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PVA and EDTA grafted superparamagnetic Ni doped iron oxide nanoparticles prepared by constant current electrodeposition for biomedical applications

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ABSTRACT

In this paper, a rapid and room temperature electrochemical method is introduced in preparation of Ni doped iron oxide nanoparticles (Ni-IONs) grafted with ethylenediaminetetraacetic acid (EDTA) and polyvinyl alcohol (PVA). EDTA/Ni-IONs and PVA/Ni-IONs samples were prepared through base electro-generation on the cathode surface from aqueous solution of iron(II) chloride, iron(III) nitrate and nickel chloride salts with EDTA/PVA additive. Uniform and narrow particle size Ni-IONs with an average diameter of 15 nm was achieved. Ni doping into the crystal structure of synthesized IONs and also surface grafting with EDTA/or PVA were established through FT-IR and EDAX analyses. The saturation magnetization values for the resulting EDTA/Ni-IONs and PVA/Ni-IONs were found to be 38.03 emu/g and 33.45 emu/g, respectively, which proved their superparamagnetic nature in the presence of applied magnetic field. The FE-SEM observations, XRD and VSM data confirmed the suitable size, crystal structure and magnetic properties of the prepared samples for uses in biomedical aims.

Keywords: *Electrochemical Synthesis; Iron Oxide; Nanoparticles, Ni Doping; Surface Grafting*

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INTRODUCTION

Iron oxide nanoparticles (IONs) have tremendous attention for their excellent physicochemical characteristics and engaged biomedical uses. Recently, IONs have been investigated as the diagnostic and therapeutic agents in magnetic hyperthermia, bio-sensing, cancer therapy, etc. [1-3]. IONs are a proper medical nano-material type due to their high surface area, superparamagnetic nature, and high magnetization values [4]. Among IONs, magnetite (Fe_3O_4) is promising case for *in vivo* and *in vitro* therapies [5]. In this regard, development of new synthesis routes for fabrication of the naked and coated IONs are very interesting [6]. The most common methods applied to produce high-quality IONs are hydrothermal, thermal decomposition and co-precipitation protocols [7,8], which need to special pressure and temperature synthesis conditions. Recently, electrochemical synthesis has been also mentioned as a simple and non-expensive method for fabrication of pure and metal-ion doped IONs [9-16]. Electrochemical synthesis provides a low cost and simple route to prepare nano-materials [17-20].

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It was reported that surface modification plays critical role in the development of any magnetic NPs platform for biomedical uses. In this regard, coating with polymers (like PEG, PVA, PEI and chitosan [21-26]) are preferred due to the (1) balance the magnetic interaction and (2) improve the biocompatibility [23,24]. Up now, metal ion doping has been also used to improve the superparamagnetic behavior of IONs [27,28]. Here, PVA- and EDTA- grafted Ni doped IONs (i.e. PVA/Ni-IONs and EDTA/Ni-IONs) were prepared using a novel one-pot electrochemical strategy. The galvanostatic Cathodic was chosen for the preparation of samples. In this method, the morphology and crystal structure of the products could be easily changed through altering the applied current, potential and electrolyte composition [29-31]. The prepared samples were analyzed using FT-IR, FE-SEM, XRD and VSM techniques.

EXPERIMENTAL PROCEDURE

Materials

Iron (III) nitrate nonahydrate ($Fe(NO_3)_3.6H_2O$), Iron (II) chloride tetrachloride ($FeCl_2.4H_2O$), nickel chloride hexahydrate ($NiCl_2 .4H_2O$), polyvinyl alcohol (PVA) and (ethylenediaminetetraacetic acid disodium salt dehydrate (EDTA-Na_2, 99.9%) were purchased from Sigma-Aldrich. The graphite plates and stainless steel sheets (316L) were provided from local companies.

