



Synthesis of FeCo-reduced graphene oxide composite and its magnetic and adsorption properties



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ABSTRACT

The composite of FeCo and reduced graphene oxide (RGO) was synthesized via a facile ultrasonic chemical method. FeCo nanoparticles were firmly dispersed on the surfaces of RGO nanosheets. Interestingly, the graphene scrolls were also observed in the composite. The composite exhibited excellent magnetic property with specific saturation magnetization (M_s) of 78.06 emu/g and strong adsorption for Rhodamine-B. The comprehensive properties made the composite have great potential applications in water treatment.

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1. Introduction

Recently, considerable efforts have been made toward the development of magnetic nanoparticles decorated graphene owing to their potential applications in various fields, the energy storage [1], electromagnetic wave absorption [2] and water purification [3], to name a few. The synergetic effects between graphene and the magnetic nanoparticles make the composites exhibit the superior properties [4,5]. (1) The magnetic nanoparticles are beneficial to prevent graphene nanosheets from aggregation and maintain their high surface area [6]. (2) The nanoscale surface of graphene could prevent the agglomeration of these magnetic nanoparticles [7]. (3) Pristine graphene was found to be non-magnetic [8], but decorating magnetic nanoparticles onto graphene would introduce the significant magnetic characteristic.

FeCo alloys, with good thermostability, high saturation magnetization and magnetic permeability, were among the most promising soft magnetic materials [9,10]. Based on its outstanding properties, FeCo alloys have been widely used in high frequency power applications [11], magnetic resonance imaging [12], magnetoelastic soft actuators [13] and recently in the nano-medicine applications [14]. It has been reported that the salts of FeCo could integrate with graphene oxide (GO) by the electrostatic interaction [15]. And very recently, Deng et al. also

reported that FeCo nanoparticles encapsulated in graphene nanosheets exhibited highly active reduction of oxygen [16]. To our best knowledge, other microstructure of the composite of FeCo and reduced graphene oxide (RGO) have been rarely researched.

Herein, we developed a facile ultrasonic chemical method to successfully synthesize the FeCo-RGO composite. And the FeCo-RGO composite was investigated for its unique microstructure including the scrolled structure, the magnetic and adsorption properties for organic dyes.

2. Experimental

2.1. Materials

Graphite was purchased from Sinopharm Chemical Reagent Co., Ltd. All chemicals were of analytical reagent without further note and used as received.

2.2. Syntheses of FeCo-RGO composite

GO was synthesized by a modified Hummers starting from graphite powder [17]. Firstly, 0.3336 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and 0.1904 g of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ were dissolved into 4 ml of deionized water and the mixture was stirred at 30 °C for 30 min to form a homogenous solution. Then, 5 g NaOH in 10 ml in deionized water was slowly added into the solution under vigorous stirring. The as-synthesized $\text{Fe}_{60}\text{Co}_{40}$ mixture was added into 20 ml of an aqueous GO suspension (1 mg/ml). After that, 10 ml of hydrazine hydrate was added to reduce graphene oxide, iron ions and cobalt ions

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simultaneously under magnetic stirring. Lastly, the solution was treated in a 60 °C water bath for 5 h with the help of ultrasonic dispersion. The final product followed by magnetic separation was washed repeatedly with deionized water and ethanol (near neutral) and then dried at 80 °C for 24 h. As a comparison, pristine Fe₆₀Co₄₀ nanoparticles and pristine RGO were also produced under the same experimental condition without GO and the metallic salts, respectively. In the adsorption experiment, composite sample was made by mixing of FeCo-RGO composite (0.19 g) and 20 ml Rhodamine-B solution (2.5 ppm).

2.3. Characterization techniques

The morphology of the composite was measured using a Hitachi S-4800 field emission scanning electron microscope (SEM). The structure of the synthesized material was investigated by X-ray diffraction (XRD) equipped with Cu K α source ($\lambda = 1.54187 \text{ \AA}$), scan range 10–90°. The Raman spectrum was carried out using a Renishaw InVia Raman microscope with 532 nm line of an Ar ion laser as an excitation source. X-ray photoelectron spectroscopy (XPS, PHI-5702, Physical Electronics) were obtained by monochromated Al K α irradiation. The magnetization was characterized by a vibrating sample magnetometer (VSM). The photoluminescence (PL) spectra were determined by an FLS-920T fluorescence spectrophotometer.

