

Photocatalytic Degradation Study of Pantoprazole Sodium using NiO/CoFe₂O₄ Composite Prepared Using Chemical Co-Precipitation Technique

Submitted in partial fulfilment of the requirements for the award of
Bachelor of Technology degree in Chemical Engineering
by

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**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
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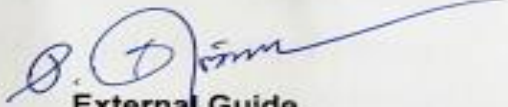
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BONAFIDE CERTIFICATE

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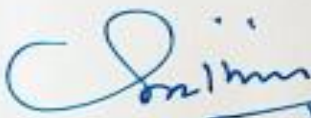
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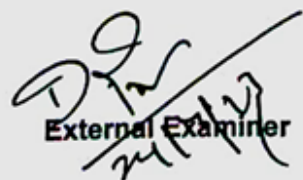
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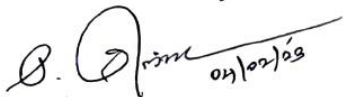
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PROJECT COMPLETION CERTIFICATE

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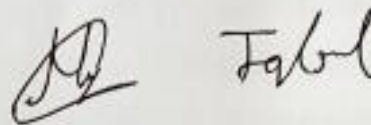


Declaration

We Manikandan N and Mohmed Iqbal A hereby declare that the Project Report entitled "Photocatalytic Degradation Study of Pantoprazole Sodium using NiO/CoFe₂O₄ Composite Prepared using Chemical Co-Precipitation Technique" done by me under the guidance of Dr. A. Annam Renita M.Tech., Ph.D., (Internal) and Dr. P. Senthil Kumar M.E., Ph.D., (External) at SSN college of Engineering, Kalavakkam, Tamil Nadu is submitted in partial fulfilment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

DATE: 18-04-2023

PLACE: CHENNAI



SIGNATURE OF THE CANDIDATES

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ABSTRACT

Organic pollutants from pharmaceutical industries and hospital effluents are a foremost wastewater issue because they have complex structures. Photocatalysis is a capable wastewater treatment technique, which is employed to degrade organic and inorganic substances under illumination of visible light in the existence of a photocatalyst. In this Project, NiO/CoFe₂O₄ binary composites were fabricated via the chemical precipitation technique in three different ratios namely NCF1, NCF2 and NCF3. The obtained products were characterized by applying various analytical routes like XRD, UV-DRS, FTIR, PL spectra and FESEM with EDX, EPR, etc. The optical energy gaps of NiO/CoFe₂O₄ nanocomposites were slightly decreased with varying NiO concentrations. The oxygen vacancies and surface defects were examined by PL spectroscopy. These emission peaks gradually increased and decreased with enhancements in NiO concentration. The obtained outcomes showed that optimal binary composite (NCF1). The experiment was carried out for the degradation of Pantoprazole Sodium which is a type of drug that is commonly used to treat gastrointestinal problems. The degradation was optimized by varying the catalyst dosage, pH and pollutant concentration. The maximum degradation of 86.24% is achieved for the optimal conditions (catalyst dosage - 500 mg, pollutant concentration – 15 ppm, pH – 12, time = 150 min). The COD removal was observed as high as 89 ± 3% within 120 min. Subsequently, the NiO/CoFe₂O₄ exhibited high photocatalytic performance outstanding stability which could be possible candidate for pharmaceutical contaminant degradation.

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LIST OF ABBREVIATIONS

AOPs	Advance Oxidation Processes
WWTP	Waste Water Treatment Plants
UV light	Ultra Violet light
PL spectra	Photo Luminescent Spectra
UV-DRS	Ultra Violet Diffuse Reflectance Spectroscopy
FTIR spectroscopy	Fourier Transform Infrared Spectroscopy
FESEM	Field Emission Scanning Electron microscopy
EDAX	Energy Dispersive X-Ray Analysis
PPM	Parts Per Million
EPR spectroscopy	Electron Paramagnetic Resonance Spectroscopy
NCF1	Nickel oxide Cobalt Ferrite (2:3.5)
NCF2	Nickel oxide Cobalt Ferrite (2.5:3.5)
NCF3	Nickel oxide Cobalt Ferrite (3:3.5)

CHAPTER – 1

INTRODUCTION

In recent periods due to various environmental issues, pharmaceutical contaminants have resulted as a possible risk to the human and animal health (Zaied et al., 2020). The major sources for these contaminants were found to be from inappropriate disposal of drugs, hospital effluents, human excrements and pharmaceutical manufacturers. Many types of these contaminants such as analgesics, antiplatelet and antibiotics were identified frequently in water bodies (Desbiolles et al., 2018). Organic contaminants like personal care products and pharmaceuticals were detected in 139 U.S. streams and their presence is confirmed in 80% of them. A few contaminants have been discovered in wastewaters in the μgL^{-1} range, but concentrations in natural rivers and streams are in the tens μgL^{-1} range, leading to the dilution effect (Desbiolles et al., 2018; Mansour et al., 2012).

Pharmaceutical chemicals are often released into the natural bodies via aquaculture effluent, sewage, solid waste leachate and industrial wastewater (Dey et al., 2019). Yet, the primary contaminants are industrial pharmaceutical effluents. Additionally, residential sewage is regarded as being among the most significant sources of pharmaceutical chemicals (Li, 2014). Certainly, wastewater flows through the sewage system to wastewater treatment facilities, and part of it reaches the environment directly by spreading pharmaceutical contaminants. Due to their molecular complexity of the pharmaceutical compounds and differences in their characteristics, such as polarity, stability, ionisation and solubility and which also depend on environmental properties, experimental challenges in comprehending the relationship of pharmaceutical contaminants with the environment may be discovered (González-González et al., 2022).

Even though they are present in trace level their synergic effect, chemical activity and microbial resistance are still not defined (Kyzas et al., 2015). The occurrence of these contaminants may result in ailments that disrupts the normal metabolic activity of humans. Persistent exposure to these drugs can cause nausea, skin rashes, hair loss, vomiting, infertility and can also be carcinogenic (Zenker et al., 2014). Most of the wastewater treatment plants (WTP) focus on removing these

contaminants at a micro level but still a large number of reactive components are dissolved in water and easily distributed to ecosystem, hence it is necessary to remove them from nanoscale (Majumder et al., 2019).

1.1 PANTOPRAZOLE SODIUM – A PERSISTENT POLLUTANT

Pantoprazole Sodium ($C_{16}H_{14}F_2N_3NaO_4S$) is a type of drug commonly used in the treatment of gastrointestinal problems. But long-term ingestion and exposure of this drug can induce hypomagnesaemia, nephritis etc. Raha et al., used g- $C_3N_4/Fe_2O_4/ZnO$ photocatalyst and reported the maximum degradation efficiency of pantoprazole as 97.09% at an optimal dosage of 0.8 gL^{-1} at a constant pH of 5 for the duration of 240 min beyond which no degradation occurred significantly (Raha & Ahmaruzzaman, 2020). It is reported that nearly 70 to 95% of pantoprazole consumed is defecated and urinated (Kosma et al., 2016).

