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Nanoparticle formation mechanism by using green synthesis for optical applications

A graduation project submitted to the Department of Physics in partial fulfillment of the requirements for the degree of Bachelor of Science in Applied Physics

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ACKNOWLEDGMENT

"إن أجمل إحساس يمكننا تجربته هو الغموض، فهو مصدر كل فنٍ وعلمٍ حقيقي"

ألبرت أينشتاين

الحمد لله الذي وهبني الشغف لاكتشاف أسرار هذا الكون، ومنحني القوة والإصرار لإتمام هذا المشروع الذي كان بالنسبة لي أكثر من مجرد متطلب أكاديمي.

لطالما كانت الفيزياء بالنسبة لي لغة الطبيعة، ذلك المفتاح الذي يفتح أبواب الفهم العميق لكل ما يدور حولنا، من أدق الجسيمات إلى أعظم المجرات. إن خوضي في هذا المجال جعلني أدرك أن كل ظاهرة، مهما بدت بسيطة، تحمل في طياتها معجزة علمية تنتظر من يكتشفها.

أتقدم بخالص الشكر والامتنان لوالديّ العزيزين، اللذين كانا دائمًا مصدر قوتي وإلهامي، فبدعمهما اللامحدود وثقتهما بي استطعت أن أواصل السير في هذا الطريق بشغف وحب. كما لا يسعني إلا أن أخص بالشكر دكتورتي الفاضلة (تماضر الهزاني)، التي لم تبخل عليّ بعلمها وتوجيهاتها القيّمة، فكان لها أثر عظيم في توسيع آفاقي وإثراء تجربتي في هذا المشروع.

ولا أنسى زملائي وأصدقائي، الذين كانوا جزءًا من هذه الرحلة، نشارك معًا الشغف والاكتشافات، ونتجاوز التحديات بروح الفريق والدعم المتبادل.

أخيرًا، هذا الإنجاز ليس لي وحدي، بل هو ثمرة لكل من آمن بالعلم وبأن الفضول والسعي وراء المعرفة هما أساس التقدم والإبداع.

ABSTRACT

This research addresses the development of an innovative and environmentally friendly method for the synthesis of iron oxide [Fe_3O_4] nanoparticles, using a mixture of ferric chloride (III) hydrate and hydrated ferrous ammonium sulfate in a 1:2 molar ratio, along with essential oils of peppermint and castor oil. This method is rapid and efficient, as the mixture is heated at 80°C for only 10 minutes. The nanoparticles were examined using Ultraviolet-visible spectrophotometry (UV) technique to confirm formation and stability of magnetite nanoparticles. Furthermore, a mechanism for the formation of the nanoparticles was proposed based on experimental results, providing a deeper understanding of the process. This green method has great potential in the field of applied chemistry, combining speed and simplicity, making it suitable for practical applications in fields such as medicine, electronics, and environmental purification.

الملخص

يتناول هذا البحث تطوير طريقة مبتكرة وصديقة للبيئة لتخليق جسيمات أكسيد الحديد [Fe_3O_4] النانوية، باستخدام خليط من كلوريد الحديد الثلاثي المائي وكبريتات الأمونيوم الحديدية المائية بنسبة مولارية ٢:١، بالإضافة إلى الزيوت الأساسية من النعناع والخروع. تتميز هذه الطريقة بكونها سريعة وفعّالة، حيث يتم تسخين المزيج عند درجة حرارة ٨٠ درجة مئوية لمدة ١٠ دقائق فقط. تم فحص الجسيمات النانونية باستخدام تقنية كشف وامتصاص الاشعة فوق البنفسجية، لتأكيد تكوين واستقرار الجسيمات النانونية. بالإضافة الى ذلك، تم اقتراح آلية لتكوين الجسيمات النانونية بناءً على النتائج التجريبية، مما يوفر فهماً عميقاً للعملية. تعكس هذه الطريقة الخضراء إمكانات كبيرة في مجال الكيمياء التطبيقية، حيث تجمع بين السرعة والبساطة، مما يجعلها مناسبة للتطبيقات العملية في مجالات مثل الطب والإلكترونيات وتنقية البيئة.

