

Spectroscopic Evaluation of Fe–Ni Nanoalloys for Potential Applications in Magnetic Hyperthermia

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ABSTRACT

Due to the capacity for magnetic materials to produce localised heat under an externally imposed alternating magnetic field, magnetic hyperthermia has become a promising additional technique for cancer treatment, potentially avoiding undesired thermal exposure of surrounding healthy tissues. Because composition and synthesis circumstances can modify the structural, magnetic, and physicochemical properties of iron-nickel (Fe–Ni) alloys, they are particularly interesting. In the current investigation, an electrodeposition process was used to create a Fe–Ni-based coating on a copper substrate in order to examine its properties and assess its potential for use in future magnetic hyperthermia applications. Before electrodeposition, the copper substrates were manually polished, chemically etched, and ultrasonically cleaned to enhance coating adherence and surface quality. Under carefully regulated experimental conditions, electrodeposition was performed at a constant potential of 2 V. UV-visible spectroscopy was used to examine the optical response of the deposited material over a wavelength range of roughly 320–800 nm. The near-ultraviolet part of the spectrum showed very weak absorption, whereas the visible and near-infrared sections showed wavelength-dependent increases in absorption. Around 590 nm, a noticeable absorption characteristic of about 0.047 a.u. was seen, and at 800 nm, the maximum absorbance of around 0.054 a.u. was recorded. A complex wavelength-dependent optical response of the deposited material is shown by the broad and irregular spectral behaviour. These initial results encourage more research on electrodeposited Fe–Ni-based materials;

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however, before their efficacy for magnetic hyperthermia can be determined, magnetic hysteresis, alternating-magnetic-field heating, specific absorption rate, and biological studies are needed.

Keywords: Fe-Ni Alloy; Electrodeposition; Magnetic Hyperthermia; UV–Visible Spectroscopy; Thin Films; Biomedical Applications.

Introduction

There is an ongoing need for therapeutic strategies that can increase tumour selectivity while minimising harm to healthy tissues because cancer is still a significant global health concern. Chemotherapy and radiation therapy are examples of conventional treatments that are clinically significant but may cause systemic toxicity, treatment resistance, and harm to nearby healthy cells. As a result, hyperthermia has drawn interest as an additional or supplemental therapeutic approach. To cause cellular stress, protein denaturation, membrane damage, and enhanced sensitivity to chemotherapy or radiation, hyperthermia therapy involves purposefully raising the temperature of tumour tissue, usually to 40–45 °C. However, deep or irregularly shaped tumours may be challenging for traditional heating methods to produce a consistent and localised temperature distribution. Because magnetic materials exposed to an alternating magnetic field can produce heat locally, magnetic hyperthermia has emerged as a potential solution to this constraint. Therefore, without requiring direct electrical contact with the target tissue, magnetic nanoparticles or nanostructured magnetic materials can function as remotely activated heat mediators [1].

An alternating magnetic field's magnetic energy is transformed into thermal energy during magnetic hyperthermia. Heating can be caused by Brownian relaxation, α relaxation, magnetic hysteresis losses, and interactions between nearby magnetic domains or particles, depending on the material's size, magnetic state, and surroundings. Brownian relaxation refers to the rotational movement of a magnetic nanoparticles within the surrounding liquid, whereas Neel relaxation occurs through the reorientation of the particles's magnetic movement. Energy dissipation caused by the irreversible magnetization reversal and hysteresis can become more pronounced in larger ferromagnetic particles and continuous magnetic films. As a result, the size of the particles or crystallites, phase structure, saturation magnetisation, coercivity, magnetic anisotropy, particle concentration, aggregation state, and the amplitude and frequency of the applied magnetic field all influence the heat produced by a magnetic material [2].

The specific absorption rate or specific loss power are frequently used to characterise the heating performance of magnetic materials. Under particular alternating-field circumstances, these parameters show how much heat is produced per unit mass of magnetic material. However, without reporting the magnetic-field intensity, frequency, sample concentration, heat capacity, and experimental geometry, it is challenging to compare various investigations. Furthermore, because heating also depends on coercivity, anisotropy, magnetic relaxation, and interparticle interactions, a material with high saturation magnetisation may not always show the best hyperthermia performance. Therefore, before a material can be categorised as an effective magnetic-

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hyperthermia agent, structural characterisation must be paired with magnetic and alternating-field heating data [1] [2].

