



Synthesis of Fe₃O₄ hollow nanospheres-carbon nanotubes nanocomposites for the enhancement of dielectric heating performance



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ABSTRACT

Fe₃O₄ hollow nanospheres-carbon nanotubes (Fe₃O₄ HNSs-CNTs) nanocomposites were synthesized under one-pot solvothermal treatment. The crystalline structure, size and morphology of the composite products are characterized by XRD, SEM and TEM, indicating that Fe₃O₄ HNSs are tightly wrapped by CNTs with a good distribution. The products were dispersed in silicone oil for evaluating the dielectric heating performance under microwave irradiation. The Fe₃O₄ HNSs-CNTs nanocomposite (mass ratio 1:9) exhibits the best dielectric heating performance under microwave irradiation among all products, probably because an optimized proportion of Fe₃O₄ HNSs can improve the joint heating effect where the Fe₃O₄ HNSs as bridges between CNTs strengthen the interfacial polarization. The nanocomposites can be totally separated from the liquid medium by magnetic decantation.

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1. Introduction

The materials sensitive to dielectric heating can effectively absorb the electromagnetic waves and convert them into heating, which leads to the rapid temperature increasing [1–3]. These materials include metal particles [4], ferromagnetic compounds [5], and carbon nanomaterials [6], which have been reported for many interesting applications (e.g. microwave absorption, hyperthermia therapy, pollutant degradation, reworkable adhesives) [3–10]. The major sources for dielectric heating include microwave and radio frequency, among which the microwave can achieve highly selective, instantaneous volumetric heating [11]. Dipolar, electric, and magnetic loss are the three main routes to contribute the microwave-induced heating [4,6,12,13]. Materials types and morphologies should also be considered as important factors affecting the dielectric heating performance [5,8,10,13].

Previous works demonstrated that Fe₃O₄ and carbon nanotubes (CNTs) showed good dielectric heating efficiency [5,6] and potential application for cancer treatment under hyperthermia therapy [7,8]. Fe₃O₄ with hollow spherical morphology exhibiting better heating performance was reported for the degradation of rework-

able nanocomposite adhesives to disassemble adhesive joints [10]. Furthermore, carbon nanotubes (CNTs) exhibited excellent dielectric heating performance as the temperature ≥ 1500 °C was observed in microwave fields [3,6,13]. However, the application of CNTs is restricted due to the difficulty in target heating and recycling. Fe₃O₄ hollow nanospheres (HNSs) can be easily positioned and separated in the magnetic field, but the joint dielectric heating of Fe₃O₄ HNSs-CNTs is seldom investigated.

In this work, we report a one-pot route for the synthesis of Fe₃O₄ HNSs-CNTs nanocomposites. Characterization results show that the Fe₃O₄ HNSs are tightly wrapped by CNTs with a good distribution. The dielectric heating performance is evaluated under microwave at 2.45 GHz by dispersing the as-prepared nanocomposites in liquid heating medium of silicone oil. The Fe₃O₄ HNSs-CNTs nanocomposite (mass ratio 1:9) not only shows the optimum dielectric heating performance but also can be totally separated from the liquid heating medium by magnetic decantation.

2. Experimental details

In a typical procedure, 0.450 g of CNTs (multi-walled, O.D. × L 6–9 nm × 5 μm) were dispersed in 60 mL ethylene glycol under sonication at 50 °C. 0.729 g of cetyltrimethylammonium bromide (CTAB) and 0.701 g of hexamethylenetetramine (HMTA) were dissolved in the dispersion under sonication at 50 °C. 0.175 g of

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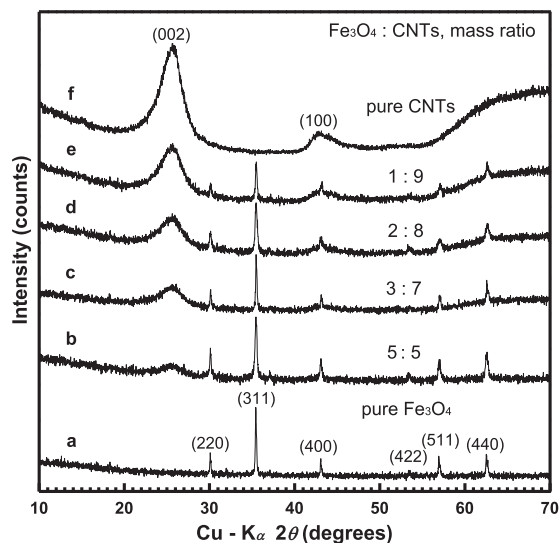


Fig. 1. XRD patterns of pure Fe_3O_4 (a), CNTs (f), and Fe_3O_4 -CNTs products with different mass ratio (b-e).

