

*Full Paper*

## **Electrochemical Synthesis of Iron Oxide Nanoparticles: Doping with Zn<sup>2+</sup> and Gd<sup>3+</sup> Cations and *In-situ* Capping with Polyethylene Glycol**

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**Abstract-** Development of a facile synthesis rout for preparation of polymer coted, metal ion doped magnetic nanoparticles, which have proper magnetic and physicochemical properties for bio-medical uses, is one of the most active research areas in advanced magnetic nano-materials. In this work, we demonstrate an easy electrochemical method for fabrication of PEG-coated Zn<sup>2+</sup> or Gd<sup>3+</sup> doped magnetic iron oxides (i.e. PEG/Zn-MIOs and PEG/Gd-MIOs). Characterization of the synthesized MIOs was carried out by X-ray diffraction (XRD), Energy-dispersive X-ray spectroscopy (EDAX), Fourier transform infrared spectroscopy (FTIR), Field-emission electron microscopy (FE-SEM), thermal gravimetric analysis (TGA), and vibrating sample magnetometry (VSM) analyses. The XRD and EDAX analyses proved that the electro-synthesized MIOs were magnetite (Fe<sub>3</sub>O<sub>4</sub>) doped with 8.6% Zn<sup>2+</sup> and/or 19.7% Gd<sup>3+</sup>. The PEG layer on the Zn-MIOs and/or Gd-MIOs surface was evidenced by FT-IR. FE-SEM showed a spherical morphology with average diameters of 20 nm and 25 nm for PEG/Zn-MIOs and PEG/Gd-MIOs, respectively. TGA data demonstrated that the PEG content of Gd-MIOs was about 10% by weight. Superparamagnetic nature of both prepared magnetic powders was verified by VSM measurements. In final, the analyses results supported that the prepared magnetic nano-particles have suitable size, crystal phase, surface layer and magnetism for bio-medical uses.

**Keywords-** Magnetic materials, Gd doping, Nanoparticles, Electrochemical synthesis, Dextran

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## 1. INTRODUCTION

Magnetic iron oxides (MIOs) are used in many state-of-the-art bio-medical applications such as tumor targeting and therapy [1-3], MRI and fluorescence) [4-6], cell labeling [7,8], hyperthermia [9], targeted drug delivery [10-12], gene therapy [13,14], cancer diagnosis [15,16], bio-separation [17,18], and bio-sensing [19]. These application fields of MIOs are due to their suitable chemical and physical properties, and unique super-paramagnetic performances. However, introducing the MIOs in the mentioned bio-medicine area requires specific magnetic characters like as low  $M_r$  and  $H_C$ , and high saturation magnetization ( $M_s$ ) [20-23]. For example, these particles are applied in magnetic hyperthermia to achieve a local heating of the tissue to treat tumors [24]. Furthermore, surface modification of MIOs by polymers [25-28], saccharides [29,30], amino acids [31-32] or other biocompatible molecules [33-35] is essentially needed to provide stability and biocompatibility for MIOs.

To fulfill these requirements, particle properties including crystal structure, particle size, and size distribution should be optimized, where all of these characters are depended to the type of synthesis procedure and the conditions applied in this procedure [36]. Magnetic properties, as the vital requirement for biomedical uses, can be tuned by the alteration of the structure, size, and size distribution of MIOs [37]. Notably, the magnetic properties of MIOs are also can be improved through metal ion doping [38-43], and so could be enhanced their ability in the biomedical uses.

In all above mentioned MIOs uses, inorganic or polymeric coating layers minimize hydrophobic interactions, thus enhancing desirable properties such as colloid dispersion and biocompatibility, and enabling the modification of surfaces with functional groups required for above mentioned applications. In this regard, various coating agents containing amine-, hydroxyl-, and carboxylic groups have been used for surface capping of MIOs [44-46]. MIOs for nano-medicines are usually referring to super-paramagnetic  $\text{Fe}_3\text{O}_4$  particles with a core size between 5 and 100 nm [47]. To date, a variety of synthetic methods such as co-precipitation, thermal decomposition, hydrothermal, solvothermal, sol-gel, electrochemical and microemulsion have been applied to produce super-paramagnetic  $\text{Fe}_3\text{O}_4$  particles [48-52]. In addition to these mentioned methods, electrochemical synthesis could be easy technique for synthesis of nanostructured materials. For instance, it was reported that magnetite NPs could be easily prepared and their surface be simply capped through cathodic deposition method [53-62]. In this paper, PEG capped  $\text{Zn}^{2+}$ - and  $\text{Gd}^{3+}$ - doped magnetic iron oxides (i.e. PEG/Zn-MIOs and PEG/Gd-MIOs) are fabricated through an electrochemical synthesis route. This route is based on the well-known base electro-generation *via* water molecule reduction on the cathode electrode [63-65]. It is worth noting that PEG-coated,  $\text{Zn}^{2+}$ /or  $\text{Gd}^{3+}$  doped magnetic NPs have not been reported until now, and this is the first report on the synthesis and characterization of this type of MIOs. The prepared MIOs are investigated through various analyses of XRD, FT-IR, FE-SEM, EDAX, TG and VSM to characterize

their physic-chemical and magnetic properties, and also their suitability for uses in the biomedical applications.

