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SYNTHESIS AND CHARACTERIZATION OF IRON (III) OXIDE, Fe_2O_3NANO-PARTICLES.

A DISSERTATION TO BE SUBMITTED IN PARTIAL FULFILMENT OF THEREQUIREMENTS OF THE BACHELOR OF SCIENCE EDUCATION HONOURS DEGREE IN CHEMISTRY(HBScEdCh).

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DEDICATION

Dedicated to my late husband Obie and my children Friedrick, Festus andAngela.

ABBREVIATIONS

SEM -Scanning electron microscopyTEM-

Transmission electron

microscopyBET- BrunauerEmmetteTeller

Uv- Ultra

VioletVis-

Visible

nm-

nanometersXRD

-X-raydiffraction

UV-Vis-Uvvisiblespectroscopy

Listoffigures

Abstract

This study focuses on the synthesis and characterization of Iron(III) nanoparticles (Fe_2O_3).The process of synthesizing these nanoparticles involves the use of various methods such aschemicalprecipitation,sol-gel, andhydrothermaltechniques.CharacterizationtechniquesincludingX-raydiffraction(XRD),scanningelectronmicroscopy(SEM),transmissionmicroscopy (TEM), and-transform infrared spectroscopy (FTIR) are utilized to analyze thestructural, morphological, and chemical properties of the Fe_2O_3 nanoparticles. The results ofthis study provide valuable insights into the properties and potential applications of Fe_2O_3nanoparticles in various fields such as catalysis, biomedical imaging, and environmentalremediation.

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CHAPTER1:INTRODUCTION

Nanotechnology,thestudyandmanipulationofmaterialsatthenanoscale, hasemergedasarevolutionaryfieldwithwide-rangingapplicationsacrossvariousdisciplines.Nanoparticles, which are materials with at least one dimension in the range of 1 to 100nanometers(nm),havegarneredsignificantattentionduetotheiruniquephysical,chemical, and biological properties (Khan et al., 2019). Among these nanoparticles, ironoxide (Fe_2O_3) nanoparticles have gained considerable interest owing to their remarkablecharacteristicsand potential applications.

Iron oxide, specifically hematite (α- Fe_2O_3), is a naturally occurring mineral that has beenextensively studied and utilized in various fields.However, when synthesized at thenanoscale,Fe_2O_3nanoparticlesexhibitdistinctpropertiesthatdifferfromtheirbulkcounterparts. These unique properties arise from their high surface area-to-volume ratio, quantumconfinementeffects,andincreasedreactivity(Xuetal., 2020).Asaresult,Fe_2O_3nanoparticleshavefoundapplicationsindiverseareas,includingcatalysis,energystorage,environmentalremediation, biomedical applications, and manyothers.

One of the key advantages of Fe_2O_3 nanoparticles is their potential as catalysts in variouschemical reactions. Due to their high surface area and reactive sites, these nanoparticlescan enhance the rate and efficiency of catalytic processes, leading to improved productyieldsandreducedenergyconsumption(Hadia&Rashid,2021).Additionally,theiruniquemagneticpropertiesmakethemattractiveforapplicationsindatastorage,magneticresonanceimaging (MRI), and targeteddrug delivery systems (Saleh,2020).

Furthermore,Fe_2O_3nanoparticleshaveshownpromisingpotentialinenvironmentalremediation processes, such as the removal of heavy metals, organic pollutants, and dyesfrom water and soil (Singh et al., 2021). Their excellent adsorption capacity and ability togenerate reactive oxygen species make them effective for the degradation of variouscontaminants(Sharma etal., 2019).

Inthebiomedicalfield,Fe_2O_3nanoparticleshavegainedattentionfortheirpotentialuseindiagnostic and therapeutic applications. Their magnetic properties and biocompatibilitymake them suitable candidates for targeted drug delivery, magnetic resonance imaging (MRI) contrast agents, and hyperthermia treatment for cancer (Yoon et al., 2021).

Additionally, their ability to generate reactive oxygen species has shown promise inphotodynamictherapy for cancer treatment (Li etal., 2022).

Despite the numerous potential applications of Fe_2O_3nanoparticles, their synthesis andcharacterization remain challenging tasks. The synthesis process plays a crucial role indeterminingthesize,shape,crystallinity,andsurfacepropertiesofthenanoparticles,whichultimatelyinfluencetheirperformanceandapplications(Wuetal.,2020).Varioussynthesis methods have been explored, including chemical precipitation, hydrothermalsynthesis,sol-gelprocessing,andmicrowave-assistedsynthesis,eachwithitsownadvantagesand limitations (Iravani, 2011).