Preparation of PVA/Ni-IONs and EDTA/Ni-IONs

The cathodic electro-deposition was applied as a preparation route for the fabrication of PVA/Ni-IONs and EDTA/Ni-IONs samples. The deposition bath solution was the iron(II) chloride/iron(III) nitrate/nickel chloride salts (0.2g:0.5g:0.05g, respectively) dissolved in 200 cc deionized H_2O. In the deposition of PVA/Ni-IONs, polyvinyl alcohol (0.1g) was added into the above mentioned deposition bath and stirred for 10min, and the electro-synthesis run was then performed. For preparation of EDTA/Ni-IONs sample, ethylenediaminetetraacetic acid disodium salt (0.1g) was added into the electrolyte and stirred for 10min. The electrochemical set up was composed of two-electrode system containing a 4cm*2cm stainless steel cathode centered between two graphite plates (4cm*2cm). In the deposition of both samples, constant current deposition mode (i= 5 mA cm^{-2}), deposition time of 30 min and T=25°C were the electro-synthesis conditions. After 30min deposition in the above mentioned electrochemical set-up, a black film was deposited on the steel cathode in both deposition syntheses. At the end of deposition time, the cathode sheet was removed from the deposition bath and washed with ethanol several times. Then, the black film was scraped from the cathode and dispersed in the 50 cc ethanol (96%), and centrifuged at 3000rpm for 5min (to remove the free- and unattached PVA/or EDTA onto the IONs). In final, the black powder was collected from the ethanol solution by magnet and dried at 70 °C (in vacuum oven) for 20min. The obtained products were labeled EDTA/-Ni-IONs and PVA/Ni-IONs samples and analyzed through various techniques.

Characterization techniques

The XRD patterns of the fabricated samples were collected at room temperature using a Phillips PW-1800 X-ray diffractometer equipped with a Cu Kα radiation source (λ = 0.154056 nm). The IR spectra were provided using Bruker Vector 22 IR spectrometer in the frequency range 4,000–400 cm^{-1}. Field-emission Scanning Electron Microscope (FE-SEM, model Mira 3-XMU with accelerating voltage of 100 kV), attached with Energy Dispersive X-ray Spectroscopy (EDAX, for measuring elemental composition) were used for observing the surface morphology and collecting elemental data. Magnetization measurements were performed at room temperature with vibrating sample magnetometer (VSM, Lakeshore 4710).

RESULTS AND DISCUSSION

Crystal structure

Fig. 1 shows the resulted patterns for the prepared EDTA/-Ni-IONs and PVA/Ni-IONs samples from XRD analysis. In both XRD patterns, the diffraction peaks of (111), (220), (311), (400), (422), (511), (440) and (533) are observed at 2theta of 18.35°, 31.06°, 36.24°, 43.18°, 53.81°, 57.02° and 74.24°, respectively. These peaks in the diffraction patterns correspond to the inverse cubic spinel structure of the magnetite phase with a lattice parameter a_0 = 8.394 Å. The identification of the crystalline phase was performed by comparing our results with the PDF card (85-1436). As the chemical nature of both samples are the same, i.e. Ni cations doped iron oxide. Hence, it is expected that the XRD patterns of both samples are identical with some differences in the peak intensities. Our XRD data proved this fact, where the magnetite

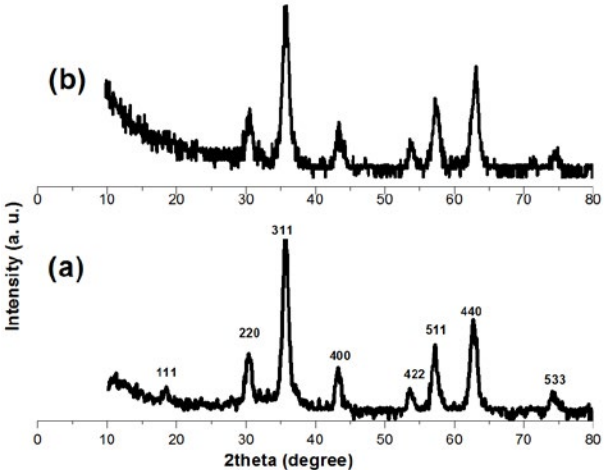


Fig. 1. XRD patterns of the prepared (a) EDTA/Ni-IONs and (b) PVA/Ni-IONs samples.

crystal structure is observed for both samples. Notably, the surface coat on the surface of Ni-IONs has no essential change on the XRD patterns of Ni-IONs. The Debye–Scherrer formula was used to obtain the average diameter from the most intense peak in Figs. 1a-b (i.e. (311) diffraction peaks), and yielded the $D_{(hkl)}$ values of 9.4nm and 10.1nm for the fabricated EDTA/-Ni-IONs and PVA/Ni-IONs samples, respectively.