The pantoprazole residues were found to be in concentration of about $0.18\text{ }\mu\text{g L}^{-1}$ in domestic waste waters (Della Rocca et al., 2021). Hence it is highly important to eradicate these residues from the contaminated effluent water. Yet, there has been minimal research about how pantoprazole breaks down chemically in pharmaceutical effluent. Thus, it is crucial to do study on how pantoprazole sodium degrades in aqueous solutions. Pharmaceutical effluent is readily harmful to traditional biological processes because of its high toxicity, carcinogenicity, and structural stability. Imran Ali and colleagues used a ferrous nano composite as an adsorbent to purge water of Pantoprazole contamination. They discovered that Pantoprazole may be economically and widely removed from water via adsorption. (Ali et al., 2016; Gan et al., 2017).

Polymorphism, Particle size, crystal structure and surface area are all variables that impact drug photodegradation in the solid state. Photo-unstable drugs have been stabilised utilising coated tablets, complexation with cyclodextrins, encapsulation in phospholipid bilayers and using coloured gelatine capsules. Photodegradation occurs when pharmaceutical compounds absorb light or when they are diluted by excipients; even coating can impair photostability (Dhurke et al., 2013; Glass et al., 2004). Gan et al (Gan et al., 2017) performed the degradation study of pantoprazole sodium using magnetic nanosized Fe_3O_4/CeO_2 composite heterogeneous catalyst prepared using combined oxidation and precipitation

technique and found nearly 98% of the pantoprazole was degraded within 120 minutes under the optimal conditions of H_2O_2 - 30.0 mM, pH - 3.3, pollutant concentration - 20.0 mg L^{-1} , catalyst dosage - 2.0 g L^{-1} and temperature as 35°C and proposed the degradation pathway based on $\bullet\text{OH}$ mechanism.

1.2 ADVANCE OXIDATION PROCESSES

As compared to conventional treatment techniques, tertiary processes such as advance oxidation have been shown to be effective in the elimination of organic contaminants. The conventional waste water treatment techniques widely used among the WWTPs. Even though their performance is proven to be effective in some cases, still each of the treatment methods has certain limitations. As an alternative to conventional treatment techniques advanced oxidation processes were established to be effective in organic contaminant removal. AOPs are characterized as methods that depend on the production of highly oxidizing radicals, primarily OH radicals, to convert hazardous organic pollutants into less harmful ones.

There is a lot of interest in using NPs in wastewater treatment. It offers various potential benefits, including high-efficiency treatment due to its enormous surface area and enhanced chemical characteristics. Furthermore, the low cost, great reusability, and accessibility of excellent recovery of nanoparticles after usage make them more desirable (Jayakaran et al., 2019). Advanced oxidation processes (AOPs) use cutting-edge, eco-friendly methods to clean wastewater and improve anti-pollution technology operations (Miklos et al., 2018). AOPs have an extensive range of natural and environmental applications, including soil remediation, air pollution reduction, and water and wastewater treatment (i.e., pathogens, removal of inorganic and organic pollutants).

AOPs are utilised to reduce pollution brought on by the lingering occurrence of pharmaceutical substances in water supplies during the past ten years (Klavarioti et al., 2009). A vast variety of inorganic and organic chemicals that are impervious to current treatment procedures can be quickly oxidised and degraded using AOPs, which are extremely effective new technologies. Up until the target contaminant species are entirely converted to CO_2 , H_2O , and mineral acids (particularly if the pollutant consists of halogens), AOPs produce in-situ transitory species (mostly

hydroxyl radicals) that contribute in the degrading process. In some reactions, harmless salts can mineralize, but this is entirely dependent on the contaminant. AOPs are able to oxidise a variety of contaminants in water, air, and polluted soil by using variety of combinations of sonolysis, ozone, ultraviolet (UV) light, hydrogen peroxide, and photocatalytic treatment (Joseph et al., 2009). Selected AOPs have been developed and demonstrated to have industrial uses throughout the past few decades which includes i) U-V H₂O₂ process, ii) Fenton process, iii) Photocatalysis, iv) Sonolysis.

1.3 PHOTOCATALYSIS

Among the other treatment methods, photocatalysis is proven to be more effective and efficient as the degradation mechanism uses photonic energy as the driving force (Ponraj et al., 2017). Photocatalysis is a process or activity that takes place when a light source interacts with the surface of a substance (especially, a semiconductor material). It is essential that at least two reactions occur concurrently during this activity, an oxidation reaction using photogenerated holes and a reduction reaction using photogenerated electrons. The photocatalyst species itself should not change at all during the procedure, therefore the two processes indicated above must be precisely synchronised to occur at the same time.

Photocatalysis employing semiconductors is found to be one of the growing research areas as the number of publications increased substantially from 2009-2017 (Majumdar & Pal, 2020). In the field of semiconductor photo-catalysis, doped or undoped Nano-structural materials are often utilised to develop, characterise, and execute future photo catalytical applications. Particle sizes and shapes are heavily taken into account in such procedures. Porous metallic oxides, metallic nanoparticles, porous carbons, and their synthetic mixtures are a few examples of nanoparticles that are heavily researched for their possible use in gas storage, energy conversion/storage devices, and electro- and photocatalysis.

A special class of chemical processes is represented by photocatalysis. It makes use of the light's energy to propel processes that would be challenging, if not impossible, to carry out in the dark. Photocatalysis has promise for a long-term answer to large-scale solar energy storage when utilised for thermodynamically uphill processes like photosynthesis. Existing photocatalysis demonstrations are

only performed in university laboratories, despite the long-standing interest in this process and research efforts. Lack of adequate photocatalyst materials for large-scale applications ranks as one of the main causes of the delayed progress.

Rajendra Mohan et al synthesised $\text{CoFe}_2\text{O}_4/\text{NiO}$ using co-precipitation technique and mentioned that the average crystallite size of the nanocomposite can be varied due to the changes in the weight percentage of NiO. They found as the particle size decreased the coercive field and exchange bias increased, also the effective surface area improves as the particle size decreases, increasing the effective surface anisotropy at the interface and frozen spins (Mohan et al., 2019).

CoFe_2O_4 is a highly coercive Fe-based semiconductor oxide with low toxicity, high chemical stability, moderate magnetisation, high cubic magneto crystalline anisotropy (Sonu et al., 2019). The activation of semiconductors is done by illumination of light triggering the photocatalytic reaction, The electrons from the valance band get transferred to the conduction band and holes are produced in the valence band if the energy of the photon is greater than the photocatalyst bandgap energy (E_g) (Annie Vinosha & Jerome Das, 2018). The generated charge carriers imitate reduction and oxidation reactions on the photocatalyst surface resulting in the generation of oxidizing species like superoxide and hydroxide radicals. The bandgap of the n-type semiconductor CoFe_2O_4 is 1.76 eV, the oxidation potential of CoFe_2O_4 with respect to NHE is 1.9 eV and the reduction potential is 0.14 eV (Ferdosi et al., 2019).

Nickel Oxide (NiO) is a typical p-type semiconducting material having a large E_g between conduction band and valence band making it efficient towards photocatalytic applications (Motahari et al., 2014). Various method of synthesis of the composite ternary catalysts were reported such as chemical co-precipitation method (Mohan et al., 2019), forced hydrolysis method (Artuse et al., 2008), high energy planetary ball mill technique (Kumar et al., 2018), coaxial electrospinning (Dai et al., 2020). In this research, we have successfully synthesized antiferromagnetic nickel oxide (NiO) embedded in binary ferrimagnetic cobalt ferrite (CoFe_2O_4) by co-precipitation technique employed towards the degradation of pantoprazole sodium.