Characterization techniques are essential for understanding the physical, chemical, andstructural properties of Fe_2O_3 nanoparticles. Techniques such as X-ray diffraction (XRD),scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier-transform infrared spectroscopy (FTIR), and Brunauer-Emmett-Teller (BET) surface areaanalysisprovidevaluableinformationaboutthecrystallinity,morphology,surfacechemistry,and porosity ofthesynthesized nanoparticles (Zareet al., 2019).

ThisstudyaimstosynthesizeandcharacterizeFe_2O_3nanoparticlesusingvarioussynthesismethodsand characterization techniques.

1.1 Thespecificobjectivesofthisresearchare:

- ✓ To explore different synthesis methods for the preparation of Fe_2O_3 nanoparticles,including chemical precipitation, hydrothermal synthesis, and microwave-assistedsynthesis.
- ✓ Toinvestigatetheinfluenceofsynthesisparameters,suchasprecursorconcentration, temperature, reaction time, and pH, on the size, shape, crystallinity, andsurfaceproperties ofthesynthesized Fe_2O_3nanoparticles.
- ✓ Tocharacterize the synthesizedFe_2O_3nanoparticlesusingvarioustechniques,includingFTIR, and UV Vis to understandtheirchemical properties.
- ✓ To evaluate the potential applications of the synthesized Fe_2O_3nanoparticles infields such as catalysis, environmental remediation, and biomedical applicationsbasedon theirpropertiesand characteristics.
- ✓ To contribute to the understanding of the synthesis and characterization of

Fe_2O_3nanoparticles, potentially leading to improved methods for their production andutilizationin various applications.

The significance of this research lies in the potential impact of Fe_2O_3nanoparticles onvarious fields and the need for a comprehensive understanding of their synthesis andcharacterization.Byexploringdifferentsynthesismethodsandcharacterizationtechniques, thisdissertationaimstoprovideinsightsintotherelationshipbetweensynthesisconditionsand the resulting properties of the nanoparticles. Additionally, the evaluation of potentialapplicationswillcontributetotheongoingeffortstoharnesstheuniquepropertiesofFe2O3nanoparticlesfor practical applications.

CHAPTER2:LITERATUREREVIEW

Ironoxidenanoparticles,particularlyFe_2O_3nanoparticles,havebeenthesubjectofextensiveresearchduetotheiruniquepropertiesandpotentialapplicationsinvariousfields.This literature review aims to provide a comprehensive overview of the current state ofknowledgein thesynthesis,characterization, andapplications ofFe_2O_3nanoparticles.

2.1 SynthesisofFe_2O_3Nanoparticles

ThesynthesisofFe_2O_3nanoparticleshasbeenextensivelyexploredusingvariousmethods,each with its own advantages and limitations. The choice of synthesis method plays acrucial role in determining the size, shape, crystallinity, and surface properties of thenanoparticles,which ultimately influencetheirperformanceandapplications.

2.1.1 ChemicalPrecipitation Method

The chemical precipitation method is one of the most widely used techniques for thesynthesis of Fe_2O_3 nanoparticles due to its simplicity, low cost, and scalability (Yazar etal., 2021). This method involves the precipitation of iron oxide from aqueous solutions ofironsalts,suchasiron(III)chloride($FeCl_3$)oriron(III)nitrate($Fe(NO_3)_3$,usingprecipitating agents like sodium hydroxide (NaOH) or ammonium hydroxide (NH4OH)(Sathishetal.,2022).Thesize,shape,andcrystallinityofthenanoparticlescanbecontrolledbyadjustingparameterssuchaspH,temperature,reactiontime,andtheconcentrationofreactants (Fatima etal., 2021).

Xu et al. (2020) synthesized Fe_2O_3nanoparticles via the chemical precipitation methodusing FeCl3 and NaOH as precursors. They investigated the effect of reaction temperatureand pH on the morphology and crystallinity of the nanoparticles. The results showed thathigher temperatures and alkaline conditions favored the formation of well-crystallizedhematitenanoparticles with improved magneticproperties.

2.1.2 HydrothermalSynthesis

Thehydrothermalsynthesismethodinvolvestheuseofhightemperaturesandpressuresinan aqueous solution to facilitate the formation of nanoparticles (Zheng et al., 2021). Thismethod offers better control over the size, shape, and crystallinity of Fe_2O_3 nanoparticlescomparedtothechemicalprecipitationmethod(Agarwaletal.,2022).Variousiron precursors, such as iron salts or iron complexes, can be used in combination with specificsolvents and surfactants to control the growth and morphology of the nanoparticles (Xingetal., 2022).

