Magnetic properties and kinetic roughening study of prepared polyaniline: lead ferrite, cobalt ferrite and nickel ferrite nanocomposites electrodeposited thin films

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ABSTRACT

The purpose of this study is preparation of polyaniline–lead ferrite, polyaniline–cobalt ferrite and polyaniline/nickel ferrite thin layers. The electromagnetic pollution is a serious problem in the world that can be solved by electromagnetic interference (EMI) shielding. The EMI layers can be synthesized by conductive layer composed by magnetic particles. Crystallite size of samples was studied by (XRD) analyze via Debye–Scherrer and Williamson–Hall equations. The X-ray diffraction XRD patterns confirm the crystalline structure of the samples. The surface morphology of the composite layers was investigated by scanning electron microscopy (SEM) and the effect of thickness and different composed particles was investigated. The percentage of the constituents and purity of samples was studied by energy dispersive X-ray analysis (EDX) analysis. Also, surface roughness and kinetic roughening of thin films was investigated using atomic force microscopy (AFM). Hysteresis loop of the magnetic samples were analyzed by vibrating sample magnetometer (VSM). These new easy prepared nanocomposites introduce a suitable and effective coating for electromagnetic interference (EMI) shielding.

1 Introduction

The electromagnetic pollution has been arising from mobile phones, radar systems, computers and other electronic devices. It has become electromagnetic pollution where can be compared with other pollution like water and air pollution and it is necessary to dissolve this problem [1]. Since the use of electromagnetic devices has quickly increased and publications of electromagnetic energy have grown in the frequency span used via another users, electromagnetic interposition (EMI) has become an important trouble in urban society. Electromagnetic waves can peril health similar nervousness and headaches. Therefore, different EMI shielding procedures have
been expanded for decrease the possibility of incidence of the aforesaid dangers [2].

At first, metallic components were utilized for shielding aim. Newly, carbon-based nanostructures shielding materials have expand considerable attractive due to appropriate flexibility, small density and high electrical conductivity. The carbon materials in any matrix may reason decreasing EMI shielding efficiency and make them excellent materials for commercial shielding usage. Between the conducting polymers (CPs), polyaniline (PANI) is charming causes because easy synthesis, easy doping, high chemical stability and high electrical conductivity [3–6].

The addition of nanoparticles to polymers allows the modification of the polymer’s physical and mechanical virtues. The properties of nanomaterials are various from bulk because of surface efficacy and quantum confined size [5, 6]. Magnetic materials have many applications in data storage and magnetic devices like patterned media and random access. Magnetic iron oxide nanoparticles or ferrites are effective magnetic materials for the uses in sensors, catalysis and other magnetic devices [7, 8]. Ferrites can be classified in spinel ferrites, hexagonal ferrites and garnet ferrites categories between these, spinel ferrites have substantial significance for researchers because of their high saturation magnetization, small coercivity, large magneto crystalline anisotropy and high Curie temperature. Spinel ferrites in the total formula of AFe2O4 (A is a divalent metal cation) have newly possessed a big amount of consideration. Nickel ferrite (NiFe2O4) and cobalt ferrite (CoFe2O4) are the superlative significant functional ferrites which have a vast span of applications [9–12]. Hexagonal ferrites MFe12O19 (M = Pb, Sr and Ba) are known as permanent magnetic materials with powerful anisotropy concomitant the c-axis. Furthermore, hexagonal ferrites are technologically very helpful materials in sector because of their application in permanent constant magnets and microwave devices [13].

Due to the widespread applicability of ferrites, different synthesis path has been expanded since their fabrication. These contain the sol–gel, coprecipitation, hydrothermal and microemulsion, microwave treatment [14–16]. Between the different techniques reported, microwave combustion the method is maybe chosen for homogeneity, high purity and reformed specifications. The microwave energy is an inner one equipment of heat energy generation and transformation. In result, the morphology, shape, crystallite size, and other properties are almost variation by changed microwave energy as heating source [15].

Electrodeposition is an interesting technique for deposition of conductive and semi conductive layers. Electrodeposited films can be produced by controlling either the deposition potential or current density in electrolyte. Three electrodes that are used in electrodeposition: (1) a working electrode (WE) on substrate is deposited, (2) a secondary electrode (SE) to complete the electrical circuit and (3) a reference electrode (RE) to control the potential between the WE and the electrolyte [17, 18].