Abbreviation &Acronym

Ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, AR)

Sodium hydroxide (NaOH)

Ferric ammonium sulfate hexahydrate ($(NH_4)_2.Fe(SO_4).6H_2O$)

Ultraviolet-visible spectrophotometry (UV)

CHAPTER 1

Theoretical Background

1.1 Introduction

Biomedical research focuses on developing and enhancing modern technologies, materials, and methodologies to improve healthcare quality and clinical outcomes. However, this research faces multiple challenges, such as inefficient drug delivery, limited targeting of affected tissues, and adverse effects from conventional treatments. The advancement of nanomaterials presents a promising solution to these problems by enabling precise drug delivery to specific cells or tissues, enhancing efficacy while minimizing side effects [1, 2].

Nanoparticles can be designed with optimal dimensions, shapes, and surface properties, allowing for controlled drug release, targeted delivery, and improved biocompatibility. This precision in design enables effective treatments for diseases such as cancer, cardiovascular disorders, and infections, addressing limitations associated with traditional therapeutic approaches [1, 2].

Magnetic Nanoparticles Iron oxide-based magnetic nanoparticles, particularly magnetite (Fe_3O_4), have garnered significant attention due to their unique properties. Some of their key advantages include:

- **Low toxicity:** Magnetite nanoparticles are biocompatible and considered non-toxic.

- **Ease of synthesis:** They can be produced using simple and cost-effective methods.

- **Efficient separation:** Due to their magnetic properties, they can be easily manipulated and isolated using an external magnetic field.

- **Light absorption:** They possess a high ability to absorb light across the solar spectrum.

- **Reusability:** These nanoparticles can be recovered and reused multiple times without significant loss of efficiency or performance [3, 4, 5, 6].

Iron oxide nanoparticles have gained increasing significance in various industrial and biomedical applications due to their unique properties. These nanoparticles exhibit remarkable catalytic activity, high adsorption capabilities for wastewater treatment, and utility in dye technologies, magnetic recording devices, gas sensors, and biomedical applications [7].

In recent years, green synthesis methods have emerged as an eco-friendly and cost-effective approach for producing metal nanoparticles. These methods rely on natural resources such as plant extracts, microorganisms, and biomolecules to reduce metal ions and facilitate nanoparticle formation under mild conditions, eliminating the need for harsh chemicals or elevated temperatures [8].

Green synthesis employs plant extracts or microorganisms (such as bacteria and fungi) as primary materials for nanoparticle production. This process occurs under environmentally benign conditions, such as ambient temperature and atmospheric pressure, minimizing the risks associated with traditional chemical synthesis methods as shown in figure 1.1. Moreover, green synthesis is more sustainable, as it avoids toxic by-products commonly generated through conventional chemical approaches and is cost-effective due to lower energy consumption [9].

Advantages of Green Synthesis:

- **Environmental Benefits:** Avoids the use of toxic chemicals and reduces hazardous waste generation.

- **Economic Efficiency:** Lower energy and raw material consumption lead to reduced production costs.

- **Biocompatibility:** Produces non-toxic nanoparticles suitable for biomedical and pharmaceutical applications.

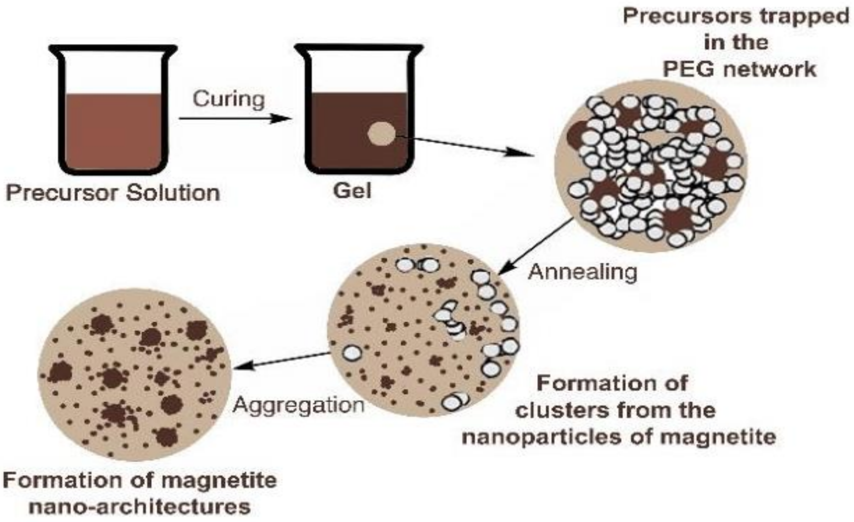

Figure 1.1: Schematic illustration showing the possible formation mechanism of the as-synthesized magnetite nano-architectures [10].