CHAPTER – 2

LITERATURE SURVEY

(Naghizadeh et al., 2019) examined the possibility of ZnO/CoFe₂O₄ magnetic nanocomposite in degradation of imidacloprid entirely to in order to achieve safe and sustainable pollutant free water supply. They were trying to investigate and improve method and percentage of imidacloprid degradation parameters like pollutant concentration, photocatalyst dosage, radiation time, temperature and pH and found that nearly 92.3 % of imidacloprid is degraded within 30 min of visible irradiation. (X. Wang et al., 2022) synthesized recyclable and efficient p-n-p type heterojunction photocatalyst CoFe₂O₄/CoFe₂S₄/MgBi₂O₆ prepared using hydrothermal technique for the elimination of cefotaxime sodium under irradiation of visible light and found that 82.7% of the contaminant is completely mineralized within 60 min for the optimal catalyst dosage of 0.5 mg/L.

(F. Wang et al., 2018) a ternary novel photocatalyst with greatly increased photocatalytic response and broad-spectrum (visible, ultraviolet, and near infrared) photocatalytic activity, made of single atom-distributed Ag and carbon quantum dots co-loaded with g-C₃N₄ nanostructures, SDAg-CQDs/UCN degraded at a rate that was ten times faster than UCN. The electron separation, quantum efficiency, and up-converted PL properties of the CQDs, as well as their reduced energy gap and Ag SPR effects, were all found to be advantageous. According to the experimental results, transfer capacity was responsible for about 90% of Naproxen degradation under visible light over a duration of 25 minutes.

Ternary novel photocatalyst Ag₂Co₃/CeO₂/AgBr was successfully prepared using combined co-precipitation and hydrobromic acid corrosion method by (Wen et al., 2018), the reactive oxidizing species holes, superoxide anion, and hydroxide radical were the key active groups that influenced the photocatalytic degradation of Levofloxacin under visible light illumination, they observed more that 87.63% of the contaminant is successfully degraded into smaller components within 40 min for an optimal catalyst dosage of 25 mg/L.

(Davari et al., 2021) evaluated the optimum metronidazole degradation parameters and determined the objective to achieve high efficiency with minimal photocatalyst dosage and low energy usage. They also tested the reusability of the synthesised photocatalyst in the elimination of metronidazole. The metronidazole mineralization experiment was performed using the optimal conditions obtained in the UV degradation analysis (photocatalyst concentration: 1 g/l, illumination time: 90 minutes, pollutant concentration: 60 mg/l, pH = 10 and H₂O₂ concentration: 40 mg/l). Metronidazole degradation efficiency is found to be around 80% under these conditions.

The researcher (El-Fawal et al., 2020) prepared tertiary heterojunction AgFeO₂/G@Cu₂(BTC)₃ photocatalysts with reduced bandgaps as low as 1.95 to 1.72 eV. Under direct sunlight irradiation, AgFeO₂/G@Cu₂(BTC)₃ nanocomposite was tested for its photocatalytic performance in experimental pharmaceutical industry effluent from Egypt and to degrade its organic pollutants. In their experiment, 2.5 g of AgFeO₂/G@Cu₂(BTC)₃ were distributed in 500 mL of pharmaceutical wastewater (without previous treatment) regardless of the addition of 0.25% (Wt./V) H₂O₂ to determine how well the synthesised nanocomposite could produce photocatalysis/AOPs hybrid treatment. Finally, 92.9% of amoxicillin was mineralized within 150 min of sunlight illumination along with 87.1± 2.9 % of COD removal for a period of 7 hours.

A persistent pharmaceutical pollutant sulfamethoxazole degradation using a novel binary n-n type photocatalyst WC/BiOCl was studied by (Cao et al., 2021). Several experimental and computational approaches were used to characterise the photoelectric and physical attributes of WC/BiOCl. The decrease of BiOCl clustering and, more importantly, the development of an intermediate electrical field between WC and BiOCl are made possible by the development of a tight interfacial connection between WC and BiOCl. It is intriguing to learn that the WC layers can serve as electron transfer and storage sites as well as help reduce interfacial charge transfer resistance. After then, the holes assist in breakdown of sulfamethoxazole with the help of WC/BiOCl. Moreover, the WC/BiOCl photocatalyst has much more reactive sites than pristine BiOCl and absorbs more incoming photons. The study

revealed that the optimum dosage of catalyst is 0.4 g/L resulted in more than 95 % of elimination of the sulfamethoxazole under solar light irradiation within 20 min.

(Tang et al., 2020) performed the photodegradation on neonicotinoid under visible light source using the novel g-C₃N₄/WO₃/AgI with dual Z-scheme prepared using electrostatic selective deposition method. They reported that in a typical heterojunction, photogenerated electrons move in opposite directions from the conduction band of g-C₃N₄ and AgI to the conduction band of WO₃, while photogenerated h⁺ transfers in opposite directions from the valence band of WO₃ to the valence band of g-C₃N₄ and AgI. Since conduction band of WO₃ edge location in this carrier transport mode is lower than the typical reduction potential of O₂/H₂O₂, it cannot be converted from O₂ to H₂O₂ using conduction potential energy of WO₃. Therefore, the higher degradation efficiency of about 95.2 % degradation was observed within 5 min of visible light irradiation.

(Asadzadeh Patehkhori et al., 2021) synthesized ternary chitosan–TiO₂–ZnO over graphene using sol-gel assisted method for degradation of tetracycline and found more that 97% of the pollutant is degraded within 3h of UV irradiation, the possible mechanism proposed was, when photocatalytic processes employ the irradiation of light with a suitable wavelength and adsorption of photon energy appropriate to the bandgap of TiO₂, the electrons in the TiO₂ valence band are stimulated. Since TiO₂ (3.20 eV) has a narrower bandgap than ZnO (3.37 eV), electrons are driven to the CB of ZnO, parting h⁺ with positive charge in the TiO₂ VB. The solid supports CS and Gr provides a large response to light region by offering a broad surface and, as a result, greater distribution of consumed metal oxides.

CHAPTER – 3

AIM AND SCOPE OF THE PROJECT

3.1 AIM:

- To synthesize the Nickel oxide-cobalt ferrite composite ($\text{NiO/CoFe}_2\text{O}_4$) photocatalyst using Chemical Co-precipitation method and Characterization.
- The selection of the efficient composite among the other variants (NCF1, NCF2, NCF3) and optimization
- Utilization of polycrystalline catalysts to enhance the optical absorption and reducing the recombination rate of electron-hole charge carriers.
- Optimization of degradation parameters (pH, Catalyst dosage, Pollutant concentration) to achieve higher removal rates.

