently been synthesized via hydrothermal and sol-gel methods. In the hydrothermal methods, the BFO precursors were introduced into the dispersed aqueous graphene or graphene oxide, before a hydrothermal reaction to obtain BFO-graphene nanocomposites [36–39]. In the sol-gel method, the sol comprising precursors and graphene oxide was dried, and the resultant product was then thermally treated in  $N_2$  atmosphere to obtain the BFO-graphene nanocomposite [21]. However, it should be noted that the hydrothermal method and high temperature thermal treatment processes may destroy the graphene lattice [40]. In addition, doping carbon materials with a heteroatom, such as B, N, or S, can enhance the pseudocapacitance by controlling the electronic properties and chemical reactivity, thereby improving the efficiency of the doped graphene [41–44]. Thus, nitrogen-doped graphene (NG) may be a suitable electrode material for applications in photocatalysis. Previously, Li et al. reported the enhanced photocatalytic activity of bismuth ferrite combined with NG and prepared via the hydrothermal method [49,50]. Moreover, the photocatalytic performance depends on the shape and size of the nanoparticles in many other nanocomposites, such as  $LaFeO_3$  [51],  $GdFeO_3$  [52],  $LuFeO_3$  [53],  $PrFeO_3$  [54], and  $YFeO_3$ .

According to previous research, the facile synthesis of nanocomposite systems is highly desirable and an appropriate efficient carbon-based material is required to improve the photocatalytic efficiency.

In this study, we evaluated the effects of the nitrogen concentration in nanocomposites prepared via a sol-gel method on their

photocatalytic activity. We found that BFO nanoparticles can be supported on NG by combining BFO nanoparticles and NG in absolute ethanol solution, before thermal drying. NG has great advantages due to its higher specific capacitance compared with pure graphene and great durability. Moreover, its electronic properties in the “activation region” are enhanced by the introduction of nitrogen atoms in the carbon lattice. However, its efficiency is not good [45–48] so we tested different concentration of NG to try to improve the photocatalytic performance. The photocatalytic activities of the as-prepared BFO/NG nanocomposites were determined based on the degradation of methyl orange (MO) under irradiation by simulated sunlight. The production of  $\cdot OH$  radicals on the irradiated BFO/NG nanocomposites was detected using the photoluminescence (PL) technique.

## 2. Experimental

### 2.1. Synthesis of BFO nanoparticles

BFO nanoparticles were prepared via a sol-gel method, as described previously [55]. Briefly, 15 mmol  $Bi(NO_3)_3 \cdot 5H_2O$  was dissolved in 36 mL of ethylene glycol ( $HOCH_2CH_2OH$ ) to form a transparent solution. Next, 15 mmol  $Fe(NO_3)_3 \cdot 9H_2O$  was added to the solution and stirred for 1 h until a brownish red colloidal sol was obtained. The sol was dried at  $80^\circ C$  for 48 h in a vacuum oven and the BFO xerogel powder was produced. The xerogel was heated at  $400^\circ C$  for 30 min to eliminate  $NO_3^-$  and organic compounds. The remaining powder was then maintained at  $500^\circ C$  for 30 min. Finally, the as-synthesized sample was washed with deionized water and absolute alcohol several times, before drying overnight at  $80^\circ C$ . The synthesis of BFO via the sol-gel method is shown in Fig. 1.

### 2.2. Synthesis of BFO/NG nanocomposites

Fig. 2 shows a schematic to illustrate how the BFO nanoparticles were successfully anchored on the NG. The NG used in this study was purchased from XF Nano, INC Advance Materials Tech Co. Ltd. The BFO nanoparticles and NG were dispersed in ethanol solution and

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