The Superlative efficient method to fabrication conducting polymers is via electrochemical polymerization. Electrochemical polymerization technique has well control on the morphology and thickness of the coatings via controlling current density and potential. In addition, Electrochemical is an economical method [19]. Electrodeposition of polyaniline is an economical and environmental technique. Electrodeposition process can be environmental because no toxic oxidant or surfactant is needed to polymerization of solution. The electrodeposition of polyaniline is dependent on different factors like pH, temperature, concentration and substrate material texture [20–22]. Polyaniline–lead ferrite, polyaniline–cobalt ferrite and polyaniline/nickel ferrite thin layers were synthesized by electrodeposition methods. These new easy prepared nanocomposites introduce a suitable and effective coating for electromagnetic interference (EMI) shielding.

2 Experimental

The materials that were used to synthesize of nanocomposites are lead acetate (Pb(C2H3O2)2), cobalt-sulfate hydrate (CoSO4·H2O)7, nickel sulfate (NiSO4·6H2O), sodium hydroxide (NaOH) and iron(III) nitrate nonahydrate (Fe(NO3)3·9H2O) were purchased from Sigma-Aldrich or Merck (Germany, Berlin). Aniline (C6H5NH2) that was distilled before use was purchased from Merck. All the chemicals were used as received without further purification, the purity of the all materials were about 99%.

The structure of the samples was analyzed by X-ray diffraction (Philips, X’PertPro), with CuKα
radiation ($\lambda = 0.154$ nm). The percentage of ingredients was studied by X-ray energy scattering analysis (EDX). The surface morphology of the thin films was examined by scanning electron microscope equipped with EDX detector by LEO model VP 1450 (Germany). The surface roughness analysis of the layers was studied by atomic force microscopy manufactured by Ara Research Company (Iran). The room temperature hysteresis magnetic loop was investigated using the VSM device (Meghnatis Kavir Kashan Company, Iran).

2.1 Preparation of ferrite nanoparticles

In this study, nanoparticles of cobalt, lead and nickel ferrite were synthesized by microwave irradiation. The process steps for each of these are as follows.

2.2 Preparation of cobalt ferrite nanoparticles

For synthesized cobalt ferrite, 0.2 g of cobalt-sulfate hydrate and 1 g of iron nitrate $\text{9H}_2\text{O}$ were combined into 200 ml of deionized water. The solution was stirred for 30 min with a magnetic stirrer at temperature of 100 $^\circ$C. Then the pH of the prepared solution was increased to 10 by incorporate 20 ml of 1 M sodium hydroxide solution. The solution was cooled to room temperature for 10 min. Then the solution was irradiated under microwave irradiations (10 min). The cobalt ferrite precipitate was washed and separated by centrifugation and completely dried at 80 $^\circ$C. Schematic of cobalt ferrite nanoparticle synthesis is shown in Fig. 1.

2.3 Synthesis of lead hexa-ferrite nanoparticles

0.1 g of Pb($\text{C}_2\text{H}_3\text{O}_2$)$_2$ and 1.5 g of Fe(NO$_3$)$_3$9H$_2$O were mixed together into 50 ml of deionized water. The solution was stirred for 5 min with a magnetic stirrer. The pH of the solution by adding 15 ml of 1 M NaOH solution was increased to 10. The solution was then microwave irradiated for 5 min. The PbFe$_{12}$O$_{19}$ precipitate was washed and centrifuged and completely dried at 80 $^\circ$C. The powder was then placed in the electronic furnace for 2 h at 800 $^\circ$C. Figure 2 indicates schematic synthesis steps of lead ferrite nanoparticle.

2.4 Preparation of nickel ferrite

Nickel ferrite nanoparticles were prepared by combination of 0.1 g of nickel sulfate and 0.31 g of iron(III) nitrate nonahydrate into 50 ml of deionized water. The mixed solution was stirred for 5 min and pH of the resulted solution was increased to 10 (by sodium hydroxide solution). The final solution was then irradiated under microwave for 5 min. The nickel ferrite precipitate was separated by centrifugation and dried completely at 80 $^\circ$C. The nickel ferrite powder was then calcinated for 2 h at 500 $^\circ$C. Figure 3 illustrates schematic synthesis of nickel ferrite nanoparticles.

2.5 Electrodeposition of polyaniline

Aniline thin films were deposited on copper substrate by cyclic voltammetry technique. For electrodeposition of polyaniline thin layers, 0.2 M of sulfuric acid solution and 0.1 M of aniline were used as electrolyte. The deposition was performed using cyclic voltammetry (number of cycles 10 and 5, applied voltage at this stage was about $-0.2$–$1.2$ V). Schematic of polyaniline thin film synthesis steps is shown in Fig. 4.

