Thesis: In-Situ Synthesis of Nickel Ferrite Nanoparticles in Textiles

Abstract

This research explores the in-situ synthesis of nickel ferrite (NiFe2O4) nanoparticles within textiles, resulting in magnetically responsive fabrics. The method uses Fe(NO3)3·9H2O, Ni(NO3)2·6H2O, hydrazine, and hex-amine under controlled conditions (pH ~11, 100°C, 1 hour). To enhance magnetic properties, barium and cobalt nitrates were introduced. Though the material showed super-paramagnetic behavior, permanent magnetization was not achieved due to the absence of a 1 Tesla magnetic field. Magnetic crystals and powders were successfully recovered from wastewater, closing the loop for a more sustainable process. The nanoparticles were analyzed using VSM, SEM, XRD, and FTIR, confirming the formation of spinel NiFe2O4 with super-paramagnetic properties.

Chapter 1: Introduction

Textiles have traditionally served passive roles in daily life. However, recent advances in material science and nanotechnology have enabled the creation of smart textiles that respond to magnetic fields. Among these, magnetic textiles embedded with nanoparticles such as NiFe2O4 offer new capabilities in adaptive fashion, biomedical applications, and soft robotics.

This thesis focuses on the in-situ synthesis of NiFe2O4 nanoparticles directly within fabric substrates and explores methods to enhance their magnetic behavior, along with sustainable recovery of magnetic residues from wastewater.