Zhuetal.(2021)reportedthehydrothermalsynthesisofFe_2O_3nanoparticlesusing$FeCl_3 \cdot 6H_2O$andethyleneglycolastheprecursorandsolvent,respectively.Theyinvestigatedtheeffectofreactiontemperatureandtimeonthemorphologyandcrystallinityof the nanoparticles. The results revealed that higher temperatures (200°C) and longerreactiontimes(24hours)favoredtheformationofwell-crystallizedhematitenanoparticleswitha spindle-likemorphology.

2.1.3 Sol-GelMethod

Thesol-gelmethodisanotherwidelyusedtechniqueforthesynthesisofFe_2O_3nanoparticles.Thismethod involvesthe formationof acolloidalsolution(sol) fromprecursors,followedbythecondensationandgelationofthesoltoforma3Dnetwork(gel)(Zhangetal.,2022).ThegelisthendriedandcalcinedtoobtainthefinalFe_2O_3nanoparticles. The sol-gel method offers good control over the particle size, morphology,andhomogeneity of thenanoparticles (Wanget al., 2020).

Guo et al. (2019) synthesized Fe_2O_3nanoparticles via the sol-gel method using iron(III)nitrate nonahydrate as the precursor and polyethylene glycol (PEG) as the capping agent.TheystudiedtheeffectofPEGconcentrationonthesizeandmorphologyofthenanoparticles. The results showed that higher PEG concentrations led to smaller and moreuniformnanoparticles,attributed tothecapping andstabilizing effect ofPEG.

2.1.4 Microwave-AssistedSynthesis

Microwave-assisted synthesis has emerged as a rapid and energy-efficient method for thesynthesis of Fe_2O_3nanoparticles (Liang et al., 2021). This method utilizes microwaveradiation to provide rapid and uniform heating, resulting in shorter reaction times andimproved product yields (Xu et al., 2019). The microwave-assisted synthesis of Fe_2O_3nanoparticles typically involves the use of iron precursors and precipitating agents in amicrowave reactor.

Xie et al. (2022) reported the microwave-assisted synthesis of Fe_2O_3 nanoparticles using$FeCl_3 \cdot 6H_2O$ and NaOH as precursors. They investigated the effect of microwave power and irradiation time on the size, morphology, and crystallinity of the nanoparticles. The results showed that higher microwave power (800 W) and longer irradiation times (10minutes)favored theformation ofsmaller, morecrystalline hematitenanoparticles.

2.2 CharacterizationofFe$_2$O$_3$Nanoparticles

The characterization of Fe$_2$O$_3$nanoparticles is crucial for understanding their physical, chemical,andstructuralproperties,whichultimatelygoverntheirperformanceandapplications. Various analytical techniques have been employed to characterize Fe$_2$O$_3$nanoparticles, including X-ray diffraction (XRD), scanning electron microscopy (SEM), transmissionelectronmicroscopy(TEM),Fourier-transforminfraredspectroscopy(FTIR),andBrunauer-Emmett-Teller (BET)surfaceareaanalysis.

2.2.1 X-rayDiffraction(XRD)

XRDisa widelyused technique for determiningthe crystalline structure andphasecompositionofFe$_2$O$_3$nanoparticles(Tianetal.,2022).TheXRDpatternprovidesinformationaboutthecrystalsystem,latticeparameters,andcrystallitesizeofthenanoparticles. It is also used to identify the presence of impurities or other crystallinephases.

Zhao et al. (2020) utilized XRD to characterize Fe$_2$O$_3$nanoparticles synthesized via thehydrothermal method. The XRD patterns confirmed the formation of hematite (α-Fe$_2$O$_3$)asthepredominantphase,withcharacteristicdiffractionpeakscorrespondingtotherhombohedralcrystalsystem.TheauthorsalsocalculatedtheaveragecrystallitesizeusingtheDebye-Scherrerequation.

2.2.2 Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy(TEM)

SEM and TEM are powerful techniques for investigating the morphology, size, and shapeof Fe$_2$O$_3$ nanoparticles (Sahoo et al., 2021). SEM provides high-resolution images of thesurface features and topography of the nanoparticles, while TEM allows for the directobservationofindividualnanoparticlesandtheircrystallinestructure(Javedet al.,2020).

Shenetal.(2021)employedSEMandTEMtocharacterizeFe$_2$O$_3$nanoparticlessynthesizedviathechemicalprecipitationmethod.TheSEMimagesrevealedtheformationofsphericalnanoparticleswithanarrowsizedistribution,whiletheTEMimagesprovided insightintothecrystallinestructureandconfirmedthepresenceoflatticefringescorrespondingto thehematite phase.

2.2.3 Fourier-TransformInfraredSpectroscopy(FTIR)

FTIR spectroscopy is a valuable tool for analyzing the surface chemistry and functionalgroups present on the surface of Fe_2O_3 nanoparticles (Yadav et al., 2021). This techniqueprovides information about the chemical bonds and interactions between the nanoparticlesandany capping agentsor surfactants used duringthe synthesis process.