## 2. MATERIALS AND METHODS

### 2.1. Electrochemical deposition of PEG coated IONs

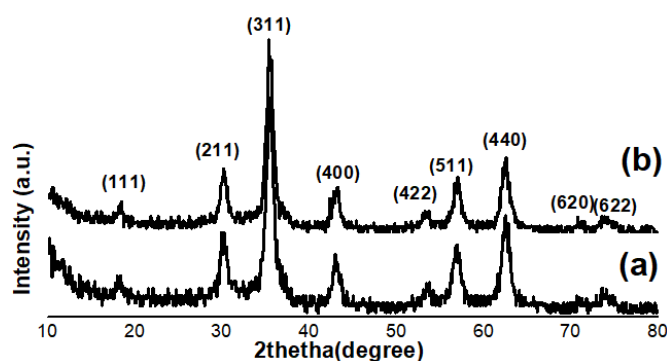
All chemicals including zinc chloride ( $\text{ZnCl}_2 \cdot 4\text{H}_2\text{O}$ , 99.5%), iron(III) nitrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ , 99.95%), gadolinium chloride ( $\text{GdCl}_3 \cdot 6\text{H}_2\text{O}$ , 99.8%), iron(II) chloride ( $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ , 99.5%), and PEG ( $M_w=70000$ ) were purchased from the Sigma Aldrich Company and used without purification. The electrochemical set-up was constructed using one steel sheet (as cathode side), two graphite sheets (as anode side) and a plexi holder. The size of these sheets was 2 cm\*5 cm. Both steel and graphite sheets were mounted on the plexi holder, and immersed into the electro-deposition bath. The electrolyte solution was prepared as follow; first, 0.8 g iron(II) chloride, 1.8 g iron(III) nitrate, 0.4 g zinc chloride or 0.6 g gadolinium chloride were dissolved in the 500 cc distilled water. And 0.5 g PEG was then added into this electrolyte solution and stirred for 1 h. The obtained solution was used as the electro-deposition bath in the electrochemical synthesis runs. The electrochemical parameters for preparation of both PEG/Zn-MIOs and PEG/Gd-MIOs samples were chosen to be: bath temperature=25 °C, current density=5 mA/cm<sup>2</sup>, pH=4.2-5, and deposition time=30 min. After each electrochemical deposition, both electrodes (anode and cathode) were removed from the electrochemical cell and a black film was observed on the cathode side (i.e. steel sheet). The deposited film was washed with ethanol and collected from the steel surface. The obtained wet powder was then dried in oven for 2 h at the temperature of 80 °C. In final, the prepared dry product was analyzed by different techniques.

### 2.2. Sample characterization

The X-ray diffraction (XRD, Phillips PW-1800) patterns of the fabricated samples were recorded using Cu K $\alpha$  radiation. The surface morphology of the synthesized powders were observed through field-emission scanning electron microscopy (FE-SEM, Mira 3-XMU with accelerating voltage of 100 kV) and also their elemental compositions were determined by Energy Dispersive Spectroscopy (EDS). The FTIR spectra was collected at a resolution of 4 cm<sup>-1</sup> from 400 to 4000 cm<sup>-1</sup> by a Bruker Vector 22 Fourier transformed infrared spectroscope. Thermal analysis (DCS-TG) was performed in N<sub>2</sub> atmosphere between room temperature and 500 °C at a heating rate of 5 °C min<sup>-1</sup> using a thermoanalyzer (STA-1500). Also, using vibrating sample magnetometer (VSM, model Lakeshore 4710), the magnetic data i.e. saturation magnetization, remanent magnetization and coercivity for the prepared PEG coated Zn- and Gd doped IOs were obtained in the range of -20000 to 20000 Oe.

