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# Galvanostatic Deposition of Magnetite Nanoparticles for Biomedical Applications: Simple Preparation and Surface Modification with Polyethylenimine and Polyvinyl Chloride

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**Abstract**- In this work, PEI and PVC grafted Ni doped superparamagnetic iron oxide (i.e. PEI/Ni-SPIOs and PVC/Ni-SPIOs) were synthesized on steel sheet though galvanostatic (constant current) deposition mode. Structural and morphological properties of the fabricated PEI/Ni-SPIOs and PVC/Ni-SPIOs samples were studied and the results indicated the successful synthesis of polymer grafted iron oxide nanoparticles. The size of prepared particles was about 20 nm. Thermogravimetric data showed 7.2 wt % PEI and 6 wt % PVC coated onto the surface of Ni-SPIOs particles. The magnetite crystal phase of samples was proved via XRD and IR data. In addition, the obtained results from vibrating sample magnetometer analysis showed that the fabricated samples exhibit low residual magnetization values (i.e. $Mr$=0.95 and $Mr$=0.53 emu/g, respectively, for PVC and PEI grafted Ni-SPIOs), which revealed their suitability for biomedical uses.

**Keywords**- Galvanostatic Deposition, Magnetite, Nanoparticles, Biomedical Applications

## 1. INTRODUCTION

Among different nano-particulate materials, magnetic nanoparticles (MNPs) exhibit the potential applications in the biomedical fields and are proper therapeutic and diagnostic agents to a specific region in the body with an external magnetic field manipulation [1]. In

these application fields, magnetic particles with large magnetization are required, where they could respond to externally applied magnetic fields at physiological temperatures.

Superparamagnetic iron oxides (SPIOs), such as magnetite and maghemite [2,3], show relatively higher saturation magnetization (*Ms*) with zero residual magnetization (*Mr*) and negligible coercivity (*Hci*), and accomplish other essential requirements including low toxicity, biocompatibility, and surface functionalization capabilities for various diagnostic and theranostic uses like as magnetic gene therapy, drug carriers, contrast agent in magnetic resonance imaging (MRI) and magnetic resonance angiography (MRA), magnetic immunoassay, hyperthermic therapy, and magnetic drug targeting [4-10]. Hence, the major magnetic characters of MNPs in the theranostic applications are Ms, Mr, Hci, Curie temperature (Tc), magnetic anisotropy energy density (K), and blocking temperature (Tb). It was clearly revealed that these parameters are directly related to the nanoparticles type, size, shape, composition, and surface coat [11-13]. Particle size has an important role in many magnetic biomedical uses including magnetic hyperthermia and drug delivery [14-16]. Over the past decades, many efficient synthesis procedures have been reported to prepare stable mono-dispersed MNPs with controlled the size/shape, which the most common methods are coprecipitation, hydrothermal synthesis, thermal decomposition and sonochemical synthesis [17-20].

Surface modification has also an important role for theranostic and diagnostic uses, which provides colloidal stability and biocompatibility of SPIOs [21]. For biocompatibility, the surface coat of MNPs should shields the magnetic core from oxidation and corrosion, and prevents any toxic ion leakage from magnetic core into the biological environment [22].

Up now, various surface modification strategies like as grafting or coating with both organic and inorganic materials have been developed [23]. Organic molecules include small organic molecules, macromolecules or polymer and biological molecules. They provide various highly reactive sites such as amino, carboxyl and aldehyde groups. Polymer-coating materials can be classified into natural and synthetic. Also, the surface coating could affect the magnetic properties of SPION. For example, the surface coating of SPIONs with both natural (e.g. chitosan [24], starch [25,26] and dextran [27]) and synthetic polymers (like as PVP [28,29], PEG [30] and alginate [31]) has been carried out and the ad-layer effects on the physic-chemical properties of SPIOs have been reported.