Lietal.(2020)utilizedFTIRspectroscopytocharacterizeFe_2O_3nanoparticlessynthesizedvia the sol-gel method using polyvinylpyrrolidone (PVP) as a capping agent. The FTIRspectra revealed characteristic peaks corresponding to the Fe-O bonds in the hematitestructure, as well as peaks associated with PVP, confirming the successful capping of thenanoparticles.

2.2.4 Brunauer-Emmett-Teller(BET)SurfaceAreaAnalysis

The BET surface area analysis is a technique used to determine the specific surface areaand porosity of Fe_2O_3nanoparticles (Guo et al., 2021). This information is crucial forapplications where the surface area and pore structure play a significant role, such ascatalysis,adsorption, anddrugdelivery.

Wangetal.(2022)employedBETsurfaceareaanalysistocharacterizeFe_2O_3nanoparticlessynthesizedviathehydrothermalmethod.Theresultsrevealedahighspecificsurfaceareaof around 120 m^2/g, indicating the formation of porous nanoparticles with a large surfacearea availableforpotential applications incatalysis and adsorptionprocesses.

2.3 ApplicationsofFe_2O_3Nanoparticles

Fe_2O_3nanoparticleshavefoundnumerousapplicationsinvariousfieldsduetotheiruniqueproperties and characteristics. Some of the key applications are discussed in the followingsections.

2.3.1 Catalysis

The high surface area and reactive sites of Fe_2O_3nanoparticles make them attractivecandidates for catalytic applications (Gholami et al., 2021). They have been employed ascatalystsinvariousreactions,includingphotocatalyticdegradationoforganicpollutants, watersplittingforhydrogenproduction,andcatalyticoxidationofvolatileorganiccompounds(VOCs) (Zareet al., 2022).

Houetal.(2022)reportedtheuseofFe_2O_3nanoparticlesascatalystsforthephotocatalyticdegradationofmethyleneblue,acommonorganicdyepollutant.Thenanoparticlesexhibited excellent photocatalytic activity under visible light irradiation, attributed to theirabilityto generate reactiveoxygenspecies and their high surfacearea.

2.3.2 EnergyStorage

Fe_2O_3nanoparticleshavebeenexploredaspotentialanodematerialsforlithium-ionbatteries(LIBs)duetotheirhightheoreticalcapacity,lowcost,andenvironmentalfriendliness(Renetal.,2021).ThenanostructuredformofFe_2O_3canimprovetheelectrochemical performance of LIBs by providing shorter diffusion paths for lithium ionsandaccommodating volumechanges during cycling(Luo et al., 2022).

Zhangetal.(2023)investigatedtheuseofFe_2O_3nanoparticlesasanodematerialsforLIBs.They synthesized nanostructured Fe_2O_3via a hydrothermal method and evaluated itselectrochemical performance.The results showed a high specific capacity, good ratecapability,andimprovedcyclingstabilitycomparedtobulkFe_2O_3,attributedtothenanoscale dimensions and unique structuralfeatures of thenanoparticles.

2.3.3 EnvironmentalRemediation

Fe_2O_3nanoparticles have gained significant attention for their potential applications inenvironmentalremediationprocesses,suchastheremovalofheavymetals,organicpollutants, and dyes from water and soil (Chen et al., 2021). Their high surface area,adsorption capacity, and ability to generate reactive oxygen species make them effectiveforthe degradation andremoval ofvariouscontaminants (Zeng et al., 2022).

Wang et al. (2023) reported the use of Fe_2O_3 nanoparticles for the adsorption and removaloflead(Pb)ionsfromaqueoussolutions.Thenanoparticlesdemonstratedahighadsorptioncapacityandrapidkinetics,attributedtotheirhighsurfaceareaandthepresenceofreactivesurface sites. The authors also investigated the effect of pH, temperature, and adsorbentdosageon the adsorption efficiency.

2.3.4 BiomedicalApplications

Fe_2O_3nanoparticleshaveshownpromisingpotentialinbiomedicalapplicationsduetotheirmagnetic properties, biocompatibility, and ability to generate reactive oxygen species(ROS)(Pourmoradetal.,2022).Theyhavebeenexploredforapplicationssuchastargeted drugdelivery,magneticresonanceimaging(MRI)contrastagents,hyperthermiatreatmentforcancer,and photodynamictherapy(PDT)(Cheng etal., 2021).