1.2 Literature review

Therefore, Biological synthesis method of nanoparticles, particularly green synthesis has been making considerable headway over the past few years. This method adheres to the principles of green chemistry, which seek the design of chemical processes and substances that minimize the amount of harmful materials used. Eco-friendly approaches exist to synthesize metal oxide nanoparticles using green synthesis [11]. For example, Yossefi et al [12] synthesized the nanoparticles (Fe_3O_4) using a simple co-precipitation method, using Garcinia mangostana fruit peel extract a natural stabilizer. The nanoparticles were primarily for magnetic hyperthermia and cytotoxic properties. The magnetic nanofluids exhibited excellent heating under magnetic field. Data in the cytotoxicity assays showed that the material was more toxic to HCT116 colon cancer cells than to CCD112 normal colon cells. The 10wt. Fe_3O_4 nanoparticles exhibited better selectivity for cancer cells, as indicated by their lower IC50 value (99.80 ug/mL) in HCT116 colon cancer cells compared to 140.80 g/mL in CCS112 colon cells.

CHAPTER 2

Experimental and Techniques

2.1 Ultraviolet-visible spectrophotometry

Ultraviolet-visible Spectroscopy is the scientific study of the interaction between light or more broadly, electromagnetic radiation and matter. This interaction reveals a fundamental phenomenon in which energy is absorbed or emitted by matter in discrete quantities known as quanta. Across the electromagnetic spectrum, both absorption and emission processes occur, each offering distinct insights into the structural and compositional characteristics of the materials studied [13].

Among the various spectroscopic methods, ultraviolet (UV) spectroscopy as shown in figure 2.1 emerges as a particularly powerful analytical tool. It utilizes radiation in the ultraviolet and visible regions, typically within the wavelength range of 200 to 800 nanometers. This technique is remarkably versatile, capable of analyzing both colorless and colored compounds. Fundamentally, UV spectroscopy measures the specific wavelengths of UV or visible light absorbed or transmitted by a sample, relative to a reference or blank. This absorption behavior is intimately related to the molecular composition of the sample, thus providing valuable information about its constituents and their concentrations [13].

Figure 2.1 Ultraviolet-visible Spectroscopy (UV).

The results of UV spectroscopic analysis are typically presented in the form of absorption of spectra graphical representations that quantitatively and visually reflect the interaction of light with matter. This enables scientists to investigate molecular structures, identify compounds, and perform precise quantitative analyses. The present study seeks to underscore the critical role of UV spectroscopy in analytical chemistry, molecular characterization, and modern scientific research at large [13].

UV Absorption and Emission Spectra:

• Absorption spectra are produced when electrons within a molecule or ion are promoted from a lower energy level to a higher one.

• Emission spectra arise when electrons return from an excited state to a lower energy level, releasing energy in the form of radiation.

Ultraviolet radiation possesses sufficient energy to excite valence electrons from their ground state orbitals to higher energy levels such as excited or anti-bonding orbitals. This electronic transition manifests as a measurable absorption event, forming the basis of UV spectroscopic detection [13].

2.2 Sample preparation method

This study describes a method for synthesizing magnetite nanoparticles using a green chemistry approach involving the use of Castro-mint oil extract as a reducing and stabilizing agent. The stepwise synthesis processes have been prepared by Dr. Tameder's group before.

Preparation of Iron salt solution: A solution containing iron chloride salt ($FeCl_3.6H_2O$) in molar ratio 1:2 was prepared by dissolving 0.50 g of $((NH_4)_2Fe(SO_4)_2\cdot 6H_2O)$and 1.11 g of ($FeCl_3.6H_2O$)in 100 mL of deionized water contained in a 250 mL beaker.

Heating and stirring: The iron salt solution was heated to 80°C under mild stirring using a magnetic stirrer and under atmospheric pressure.

Addition of Castro-mint oil:

After 30 minutes of heating, 5 mL of an aqueous solution of Castro-mint oil was added to the iron salt solution. The addition of Castro-mint oil caused the color of mixture to change from yellowish to reddish-brown, indicating the reduction of iron ions and magnetite nano particles.

Precipitation of magnetite: After 10 minutes of adding Castro-mint oil extract to the iron salt solution,20 mL of sodium hydroxide (NaOH) aqueous solution was added to the mixture at a rate of 3 mL/min to precipitate the magnetite nanoparticles uniformly. The reddish-brown mixture turned into a darker color, and suspended nanoparticles were obtained.

