Available online at www.sciencedirect.com
ScienceDirect
Ceramics International 42 (2016) 3407–3410
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

# Novel route for the preparation of cobalt oxide nanoparticles/reduced graphene oxide nanocomposites and their antibacterial activities

Edreese Alsharaeh*, Yasmin Mussa, Faheem Ahmed, Yazeed Aldawsari, Mohammed Al-Hindawi, Garwin Kim Sing

*College of Science & General Studies, Alfaisal University, Riyadh, Saudi Arabia*

Received 26 September 2015; received in revised form 25 October 2015; accepted 26 October 2015
Available online 30 October 2015

**Abstract**

In this work, we report a simple one step microwave irradiation method for the synthesis of cobalt oxide ($Co_3O_4$) nanoparticles and their reduced graphene oxide (RGO) based nanocomposites as an antibacterial agent against microorganism. X-ray diffraction (XRD), and Raman studies revealed the formation of crystalline $Co_3O_4$ nanoparticles supported onto the RGO sheets. Ultraviolet–visible (UV–vis) spectra showed the characteristic absorption peaks of $Co_3O_4$ nanoparticles and RGO. From field emission scanning electron microscopy (FESEM) analysis, nanoporous structure was observed for $Co_3O_4$/RGO nanocomposites. Antibacterial activity of these nanocomposites against gram-negative bacterium *Escherichia coli* (*E. coli*) was carried out. It was observed that $Co_3O_4$ nanoparticles supported on RGO sheets showed antibacterial activity against *E. coli* in contrast to the GO, RGO sheets and $Co_3O_4$ nanoparticles itself which did not show any antibacterial activity against *E. coli*. The presented approach to prepare graphene based nanocomposites within few minutes using the cheap, cost effective and environmental friendly microwave irradiation method will be a track to bridging between laboratory and industry for future biomedical applications.


*Keywords:* B. Nanocomposites; Reduced graphene oxide; X-ray techniques; Raman

## 1. Introduction

Antibacterial agents are very important in water disinfection, textile industry, medicine, and food packaging [1]. The most commonly available antibacterial agents in the medical community are antibiotics [2]. The development of antibiotic resistant bacteria as a result of the broad use and abuse is becoming a major problem. This antibacterial resistant strain can lead to infectious diseases that were under control for years. Today, materials in the nanosize are one of the novel antibacterial agents. In vivo and in vitro animal models have shown the effectiveness of these nanomaterials in treating infectious diseases and also the diseases caused by antibiotic resistance bacteria [3].

Graphene based nanomaterials have highly induced promising advances in, e.g., biology and medicine including cancer cell targeting, imaging, and therapy, drug delivery, antiviral, bactericidal etc. [4,5]. Nowadays, novel graphene oxide (GO) and reduced graphene oxide (RGO)-based nanocomposites using cobalt oxide ($Co_3O_4$) nanoparticles have been given much attention in a variety of biomedical applications [6,7]. $Co_3O_4$ nanoparticles are magnetic p-type semiconductors with a cubic structure [8] and nanosize $Co_3O_4$ were found to have a number of applications [9–11].

$Co_3O_4$ nanoparticles have been synthesized using different methods such as co-precipitation, hydrothermal synthesis, thermal decomposition, reduction etc. [12–15]. Synthesis of $Co_3O_4$/RGO nanocomposites is usually achieved by in situ reduction of cobalt salt precursors or assembly of the nanoparticles on the GO surface [16]. However, some problems with the approaches reported so far, such as poor control over the size, size distribution, and location of nanoparticles on the GO sheets. All these drawbacks make the use of magnetic nanoparticles/RGO nanocomposites in biomedical and other fields difficult.

*Corresponding author. Tel./fax: +966 55 215 7739.
*E-mail address:* ealsharaeh@alfaisal.edu (E. Alsharaeh).

http://dx.doi.org/10.1016/j.ceramint.2015.10.135
0272-8842/

To solve this issue, in this work, $Co_3O_4$ and $Co_3O_4$/RGO nanocomposites were synthesized using a novel, cost-effective, and environmental friendly microwave irradiation (MWI) assisted solution route and their antibacterial activities toward *Escherichia coli* was reported.

## 2. Materials and methods

In a typical synthesis of $Co_3O_4$/RGO nanocomposites, 920 mg of CTAB was mixed with 800 ml of distilled water and 8% (2 mg/ml) GO (synthesized by the hummer's method [17]) in a 1000 ml beaker. After 30 min of continuous stirring, a mixture of 4.6 g of cobalt acetate in 333 ml distilled water was added drop wise to the solution. Next, 1 ml of the reducing agent hydrazine hydrate was added to the solution. The solution was heated under microwave irradiation (KENWOOD; 740 W) for 20 min with a power of 100 W; then the product was washed several times by distilled water and ethanol and then was dried at 80 °C overnight. The dried sample was ground and calcined at 600 °C for 2 h. A black colored product was obtained. In addition, pure $Co_3O_4$ nanoparticles and RGO were synthesized by the same method.

The X-ray diffraction (Philips-PW 1729, Holland) of the material was investigated with Cu radiation (1.54430 Å). The XRD patterns were recorded in the scan ranging from 5 ° to 60 ° of 2$\theta$, with a step size of 0.02° and scan speed of 2°/min. The Raman spectra were measured using a Bruker FRA106/S FT-Raman module. A field emission scanning electron microscope (FESEM, FEI Quanta 200) was employed to study the morphology of the product. Room temperature optical absorption spectra were recorded in the range of 200–800 nm using a UV–vis spectrophotometer (Agilent-8453).