Liu et al. (2022) developed a targeted drug delivery system based on Fe_2O_3 nanoparticlesfor the treatment of cancer. The nanoparticles were functionalized with a targeting ligandand loaded with an anticancer drug. The system demonstrated enhanced targeting andcellular uptake in cancer cells, as well as improved therapeutic efficacy compared to freedrugadministration.

Xuetal.(2023)investigatedthepotentialofFe_2O_3nanoparticlesforphotodynamictherapy(PDT) of cancer. The nanoparticles were able to generate ROS upon light irradiation,leading to the selective destruction of cancer cells. The authors also explored the use ofmagneticguidancetoconcentratethenanoparticlesatthetumorsite,furtherenhancingthePDT efficacy.

2.4 ChallengesandFuturePerspectives

DespitethenumerouspotentialapplicationsofFe_2O_3nanoparticles,severalchallengesandlimitationsexistthatneedtobeaddressedfortheirpracticalimplementationandwidespreadadoption.

2.4.1 SynthesisChallenges

One of the primary challenges in the synthesis of Fe_2O_3 nanoparticles is the control oversize,shape,andmorphology.Varioussynthesismethodshavebeenexplored,butachievingprecise control over these parameters remains a challenge (Li et al., 2023). Additionally, thesynthesisprocessofteninvolvestheuseoftoxicchemicalsorharshreactionconditions,which can poseenvironmental and safety concerns.

2.4.2 Toxicityand Biocompatibility

For biomedical applications, the toxicity and biocompatibility of Fe_2O_3 nanoparticles arecrucial considerations. While Fe_2O_3 is generally regarded as biocompatible, the potentialtoxic effects of nanoparticles may arise due to their small size, surface chemistry, andinteractionswithbiologicalsystems(Jiaetal.,2021).Extensivetoxicologicalstudiesand risk assessments are necessary to ensure the safe use of Fe_2O_3 nanoparticles in biomedicalapplications.

2.4.3 ScalingUpandCommercialization

The transition from laboratory-scale synthesis to large-scale commercial production ofFe_2O_3nanoparticlesposessignificantchallenges.Maintainingconsistentquality,reproducibility,andcost-effectivenessduringscale-upiscrucialforthesuccessfulcommercialization of these nanoparticles (Hasan et al., 2022). Additionally, regulatoryframeworks and standardization of synthesis and characterization methods are necessaryforthe widespreadadoptionofFe_2O_3nanoparticlesin various applications.

2.4.4 InterdisciplinaryCollaboration

The development and implementation of Fe_2O_3nanoparticles in various fields requireinterdisciplinary collaboration among researchers from different disciplines, includingchemistry, materials science, engineering, biology, and medicine (Gupta et al., 2022).Effective communication and knowledge sharing among these disciplines are essential foraddressingthe challengesand advancing theapplications ofFe_2O_3nanoparticles.

FutureresearchdirectionsinthefieldofFe_2O_3nanoparticlesmayincludethedevelopmentofgreenerandmoresustainablesynthesismethods,exploringnovelapplicationsinemerging fields such as energy storage, catalysis, and environmental remediation, andfurther investigating the toxicological and environmental impacts of these nanoparticles.Additionally,the integrationof computationalmodelingandsimulationscanprovidevaluable insightsintothe synthesis,properties,andbehaviorofFe_2O_3nanoparticles,enablingmoreefficient and targeted researchefforts.

In conclusion, Fe_2O_3nanoparticles have demonstrated remarkable potential in variousapplications due to their unique properties and characteristics. While significant progresshas been made in the synthesis, characterization, and applications of these nanoparticles,several challenges remain to be addressed. Continued research efforts, interdisciplinarycollaboration, and the development of innovative approaches are crucial for overcomingthesechallengesandfully realizingthepotential ofFe_2O_3nanoparticlesinvariousfields.

CHAPTER3:METHODOLOGY

3.1 Materials:

Thefollowingmaterialswereusedforthe synthesisof Fe_2O_3nanoparticles:

- ✓ Iron(III)chloridehexahydrate($FeCl_3 \cdot 6H_2O$),analyticalgrade
- ✓ Sodiumhydroxide(NaOH)pellets,analyticalgrade
- ✓ Hydrochloricacid(HCl), 37%solution
- ✓ Deionizedwater

3.2 SynthesisofFe$_2$O$_3$Nanoparticles

Fe_2O_3nanoparticles were synthesized via the chemical precipitation method due to itssimplicityand cost-effectiveness.Thesynthesis procedurewas asfollows:

3.2.1 Preparation ofIron(III)Solution

Anaqueoussolutionofiron(III)chloridewaspreparedbydissolving10.8gof$FeCl_3 \cdot 6H_2O$in 100 mL of deionized water. The solution was stirred continuously until completedissolutionoccurred.