3.2 SCOPE:

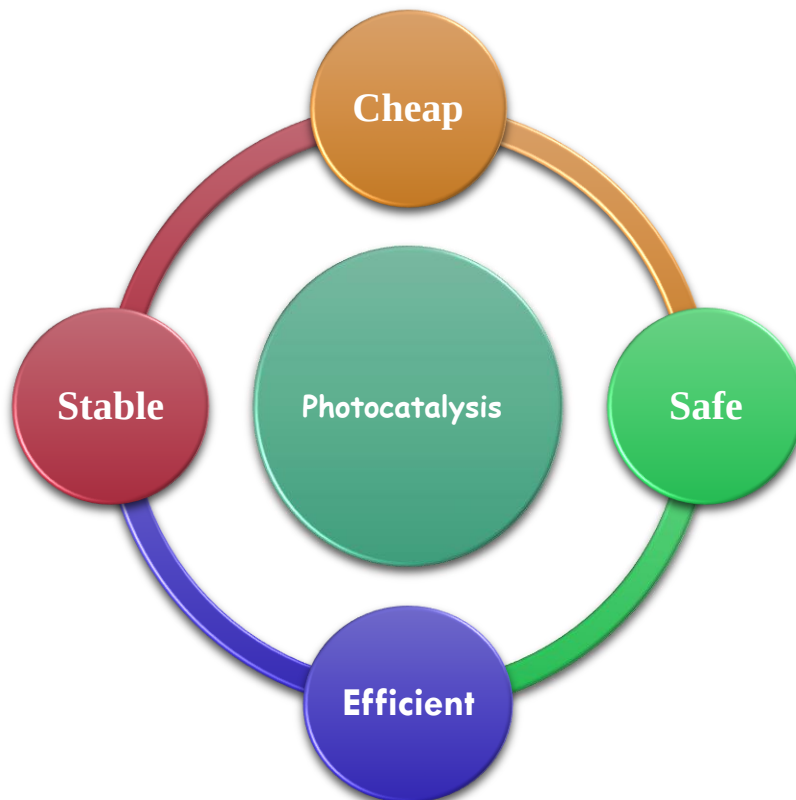


Fig 3.1: Key advantages of photocatalysis

The engineering application of adjusted binary composite photocatalysts is expected in the future owing to its greater photodegradation behaviour, photostability, and eco-friendly nature. However, further alterations are crucial to increase its photodegradation performance under natural solar illumination by reducing its energy gap as well as diminishing the charge recombination losses.

Several reports have established that decreasing the probability of e^-h^+ recombination percentage is one of the utmost significant parameters which influence the photodegradation behaviour utilizing the binary composite materials. Several chemical additives and modification methods have been formulated to lower the e^-h^+ recombination process and prolonged the absorbance response of binary composite materials to the visible region by employing metal ions modification, RE-doping, co-doping, non-metal doping, and binary materials with composites.

Numerous scientists have been endeavouring to prepare a greater surface area of photocatalyst and a sunlight/visible-driven photocatalytic reaction for effectual degradation performance. By this way, adding hole scavengers (electron mediators) or e^- donors with photocatalyst can more expand the photodegradation behaviour by interacting with h^+ (VB) irreversibly to forbid the recombination rate of charge carriers and also inhibit the backward process (i.e., nonmetal additives).

Incessant endeavours have been carried out to attain the solar/visible light-driven Nickel oxide catalyst by incorporating e^- donors together with Nickel oxide catalyst is essential (co-metal doping, RE-metal doping route). Also, lesser energy gap semiconductor materials can inject e^- to the conduction band of large E_g materials, causing in effectual e^-h^+ separation and greater removal performance (spinel materials). Therefore, the band gap and charge carrier carry-over should be regarded carefully when planning such photocatalytic elimination systems for pharmaceutical pollutants, phenolic compounds, organic dyes, and some other contaminants.

CHAPTER – 4

MATERIALS AND METHODS

4.1 CHEMICALS AND REAGENTS:

Ferric (III) nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) (98% pure), 0.8 M cobalt chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$), sodium hydroxide (NaOH), pantoprazole sodium ($\text{C}_{16}\text{H}_{14}\text{F}_2\text{N}_3\text{NaO}_4\text{S}$), ethanol ($\text{C}_2\text{H}_6\text{O}$), deionized water, Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$), Hydrogen peroxide (H_2O_2), Concentrated sulphuric acid (H_2SO_4).

4.2 SYNTHESIS OF COBALT FERRITE (CoFe_2O_4):

Nanostructured CoFe_2O_4 were synthesized using co-precipitation technique. A stoichiometric quantity of 0.9 M ferric (III) nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$) (98% pure) and 0.8 M cobalt chloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) were used as precursors. The ferric (III) nitrate nonahydrate was dissolved in 100 mL of deionized water to get a clear solution and the $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ was dissolved in another beaker with 100 mL of deionized water. The cobalt chloride hexahydrate solution was added to the ferric (III) nitrate nonahydrate solution drop by drop under vigorous stirring. 21 g of NaOH pellets were added to 100 mL of deionized water and mixed thoroughly, the NaOH solution is then slowly added to the binary mixture as the precipitation occurred immediately changing the colour of the solution to dark brown with initial pH of 12.2. The complete reaction was carried out for a period of 4 h. The resultant precipitate is calcined at a temperature of 700°C for 5 hours after being repeatedly rinsed with deionized water and ethanol. It is established that the resultant CoFe_2O_4 is strongly magnetic in nature.

4.3 SYNTHESIS OF NICKEL OXIDE (NiO):

1.8 M nickel (II) chloride hexahydrate ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) and 4.5 M sodium hydroxide (NaOH) were used as starting materials. $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ is dissolved in 100 mL of deionized water in a stoichiometric ratio. After being dissolved in 100 mL of deionized water, the NaOH pellets were added to the solution dropwise and continuously stirred. The original pH of the mixture was estimated to be 12. The

reaction was carried out for a period of 4 h. The precipitate is repeatedly rinsed with deionized water and ethanol before being calcined for four hours at 500°C.

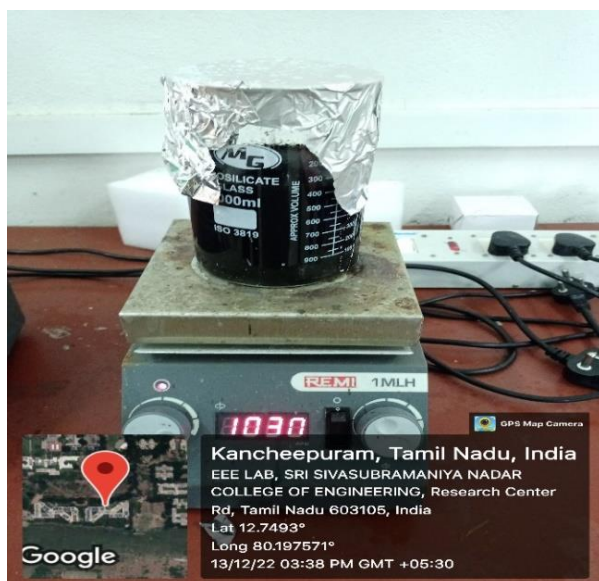


Fig 4.1: Synthesis of CoFe_2O_4 by chemical co-precipitation technique



Fig 4.2: Synthesis of NiO using chemical co-precipitation method

4.4 SYNTHESIS OF BINARY COMPOSITE:

The synthesis process is done in three different ratios of $\text{NiO}:\text{CoFe}_2\text{O}_4$ (2:3.5, 2.5:3.5 and 3:3.5). Initially in a 500 mL beaker, 2 g of as prepared nickel oxide catalyst is mixed with 3.5 g of as prepared cobalt ferrite composite, the mixture is dispersed into along with the equimolar amount of ethanol and water followed by continuous stirring. Similarly, in a combination of 2.5 g of as-prepared nickel oxide catalyst and 3.5 g of as-prepared cobalt ferrite composite, equimolar amounts of ethanol and water are added, and the mixture is continuously stirred. Finally, 3 g of as-prepared nickel oxide catalyst and 3.5 g of cobalt ferrite composite is separately dispersed in the equimolar amounts of ethanol and water in a 500 mL beaker followed by continuous stirring. The samples are then dried for 24 hours followed by grinding into fine particles.