FT-IR

The surface chemical structures of the EDTA/Ni-IONs and PVA/Ni-IONs samples were characterized by Fourier transform infrared (FTIR) spectroscopy (Fig. 2). This was used to prove the EDTA and PVA is grafted onto the Ni-IONs. In both spectra, the IR absorption bands at about 592 cm^{-1} and 554 cm^{-1} are attributed to Fe–O stretching vibration for the IONs [32,33]. Also, the broad bands at about 3400-3450 cm^{-1} are ascribed to the –OH groups of water, PVA and EDTA, and those originally on the surface of the IONs [34]. These data verified the magnetite structure for both EDTA/Ni-IONs and PVA/Ni-IONs samples.

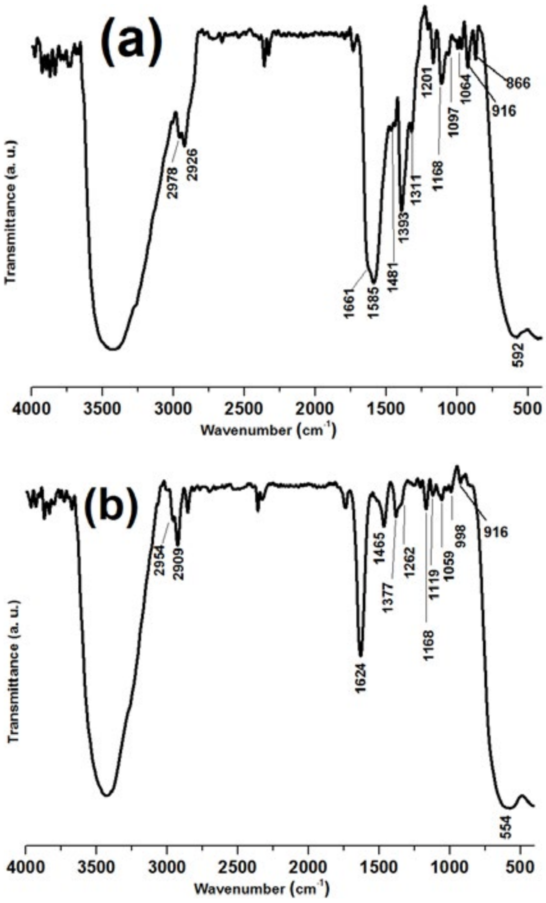


Fig. 2. IR spectra of the prepared (a) EDTA/Ni-IONs and (b) PVA/Ni-IONs samples.

For the EDTA grafted Ni-IONs sample, the following IR bands are also observed in Fig. 2a; (1) C–N and C–C stretching vibrations at 1097 cm^{-1} and 916 cm^{-1}, respectively [34], (2) C-O scissoring vibration at 1661 cm^{-1} [35], (3) -CH_2 and C-H stretching and waging vibration modes at 1393 cm^{-1}, 1311 cm^{-1} and 1168 cm^{-1} [36], (4) N–C-H asymmetric stretching at 2862 cm^{-1} [37,38], (5) C=O and C-OH stretching vibrations at 1585 cm^{-1} and 1481 cm^{-1}, respectively [36,38] and (6) the C-H stretching of CH_2 groups at 2978 cm^{-1} and 2926 cm^{-1} [36-38]. These IR results clearly indicated the EDTA is grafted onto Ni-IONs surfaces.