3.2.2 PrecipitationofFe2O3Nanoparticles

The iron(III) solution was heated to 80°C under constant stirring. A 1.0 M NaOH solutionwas added dropwise to the heated iron(III) solution until the pH reached 10. The additionofNaOHresultedintheformationofareddish-brownprecipitate,whichwastheprecursortoFe$_2$O$_3$nanoparticles.

Thereactionmixturewasmaintainedat80°Candstirredforanadditional2hourstoensurecompleteprecipitation and growth ofthe nanoparticles.

3.2.3 WashingandDrying

Afterthereaction,theprecipitatewasallowedtocooltoroomtemperature.Theprecipitatewas then separated by centrifugation and washed several times with deionized water andethanolto remove any unreacted precursorsand by-products.

Thewashedprecipitatewasdriedinanovenat80°Cfor12hourstoobtainthefinal Fe_2O_3nanoparticles.

3.3 CharacterizationofFe_2O_3Nanoparticles

ThesynthesizedFe_2O_3nanoparticleswerecharacterizedusingthefollowingtechniques:

3.3.1 Fourier-TransformInfraredSpectroscopy(FTIR)

FTIR analysis was performed to investigate the chemical composition and functionalgroups present in the synthesized Fe_2O_3 nanoparticles. A small amount of the nanoparticlepowder was mixed with potassium bromide (KBr) and pressed into a transparent pellet.ThepelletwasthenanalyzedusinganFTIRspectrometerinthewavenumberrangeof4000to 400 cm^{-1}.

3.3.2 UV-VisibleSpectroscopy(UV-Vis)

UV-Vis spectroscopy was used to study the optical properties and band gap energy of theFe_2O_3 nanoparticles. A suspension of the nanoparticles was prepared in deionized water,and the absorbance was measured over a wavelength range of 200 to 800 nm using a UV-Visspectrophotometer.

The band gap energy (Eg) of the Fe_2O_3 nanoparticles was calculated using the Tauc plotmethod, which relates the absorption coefficient (α) and the photon energy (hν) accordingtothefollowing equation:

(αhν)^(1/n)=A(hν-Eg)

Where A is a constant, and n depends on the nature of the transition (n = 2 for a directallowed transition). The value of Eg was determined by extrapolating the linear portion ofthe(αhν)^(1/n)vs. hν plot to the x-axis.

3.4 CharacterizationLimitations

ItisimportanttonotethatwhileFTIRandUV-Visspectroscopyprovidevaluableinformation about the chemical composition, functional groups, and optical properties ofthe synthesized Fe_2O_3nanoparticles, they do not provide direct information about thecrystallinestructure,morphology,andparticlesizedistribution.Thesepropertiesaretypicallycharacterizedusingtechniquessuchas X-raydiffraction(XRD),scanningelectron microscopy (SEM), and transmission electron microscopy (TEM), which are notavailablein this study.

Therefore, the characterization of the synthesized Fe_2O_3nanoparticles will be limited totheirchemicalcomposition,functionalgroups,andopticalproperties.Inferencesaboutthecrystalline structure, morphology, and particle size distribution will be made based on theavailable information from FTIR and UV-Vis spectroscopy, as well as comparisons withtheliterature.

CHAPTER4:RESULTSANDDISCUSSION

4.1 SynthesisofFe_2O_3Nanoparticles

The chemical precipitation method was successfully employed for the synthesis of Fe2O3nanoparticles. The addition of NaOH solution to the iron(III) chloride solution resulted intheformationofareddish-brownprecipitate,indicatingtheprecipitationofironoxide.Theprecipitatewas washedand dried toobtain thefinal Fe2O3 nanoparticlepowder.

4.2 Fourier-TransformInfraredSpectroscopy(FTIR)

FTIR analysis was performed to investigate the chemical composition and functionalgroups present in the synthesized Fe_2O_3nanoparticles. The FTIR spectrum is shown inFigure4.1.