Due to their magnetic behaviour and somewhat established biomedical relevance, iron-based magnetic nanoparticles, especially iron oxides, have been extensively studied for magnetic hyperthermia. However, because alloying can offer more control over saturation magnetisation, coercivity, Curie temperature, and magnetic anisotropy, metallic and bimetallic magnetic systems have also drawn interest. Iron-nickel alloys are particularly intriguing among these systems because their crystallographic and magnetic properties can be altered by varying the Fe composition, synthesis conditions, and post-deposition treatment. The relative contributions of these phases can significantly influence the magnetic response of Fe-Ni alloys, which can form face-centered cubic, body-centered cubic, or hybrid FCC-BCC structures [3].

A recent work showed that a composition-dependent structural transition from a primarily FCC structure to coexisting FCC and BCC phases was generated by varying the iron concentration of Fe-Ni nanoalloys. The produced nanoalloys showed low coercivity, comparatively strong saturation magnetisation, and soft-ferromagnetic behaviour. An optimised Fe-Ni ferrofluid produced a highest reported specific absorption rate of 236.03 W g^{-1} at a concentration of 2.5 mg mL^{-1} , when exposed to alternating magnetic field, allowing the material to reach temperature range required for hyperthermia. Although the authors stressed the need for more thorough biological validation, the study also included preliminary cytocompatibility testing. These results encourage more research into Fe-Ni-based materials for magnetic hyperthermia and offer concrete proof that the composition and crystalline phase of Fe-Ni alloys can affect their magnetic heating behaviour [4].

Fe-Ni alloys can be made using a variety of physical and chemical processes, including as hydrothermal synthesis, chemical reduction, thermal decomposition, mechanical alloying, sputtering, and electrodeposition. Among these methods, electrodeposition is appealing because it allows the direct production of alloy coatings on conductive substrates, is reasonably priced, and can be carried out at a moderate temperature and pressure. Electrolyte composition, metal-ion concentration, current density, applied potential, deposition duration, bath temperature, pH, agitation, complexing agents, and substrate characteristics can all be used to modify the thickness, composition, and microstructure of electrodeposited films. Therefore, electrodeposition can be used to create nanocrystalline Fe-Ni coatings with specific structural and magnetic properties [4][5].

Nevertheless, the simultaneous electrodeposition of nickel and iron is a difficult procedure. The less noble iron component may be deposited preferentially in comparison to nickel in Fe-Ni complexes, which frequently display aberrant code position. As a result, the nominal Fe ratio in the electrolyte may be very different from the composition of the deposited layer. This behaviour can be affected by local pH variations, complex formation, mass transport, hydrogen evolution, and adsorption of iron-containing hydroxyl species. Recent studies have demonstrated that the iron content, grain shape, surface roughness, crystalline phase, and magnetic characteristics of electrodeposited Ni-Fe films can be changed by electrolyte composition, boric acid concentration, chloride ions, complexing agents, and current density. While lower

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current densities may prefer compact and fine-grained coatings, higher current densities may potentially promote grain expansion or result in dendritic formations [5][6].

The nucleation and development of electrodeposited Fe-Ni coatings are also significantly influenced by the substrate. Adhesion, elemental distribution, crystallographic texture, and deposit morphology can all be altered by variations in substrate composition, crystal structure, surface energy, roughness, and conductivity. According to Bündostocka et al., substrate-dependent variations in alloy composition and crystallographic texture were observed in Fe-Ni films formed under otherwise comparable conditions. This suggests that while analysing the structural and functional behaviour of electrodeposited Fe-Ni films, the substrate needs to be reported and taken into account [4][7].

Therefore, the goal of the current work was to create an electrodeposited Fe-Ni alloy coating and use X-ray diffraction and UV-visible spectroscopy to examine its structural and optical properties. The crystalline phase and average crystallite size were determined using XRD, and the wavelength-dependent optical response of the deposited material was investigated using UV-visible spectroscopy. The study assesses the Fe-Ni coating's usefulness as a potential material for further research on magnetic hyperthermia and offers preliminary evidence about its production and nanocrystalline properties. To determine its true magnetothermal and biomedical performance, specific absorption rate calculations, magnetic hysteresis tests, alternating-magnetic field heating trials, and biological testing are still required.

EXPERIMENTAL SET-UP:

Following steps have been followed during deposition process.

Choice of Substrate

Selection of suitable substrate is fundamental for obtaining excellent thin film and depend on the deposition method used. Commonly used substrates include ITO coated glass substrates as well as metallic substrate such as copper and titanium[8]. Substrate should be economical, easily accessible, and available in suitable crystal sizes. It should possess appropriate. Substrate can cut simply and polished. Substrates must also withstand even in the elevated temperatures[9]. Substrates should have the ability to bear high temperatures. Copper substrate was selected because of its dielectric properties, thermal expansion characteristics, and lattice compatibility with the deposited film. The substrate should also be easy to cut and polish and should remain stable at elevated temperatures [9] of its good mechanical strength, electrical properties, low resistivity, high melting point, good scrap values, availability, and self-diffusion characteristics. These properties make copper a suitable material for thin film deposition. Pure copper has resistivity $1.679 \times 10^{-8} \Omega\text{m}$ at 20° temperature while conductivity $5.95 \times 10^7 (\Omega\text{m})^{-1}$.