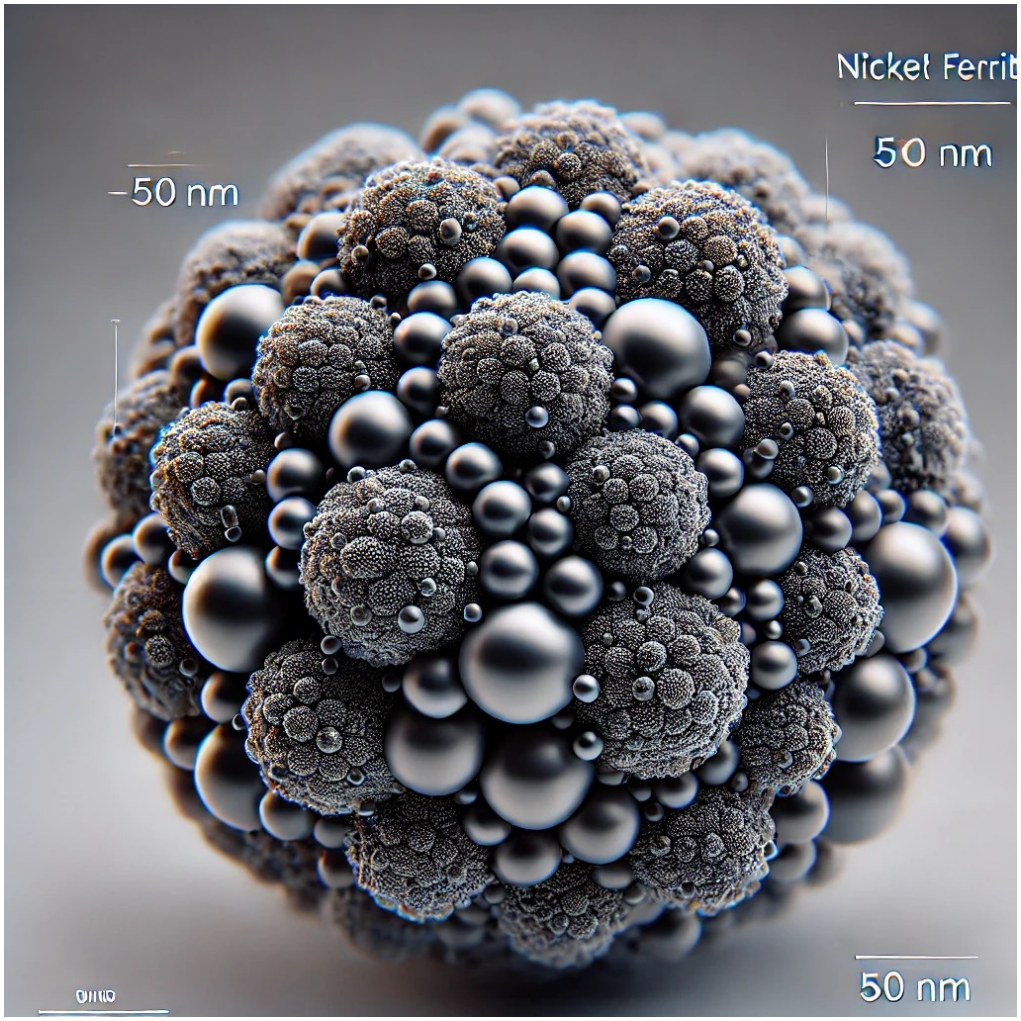
Chapter 2: Literature Review

The development of magnetic textiles through in-situ synthesis of nanoparticles is an emerging area in smart textiles and functional materials. Magnetic textiles have applications in wearable electronics, electromagnetic shielding, energy storage, biomedical textiles (drug delivery, hyperthermia treatment), and soft robotics. The integration of magnetic nanoparticles (MNPs) into textile fibers enhances their functionality while maintaining flexibility and breathability.

This section reviews previous studies on the in-situ synthesis of magnetic nanoparticles on textiles, discusses suitable textile substrates, and identifies potential nanoparticle candidates and synthesis techniques.

Magnetic Nanoparticles For Textile Applications

Magnetic Materials and Their Categories



1. Ferromagnetic Materials:

- Strong magnetic properties, permanently magnetized.
- Examples: Iron (Fe), Cobalt (Co), Nickel (Ni).

2. Paramagnetic Materials:

- Weakly attracted to magnetic fields, no permanent magnetization.
- Examples: Aluminum (Al), Platinum (Pt).

3. Super-paramagnetic Materials:

- Behave like ferromagnets in a magnetic field, but don't retain magnetization.

- Examples: Fe_3O_4, γ-Fe_2O_3.

4. Diamagnetic Materials:

- Weakly repelled by magnetic fields.
- Examples: Bismuth (Bi), Copper (Cu), Graphite.

Common Magnetic Nanoparticles (MNPs)

Nanoparticle	Properties	Applications in Textiles
Fe_3O_4 (Magnetite)	Strong magnetism, biocompatible	Wearable sensors, EMI shielding, medical textiles
γ-Fe_2O_3 (Maghemite)	High saturation magnetization, oxidation-resistant	Smart textiles, energy storage fabrics
$CoFe_2O_4$ (Cobalt Ferrite)	Hard magnetic, chemically stable	Data storage, actuators, magnetic-responsive fabrics
$NiFe_2O_4$ (Nickel Ferrite)	Corrosion-resistant, moderate magnetism	Anti-counterfeiting textiles, soft robotics

Textile Substrate Selection

Key Properties For Textile Selection:
- High Surface Area
- Hydrophilicity or Surface Reactivity
- Thermal and Chemical Stability

Textile	Advantages	Challenges
Cotton	High surface area, hydroxyl groups aid nanoparticle binding	Poor durability in harsh conditions
Polyester	Good mechanical strength, thermal stability	Requires surface activation
Nylon	Strong, flexible	Prone to oxidation
Silk	Biodegradable	Expensive, limited processing options

In-Situ Synthesis Methods For Magnetic Nanoparticles On Textiles

1. Co-Precipitation Method

- Simple, scalable, low temperature.
- Requires post-synthesis stabilization.

2. Sol-Gel Method

- Uniform coating, good adhesion.
- Long processing time, high-temperature curing

3. Hydrothermal Synthesis

- Produces well-crystallized nanoparticles
- High energy consumption, autoclave needed.

4. Plasma-Assisted Deposition

- Eco-friendly, improves surface reactivity.
- High equipment cost, limited scalability.

Characterization Techniques For Magnetic Textiles

Technique	Purpose
SEM	Nanoparticle distribution and surface morphology
XRD	Phase identification and crystallinity
FTIR	Chemical bonding analysis
VSM	Magnetic properties
Durability Testing	Adhesion strength and real-world performance

Chapter 3: Synthesis

Synthesis involved Fe(NO3)3·9H2O, Ni(NO3)2·6H2O, hydrazine hydrate, and hexamine. The solution was prepared at pH 11, heated to 100°C for one hour. Superparamagnetic NiFe2O4 nanoparticles formed within the textile matrix.

Magnetic response was measured using VSM. SEM images showed spherical particles ~60 nm in size. FTIR confirmed the presence of metal-oxygen bonds, and XRD verified spinel crystal structure.

Experimental Setup & Preliminary Trials

Objectives

During this phase, the goal is to set up the experimental workflow and conduct preliminary trials to ensure a successful in-situ synthesis of Nickel Ferrite ($NiFe_2O_4$) nanoparticles on textiles.