2.6 Polyaniline/lead ferrite, nickel ferrite and cobalt ferrite nanocomposites

In this study, electrodeposition was used to prepare polyaniline–lead ferrite, polyaniline–nickel ferrite and polyaniline–cobalt ferrite composites. For this purpose, a copper layer was used as the substrate. The substrates were prepared by two-step mechanical and electrochemical polishing. Electroporation is performed in an electrochemical cell containing working electrode, secondary electrode and reference electrode. 1 g of each ferrite was added to 100 ml electrolyte include 0.1 M of aniline and 0.2 M of sulfuric acid solution separately. The deposition was performed at five cycles and by cyclic voltammetry (CV) for the voltage of $-0.2$ to $1.2$ V. For investigation of nanoparticles concentration in electrolyte polyaniline–cobalt ferrite nanocomposites prepared with to different 0.1 or 1 g cobalt ferrite in the same electrolyte. Figure 5 illustrates the schematic of
Fig. 1 Schematic of cobalt ferrite nanoparticle synthesis steps

Fig. 2 Schematic of lead ferrite nanoparticle synthesis steps
polyaniline–lead ferrite, nickel ferrite and cobalt ferrite nanoparticles thin film synthesis.

3 Results and discussion

3.1 X-ray diffraction

Figure 6 shows the X-ray diffraction pattern of cobalt ferrite nanostructures in the range about 10°–80°. The X-ray diffraction pattern shows CoFe$_2$O$_4$ nanoparticles peaks that have a suitable agreement with standard peaks with cubic standards (JCPDS: 01-1121, space group: Fd-3 m, space group number: 227, $a = b = c$: 8.39 A) [23].

Figure 7 indicates the pattern of nickel ferrite nanoparticles, the results have suitable accordance with cubic standards (JCPDS: 74-1913, FCC, space group: Fd-3 m, space group number: 227, $a = b = c$: 8.258 A). XRD pattern of lead hexa-ferrite is shown in Fig. 8, that have agreement with hexagonal standards (JCPDS: 17-0660, space group: P6$_3$/mmc, space
Fig. 5 Schematic of polyaniline–ferrite nanoparticles thin film synthesis steps

Fig. 6 XRD pattern of cobalt ferrite nanoparticles

Fig. 7 XRD pattern of nickel ferrite nanoparticles
group number: 194, \(a\), \(b\): 5.88, \(c\): 23.02 A) [24, 25]. XRD pattern of polyaniline/cobalt ferrite, polyaniline/nickel ferrite and polyaniline/lead ferrite are depicted in Fig S1, S2 and S3, respectively, that approve formation of nanocomposite and presence of nanoparticles in poly aniline matrix (on the copper substrate).

The general technique for calculation of the crystallite size is Debye–Scherrer formula, Eq. (1).

\[
D = \frac{0.9\lambda}{\beta \cos \theta}
\]  

That \(\beta\) is full width half maximum, \(\lambda\) is the X-ray wavelength (1.54 Å), \(\theta\) is diffraction angle and \(D\) is crystallite size [25].

The crystallite size of cobalt ferrite, nickel ferrite and lead hexa-ferrite nanoparticles were calculated, 16, 21 and 42 nm, respectively.

The Williamson–Hall method permits to specify the magnitude of the crystallite size and strain of the crystal lattice. The Williamson–Hall can be written like the Eq. (2):

\[
\beta \cos \theta = 4\varepsilon \sin \theta + \frac{0.9\lambda}{D}
\]  

That \(\varepsilon\) is lattice strain and other parameters are like Eq. (1) [26]. \(\varepsilon\) and \(D\) can be computed from the slope and intercept of the \(\beta \cos \theta\) and \(4 \sin \theta\) plots, respectively. Williamson–Hall linear fit curve for cobalt ferrite, nickel ferrite and lead ferrite nanoparticles are shown in Fig. 9. The crystallite size of CoFe2O4, NiFe2O4 and PbFe12O19 nanoparticles via Williamson–Hall method were calculated 11, 23 and 46 nm, respectively. Also, the strain of lattice for CoFe2O4, NiFe2O4 and PbFe12O19 nanoparticles were obtained 0.004, 0.012 and 0.009, respectively.