Cleaning the Substrate:

Proper substrate cleaning is essential for achieving continuous and high-quality coatings with good adhesion. Surface contamination can arise from intrinsic sources, such as native oxides, as well as extrinsic sources, including organic residues from the

surrounding environment. The copper sheet was polished using P2000C-grade sandpaper and then cut into 1*1cm substrates. The substrates were flattened to obtain a smooth and uniform surface. After etching, the substrates were removed from the HCl solution and thoroughly rinsed with distilled water following an initial rinse with tap water. The substrates were then placed in a beaker containing acetone and treated in an ultrasonic bath for 10-12 minutes. This treatment helps to remove organic contaminants from the substrate surface through ultrasonic vibrations. The above-mentioned cleaning method is much more effective for producing high-quality thin films with better adhesive, texture and structural properties of deposits.

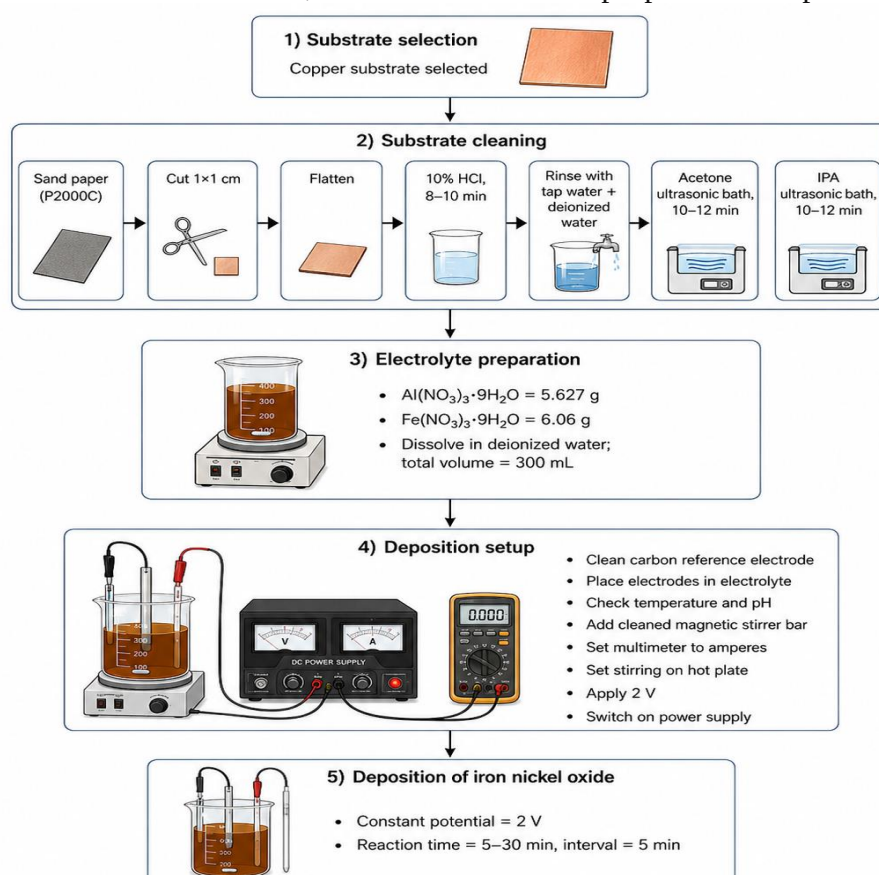


Figure 1: Experimental Flow chart for Electrodeposition of Iron-Nickel Thin Films

Electrolyte Preparation:

The required compounds were accurately weighted using a weighted machine. All the compounds were dissolved in distilled water to prepare electrolyte solution with a final volume of 300ml. Iron nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were taken 6.06 grams.

Deposition Process:

First, clean the electrode (carbon electrode) properly. Then, pour the electrolyte solution into the beaker containing the electrodes and check the temperature and pH of the electrolyte solution before starting the deposition process. Clean the magnetic stirrer

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with IPA (isopropyl alcohol) using tweezers and place it in the beaker. Set the multimeter to the amperes (A) mode and adjust the hotplate to the stirring setting. Set the voltage to 2 volts, then turn on the power supply. Once the power supply is switched on, the deposition process will begin.