Cooling and purification: The mixture was allowed to cool to room temperature as shown in figure 2.2. Magnetite nanoparticles were obtained by decantation, dilution with distilled water, and centrifuged at 4000 revolutions per 5 minute to remove heavy biomaterials from the Castro-mint oil extract as shown in figure 2.3.

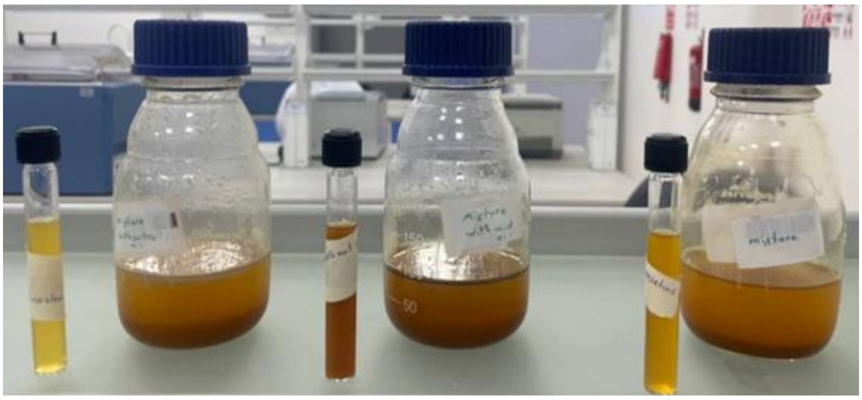

Figure 2.2 Images showing the three mixtures.

Figure 2.3 Images of the centrifuge machine and the centrifuged nanoparticles.

Division of magnetite nanoparticles: The magnetite nanoparticles were divided by centrifuged and let them dry in the oven for 14 hours at 80°C for UV-vis analysis as shown in figure 2.4.

Figure 2.4 Image of dry magnetite nanoparticles.

CHAPTER 3

Results and Discussion

3.1 Ultraviolet-visible Analysis.

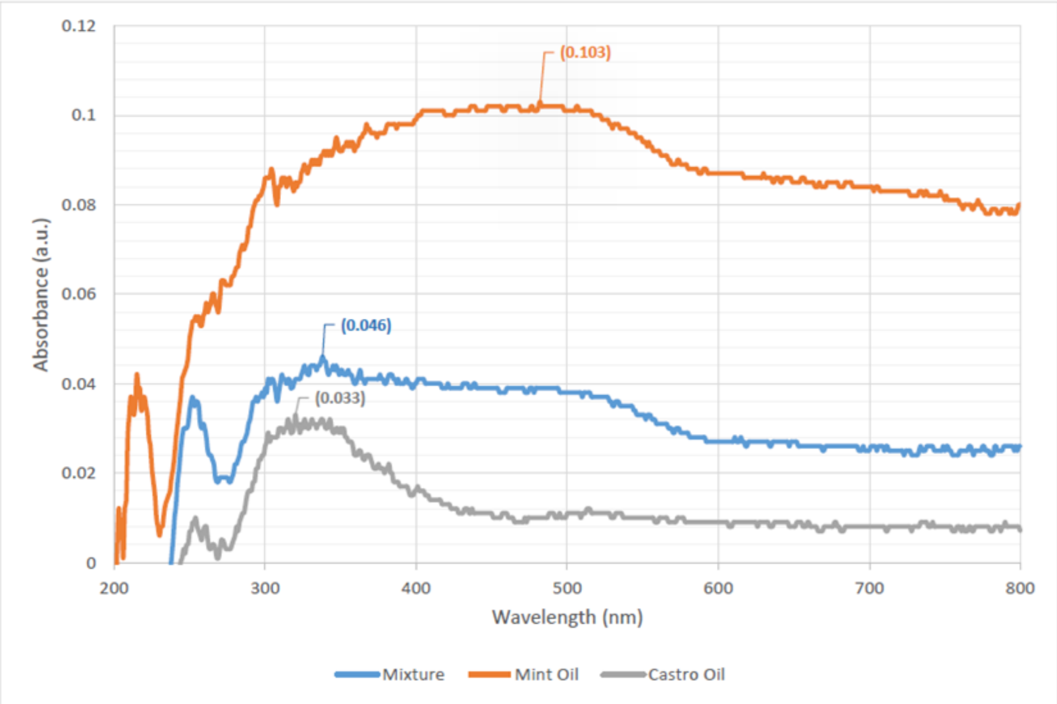

Figure 3.1 UV-vis absorption spectra of nanoparticles.

