



Regular Article

Fabrication of smart magnetite/reduced graphene oxide composite nanoparticles and their magnetic stimuli-response



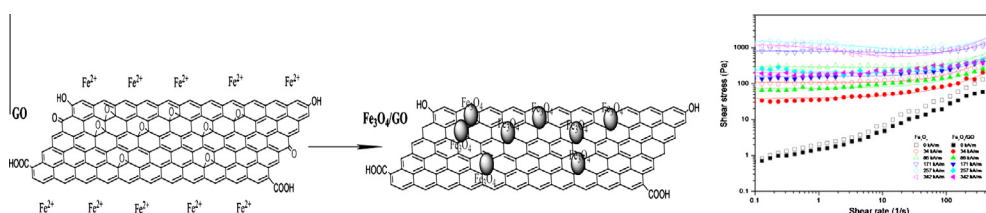
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GRAPHICAL ABSTRACT

A MR materials composed of $\text{Fe}_3\text{O}_4/\text{GO}$ composite particles were fabricated through in situ chemical deposition. When dispersed in silicone oil, their magnetorheology properties were measured by using a rotational rheometer at various magnetic field strengths under steady flow and dynamic oscillation tests.



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ABSTRACT

Novel $\text{Fe}_3\text{O}_4/\text{reduced graphene oxide (RGO)}$ composite nanoparticles were synthesized and confirmed by FT-IR spectra as good candidates for magnetic stimuli-responsive magnetorheological (MR) materials. The morphology of $\text{Fe}_3\text{O}_4/\text{RGO}$ was observed by both scanning and transmission electron microscopy and their sedimentation stability improved due to a decreased density of the synthesized composites. The MR performance of the $\text{Fe}_3\text{O}_4/\text{RGO}$ -based fluid was investigated with a rotational rheometer, and the Cho-Choi-Jhon model of the rheological equation of state was adopted to explain their performances for the entire shear rate region.

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1. Introduction

Graphene oxide (GO) possesses a layer structure containing phenolic, carboxyl and epoxide groups introduced by oxidation, contrasting with the pristine surface of graphene [1]. The presence of these functional groups, namely high amounts of hydroxyls and epoxides, allows GO to disperse well in water, favoring the

combination with other materials in water [2,3]. GO has attracted attention in various industrial applications due to unique mechanical properties, low molecular weight and favorable hydrophilic properties. In addition, not only the magnetic properties of GO have been theoretically investigated [4], but the application of GO-based magnetic materials as magnetorheological (MR) fluids has also been reported [5].

Recently, the potential of nanocomposites containing GO or reduced GO (RGO) with magnetite (Fe_3O_4) nanoparticles has been widely studied for applications in targeted drug delivery, magnetic

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resonance imaging and heavy metal ion removal from aqueous solutions [3,6–8]. In particular, the $\text{Fe}_3\text{O}_4/\text{GO}$ composite particles have been also actively studied as an anode of lithium-ion batteries [9,10]. Given that RGO has a similar structure to GO, it can also be applied as a MR fluid. The large surface area and stability of RGO motivated us to synthesize $\text{Fe}_3\text{O}_4/\text{RGO}$ composites [3]. Fe_3O_4 has been frequently used in biosensors and water treatment due to their high biocompatibility, low toxicity, easy preparation, and special magnetic properties. Methods to synthesize graphene-based nanocomposites with Fe_3O_4 include in situ chemical deposition, hydrothermal treatment, sol-gel procedure, reduction of iron salt precursors, and self-assembly of Fe_3O_4 on GO or RGO sheets [11–14]. These hybrids could not only enhance the dispersion stability of Fe_3O_4 , but also reduce the restacking or aggregation of the GO or RGO sheets themselves [11–14]. On the other hand, it can be also noted that dual stimuli-response of both electrorheological (ER) under electric fields applied and MR characteristics from GO-coated iron oxide/silica core-shell nanoparticles has been recently reported [15].

MR fluids, as mentioned above, consist of soft magnetic micron-sized particles dispersed in nonmagnetic fluids such as hydrocarbon, silicone oil, or aqueous carrier fluids. They have been extensively investigated due to their interesting phase transition characteristics under an applied magnetic field strength, and their engineering applications in a variety of multidisciplinary areas [16–19]. Their rheological properties can vary significantly with the magnetic field strength, permitting a fine-tuning of the materials behavior [20,21]. MR fluids show characteristics of Newtonian fluids when no magnetic field is exerted. However, in the presence of a magnetic field, they exhibit a continuous, rapid, and reversible change from a fluid-like to a solid-like phase [20,21]. This is because dispersed magnetic particles can form chains, which align in the direction of the magnetic field due to the magnetic-polarization interaction, and then return to its free-flowing liquid state upon removal of the external magnetic field [22–25]. Due to their outstanding controllable mechanical characteristics with a high yield stress value, MR fluids have been adopted in various engineering devices such as shock absorbers, brakes, active dampers, and so on [26]. In addition to their high yield stress, since the magnetic field is more stable under operation than an electric one, several commercially available products based on MR fluids have been developed. Moreover, their application has been quite more extensive than that of ER fluids working under an applied electric field strength [27].