As shown in figure 2, arrangements and analysis of sample

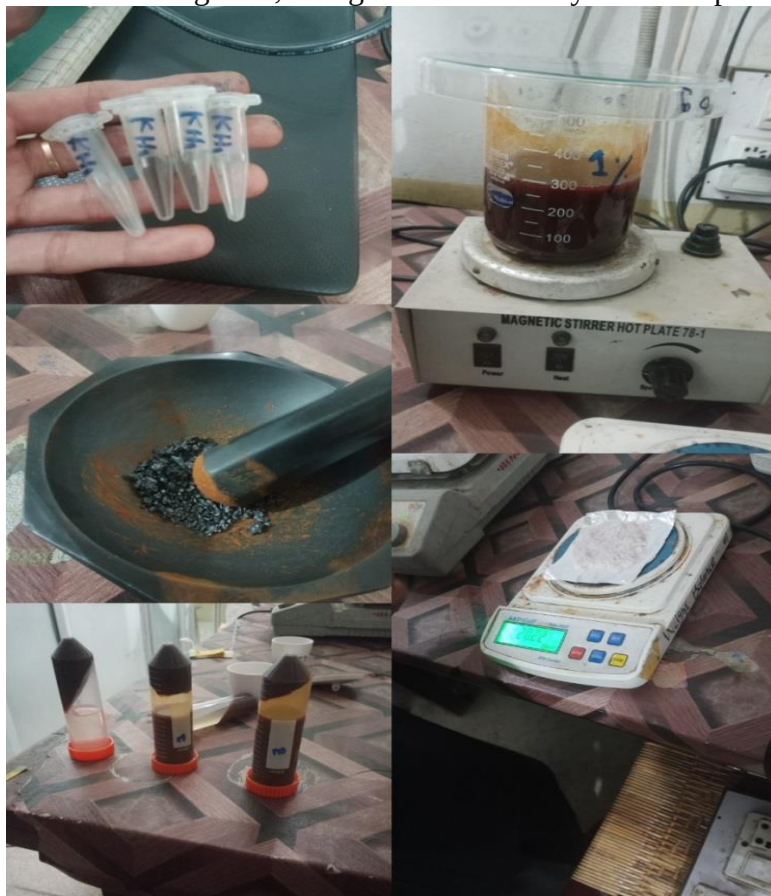


Figure 2: Experimental setup and sample procedure for NiFe_2O_4

Deposition of Iron Nickel Oxide:

Deposition process was performed at a constant potential of 2V, while the reaction time was varied from 5 to 30 minutes with a 5 minute interval.

Results and Discussions

UV-Analysis

Figure 2 shows the Fe–Ni alloy's UV-visible absorption spectra over a wavelength range of roughly 320–800 nm. The absorbance is extremely low and slightly negative at shorter wavelengths, between around 320 and 380 nm, suggesting very little optical absorption in this area. The absorbance starts to progressively increase as the wavelength rises over 380 nm. The absorbance increases noticeably between 400 and 470 nm, reaching roughly 0.019 a.u. After that, there is a slight decrease about 500 nm, and then there is a significant increase between 500 and 590 nm. This gradual rise

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suggests that, in comparison to the near-UV region, the Fe–Ni alloy interacts with incident light more strongly in the visible wavelength range.