The antibacterial activity of the product was studied against the gram negative bacterial strain *E. coli* HB101 K12 (Biorad, Hercules, CA). The bacteria were cultured in Luria-Bertani (LB) nutrient broth at 37 °C for 18 h and harvested while in the exponential log phase. Zone of inhibition was measured by making a hole at the center of LB culture medium plate, then 100 mg of $Co_3O_4$ nanoparticles, and ($Co_3O_4$/RGO nanocomposites) was placed in the hole. 100 µl of *E. coli* in mid-log phase was spread evenly on LB culture medium plates. The plates were incubated at 37 °C overnight and examined the next morning.

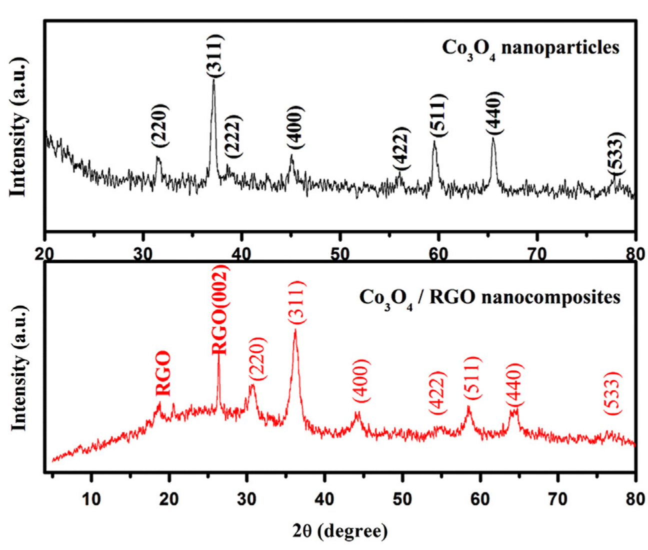


Fig. 1. XRD pattern of $Co_3O_4$ nanoparticles and $Co_3O_4$/RGO nanocomposites (8% RGO).

## 3. Results and discussion

XRD analysis was done to study the crystal phase of the $Co_3O_4$ nanoparticles and Co3O4/RGO nanocomposites as shown in Fig. 1. The diffraction peaks at 2$\theta$ of 31.55°, 37.23°, 38.54°, 45.04°, 56.09°, 59.50°, 65.68° and 77.71° can be indexed as the (220), (311), (222), (400), (422), (511),

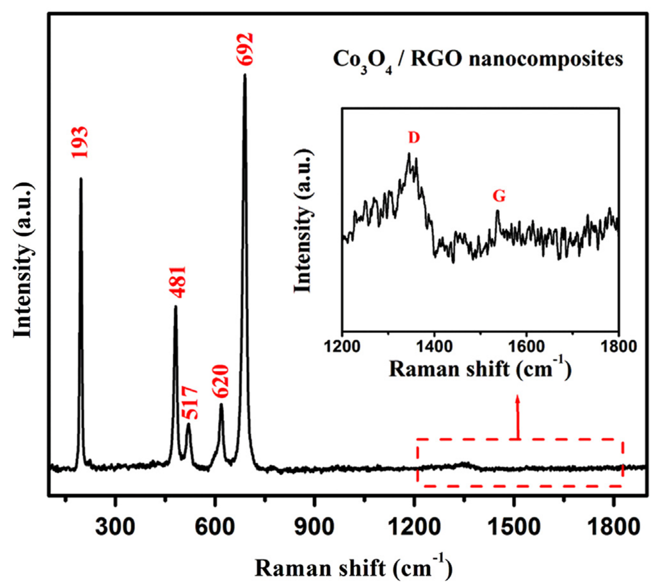


Fig. 2. Raman spectrum of $Co_3O_4$/RGO nanocomposites (8% RGO).

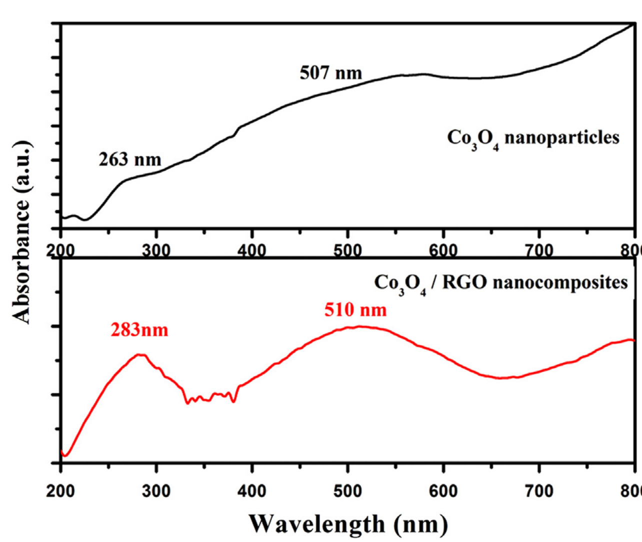


Fig. 3. UV–vis absorption spectra of $Co_3O_4$ nanoparticles and $Co_3O_4$/RGO nanocomposites (8% RGO).