Far from particle size and surface modification, the metal ion doping has also important effect on to the magnetic properties. In fact, magnetic properties of widely used magnetite($Fe_3O_4$) with its spinel structure of $[Fe^{3+}]A[Fe^{3+}Fe^{2+}]BO_4$ can be changed if other magnetic atoms such as Ni, Co, Mn, and so on are substituted at the tetrahedral or octahedral sites of the spinel structure [32]. The preparation route, concentration and dopant nature, and post synthesis processes have shown to profoundly affect the magnetic characters of obtained SPIOs. For example, it was shown that among the cobalt ferrite, nickel ferrite and manganese

ferrite, the last one shows the highest *Ms* value [33]. Furthermore, the doping of metal cations in magnetite crystal structure has been investigated during the electrochemical synthesis of iron oxide and it was reported that the doping of various metal ions [34-46], including cobalt, manganese, dysprosium, zinc and gadolinium, could be achievable during the cathodic deposition of SPIOs. The magnetic studies have been also shown that the superparamagnetic characters of SPIOs is improved via metal ion doping. In this regard, Ni doped SPIOs have been fabricated through co-precipitation method using aqueous solution of $FeCl_2{\cdot}4H_2O$ and $Ni(AcO)_2{\cdot}4H_2O$ [47], and results of this work have confirmed the partial substitution of the $Fe^{2+}$ cations by $Ni^{2+}$ atoms in the octahedral sites and superparamagnetic behavior of doped sample with anhysteretic magnetic response at RT. Furthermore, Gd-doped SPIOs with composition of $Gd_{0.02}Fe_{2.98}O_4$ have been also fabricated by Drake et al. [48], and their ability in tumour therapy through magnetic fluid hyperthermia (MFH) has been studied [48]. The results of this work have indicated that the specific power adsorption rate (SAR) value of doped SPIOs was about 4 times higher than those of undoped SPIOs. Also, the mouse models treated with Gd doped SPIOs displayed much slower tumour growth as compared with standard iron oxide or saline solution [48].

Galvanostatic deposition, as an electro-synthesis method, has been applied for the preparation of metal oxides and hydroxides at nano-scale, and it was reported that the $M_xO_y$ and $M(OH)_y$ nanoparticles (M=yttrium, zirconium, nickel, iron and manganese) could be simply fabricated through this method [49-57]. Here we applied galvanostatic deposition for preparation of nickel doped magnetite nanoparticles and their surface grafting with PEI and PVC polymers. The prepared polyethylenimine grafted $Ni^{2+}$ doped superparamagnetic iron oxide (PEI/Ni-SPIOs) and polyvinyl chloride grafted $Ni^{2+}$ doped superparamagnetic iron oxide (PVC/Ni-SPIOs) nano-particles are analyzed through by VSM, FE-SEM, TG, FT-IR and XRD techniques.

## 2. EXPERIMENTAL PROCEDURE

### 2.1. Materials and methods

All chemicals including $FeCl_2$ .$4H_2O$), $Fe(NO_3)_3$ .$6H_2O$, $NiCl_2$ .$6H_2O$, polyethylenimine (PEI, Mw~25,000) and polyvinyl chloride (PVC, $(C_2H_3Cl)_n$, Mw~233,000) were purchased from Sigma-Aldrich and used as received. The galvanostatic electrochemical method [58-61] was chosen for the preparation of the PEI and PVC grafted iron oxides. This method has simplest procedure for preparation of nanomaterial, which used a two-electrode system and mild deposition conditions [62]. For deposition of PVC grafted nickel ions doped iron oxide, the electrolyte solution was composed of 2.4 g iron(III) nitrate, 1 g iron(II) chloride, 0.2 g nickel chloride and 0.5 g polyvinyl chloride dissolved the in one litter deionized water. Also, in the electro-synthesis of PEI grafted nickel ions doped iron oxide, the same electrolyte

composition was used and just the polyvinyl chloride was replaced with polyethyleneimine. The deposition processes, for both samples, were carried out by applying a typical current density of 10 mA $cm^{-2}$ for 30 min. In both electrolytes, black films were observable on the cathode electrode at the end of deposition time. The electrodes were removed from the electrolytes and rinsed with the distilled, and dried at oven (in T=80 $^{o}$C). In the last step, the dried films were collected from the electrode surface in the powder form and were named PVC/Ni-SPIOs and PEI/Ni-SPIOs.

### 2.2. Characterization techniques

XRD patterns were obtained on PW-1800 base diffractometer, operating with Cu-Kα radiations (λ=0.15406 nm). The FT-IR spectra of both prepared Ni-SPIOs were acquired using Bruker Vector 22 IR instrument. Thermogravimetric analysis (TGA) of the PVC grafted Ni-SPIOs was performed using a PerkinElmer Pyris instrument. TGA measurements were made from room temperature to 700 °C at a heating rate of 10 °C $min^{-1}$. Observing the surface morphology of Ni-SPIONs samples were done through field-emission scanning electron microscope (FE-SEM, model Mira 3-XMU). Magnetization measurements were performed at room temperature with vibrating sample magnetometer (VSM). The isothermal magnetization (M) versus the applied magnetic field (G) measurement was performed at RT with Lake Shore 7307 instrument.