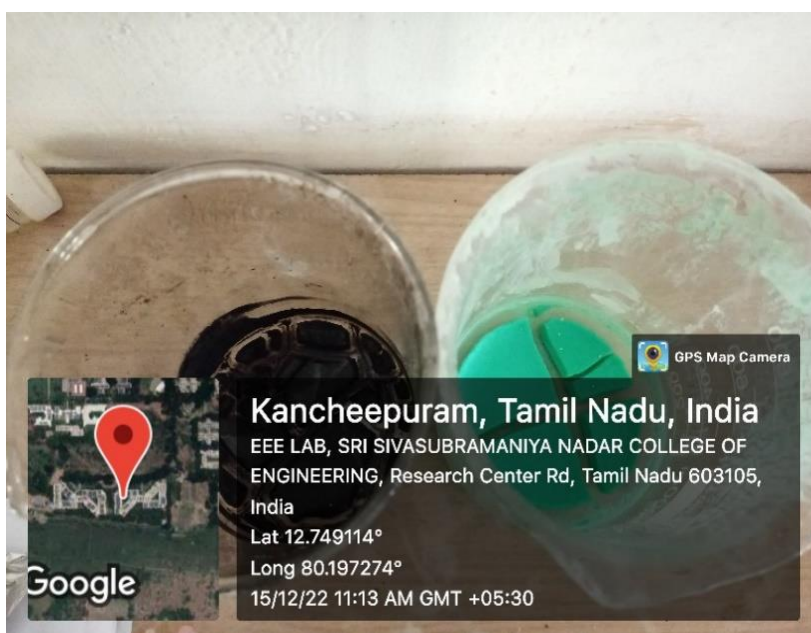


Fig 4.3: NiO and CoFe_2O_4 samples after drying under 105°C



Fig 4.4: Synthesis of binary composites by co-precipitation method



Fig 4.5: Calcination of the binary composites and pure samples

4.5 PHOTOCATALYTIC EXPERIMENTAL SETUP:

The degradation of the prototype persistent organic water pollutant pantoprazole sodium utilising the photocatalyst under visible irradiation was performed to evaluate the photocatalytic activity of the developed binary composite photocatalyst. The reaction was carried out for 150 min initially at room temperature

without any external heat source. For the photocatalytic reaction, the reaction mixtures were made separately with varying parameters containing a given amount of photocatalysts ranging from 300 mg, 500 mg and 700 mg distributed in a 150 mL aqueous solution containing a certain predetermined pantoprazole sodium concentration in range of 15 ppm, 30 ppm and 50 ppm concentration at a fixed pH of 4,6 and 12 for separate reaction conditions was kept under the visible light photocatalytic reactor under constant mechanical stirring. The samples were collected every 15 minutes from the initial concentration until the reaction was completed, then the obtained samples were centrifuged and collected for further analysis. The Pantoprazole absorption was analysed at 212 nm using the UV-vis spectroscopy (JASCO V-630-C220761148).

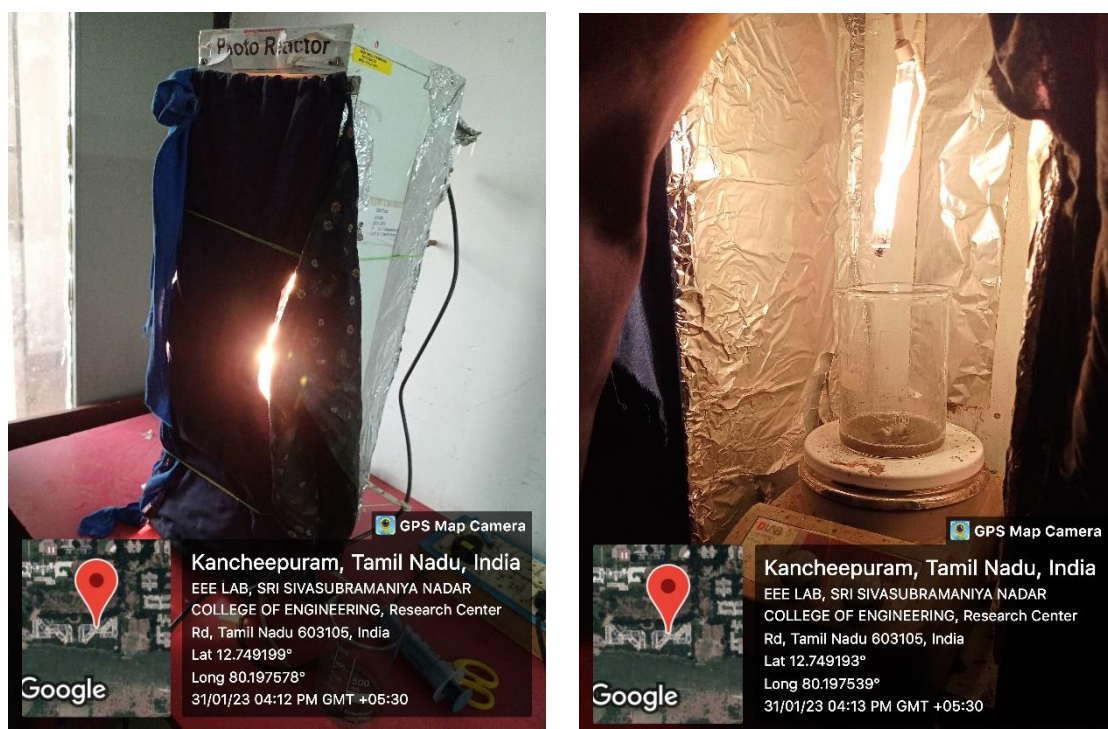


Fig 4.6: Photocatalytic reactor setup for degradation of pantoprazole sodium

CHAPTER – 5

RESULTS AND DISCUSSION

5.1 X- RAY DIFFRACTION (XRD):

X-ray diffractograms of all the samples shown in figure (5.1) was performed for the NiO/CoFe₂O₄ composite prepared using chemical co-precipitation method. It is a non-destructive method that gives thorough details on a crystallographic structure of the material, chemical structure, and physical characteristics. The characteristic peaks for both cobalt ferrite and nickel oxide are detected without any detection of extra peaks. The presence of the active components and their synergic effect is analysed from the peak crystallography. It is observed that the peak becomes sharper and crystallises well as NiO concentration rises. All of the peaks coincided with the ferrite and oxide components, demonstrating the samples phase formation and purity.

The peaks were indexed using the JCPDS Card Nos. 00-022-1086 and 03-065-2901 and matched with the Fd-3m (227) space group, containing the (220), (311), (422), (511), and (440) crystal plans of the cubic spinel cobalt ferrite phase and the (111), (200), and (220) crystal plans of the cubic spinel nickel oxide phase, respectively. The coherent volume in the material surrounding the particular peak of diffraction correlates to the crystallite size. It may also refer to the thickness of polycrystalline thin films and bulk material, as well as the size of the grains in a sample of powder. Scherrer's formula was used to determine the average crystallite size (Gao et al., 2010; Joshi et al., 2014; Joshi & Kumar, 2016).

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

In this equation, D stands for the size of the crystallites in nanometers, K for the Scherrer's constant, which is equal to 0.9, for the wavelength of the x-ray sources, which is equal to 0.15406 nm, for the full width at half maximum (FWHM) in radians, and for the peak position in radians.