### 3.2 Morphology study of nanoparticles and thin films

Figure 10 shows the surface morphology of polyaniline thin films for five cycles of deposition that was investigated by scanning electron microscopy and Fig. 11 illustrates the surface morphology of polyaniline thin layer for 10 cycles of deposition. As depicted in Figs. 10 and 11, the grain of layers increases with increasing number of deposition cycles and thus the thickness of the layer.

Figure 12 indicates the morphology of cobalt ferrite nanoparticles; images approve formation of nanoparticles with average diameter about 40 nm. Figure 13 shows scanning electron microscopy images of lead hexa-ferrite nanoparticles, the results confirm synthesis of magnetic nanostructures with size less than 50 nm. Figure 14 illustrate SEM images of nickel ferrite; these outcomes also show all ferrites nanoparticles have the average size around 50 nm.

Figure 15 shows the surface morphology of the polyaniline–cobalt ferrite thin layer nanocomposite with 0.1 g of cobalt ferrite and Fig. 16 shows the same nanocomposite with 1 g of cobalt ferrite. In these images, as we expected nanocomposite with 1 g of cobalt ferrite have more nanoparticles in comparison to nanocomposite with 0.1 g of cobalt ferrite.

Figures 17 and 18 indicate the morphology of the polyaniline/lead ferrite nanoparticles and
polyaniline/nickel ferrite thin layer nanocomposite with addition of 1 g nanofillers, respectively.

According to SEM analysis, the ferrite nanoparticles were dispersed homogeneously in the polyaniline thin layer and the larger nanoparticles make the larger grains in the polymer matrix.

3.3 Elemental map and energy dispersive X-ray spectroscopy

Energy dispersive X-ray (EDX) analysis determined the elements of electrodeposited films. Figures 19, 20 and 21 show elemental EDX analysis for cobalt ferrite, nickel ferrite and lead ferrite nanopowder, respectively. $K_{a}, K_{B}, L_{a}$ of iron element are shown in all spectra, for cobalt ferrite also $K_{a}, K_{B}, L_{a}$ peaks of cobalt element was approved. $M_{a}, M_{B}, L_{a}$ peaks are related to lead element and finally spectra confirm presence of nickel with obvious related $K_{a}, K_{B}, L_{a}$ peaks. As we expected $K_{a}$ peak of oxygen exist in all analyses and there are no major peaks related to impurities.

Figures 22, 23 and 24 indicate EDX analysis for polyaniline/cobalt ferrite, nickel ferrite and lead ferrite thin layer composite, respectively. The $K_{a}$ of N
and C peaks belong to the polyaniline thin layer. All the peaks are as same as of the pure nanoparticles with this point that all nanocomposites are on the copper substrate that observe in the all-samples spectra. In Fig. 22 Co, O and Fe peaks are related to cobalt ferrite in composite. The Ni, Fe, and O peaks indicate nickel ferrite formation in Fig. 23. Finally, Fig. 24 displays the peaks of Pb, Fe and O for of lead ferrite nanoparticles in composite. The elemental image of polyaniline thin layer is shown in Fig. S4.

3.4 Kinetic roughening of surface

Kinetic roughening happens in systems when matter is removed from or added to a surface. The study of material in atomic scale ranges in thin films to the macroscopic scales as known kinetic roughening [27]. The roughness of a thin film depended to thickness of layer. The roughness ($w$) is expressed by Eq. (3).
that \( h \) is each point height the surface of layers, \( l \) is the length of area that roughness is measured and finally \( t \) is the deposition time or film thickness. In some systems, kinetic roughening is explained via Family–Vicsek or normal scaling. In normal scaling suppose where roughness depended to \( l \) and \( t \) in small and large scales by Eqs. (4) and (5).

\[
w(l, t) = \sqrt{\langle h(t) - \langle h(t) \rangle \rangle^2} \tag{3}
\]

\[
w(l, t) \propto l^H \text{ for } l \ll l_c \tag{4}
\]

\[
w(l, t) \propto t^\beta \text{ for } l \gg l_c \tag{5}
\]

In Eqs. (4) and (5), \( H \): Hurst coefficient, \( \beta \): growth exponent and \( l_c \): crossover length. For some other systems kinetic roughening explained via anomalous scaling rules that depended by following equations:

Fig. 13 SEM images of lead ferrite nanoparticles with a 200 nm and b 500 nm scale bar

Fig. 14 SEM images of nickel ferrite nanoparticles with a 200 nm and b 500 nm scale bar
In these equations \( \beta_{\text{loc}} \) is known as the local roughness exponent. In anomalous scaling roughness in small length depended to thickness of layers but in normal scaling not depended and when \( \beta_{\text{loc}} \) is zero anomalous scaling changed to normal scaling [28].