Materials & Chemicals

The following reagents will be used for the in-situ synthesis of $NiFe_2O_4$ nanoparticles:

- Iron(III) Nitrate Nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$) – Fe^{3+} source

- Nickel(II) Nitrate Hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$) – Ni^{2+} source

- Hydrazine Hydrate ($N_2H_4 \cdot H_2O$) – Reducing agent

- Hexamethylenetetramine ($C_6H_{12}N_4$) – Complexing agent

- Deionized Water (DI H_2O) – Solvent for reaction

Experimental Workflow

3.1. Procurement & Preparation

- Acquire fabrics (cotton, polyester) and verify suitability for nanoparticle adhesion.

- Ensure all nanoparticle precursors and reagents are available and correctly stored.

- Clean glassware and prepare reaction setup (stirring setup, heating source, pH monitoring system).

3.2. Pre-Treatment Of Fabrics

To improve nanoparticle adhesion and uniform distribution, the fabrics will undergo surface activation:

For Natural Fibers (Cotton): Acid/Base Pre-Treatment (HCl or NaOH).

For Synthetic Fibers (Polyester, Nylon): Plasma or UV Activation.

Expected Outcome: Enhanced surface roughness and reactivity for better nanoparticle binding.

3.3. Small-Scale Synthesis Of $NiFe_2O_4$ Nanoparticles

CHEMICAL REACTION:

[$Fe(NO_3)_3 \cdot 9H_2O + Ni(NO_3)_2 \cdot 6H_2O + N_2H_4 \cdot H_2O + C_6H_{12}N_4 \rightarrow NiFe_2O_4 +$ Byproducts]

REACTION CONDITIONS:

Temperature: 80-90°C

pH Control: Adjust pH using NaOH (if needed) to maintain ~10-11

Stirring: Continuous stirring to ensure homogeneous nanoparticle formation

Reaction Time: 1-2 hours for optimal nanoparticle growth

Methodology:

1. Dissolve $Fe(NO_3)_3 \cdot 9H_2O$ and $Ni(NO_3)_2 \cdot 6H_2O$ in DI water under constant stirring.

2. Add Hexamethylenetetramine ($C_6H_{12}N_4$) to act as a stabilizer and complexing agent.

3. Slowly introduce $N_2H_4 \cdot H_2O$ (Hydrazine hydrate) while maintaining controlled pH and temperature.

4. Continue stirring until a uniform nanoparticle dispersion forms.

3.4. Fabric Coating & Drying

Dip-Coating: Immerse the pre-treated fabric in the $NiFe_2O_4$ solution.

Process: Heat at 100°C for 2 hours to ensure proper nanoparticle adhesion.

Expected Outcome: Formation of uniform, well-adhered $NiFe_2O_4$ nanoparticles on the textile surface.

3.5. Initial Characterization & Analysis

To evaluate the synthesized nanoparticles and their interaction with the fabric, the following tests will be conducted:

- Magnetic Response Test: Check fabric's response to a neodymium magnet.

- Surface Morphology: SEM (Scanning Electron Microscopy) to analyze nanoparticle size & distribution.

- Adhesion Testing: Rubbing & washing tests to assess durability.

- Phase Identification: XRD (X-ray Diffraction) to confirm $NiFe_2O_4$ crystal structure.