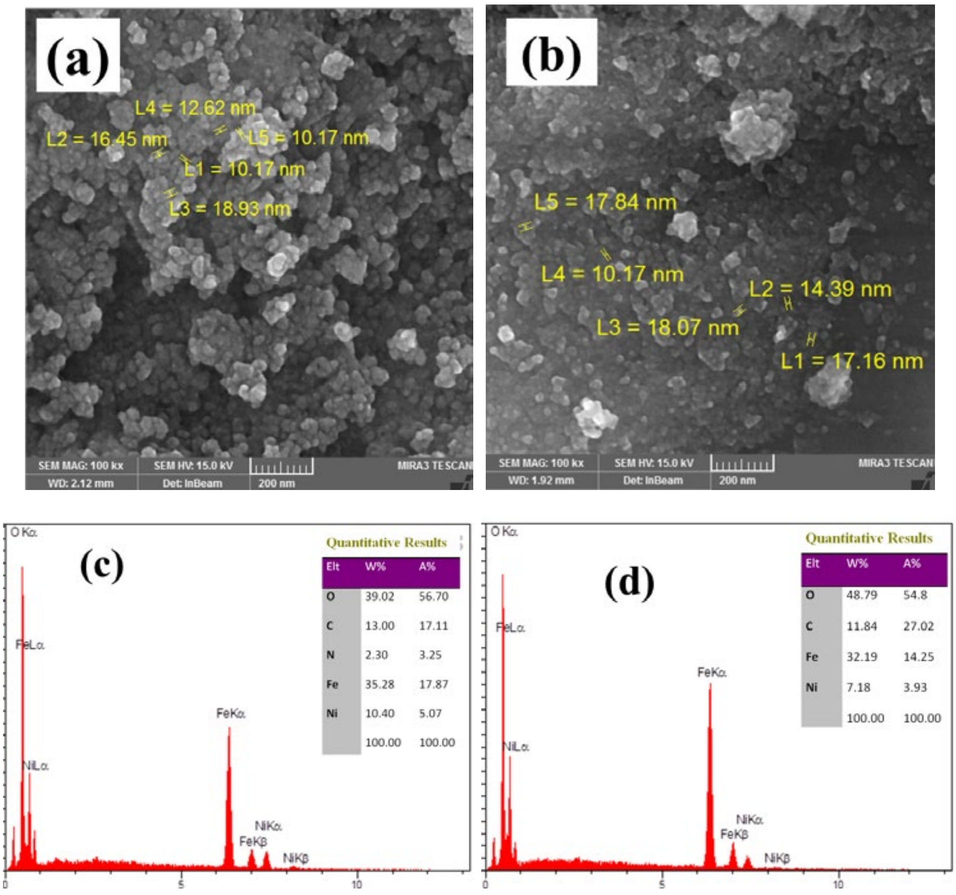
For the prepared PVA/Ni-IONs sample (Fig. 2b), the following vibrations related to PVA are observed; (i) two vibrations at 2954 cm^{-1} and 2909 cm^{-1} due to C-H in CH_2 groups [39], (ii) the twisting and wagging vibrations of the CH_3 group at 1168 and 1377 cm^{-1} [40,41], (iii) the adsorbed water,vibration band at 1624 cm^{-1} and C-C vibration at 1095 cm^{-1} [42], (iv) the stretching and waging vibration modes of CH_2 and C-H bonds at 1465 cm^{-1}, 1262 cm^{-1} and 1119 cm^{-1} [43], and (v) CH_2 rocking at 916 cm^{-1} [43,44]. These data revealed the presence of PVA onto Ni-IONs.

Morphological characterization

Morphology of the prepared samples was examined through FESEM technique and their chemical composition was detected by EDAX analysis. FESEM observations of the surface morphology of both samples showed particle texture with spherical shape (Figs. 3a and b). It was also observed the fabricated EDTA/Ni-IONs and PVA/Ni-IONs samples have a particle size range of 10-15nm (Figs. 3a-b). The EDAX plots and the extracted data are further showed in Figs. 3c and d. For the EDTA/Ni-IONs sample, the presence of oxygen, carbon, nitrogen, iron and nickel elements with weight percentage of 39.02%, 13%, 2.3%, 35.28% and 10.4% are respectively observed. From these data, the doping of iron oxide by Ni cations and their surface coating with EDTA were clearly proved. About PVA/Ni-IONs sample, the oxygen (48.79 wt%), carbon (11.84 wt%), iron (32.19 wt%) and nickel (7.18 wt%) were detected. These data revealed the PVA grafting onto iron oxide and its doping with Ni cations during electrodeposition.

Magnetic evaluation

Magnetic properties of the electrosynthesized iron oxides were studied using their M–H curves. M–H curves of EDTA and PVA grafted Ni-IONs samples are shown in Fig. 4. The saturation magnetization (*Ms*), coercivity (*Hci*) and remenance magnetization (*Mr*) values calculated from the M–H curves for both Ni-IONs samples are also listed in Table 1. The M–H curves clearly showed superparamagnetic nature of both samples



Quantitative Results

Elt	W%	A%
O	39.02	56.70
C	13.00	17.11
N	2.30	3.25
Fe	35.28	17.87
Ni	10.40	5.07
	100.00	100.00

Quantitative Results

Elt	W%	A%
O	48.79	54.8
C	11.84	27.02
Fe	32.19	14.25
Ni	7.18	3.93
	100.00	100.00

Fig. 3. FE-SEM images and EDS data of the prepared (a,c) EDTA/Ni-IONs and (b,d) PVA/Ni-IONs samples.