$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (equivalent to 0.050 g Fe_3O_4) was then added under continuous stirring until fully dissolved. The solution was transferred to a 100 mL Teflon-lined autoclave and sealed at 200 °C for 15 h. After that the black precipitate was washed with deionized water and absolute ethanol. Finally the products were dried at 80 °C for 24 h in vacuum. Fe_3O_4 HNSs-CNTs nanocomposites in different mass ratios (2:8, 3:7, 5:5, and pure Fe_3O_4 HNSs) were also synthesized by regulating the dosages of precursors and auxiliary chemicals.

The products were characterized by X-ray diffraction (XRD, PANalytical X'Pert), scanning electron microscopy (SEM, Hitachi SU-70), and transmission electron microscopy (TEM, JEOL JEM 2100F). Then the products were dispersed in silicone oil, and the dielectric heating performance was evaluated using a single mode microwave reactor (CEM, DISCOVER SP) at 50 W, 2.45 GHz for 100 s. The measuring details are shown in Fig. S1 (Electronic Supplementary Material).

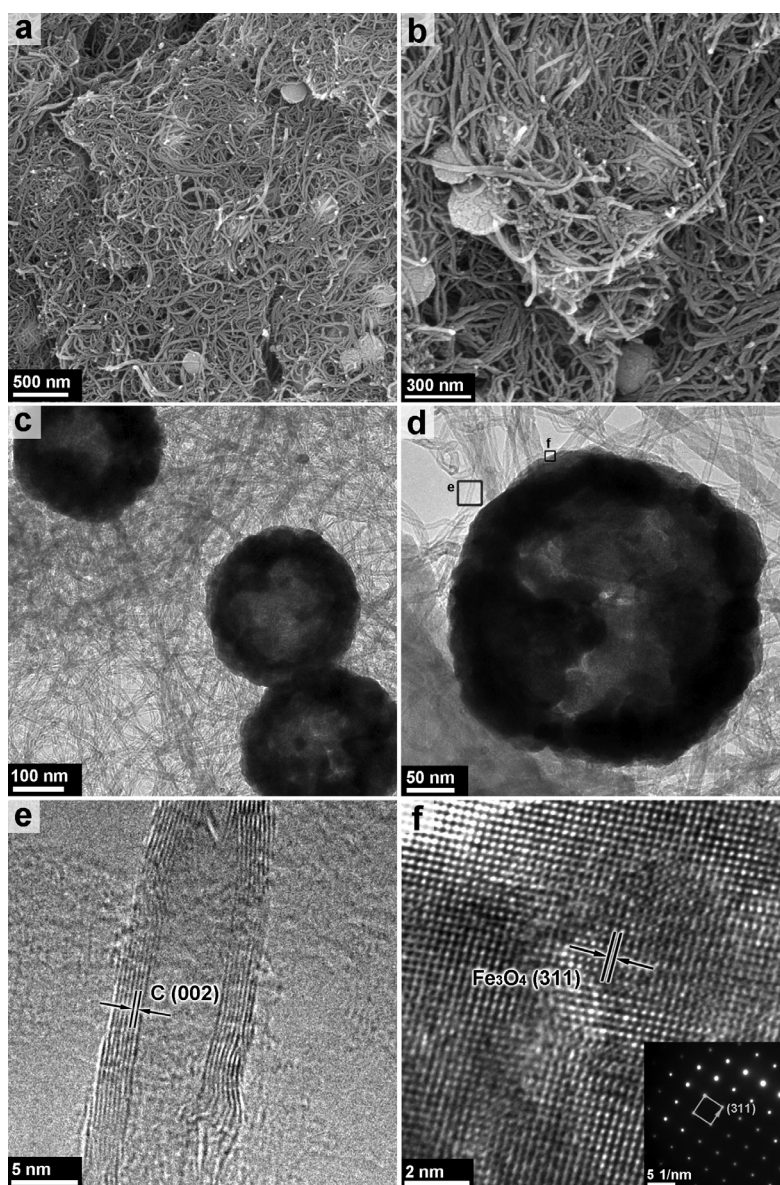


Fig. 2. FESEM (a-b) and TEM (c-f) images of Fe_3O_4 HNSs-CNTs product (mass ratio 1:9).

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