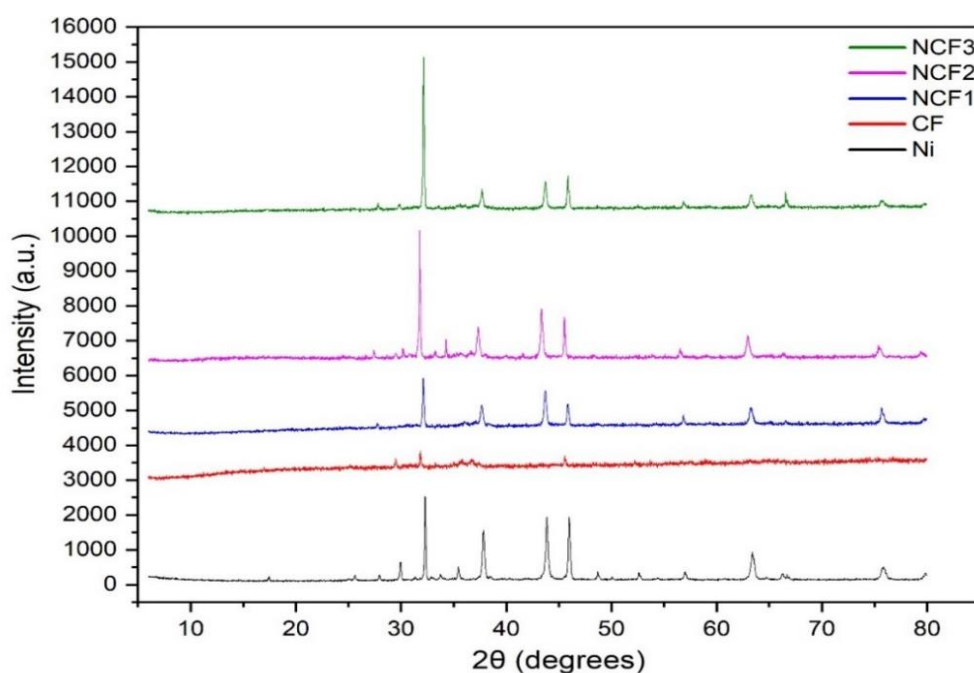


Fig 5.1: X-ray Diffraction crystallography of pure samples and composites.

Table 5.1: Grain size and crystallinity of the sample along with their band gap

S.No	Samples	Average Crystallite Grain size, D (nm)	Crystallinity %	Bandgap E_g (eV)
1	NiO (pure)	34.90173792	71.390873	3.56
2	CoFe ₂ O ₄ (pure)	1.594118567	13.2524989	2.25
3	NCF1	21.19881574	28.02046	2.45
4	NCF2	35.2428491	24.38428	2.59
5	NCF3	33.88506128	27.57033551	2.63

5.2 UV-DRS SPECTROSCOPY:

The presence of impurities was determined using the UV-DRS spectra using the instrument UV-3600Plus (A12015600584). The optical absorption spectral values mentioned in the figure (5.2) was taken between the range of 200 – 800 nm wavelength in normal mode. Because of the theoretical and practical importance of

evaluating quantities like the electrical energy band gap and magneto-optical parameters, the main focus is in the optical characteristics of ferrite nanoparticles. Data on optical absorption and percentage transmission may be used to calculate the energy and type of the transition for the optical band gap of nanoparticles (Joshi & Kumar, 2016). At 400 nm, the NCF1 exhibits a distinct and sharply visible absorption peak. In order to determine the band gap energy (E_g) of the binary composite catalyst, the wavelength value that corresponds to the location where the vertical and horizontal spectra overlap was employed.

$$E_g = \frac{hc}{\lambda} = \frac{1240}{\lambda} \text{ eV}$$

The aforementioned equation may be used to approximate the band gap energy, which corresponds to the absorption limit. Where C is the light velocity (3×10^8 m/s), E_g is the band gap energy (eV), h is the Planck's constant (6.626×10^{-34} J s), and λ is the wavelength (nm). Furtherly, Tauc's Plot is used to calculate the direct and indirect bandgap. As a response, we investigated into the optical characteristics of NiO/CoFe₂O₄ nanoparticles using UV-visible diffuse reflectance spectroscopy. The following is an expression for the optical band gap (E_g) of the material:

$$\alpha E_p = A(E_p - E_g)^q$$

, where A is a constant, E_p is the photon energy, and q can take one of two values 2 or 1/2 and is transition-dependent. Indirect electronic transition is represented by the value $q = 2$, whereas direct electronic transition is represented by the number $q = 1/2$. With the use of $(\alpha h\nu)^2$ versus photon energy ($E = h\nu$) and $(\alpha h\nu)^{1/2}$ vs photon energy plots, the direct and indirect band gaps of ferrite nanoparticles were determined, respectively. The E_g pristine NiO, CoFe₂O₄ measured in range of 3.56 eV and 2.25 eV whereas the binary composite catalysts NCF1, NCF2, NCF3 were calculated in range of 2.45 eV, 2.59 eV and 2.63 eV. NCF1 corresponds to be the optimal sample with the least bandgap when compared to other ratios promoting the higher degradation efficiency of the composite. The band gap widens with increasing Ni concentration because there is a considerable electrostatic coupling and the ionic radii of the Ni²⁺ ions are smaller than those of the Co²⁺ ions. The deeper Ni²⁺ states are created than Co²⁺ states due to the higher electrostatic contact, which

increases the energy band gap between the unoccupied and occupied states in NiO/CoFe₂O₄ composites.