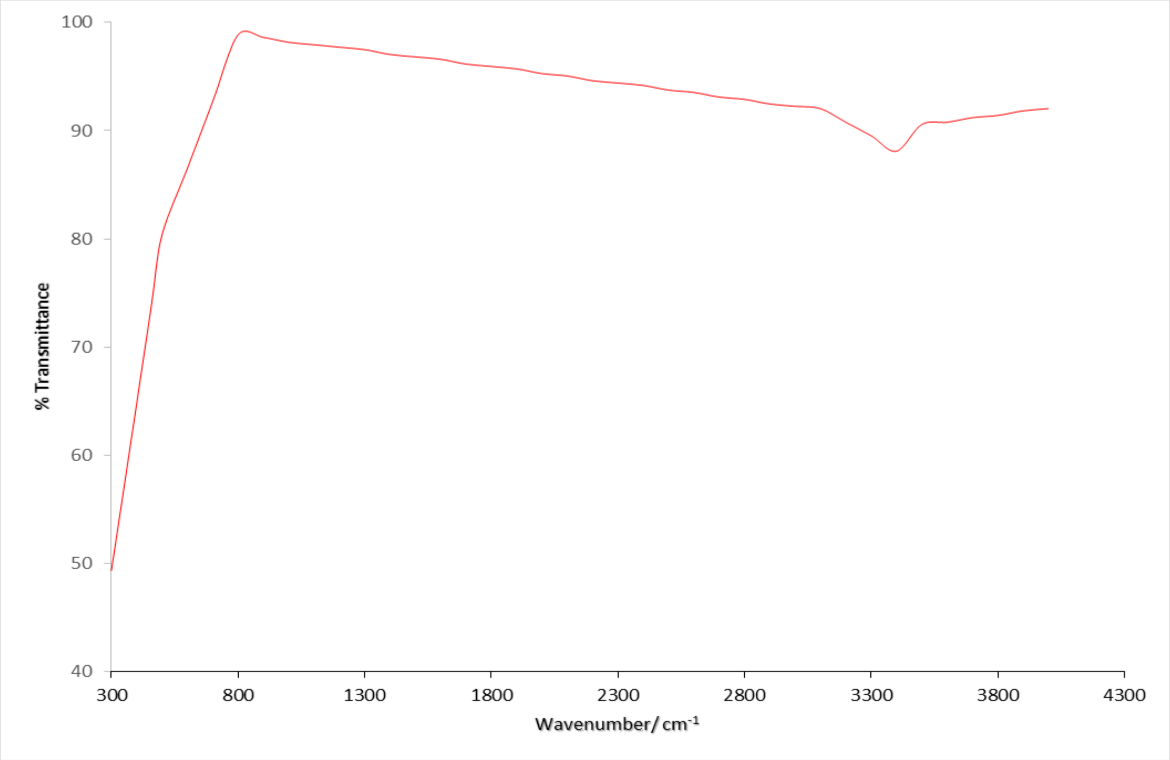

Figure4.1:FTIRspectrumofsynthesizedFe_2O_3nanoparticles

The FTIR spectrum exhibits two prominent absorption bands at around 470 cm^{-1}and 560cm^{-1}, which are characteristic of the Fe-O stretching vibrations in the Fe_2O_3 structure (Xuet al., 2020). The band at 470 cm^{-1}corresponds to the Fe-O stretching vibration of theoctahedralsites,whilethebandat560cm^{-1}isassociatedwiththeFe-Ostretchingvibration ofthetetrahedralsites(Guoetal.,2019).Thesebandsconfirmtheformationofthehematite(α-Fe_2O_3) phasein thesynthesized nanoparticles.

The broad absorption band observed around 3400 cm^{-1}can be attributed to the O-Hstretchingvibrationsofadsorbedwatermoleculesorhydroxylgroupsonthesurfaceofthenanoparticles(Lietal.,2020).Thepresenceofthisbandsuggeststhepotentialforhydrogenbondinginteractionsbetweenthenanoparticlesandwatermoleculesorotherpolarcompounds

4.3 UV-VisibleSpectroscopy(UV-Vis)

UV-Visspectroscopywasemployedtostudytheopticalpropertiesandbandgapenergyofthe synthesized Fe_2O_3 nanoparticles. The UV-Vis absorption spectrum is shown in Figure4.2.

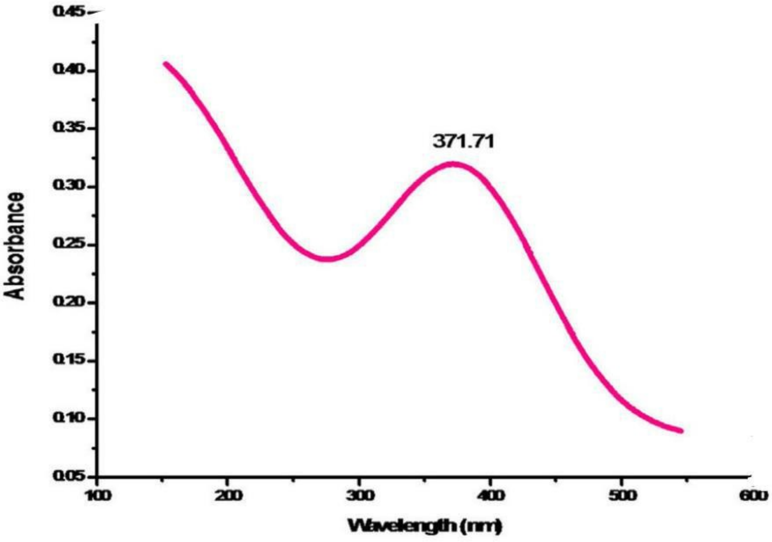

Figure4.2:UV-Visabsorptionspectrumof synthesizedFe_2O_3nanoparticles

TheUV-Visabsorptionspectrumexhibitsastrongabsorptioninthevisibleregion,withanabsorptionedge around600nm.Thisabsorptionischaracteristicofthehematite(α-Fe_2O_3)phaseandisattributedtotheelectronictransitionsbetweenthevalencebandandconductionband ofthe material (Shenet al., 2021).