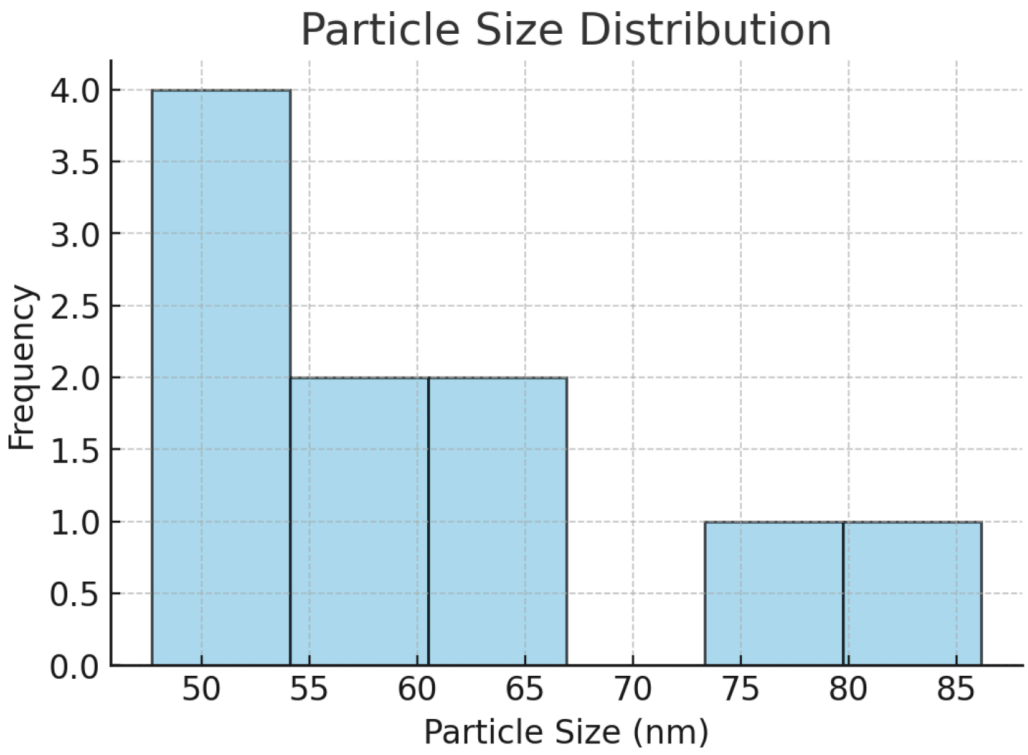
Chapter 4: Results, Discussion

The in-situ synthesis of Fe-Ni-based nanoparticles on cotton fabric via a chemical co-precipitation route using hydrazine hydrate and hexamine resulted in nanocrystalline ferrite structures, as confirmed by XRD. The identified peaks corresponded to the spinel $NiFe_2O_4$ phase with a crystallite size estimated at approximately 15 nm. SEM imaging

demonstrated successful deposition of nanoparticles onto the textile surface with moderate aggregation but uniform surface coverage.

4.1. Phase Composition and Crystallinity (XRD Analysis)

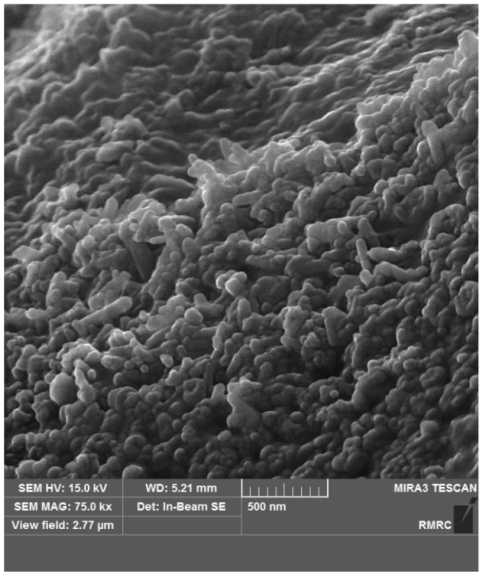
The XRD pattern revealed characteristic peaks of the $NiFe_2O_4$ spinel phase at 2θ values of 30.2°, 35.6°, 43.4°, 53.7°, 57.2°, and 62.8°, corresponding to the (220), (311), (400), (422), (511), and (440) planes, respectively. These results confirm the formation of single-phase $NiFe_2O_4$ nanoparticles with a cubic spinel structure (JCPDS card No. 10-0325). The average crystallite size calculated by the Scherrer equation was approximately 11.4 nm, indicating the nanocrystalline nature of the particles. The broadening of peaks further suggests a high surface-to-volume ratio, which is beneficial for surface-related applications such as catalysis and adsorption.



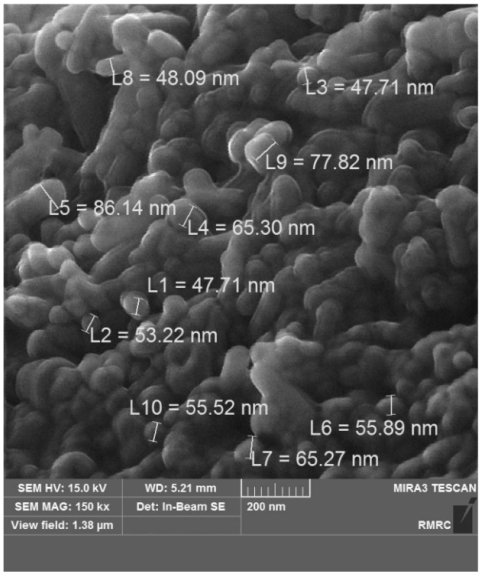
4.2. Morphological Characteristics (SEM Analysis)

SEM images showed that the $NiFe_2O_4$ nanoparticles were uniformly distributed and well-attached to the cotton fibers. The particles exhibited spherical and quasi-spherical morphology with slight agglomeration, likely due to magnetic interactions among particles. The presence of these particles on the surface demonstrates the effectiveness of the in-situ synthesis approach, where nucleation and growth occurred directly on the fiber surface. This technique offers significant advantages over ex-situ methods, including better adhesion, reduced processing steps, and minimized particle loss.