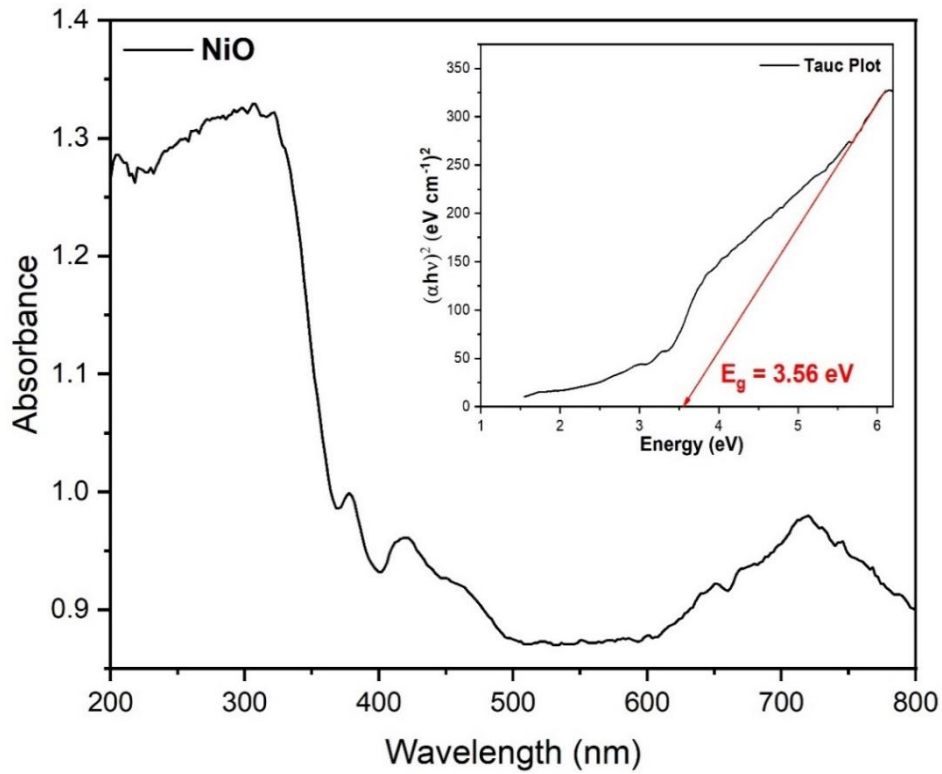


Fig 5.2: UV Diffuse reflectance spectroscopy of pristine NiO

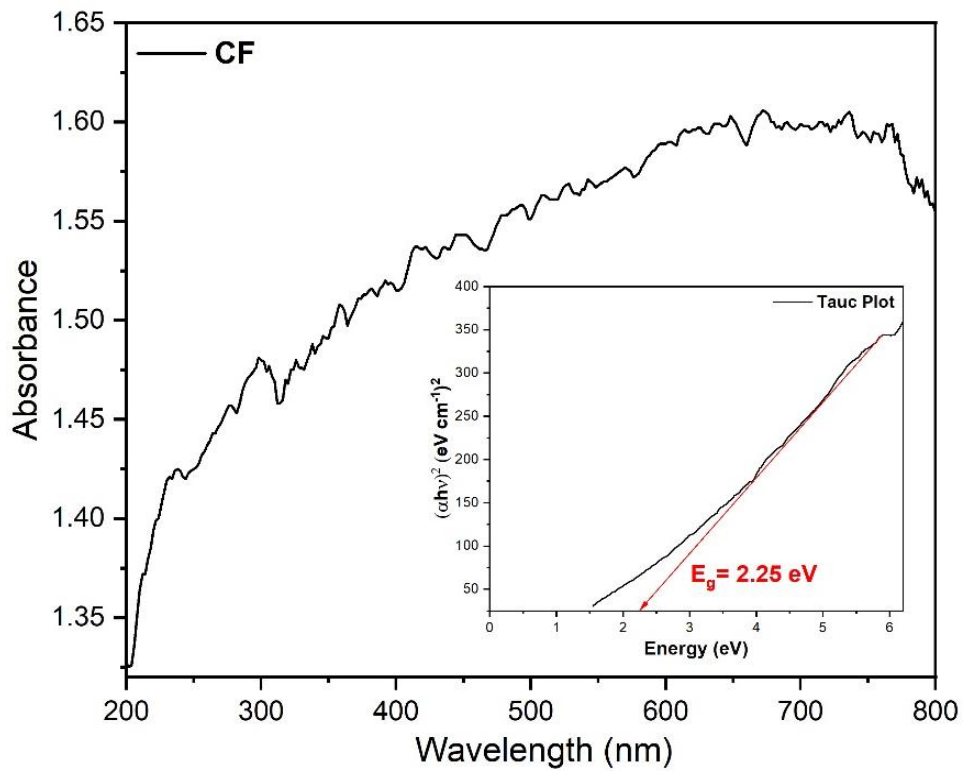


Fig 5.3: UV Diffuse reflectance spectroscopy of pristine CoFe₂O₄

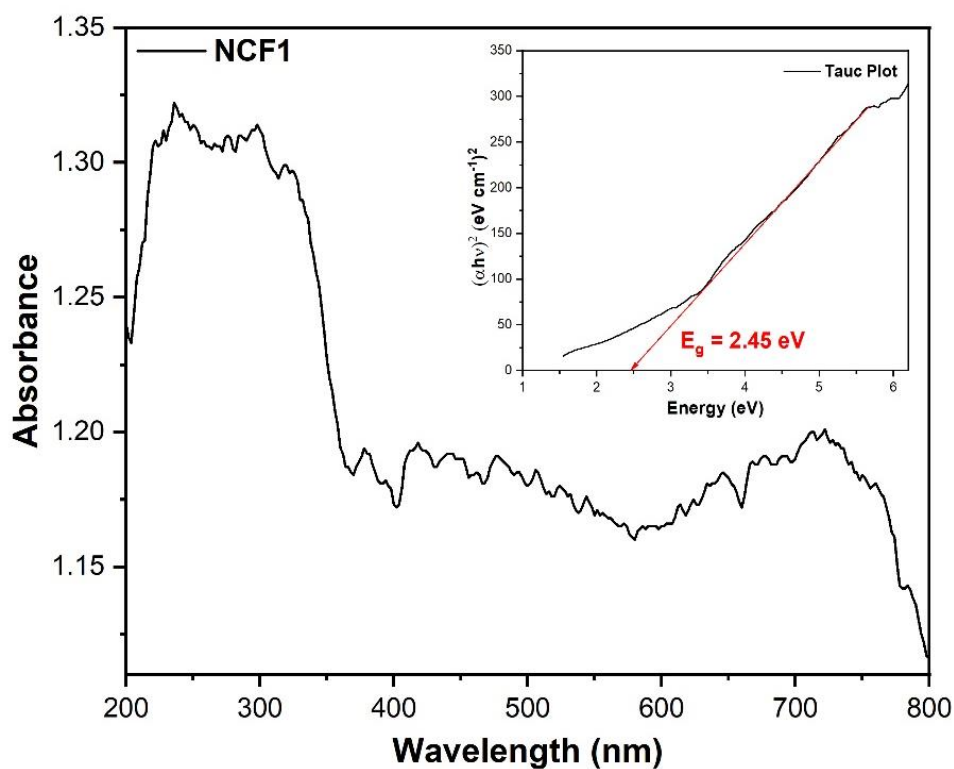


Fig 5.4: UV Diffuse reflectance spectroscopy of NCF1 composite

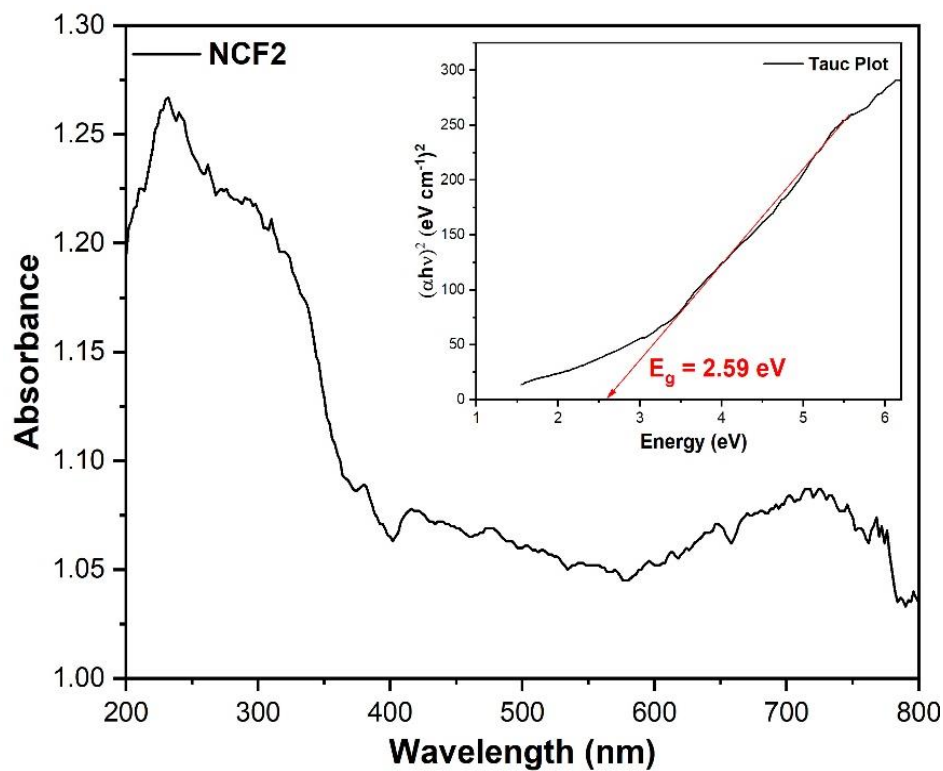


Fig 5.5: UV Diffuse reflectance spectroscopy of NCF2 composite

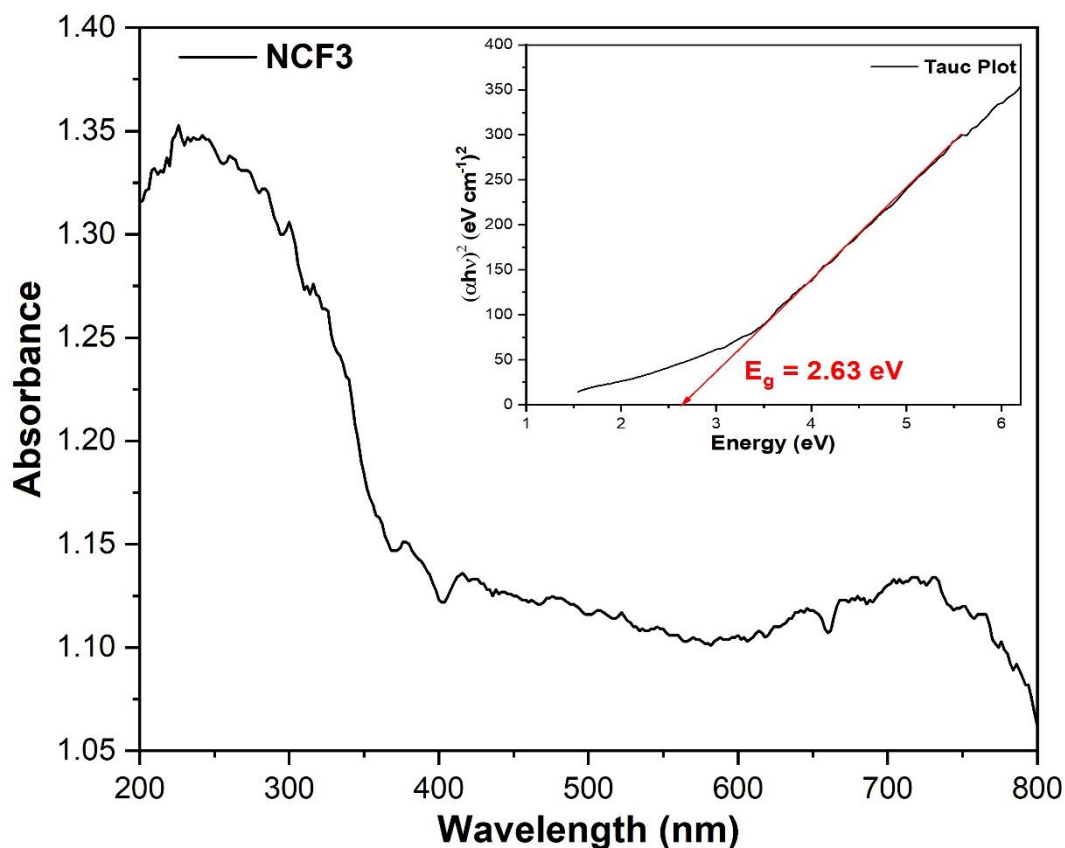


Fig 5.6: UV Diffuse reflectance spectroscopy of NCF3 composite

5.3 PL SPECTROSCOPY:

Photoluminescent spectroscopy was performed using the instrument model (LS45) to analyse the charge transfer activity of the composite photocatalyst and its capacity to separate the photogenerated charge carriers. Figure (5.7) depicts a sharp peak at 454.5 nm associated to the thorough PL emission of NCF3 because of the enhanced recombination rate of the charge carriers. In comparison with NCF3 fluorescence intensity of NCF1 and NCF2 is found to be reduced, which implies the enhanced efficiency in electron-hole pair separation of these photocatalysts. Among the other ratios of NiO/CoFe₂O₄, NCF1 has the lowest rate of recombination of photogenerated electron hole pairs, and as a result, has the lowest PL intensity. The longest carrier length and the highest effective electron-hole pair separation are also displayed. The transitions of Ni²⁺/Co²⁺ at the tetrahedral sites are responsible for the bands at 680 and 684 nanometres. The band gap is accounted for by the weak band in the 700-750 nm region, which corresponds to the band edge to edge transition.