## 3. RESULTS AND DISCUSSION

### 3.1. Physico-chemical properties

Surface morphologies of the prepared samples are presented in Fig. 3. For both deposited samples i.e. PVC/Ni-SPIOs and PEI/Ni-SPIOs, the particle-shape morphology is seen in the SEM observations (Figs. 1a and b). The average particle size for the PVC/Ni-SPIOs and PEI/Ni-SPIOs samples was found to be 20 and 25 nm, respectively.

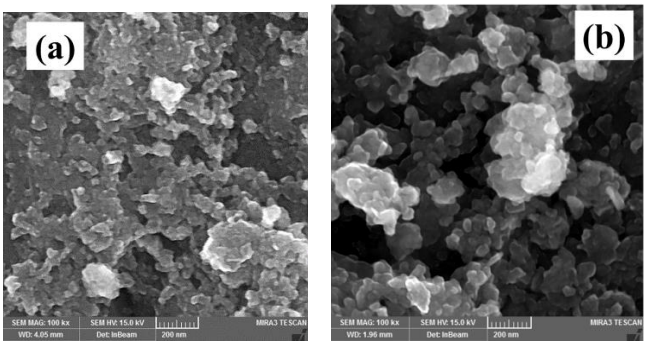


**Fig. 1.** FE-SEM images the prepared (a) PVC/Ni- SPIOs and (b) PEI/Ni-SPIOs

The XRD patterns of the prepared samples are also given in Fig. 2. It indicates that magnetite phase (i.e. $Fe_3O_4$) is the dominant phase in both samples, where the observed diffraction peaks are completely in consist with those of magnetite crystal structure. Furthermore, these XRD pattern is in agreement with reported ones in the literature [63-65]. Using the Debye-Scherrer equation for spherical particles, the PVC/Ni-SPIOs and PEI/Ni-SPIOs have an average grain size of 9.8 nm and 10.4 nm, respectively.

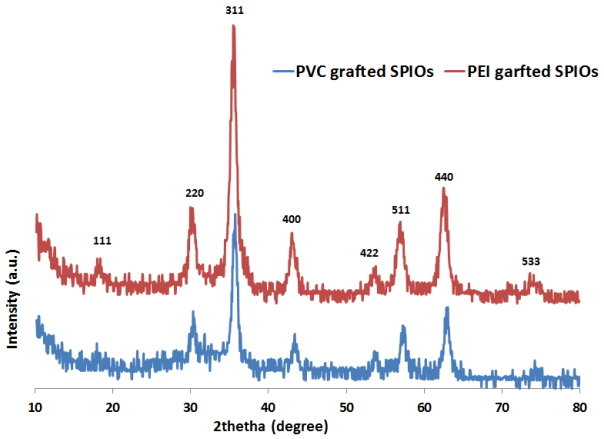


**Fig. 2.** XRD patterns of the prepared SPIOs

To confirm the grafting of surface of iron oxides particle with PVC/PEI during their galvanostatic deposition form electrolyte containing polyvinyl chloride/polyethyleneimine, the infra-red spectra of the PVC/Ni-SPIOs and PEI/Ni-SPIOs were provided in the wavenumber range of 400-4000 $cm^{-1}$. The IR spectra are shown in Fig. 3. Generally, the IR bands observed below 700 $cm^{-1}$ are related to the stretching vibrations of metal-oxygen bonds [65,66]. In the case of our sample, IR peaks are due to the iron(III)-oxygen and iron(II)-oxygen and/or nickel-oxygen vibrations. For the deposited PVC/Ni-SPIOs sample (Fig. 3a), there are IR bands located at 2935 $cm^{-1}$, 2872 $cm^{-1}$, 1472 $cm^{-1}$, 1251 $cm^{-1}$, 1190 $cm^{-1}$, 1168 $cm^{-1}$, 960 $cm^{-1}$, and 609 $cm^{-1}$, which are correspond to the asymmetric $CH_2$ stretching, symmetric $CH_2$ stretching, C-H scissoring bending, $CH_2$ deformations, C-C stretching, C-C bending, CH (out-of-plane) bending, and C-Cl stretching vibrations, respectively [65-68]. These IR results verified the presence of grafted PVC onto the Ni-SPIOs particles. In Fig. 3b, the IR spectrum of PEI/Ni-SPIOs sample exhibited the IR bands at 3396 $cm^{-1}$ (N-H stretching), 2924 $cm^{-1}$ (asymmetric C-H stretch), 2853 $cm^{-1}$ (symmetric C-H stretch), 1618 $cm^{-1}$ ($NH_2$ bending), 1525 $cm^{-1}$ (C-C stretching), 1465 $cm^{-1}$ ($CH_2$ bending), 1201 $cm^{-1}$ (C-N stretching), 1059 $cm^{-1}$ (C-C skeletal stretching), 1015 $cm^{-1}$ (C-N skeletal stretching), and 849 $cm^{-1}$ (C-H, O-H and N-H in-plane deformation vibrations) correspond to the PEI polymer [69-73], and confirmed its presence onto the Ni-SPIOs nano-particles.