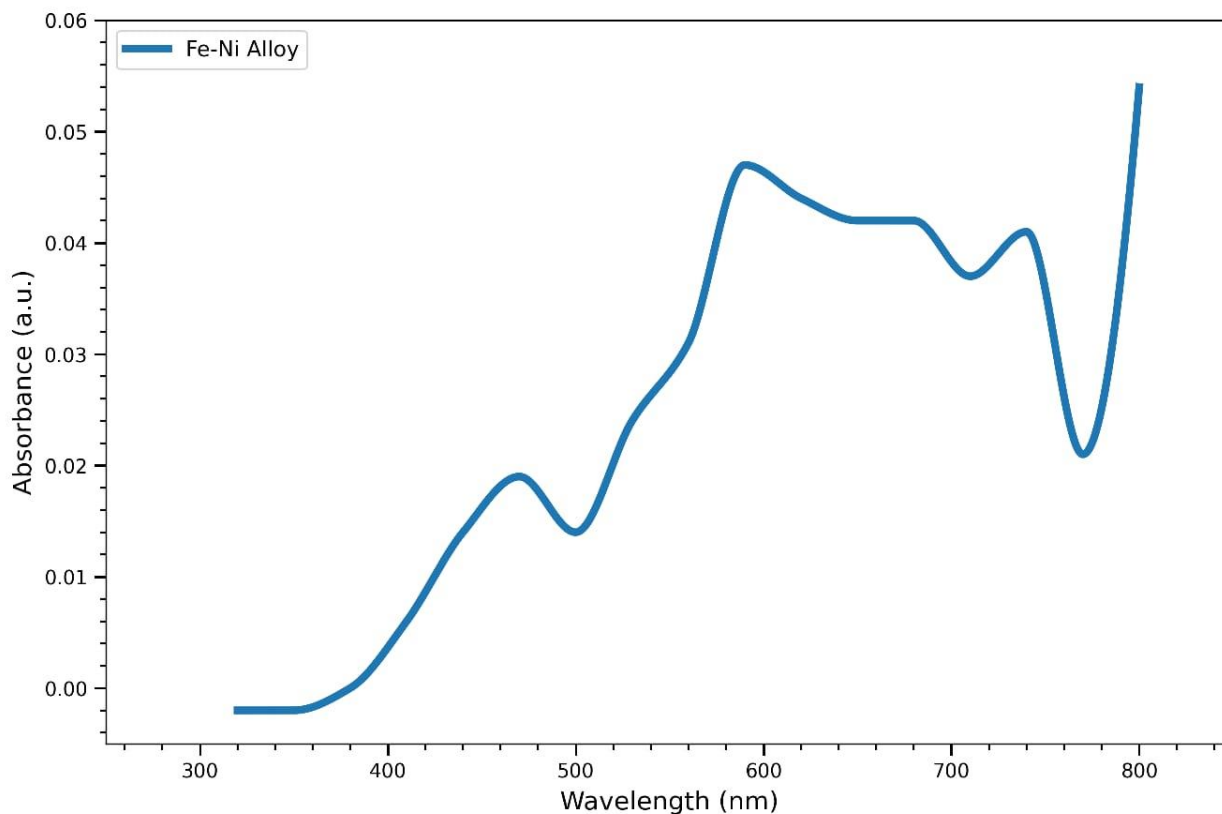


Figure 3: UV Analysis of Fe–Ni Alloy

Around 590 nm, a quite noticeable absorption characteristic emerges, with an absorbance of about 0.047 a.u. The absorbance then gradually drops and is almost constant at 0.042 a.u. between around 650 and 680 nm. Around 710 nm, there is another drop, and then there is a slight rise around 740 nm. The absorbance then rapidly increases to its highest recorded value of around 0.054 a.u. at 800 nm, with a fast decline to about 0.021 a.u. near 770 nm. These differences show that the Fe–Ni alloy's optical absorption is very wavelength-dependent and typically more noticeable in the longer wavelength range.

The electronic structure and interaction between the Fe and Ni atoms in the alloy, including wavelength-dependent electronic transitions and the contribution of free electrons characteristic of metallic materials, may be linked to the broad and irregular absorption behaviour. A complicated optical response rather than a single well-defined transition is suggested by the presence of multiple broad features and the lack of a single strong absorption peak. Overall, the spectrum shows fairly strong absorption in the visible to near-infrared range, especially between 590 and 800 nm, and very modest absorption at lower wavelengths.

Conclusion

In this work, an electrodeposition technique was used to create a Fe–Ni-based coating

on a copper substrate as a first step towards the creation of materials with potential uses in magnetic hyperthermia. To provide a clean surface appropriate for deposition, meticulous substrate preparation was used, including mechanical polishing, acid etching, and ultrasonic cleaning. After that, the deposition procedure was carried out at a constant applied potential of 2 V. The produced material's wavelength-dependent optical response was assessed using UV-visible spectroscopy. The shorter-wavelength area of the UV-visible spectrum showed very modest absorption, especially between about 320 and 380 nm. As the wavelength neared the visible and near-infrared regions, absorption generally increased. The maximum reported absorbance, around 0.054 a.u., happened at 800 nm, while a discernible absorption feature was seen at about 590 nm with an absorbance of about 0.047 a.u. A complicated optical response that might be related to the electronic properties of the deposited material is indicated by the lack of a single strong absorption peak and the presence of wide spectrum fluctuations. All things considered, the current work creates a foundation for future research and offers first characterisation of the electrodeposited material. However, appropriateness for magnetic hyperthermia cannot be confirmed by optical data alone. Therefore, comprehensive phase and structural characterisation, magnetic hysteresis measurements, saturation magnetisation and coercivity determination, alternating-magnetic-field heating experiments, specific absorption rate calculations, thermal stability assessment, and suitable cytocompatibility and biological evaluations should all be included in future research. Determining whether the synthesised material can operate safely and effectively as a magnetic heating agent for biological hyperthermia applications would require such experiments.

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