Among various magnetic materials, the excellent magnetic properties and particle sizes of soft magnetic carbonyl iron (CI) microspheres have been widely adopted for MR fluids and elastomers. However, application of the CI-based MR fluids in devices requires improvements in the fluid properties, because sedimentation of the heavy CI particles due to the significant density mismatch between the CI particles and oil medium hampers MR device operation as well as their redispersion after some time. The viscoelastic behavior of the CI-based MR fluid shows strong solid-like characteristics under an applied magnetic field strength [28]. So far, the most popular MR materials are CI particles (CD grade, BASF Germany, average particle size: 4.25 μm , density: 7.91 g cm^{-3}) due to their high saturation magnetization and appropriate particle size [29–31]. Most CI-based magnetic materials possess severe sedimentation problems due to their large density, when used as dispersed particles in MR fluids. Thus, a significant effort such as introducing additives or polymer coating technology has been carried out to prevent contact between CI particles and decreasing CI particle density in order to improve the sedimentation stability [32–34].

Compared with the complicated process of modifying CI particles, the use of other magnetic species, such as Fe_3O_4 particles

with much lower density (4.32 g cm^{-3}) but sufficiently good magnetic behavior gains an advantage [35,36]. In this work, aiming to reduce the density mismatch between Fe_3O_4 and the oil medium (0.96 g cm^{-3}) as well as the aggregation problem of nanoscaled Fe_3O_4 particles, RGO was introduced by a hydrothermal method. This method was chosen to produce $\text{Fe}_3\text{O}_4/\text{RGO}$ due to its advantages, like cost efficiency, synthesis of uniform Fe_3O_4 nanoparticles with good stability, and high magnetization.

GO was firstly prepared by oxidizing graphite with acid via the Hummers method. Then, the oxidized material was exfoliated in water through an ultrasonication treatment, followed by the reduction of the exfoliated GO by hydrazine hydrate in the solution of FeCl_3 and FeCl_2 to produce the $\text{Fe}_3\text{O}_4/\text{RGO}$ nanocomposite. Finally, the $\text{Fe}_3\text{O}_4/\text{RGO}$ particle-based MR fluid was prepared. This simple and facile process of fabricating the $\text{Fe}_3\text{O}_4/\text{RGO}$ particle is considered to be new and novel for MR community. The morphology of the $\text{Fe}_3\text{O}_4/\text{RGO}$ nanocomposites was observed via scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images and Fourier-transform infrared (FT-IR) spectroscopy. The MR performance of the synthesized $\text{Fe}_3\text{O}_4/\text{RGO}$ particle-based MR fluid was investigated as a function of magnetic field strength. The sedimentation stability was also checked by both testing the density of the $\text{Fe}_3\text{O}_4/\text{RGO}$ particles and measuring its sedimentation profile.

2. Experimental section

2.1. Materials and synthesis of the $\text{Fe}_3\text{O}_4/\text{RGO}$ composite

The GO was fabricated by a modified Hummers method, starting from graphite (Aldrich, <45 μm). Graphite powder was added to H_2SO_4 (98%), followed by the gradual addition of KMnO_4 and NaNO_3 . This solution was agitated for 2 h, and then a 30% H_2O_2 solution was added until the color of the solution turned to a brilliant brown, indicating a fully oxidized graphite. The as-obtained graphite oxide slurry was exfoliated to generate GO nanosheets by an ultrasonication process at 60 °C using an ultrasonic generator (28 kHz, 600 W, Kyungil Ultrasonic Co., Korea) for 1 h. Finally, the mixture was centrifuged, washed repeatedly with distilled water, and then finally dried in a vacuum oven.

In order to obtain the $\text{Fe}_3\text{O}_4/\text{RGO}$ composite, GO particles were then dispersed in 450 mL of water. An aqueous solution of FeCl_3 and FeCl_2 was prepared with a 2:1 mol ratio [3]. This solution was then slowly added to the GO solution at room temperature (GO = 0.700 g/450 mL, FeCl_3 = 3.2442 g/25 mL and FeCl_2 = 1.2675 g/25 mL). A 30% ammonia solution was added to the latter in order to adjust the pH to 10. The temperature of the solution was raised to 90 °C, and 10 mL of hydrazine hydrate were added under constant stirring, resulting in a black colored solution. After being rapidly stirred for 4 h, the solution was cooled down to room temperature, filtered, washed using water and ethanol several times, and finally dried in vacuum at 70 °C. For comparison purposes, Fe_3O_4 was also synthesized (FeCl_3 = 0.4055 g/25 mL and FeCl_2 = 0.1584 g/25 mL) using the same procedure, without the addition of GO.

2.2. Preparation of the MR fluid

Two MR fluids with the same particle volume fraction were prepared by dispersing Fe_3O_4 and $\text{Fe}_3\text{O}_4/\text{RGO}$ in silicone oil. To prepare these MR fluids, the concentration of Fe_3O_4 was fixed at 70 wt.% for both systems (with and without RGO). The MR fluids were immersed in a sonifier for a few minutes to obtain a homogeneous distribution of the samples.

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