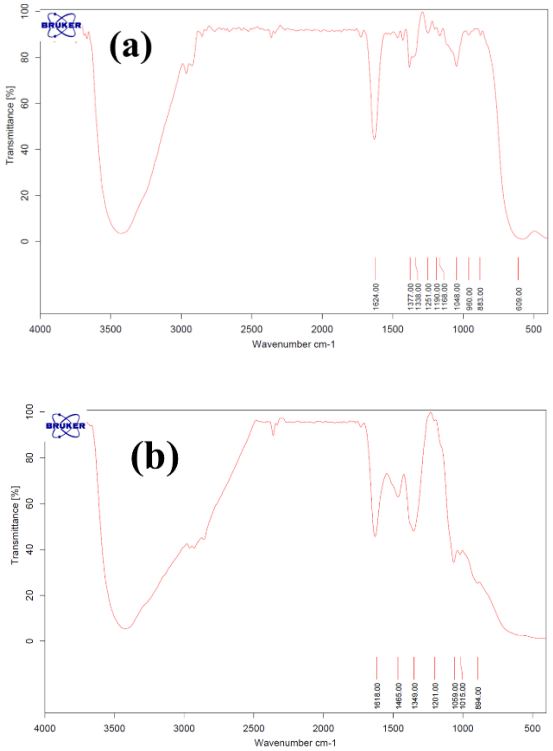


**Fig. 3.** IR spectra of the prepared (a) PVC grafted Ni- SPIOs and (b) PEI grafted Ni-SPIOs samples

Fig. 4 shows the DSC and related TG profiles for the prepared PVC+PEI/Ni-SPIOs sample at the temperature range of 25-700 ºC. The initial weight loss up to 100 °C (about 2.7%) is due to removal of physically attached $H_2O$ molecules onto Ni-SPIOs particles.

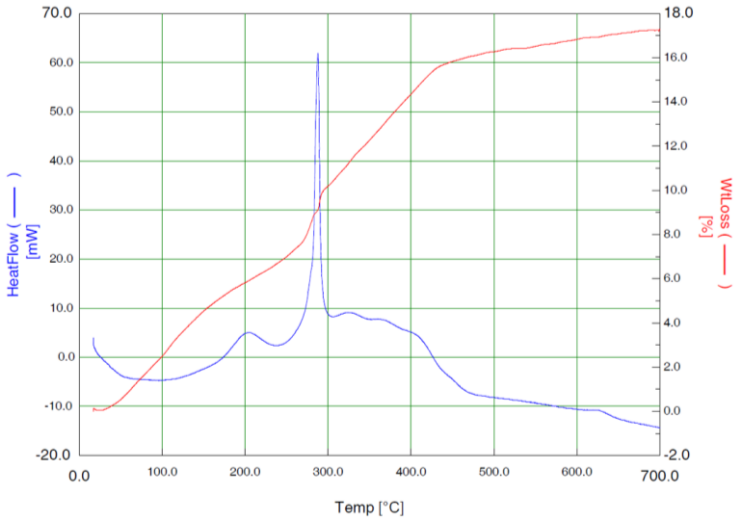


**Fig. 4.** DSC and the related TG profiles for electrosynthesized PVC/PEI grafted Ni-SPIOs

Two sharp endothermic peaks are observed in the DSC profile of sample (Fig. 4), which can be related to the decomposition of PVC polymer grafted onto Ni-SPIOs particles. In the published papers, the PVC degradation is reported to be occurred at temperature range of