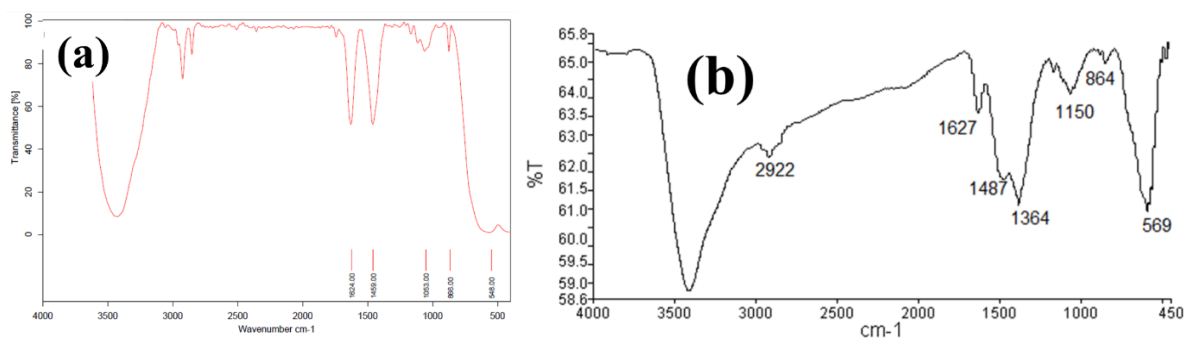
### 3. RESULTS AND DISCUSSION

Fig. 1 demonstrates XRD patterns of the PEG/Zn-MIOs and PEG/Gd-MIOs powders. As labeled in both XRD patterns, all diffractions are related to those of pure  $\text{Fe}_3\text{O}_4$  crystal phase with JCPDS card No. 01-088-0315. The calculation of average crystallite size from the Debye–Sherrer formula resulted values of 10.3 nm and 12.6 nm for PEG/Zn-MIOs and PEG/Gd-MIOs sample, respectively.

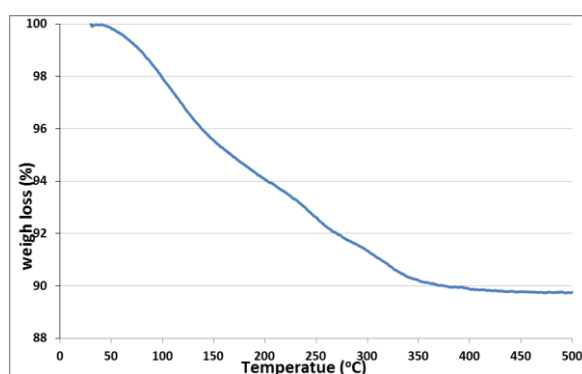


**Fig. 1.** XRD patterns of the prepared (a) PEG/Zn-MIOs and (b) PEG/Gd-MIOs samples

FT-IR analysis is suitable technique for identify the capped layer on the MIOs surface [66,67]. The IR spectra of the fabricated samples are presented in Fig. 2. For the PEG/Zn-MIOs, the IR bands of  $548\text{ cm}^{-1}$ ,  $866\text{ cm}^{-1}$ ,  $1162\text{ cm}^{-1}$ ,  $1232\text{ cm}^{-1}$ ,  $1459\text{ cm}^{-1}$ ,  $1624\text{ cm}^{-1}$ ,  $2845\text{ cm}^{-1}$ ,  $2933\text{ cm}^{-1}$  and  $3441\text{ cm}^{-1}$  are seen in Fig. 2a, which can be assigned to the following vibrations, Fe-O-Zn stretching [68,69], C-H wagging [70], C-O-C stretching [71],  $\text{CH}_2$  stretching,  $\text{CH}_2$  bending,  $\text{H}_2\text{O}$  stretching, C-H stretching [72], CH out-of-plane bending and -OH stretching [73]. From these IR results, it was concluded that Zn-MIOs has been coated by PEG layer. In Fig. 2b, IR adsorption peaks of  $569\text{ cm}^{-1}$ ,  $864\text{ cm}^{-1}$ ,  $1150\text{ cm}^{-1}$ ,  $1364\text{ cm}^{-1}$ ,  $1487\text{ cm}^{-1}$ ,  $1627\text{ cm}^{-1}$ ,  $2836\text{ cm}^{-1}$ ,  $2922\text{ cm}^{-1}$  and  $3425\text{ cm}^{-1}$  are also observable for the PEG/Gd-MIOs sample, which these peaks are related to the vibrations of following chemical bonds; Fe-O-Fe and/or Gd-O-Fe stretch vibration at  $569\text{ cm}^{-1}$  [74], C-H wagging vibration at  $864\text{ cm}^{-1}$  [71], C-O-C stretching vibration at  $1150\text{ cm}^{-1}$  [72],  $\text{CH}_2$  stretching at  $1364\text{ cm}^{-1}$ ,  $\text{CH}_2$  bending at  $1487\text{ cm}^{-1}$ ,  $\text{H}_2\text{O}$  stretching at  $1627\text{ cm}^{-1}$ , C-H stretching at  $2836\text{ cm}^{-1}$  [73], CH bending at  $2922\text{ cm}^{-1}$  [72] and -OH stretching at  $3425\text{ cm}^{-1}$ . These IR findings verified the fabrication of PEG capped Gd-MIOs by electrochemical method.