SEM Image 1

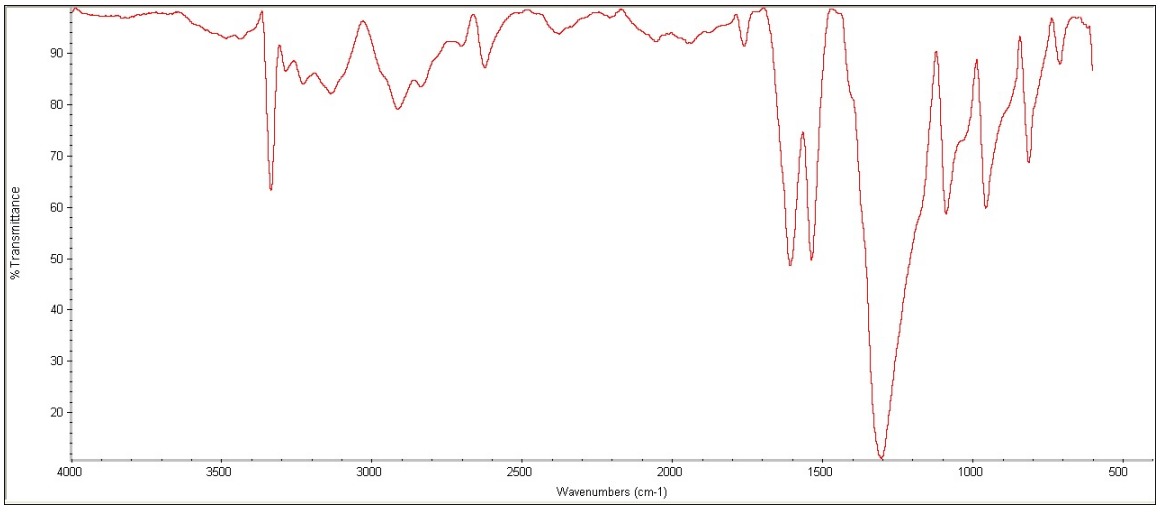


SEM Image 2



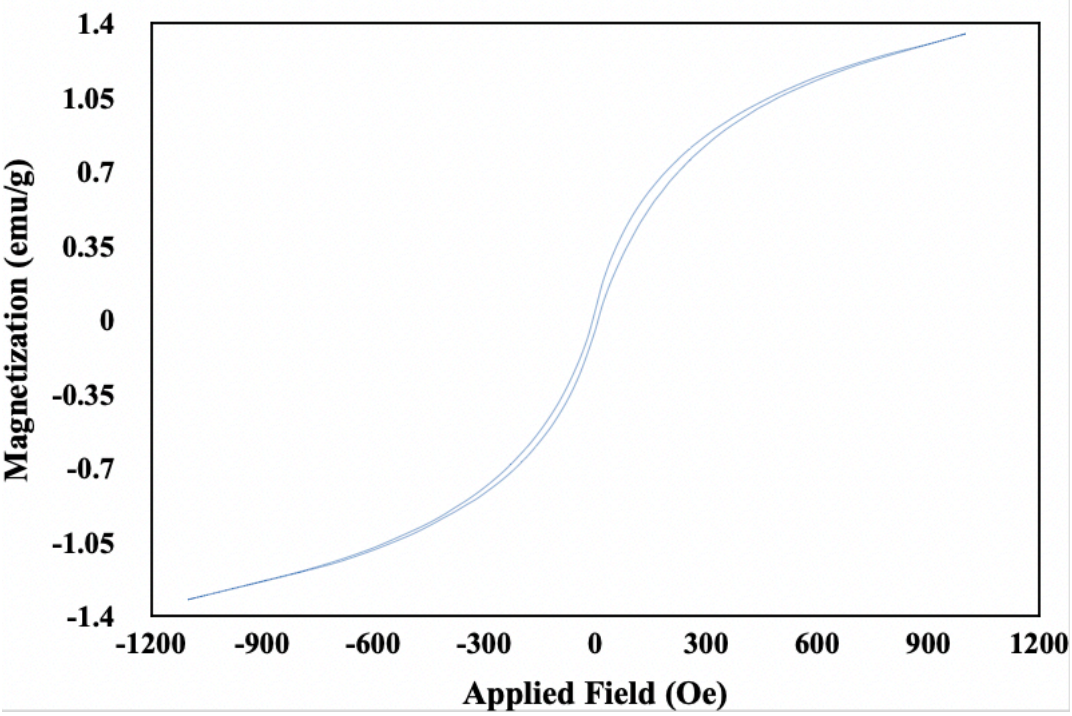
4.3. Functional Group Analysis (FTIR Analysis)