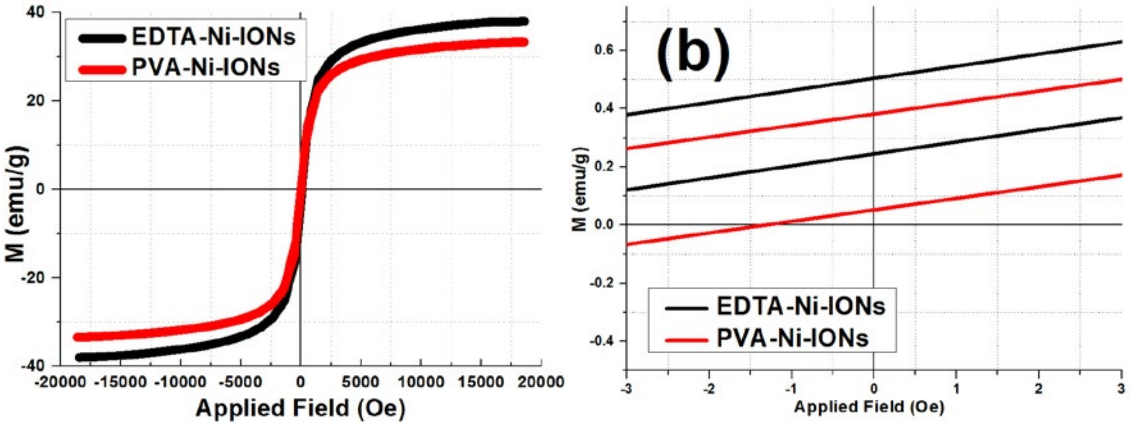


Fig. 4. Hysteresis loops for electrosynthesized (a) EDTA/Ni-IONs and (b) PVA/Ni-IONs samples.

Table 1. Magnetic data for the prepared samples.

Sample name	Ms (emu/)g	Coercivity (Hci)G	Positive (Hci) G	Negative (Hci) G	Negative Mr(emu/g)	Positive Mr(emu/g)	Retentivity Mr(emu/g)
EDTA/Ni-IONs	38.03	8.11	-11.09	-5.13	0.22	0.51	0.36
PVA/Ni-IONs	33.45	5.44	-9.34	-1.55	0.05	0.37	0.21

at 300K since *Hci* and *Mr* values are very negligible (as seen in Fig. 4b). Superparamagnetic behavior of IONs at room temperature is very useful in *in vivo* uses as they do not retain magnetization before and after exposure to an external magnetic field [45]. The superparamagnetic nature is mainly results from the small size of IONs, where their sizes (as observed in FE-SEM observations in Figs. 3a,b) are smaller than the superparamagnetic critical size (i.e. 20nm) [46]. From M-H profiles, the *Ms* values of EDTA/Ni-IONs and PVA/Ni-IONs were observed to be 38.03 emu/g and 33.45 emu/g, respectively, which are smaller than those reported to electrosynthesized bare IONs (*Ms*=82.3 emu/g [47], and bare Ni-IONs (*Ms*=47.25 emu/g [33]), which reflect the reduction of magnetite portion in the coated samples due to non-magnetic EDTA/or PVA layer. However, the observed Ms values showed the proper magnetic response of our samples in the presence of an applied field. Furthermore, the prepared samples exhibited negligible Mr values (*Mr*=0.36 emu/g for EDTA/Ni-IONs and Mr=0.21 for PVA/Ni-IONs), which verified their initial suitability for biomedical uses like as hyperthermia.

CONCLUSION

In summary, Ni^{2+} doped iron oxide nanoparticles were successfully synthesized using the cathodic deposition and their surface was *in situ* grafted with EDTA and PVA agents, leading to formation of EDTA/Ni-IONs and PVA/Ni-IONs. The XRD data proved phase purity of the electrosynthesized IONs. FTIR results confirmed the chemical composition of the samples, and VSM evaluation exhibited the superparamagnetic nature for the prepared Ni-IONs.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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