**Fig. 2.** IR spectra of the fabricated the prepared (a) PEG/Zn-MIOs and (b) PEG/Gd-MIOs samples

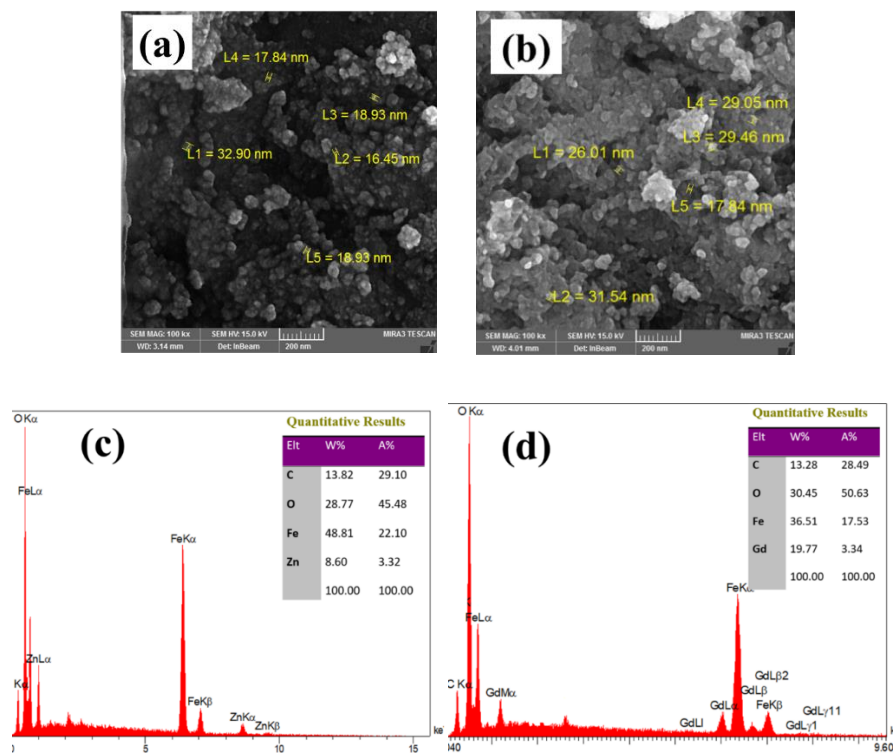


**Fig. 3.** TG curve of the fabricated PEG/Gd-MIOs sample

TG profile of the PEG/Gd-MIOs sample is shown in Fig. 3. At the temperatures below 100 °C, about 2% weight loss is seen on the TG curve, which is due to the evaporation of the adsorbed H<sub>2</sub>O onto the MIOs surface [27,35]. After this step, a sharp weight loss (~9.25%) is seen on the TG profile at the temperatures between 100-380 °C, indicating the PEG decomposition [75]. Total weight loss of PEG/Gd-MIOs sample was 11.25%.