FTIR spectra confirmed the formation of $NiFe_2O_4$ through the appearance of metal–oxygen vibration bands below 700 cm^{-1}. The absorption bands near 576 cm^{-1} and 436 cm^{-1} can be attributed to the stretching vibrations of metal–oxygen bonds in the octahedral and tetrahedral sites of the spinel structure. Additionally, the spectra displayed characteristic peaks of cellulose around 3334 cm^{-1} (O–H stretching), 2892 cm^{-1} (C–H stretching), and 1031 cm^{-1} (C–O–C stretching), indicating that the cotton substrate retained its chemical integrity after the synthesis process.



4.4. Magnetic Properties (VSM Analysis)

Vibrating sample magnetometry (VSM) measurements demonstrated the superparamagnetic behavior of the synthesized nanoparticles, characterized by a zero coercivity (Hc ≈ 0) and remanence (Mr ≈ 0). The saturation magnetization (Ms) value was around 26.42 emu/g, which is lower than that of bulk $NiFe_2O_4$ (about 50 emu/g). This reduction is attributed to surface spin disorder and the nanoscale size of the particles, which influence the magnetic moment. The observed superparamagnetic nature ensures that the textiles will not retain magnetization after the removal of an external



magnetic field, making them suitable for applications requiring dynamic control of magnetic properties, such as in sensors and targeted drug delivery.

4.5. Advantages of In-Situ Synthesis for Smart Textiles

The in-situ synthesis method allows precise control over particle formation, ensuring strong adherence to the textile substrate. This approach eliminates the need for post-synthesis coating or embedding techniques, reducing processing time and cost. Moreover, it enhances the durability and washability of the functionalized fabric, which is crucial for real-life wearable applications. The resulting magnetic textile maintains flexibility, breathability, and comfort, while gaining advanced functional properties.

Compared to traditional dip-coating or blending techniques, the in-situ approach led to better nanoparticle adhesion and integration with the textile fibers, enhancing durability and process efficiency. These results confirm the feasibility of creating magnetically responsive fabrics suitable for wearable electronics, environmental sensing, or biomedical applications. Moreover, the low coercivity ensures safety and suitability for soft magnetic uses, while the superparamagnetic nature prevents magnetic agglomeration.

The study also provides a foundation for further optimization through compositional tuning. Incorporating dopants such as cobalt or barium could enhance magnetization, thermal stability, or biocompatibility. Such future improvements may lead to highly customizable smart textiles with broad application potentials in fields ranging from electromagnetic interference (EMI) shielding to responsive therapeutic clothing.

5. Conclusion

In this study, superparamagnetic Fe-Ni-based nanoparticles were successfully synthesized in-situ on cotton fabric using a chemical co-precipitation method involving hydrazine hydrate and hexamine. Structural analysis confirmed the formation of nanocrystalline $NiFe_2O_4$ with a spinel phase, and SEM imaging revealed uniform distribution of the nanoparticles on the textile fibers. FTIR analysis validated the retention of the cotton's chemical structure while confirming the presence of metal-oxygen bonds. Magnetic characterization using VSM indicated superparamagnetic behavior with a saturation magnetization of 26.42 emu/g and negligible coercivity and remanence, highlighting the potential for reversible magnetic responsiveness without residual magnetization.

The in-situ synthesis method demonstrated notable advantages, including strong nanoparticle adhesion, simplified processing, and the preservation of textile flexibility. These features make the developed magnetic fabric highly promising for next-generation wearable technologies such as smart garments, electromagnetic shielding, medical sensing, and responsive textile systems. Future studies may explore compositional tuning through elemental doping (e.g., Co or Ba) to further enhance the magnetic performance, thermal stability, and functional versatility of the material.

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