

Optimization of CVD Parameters during Synthesis of Fe@C Core-Shell Nanoparticles using Fe-Nitrate Based Precursors

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Extended Abstract

Magnetic iron nanoparticles are desired materials for various engineering applications in electronic and biomedical industries due to their soft ferromagnetic properties [1,2]. Hyperthermia, magnetic resonance imaging, drug and gene delivery are some examples of the potential biomedical applications of the magnetic iron nanoparticles [3,4]. Even though they have superior magnetic properties; they are not resistant to corrosion. Thus, core/shell nanoparticles have been developed to protect the magnetic core materials [5]. In general, carbon encapsulated magnetic nanoparticles are preferred for biomedical practice. Carbon based coatings provide large surface area, corrosion protection and energy storage. For this reason, they are desired shell materials for magnetic nanoparticles [2,3,6]. Various forms of carbon based shell are located on the magnetic nanoparticles like amorphous shell, graphene shell and graphite shell [6]. Carbon encapsulated Fe nanoparticles have been synthesized using different processes such as chemical vapor deposition (CVD), combustion synthesis and hydrothermal synthesis [7]. Amongst them, the CVD process increases the gas and powder interaction which accelerates the decomposition reactions for graphitic shell formations on the core particles [3].

In this study, precursor powders of the CVD system were Fe-nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) salts mixed with fumed silica powders in ethanol to prepare Fe nitrate loaded fumed silica powders followed by placing them inside a furnace in quartz boats (~200 mg). Hydrogen (H_2) and methane (CH_4) were used as flowing gases in the CVD system. H_2 was used for the reduction of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ to Fe and the decomposition of CH_4 led to graphitic layer formation on the Fe nanoparticles. The CVD system used in this investigation consists of a tube furnace, a vacuum pump and gas flowmeters. Two different CVD temperatures (900 and 950°C) up to a dwell time of 90 min and two different CH_4 flow rates (100 ml/min and 200 ml/min) at a constant H_2 flow rate (100 ml/min) were employed. After that, leaching with 2 M HF and 4 M HCl acid solutions were performed for the purification of powders. Fumed silica was removed with HF leaching and uncoated Fe nanoparticles were removed with HCl acid leaching. X-Ray diffractometry (XRD), Raman analysis, Scanning electron microscope (SEM), transmission electron microscope (TEM) and vibrating sample magnetometer (VSM) were used in the characterization of the synthesized powders. Raman and XRD investigations revealed that 950°C was not a suitable temperature for graphitic layer formations it causes excessive carbon decomposition. Therefore, 900°C is chosen as the optimum CVD temperature and other experiments were carried out at this temperature. TEM images reveal that the 900°C synthesized powders had core sizes varying in size between 15-40 nm and shell sizes between 3-13 nm. VSM results showed that Fe@C nanoparticles had soft ferromagnetic behavior with low saturation magnetization and coercivity values changed between 322.91-587.13 Oe and 23-88 emu/g.

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