UV-Visible spectroscopy is a valuable analytical tool for investigating the optical and electronic properties of organic and hybrid materials.

In this study, the absorbance spectra of castro oil, mint oil, and the mixture without oil were recorded in the spectral range of 200–800 nm to explore their light absorbance behavior and estimate the energy corresponding to the wavelength**.**

Figure 3.1 presents the absorbance spectra with the wavelength for the three samples, the mixture (blue color), castro oil (gray color), and mint oil (orange color).

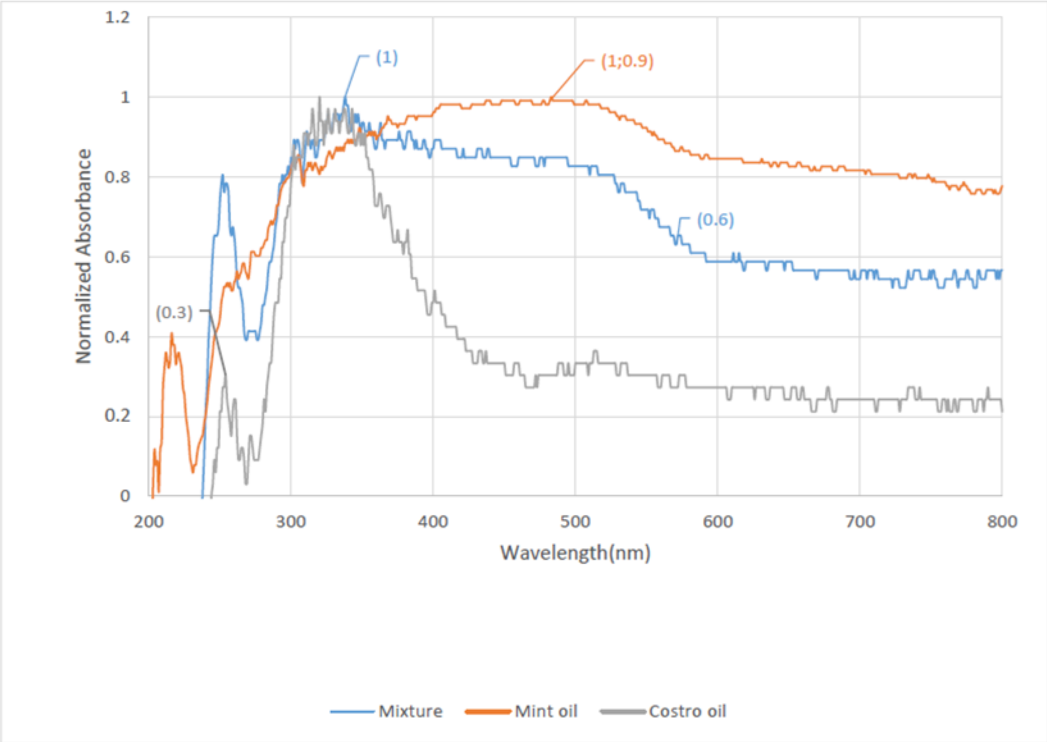

Figure 3.2 The absorbance of the three samples.

Figure 3.2 shows the absorbance normalized of the three samples. Castro oil (gray color) shows strong absorbance in UV range (200-300 nm), and after 320 nm the absorbance decreases rapidly, then almost stabilizes at around 0.3 for the rest of the visible spectrum.

Mint oil (orange color) shows steady high absorbance around 0.9–1.0 across the UV and visible regions (300–600 nm), and slight decline after 600 nm. This boarder absorbance, including some parts of visible spectrum may refer to its complex composition.

Mixture (blue color) starts similarly to mint oil but shows reduced absorbance overall. The curve between 300-700 nm has absorbance below 0.1 and then reduced to around 0.6. This shows less absorbance than mint oil, but more than castro oil in the visible region.

The wavelength of the three samples has been determined from figure 3.3 below after zoomed the range of wavelength (200-3000 nm).