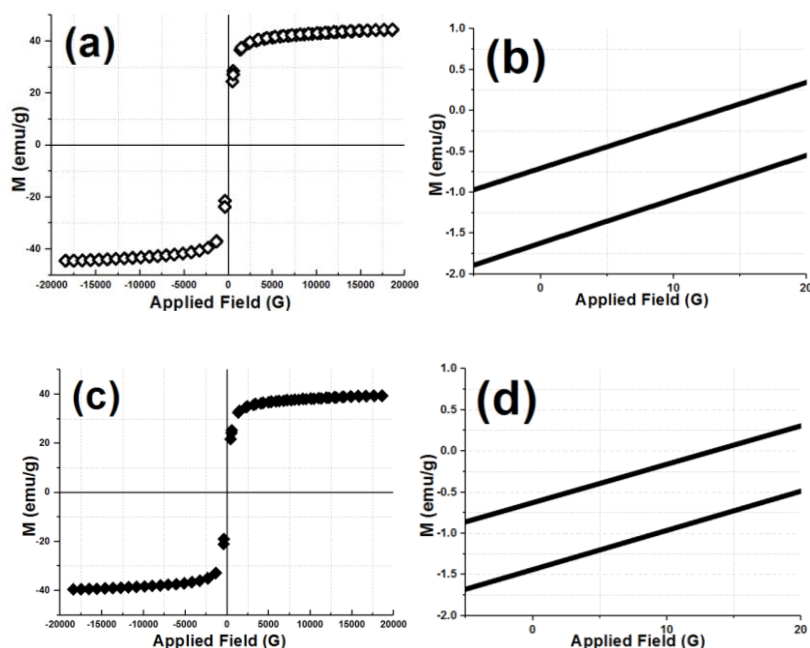
Surface morphologies of both prepared samples were observed by FE-SEM (Figs. 4a,b) and their elemental content was also analyzed *via* EDAX technique (Figs. 4c,d). The particle morphology with spherical form and good size distribution is seen for both samples (Figs. 4a,b). Notably, the Zn-MIOs are rather spherical-shape as compared with those observed for Gd-MIOs. The average particle sizes of 20nm and 25nm were measured for PEG/Zn-MIOs and PEG/Gd-MIOs powders, respectively. The elemental contents of both prepared powders were also investigated through EDAX analysis, the results are presented in Figs. 4c and d. The elemental percentages of 13.82% C, 28.77% O, 8.60% Zn and 48.81% Fe were detected for PEG/Zn-MIOs powder (Fig. 4c). The 13.28% carbon, 30.45% oxygen, 19.77% gadolinium and 36.51% iron were also measured for PEG/Gd-MIOs (Fig. 4d). From these data, the following facts are revealed; (i) the Zn element presence in EDAX data of PEG/Zn-MIOs powder proved that the iron oxide core has been doped with Zn cations, (ii) the Gd

content of PEG/Gd-MIOs is also indicated the doping of iron oxide with gadolinium cations, and (iii) the carbon content in both powders verified the presence of organic coat (i.e. PEI layer) in the fabricated samples.



**Fig. 4.** FE-SEM images and EDS data of the prepared (a,c) PEG/Zn-MIOs and (b,d) PEG/Gd-MIOs samples

Vibrating sample magnetometer (VSM) analysis was used to study the magnetic behavior of the prepared powders at the external field presence and hence identify their magnetic properties. The VSM curves of both PEG coated MIOs are presented in Fig. 5. The VSM profiles of both samples have S-like form (as seen in Figs. 5a,c) without any hysteresis loop (as seen in Figs. 5b,d), verifying their super-paramagnetic property. The saturation magnetization values of  $M_s=44.29 \text{ emu g}^{-1}$  and  $M_s=39.53 \text{ emu g}^{-1}$  are measured in Figs. 5a and c for PEG/Gd-MIOs and PEG/Zn-MIOs, respectively. These magnetization values exhibited the suitable performance of the fabricated magnetic powders at the external field, and higher than the necessary threshold for bio-medical aims. Furthermore, the PEG/Gd-MIOs showed the remanent magnetization and coercivity values of  $M_r=-1.29 \text{ emu g}^{-1}$  and  $H_{Ci}=13.64 \text{ G}$ , respectively (Fig. 5c). These values for PEG/Zn-MIOs were obtained to be  $M_r=-1.13 \text{ emu g}^{-1}$  and  $H_{Ci}=10.51 \text{ G}$  (Fig. 5d). These measured values clearly confirmed the super-paramagnetic nature of the prepared powders. Also, the negligible  $M_r$  and  $H_{ci}$  values supported the proper magnetic performance of the fabricated powders for uses in bio-medicine.



**Fig. 5.** VSM profiles for the fabricated (a,b) PEG/Gd-MIOs and (c,d) PEG/Zn-MIOs samples

#### 4. CONCLUSION

Newly formed PEG coated Zn-MIOs and Gd-MIOs of 20-25 nm diameters were fabricated using cathodic electrodeposition from PEG dissolved aqueous solution of  $\text{ZnCl}_2$ ,  $\text{Fe}(\text{NO}_3)_3$ ,  $\text{GdCl}_3$  and  $\text{FeCl}_2$  salts. The prepared nano-powders exhibited superparamagnetic behavior at the presence of external field, where PEG coted Zn-MIOs had  $M_s=39.53 \text{ emu g}^{-1}$ ,  $M_r=-1.13 \text{ emu g}^{-1}$  and  $H_{C_i}=10.51 \text{ G}$ , and PEG coted Gd-MIOs showed  $M_s=44.29 \text{ emu g}^{-1}$ ,  $M_r=-1.29 \text{ emu g}^{-1}$  and  $H_{C_i}=13.64 \text{ G}$ . In final, it was proposed that this type of iron oxide NPs have high potential for biomedical applications.

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