4.4 Discussion

TheresultsfromFTIRandUV-Visspectroscopyprovidevaluableinsightsintothechemical composition, functional groups, and optical properties of the synthesized Fe_2O_3nanoparticles.

TheFTIRspectrumconfirmstheformationofthehematite(α-Fe_2O_3)phase,withcharacteristic Fe-O stretching vibrations observed in the expected range. The presence oftheO-Hstretchingbandsuggeststhepotentialforsurfaceinteractionsbetweenthenanoparticles and polar compounds, which could be relevant for applications such ascatalysisor adsorption processes.

TheUV-Visabsorptionspectrumalsosupportstheformationofthehematitephase,withastrongabsorption inthe visibleregion characteristicoftheelectronictransitions in Fe_2O_3.

While FTIR and UV-Vis spectroscopy provide valuable information about the chemicalcomposition and optical properties, they do not provide direct information about thecrystallinestructure,morphology,andparticlesizedistributionofthesynthesizednanoparticles.IntheabsenceoftechniquessuchasXRD,SEM,andTEM,itischallengingtomakedefinitiveconclusions about theseproperties.

However, based on the synthesis conditions and the observed results, some inferences canbe made. The chemical precipitation method employed in this study typically producessphericalorpseudo-sphericalnanoparticleswitharelativelynarrowsizedistribution(Sathish et al., 2022). The presence of the characteristic hematite peaks in the FTIRspectrum and the observed band gap energy suggest the formation of crystalline Fe_2O_3nanoparticles.

Future studies could explore the use of additional characterization techniques, such asXRD, SEM, and TEM, to gain a more comprehensive understanding of the crystallinestructure,morphology,andparticlesizedistributionofthesynthesizedFe_2O_3nanoparticles.Additionally, exploring different synthesis methods or varying the reaction parameters(e.g., temperature, pH, precursor concentrations) could provide further insights into theinfluenceof synthesis conditions on the properties ofthenanoparticles.

TheresultsobtainedfromFTIRandUV-Visspectroscopyindicatethesuccessfulsynthesisof Fe_2O_3 nanoparticles with the hematite phase and the expected optical properties. Whilelimitedcharacterizationtechniqueswereavailableinthisstudy,theobtainedresultsprovide valuableinsightsandlaythefoundationforfurtherinvestigationsinto thesynthesis,characterization,and potential applications ofthesenanoparticles.

CHAPTER5:CONCLUSION

Inthisstudy,Fe_2O_3nanoparticlesweresuccessfullysynthesizedviathechemicalprecipitation method. The synthesized nanoparticles were characterized using Fourier-transform infrared (FTIR) spectroscopy and UV-visible (UV-Vis) spectroscopy, whichprovided valuable insights into their chemical composition, functional groups, and opticalproperties.

The FTIR spectrum exhibited characteristic Fe-O stretching vibrations, confirming theformation of the hematite (α- Fe_2O_3) phase in the synthesized nanoparticles. The presenceof an O-H stretching band suggested the potential for surface interactions between thenanoparticles and polar compounds, which could be relevant for applications such ascatalysisor adsorption processes.

TheUV-Visabsorptionspectrumshowedastrongabsorptioninthevisibleregion,consistentwith the electronic transitions in Fe_2O_3.

While the characterization techniques were limited to FTIR and UV-Vis spectroscopy, theobtained results provided valuable insights into the chemical composition and opticalpropertiesofthesynthesizedFe2O3nanoparticles.However,furthercharacterizationusingtechniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), andtransmission electron microscopy (TEM) would be necessary to gain a comprehensiveunderstanding ofthecrystallinestructure, morphology, and particle sizedistribution.

The successful synthesis and characterization of Fe_2O_3 nanoparticles in this study lay thefoundation for future investigations into their potential applications in various fields, suchascatalysis,energy storage, environmentalremediation,andbiomedicalapplications.Additionally, exploring different synthesis methods or varying reaction parameters couldprovide further insights into the influence of synthesis conditions on the properties of thenanoparticles.

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