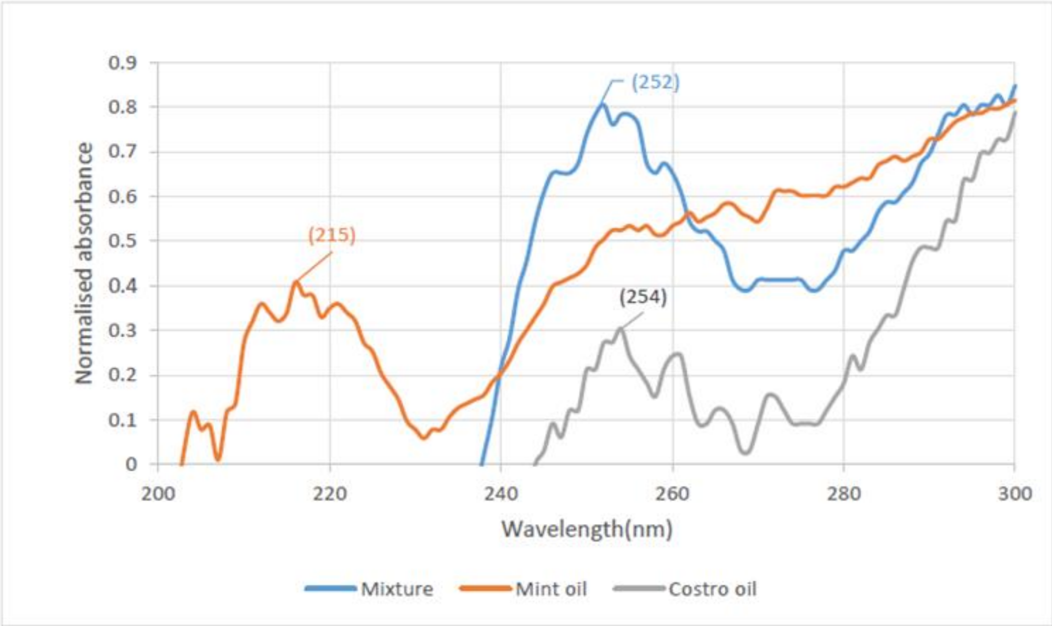

Figure 3.3 The wavelength of the three samples.

As shown in figure 3.3, the mixture (blue color) has a peak at 252 nm indicates the presence of iron oxide nanoparticles.

Castro oil (gray color) has a peak at approximately 254 nm with absorbance rises steadily from ~240 nm to 300 nm, with several small fluctuations.

Mint oil (orange color) shows a peak at 215 nm with absorbance is relatively increase and moderate between 240 nm to 300 nm. This indicates mint oil has strong UV absorbance in the lower range and a more stable absorbance at higher wavelengths.

To estimate the optical energy (E) corresponding to the wavelength (λ), the following equation has been used [14]:

$$E\ (\text{eV}) = 1240 / \lambda\ (\text{nm}) \qquad (3\text{-}1)$$

Table 3.1 provides a comparison between the three samples, showing how each wavelength corresponds to a specific energy and absorbance value.

Sample	**Absorbance** A%	**Wavelength** λ **(nm)**	**Energy** E **(eV)**
Mixture without oil	60%	252 nm	4.92 eV
Castro oil	30%	254 nm	4.88 eV
Mint oil	80%-100%	215 nm	5.77 eV

Table 3.1 A comparison between the three samples, in wavelength corresponds to the energy and absorbance value.

These results indicate that mint oil absorbs more across the visible range, possibly due to volatile compounds while castro oil is mostly transparent in the visible region. On the other hand, the mixture shows intermediate absorbance, less than mint oil and more than castro oil suggesting that blending the oils affects the molecular orbitals and alters optical behavior.

3.3 Conclusion

This study presents an innovative, sustainable, and eco-friendly approach to synthesizing magnetite (Fe_3O_4) nanoparticles using simple and natural resources. The method involves combining ammonium iron (II) sulfate hexahydrate and ferric chloride hexahydrate in a 1:2 molar ratio, along with essential oils extracted from castor and mint leaves as facilitating agents in the reaction. The mixture is heated at 80°C for only 10 minutes, making the process efficient, fast, and environmentally friendly due to its reliance on green chemistry principles. The synthesized nanoparticles were analyzed using Ultraviolet-visible spectroscopy (UV), which confirmed the formation of high-quality, ultra-small magnetite nanoparticles. The study also provides a comprehensive theoretical framework to support the experimental findings, adding further credibility to the proposed technique. This approach demonstrates significant potential across various fields. The Fe_3O_4 nanoparticles can be utilized in critical applications such as medicine for drug delivery, electronics for the development of advanced devices, and environmental remediation for improving water and soil quality. By integrating green chemistry with innovative methodologies, this research highlights the potential for creating effective and sustainable solutions with a positive impact on both the environment and society.

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