3. Results and discussion

Fig. 1(a) shows the pristine RGO nanosheets, which corrugated and agglomerated to form the irreversible coagulation. Fig. 1(b)

reveals that most of the FeCo nanoparticles (diameter ca. 80 nm) are firmly distributed on the surface of RGO nanosheets (thickness ca. 3 nm). Due to the electrostatic interaction between Fe²⁺, Co²⁺ and GO (COOH[−], OH[−]), it is believed that Fe²⁺ and Co²⁺ were attracted onto the GO surface and facilitated the reduction of FeCo nanoparticles and GO in situ simultaneously, which served as the initial nucleation sites and optimal load positions [15]. Compared with Fig. 1(a), the dispersed magnetic FeCo nanoparticles on the surface of RGO nanosheets as nanoscale spacers impede the agglomeration of RGO nanosheets [18,19]. Fig. 1(c) exhibits that the FeCo nanoparticles are covered and wrapped by the flexible RGO nanosheets. Besides that, a few RGO nanoscrolls are also detected in the samples with the diameter of ca. 2 μm and the length of ca. 10 μm (Fig. 1(d)). It could be concluded that the main growth mechanism of RGO nanoscrolls are the magnetically interacting FeCo nanoparticles and their strong adsorption on the RGO nanosheets [20]. In addition, Fig. S1 (Supplementary material) shows another four SEM images of the RGO nanoscrolls. It was also reported that graphene nanoscrolls were formed by decorating the $\gamma\text{-Fe}_2\text{O}_3$ with the diameter of ca. 38 nm [20].

Fig. 2(a) shows XRD patterns of FeCo (upline), RGO (midline) and FeCo-RGO composite (downline). The diffraction peaks of FeCo at 44.87°, 65.31°, 82.73° are attributed to the (110), (200), (211) planes of the body centered cubic structure of FeCo. A broad diffraction peak of RGO at 23.5° is assigned to the (002) plane of C. The major diffraction peak positions of FeCo-RGO composite are similar to that of the FeCo, but the intensities of the peak are relatively low. In addition, a broad diffraction peak for C (002) at 24.6° confirms the existence of RGO in the FeCo-RGO composite. The other detectable low reflections of FeCo-RGO composite would

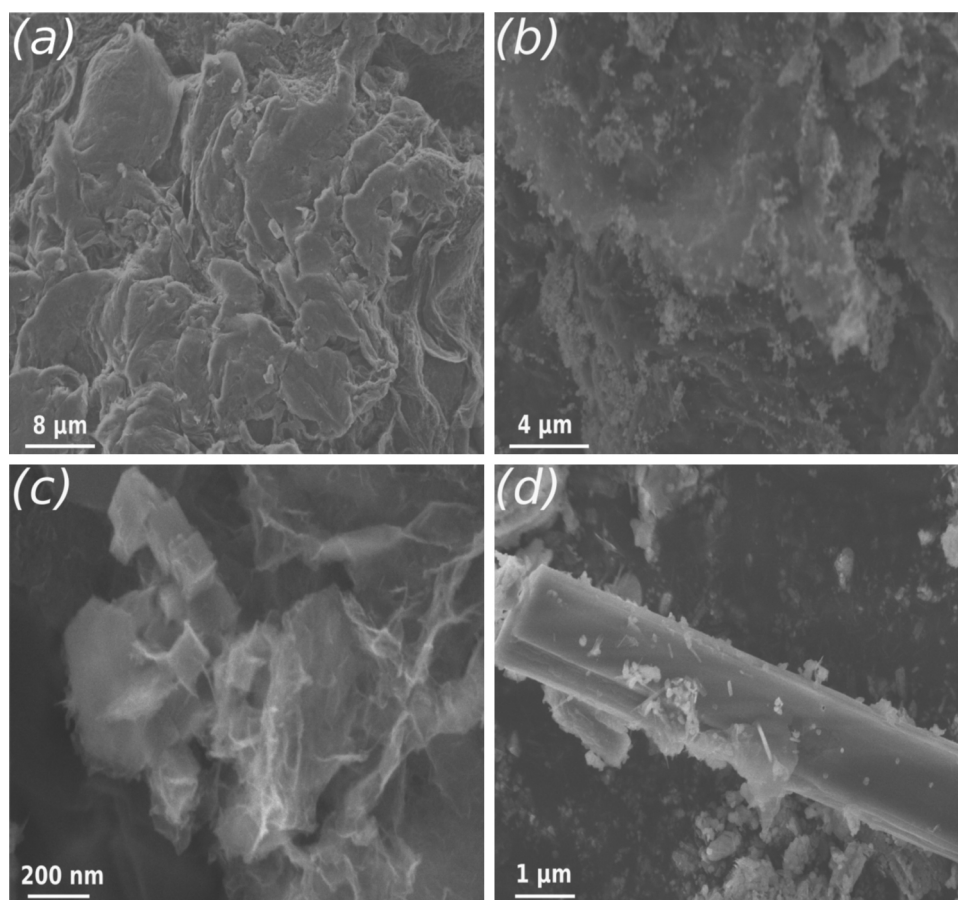


Fig. 1. SEM images of RGO (a) and FeCo-RGO composite (b–d).

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