Fig. 15 SEM images of polyaniline/cobalt ferrite thin layer composite (with 0.1 g of cobalt ferrite) with a 200 nm and b 500 nm scale bar

Fig. 16 SEM images of polyaniline/cobalt ferrite thin layer composite (1 g of cobalt ferrite) with a 200 nm and b 500 nm scale bar

\[
w(l, t) \propto t^{4l} \beta_{\text{loc}} \text{ for } l \ll l_c \tag{6}\\
w(l, t) \propto t^{6l} \beta_{\text{loc}} \text{ for } l \gg l_c \tag{7}
\]
Figure 26 shows diagram of roughness-scan length in logarithmic scale of deposited polyaniline thin films with 5 and 10 cycles. This diagram describes polyaniline thin films have an anomalous scaling behavior.

Figure 27 relates to the results of AFM analysis for polyaniline/cobalt ferrite thin layer composite with 0/1 and 1 g cobalt ferrite nanoparticles. According to these images with increases of cobalt ferrite nanoparticles density, the roughness of deposited films cycles increased, which is consistent with the SEM results.
3.5 Magnetic properties

The magnetic properties and hysteresis loop of samples were investigated via vibrating sample magnetometer at room temperature. Figures 28, 29 and 30 illustrate the vibrating sample magnetometer VSM analysis for cobalt ferrite, nickel ferrite and lead ferrite nanoparticles, respectively. According to these results, lead ferrite and cobalt ferrite are ferromagnetic materials, also lead ferrite is harder than cobalt ferrite, but nickel ferrite nanoparticles have a superparamagnetic behavior.

Magnetic properties of polyaniline–cobalt ferrite (1 g nanoparticles) thin layer composite were investigated in parallel and vertical field. The hysteresis loop of this thin film is indicated in Figs. 31 and 32 in parallel and vertical field, respectively. Comparing the results of hysteresis loop analysis for cobalt ferrite and the thin film composite show that the amount of $M_s$ and $H_c$ with the presence of polyaniline is greatly reduced. For above thin films in the parallel field state, the saturated magnetic field is less than the vertical field state. Easy axis in vertical field mode and hard axis in parallel field mode. The remanence magnetization ($M_r$), saturation magnetization ($M_s$) and magnetic coercivity ($H_c$) each of nanoparticles and composite layers explain in Table 1. As shown in Table 1, the remanence magnetization, saturation magnetization and magnetic coercivity of the thin layer composite relative to the nanoparticles have been severely reduced due to the low ratio of
magnetic particles to the polyaniline and the substrate. Also, coating of nanoparticles by polyaniline reduces the intensity of the magnetic properties of the composite.

4 Conclusions

The lead ferrite, cobalt ferrite and ferrite nickel nanoparticles were fabricated by microwave irradiation. The polyaniline/nanoferrites thin film composites was grown by electrodeposition method. XRD analysis confirmed the structure of samples, also crystallite size and strain of lattice was calculated using the Debye–Scherrer method and the Williamson–Hall method. The crystallite size of the nanoparticles was calculated by these methods consisted the grain size of samples observed by SEM. The SEM analysis found that the grains size has increased with increasing layer thickness. The EDX analysis approved the peaks related to the polyaniline each of ferrites. Kinetic roughening of polyaniline thin films has an anomalous scaling behavior.

![Fig. 23 EDX pattern of polyaniline/nickel ferrite thin layer composite](image1)

![Fig. 24 EDX pattern of polyaniline/lead ferrite thin layer composite](image2)

![Fig. 25 AFM images of thin layer of polyaniline with a 5 cycles and b 10 cycles of deposition](image3)

![Fig. 26 The roughness-scan length diagram in logarithmic scale for the thin layer of polyaniline with 5 and 10 cycles of deposition](image4)
The results of the VSM indicated lead ferrite and cobalt ferrite are ferromagnetic and nickel ferrite is superparamagnetic. Also, in composite polymer, $M_s$ and $H_c$ were reduced by adding nanoparticles to polyaniline.

The results approve these new and applicable conductive and magnetic polyaniline–lead ferrite, cobalt ferrite and nickel ferrite thin layers were synthesized by electrodeposition methods. These facile
prepared nanocomposites introduce a suitable and effective coating for electromagnetic interference (EMI) shielding.

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