200-250ºC [74,75]. Hence, the two consecutive peaks located at 215 to 285 °C are due to the PVC removal from the sample. The weight loss in this stage is observed to be 6%. After this step, there are further two continuous small endothermic peaks at the temperatures between 300-450 °C, which can be assigned to the decomposition of PEI polymer from the Ni-SPIOs particles [76,77]. The weight loss related to this event is about 7.2%. At final step, their small weigh loss (1.3%) at the temperatures of 450 up to 700 °C, is related to the phase transition of $Fe_3O_4$ into FeO [76,77]. These findings clearly verified the successful synthesis of PVC/PEI/Ni-SPIOs by our method.

### 3.2. Magnetic properties

The magnetic evolution of the electrosynthesized Ni-SPIOs samples were studied through VSM analysis. Fig. 5 presents the VSM curves of PVC grafted Ni-SPIOs and PEI grafted Ni-SPIOs samples at room temperature. From Fig. 5, it is clearly observable that bot samples behaved as a superparamagnetic material because of exhibiting reversible VSM curves, and low residual magnetization (*Mr*) and coercivity (*Hci*) values [77,78]. The magnetic data i.e. saturation magnetization (*Ms*), residual magnetization (*Mr*) and coercivity (*Hci*) values for the fabricated PVC/Ni-SPIOs were observed to be: Ms=40.42 emu/g, *Mr*=0.95 emu/g and *Hci*=8.21 G. For the PEI/Ni-SPIOs, these data are: *Ms*=49.26 emu/g, *Mr*=0.53 emu/g and *Hci*=7.05 G. It should be noted that the magnetic data have been reported to be: *Ms*=72.9 emu/g, *Mr*=1.21 emu/g for naked SPIOs [78], and *Ms*=41.47 emu/g, Mr=0.17emu/g for naked Ni-SPIOs [79]. From these data, ii is confirmed that the magnetic characters of SPIOs are enhanced with surface modification.

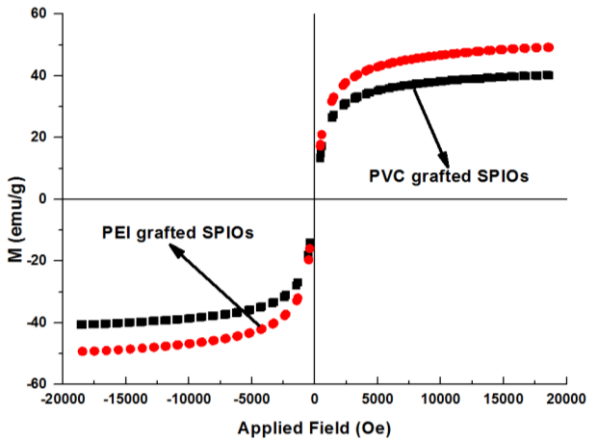


**Fig. 5.** Hysteresis loops for electrosynthesized PVC/Ni- SPIOs and PEI/Ni-SPIOs

Furthermore, the relative high saturation magnetizations (i.e. 52.98 emu/g and 48.51 emu/g, respectively, for PVC- and PEI/Ni-SPIOs samples) indicated their proper magnetic response for biomedical applications like as hyperthermia. Also, the very low residual

magnetization values (i.e. *Mr*=0.95 and *Mr*=0.53 emu/g, respectively, for PVC- and PEI grafted Ni-SPIOs revealed the excellent magnetic of these samples with removing the applied filed, which this factor is very important at many biomedical uses like as magnetic immunoassay, hyperthermic therapy, magnetic gene therapy and magnetic resonance imaging. Hence, it is concluded that the prepared SPIOs have proper physico-chemical and magnetic properties for biomedical uses.

## 4. CONCLUSION

In summary, a simple deposition method was developed for synthesis of polymer grafted metal ion doped nanoparticles with magnetite crystal structure. The IR bands related to the vibration modes of N-H, C-Cl, C-H, $NH_2$, C-C and C-N proved the presence of PVC and PEI polymer onto the surface of electrosynthesized iron oxide particles. Magnetic evolution was also confirmed the superparamagnetic behavior (i.e. low residual magnetization and negligible coercivity) of the prepared samples.

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