The intensity of the emission peaks fluctuates with the concentration of Ni^{2+} when Co^{2+} ions are replaced by Ni^{2+} ions at octahedral sites (Joshi et al., 2014). In contrast, a lower PL peak intensity results from the increased addition of NiO because fewer light-emitting electrons are trapped there. These bands could potentially be caused by defects in interface traps at grain boundaries (Joshi & Kumar, 2016).

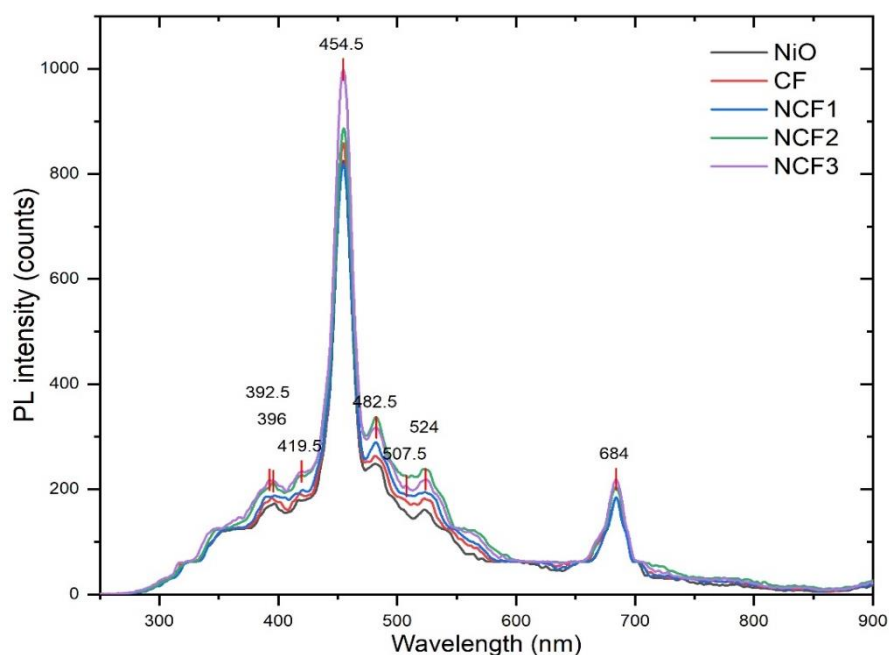


Fig 5.7: Photoluminescent spectra of pure catalyst and composites

5.4 FTIR SPECTROSCOPY:

The FTIR spectral image was taken in the range of 400 to 400 cm^{-1} wave number, Figure (5.8) illustrates the peak formation of all the samples and their corresponding transmittance value. The generated peaks provide information about the chemical bond formation between Fe and O. The spectrum revealed a narrow peak at 436 cm^{-1} indicating the presence of FeO bonds which is predominantly influencing the properties of the composite catalyst which is absent in case of pristine NiO, the progressive peak observed at 1420 cm^{-1} indicates the OH^{-1} bending due to the metal oxide vibrations. The peaks extending at 987 cm^{-1} and 2352 cm^{-1} denotes the presence of NiO bonds significantly which is absent in case of CoFe_2O_4 .

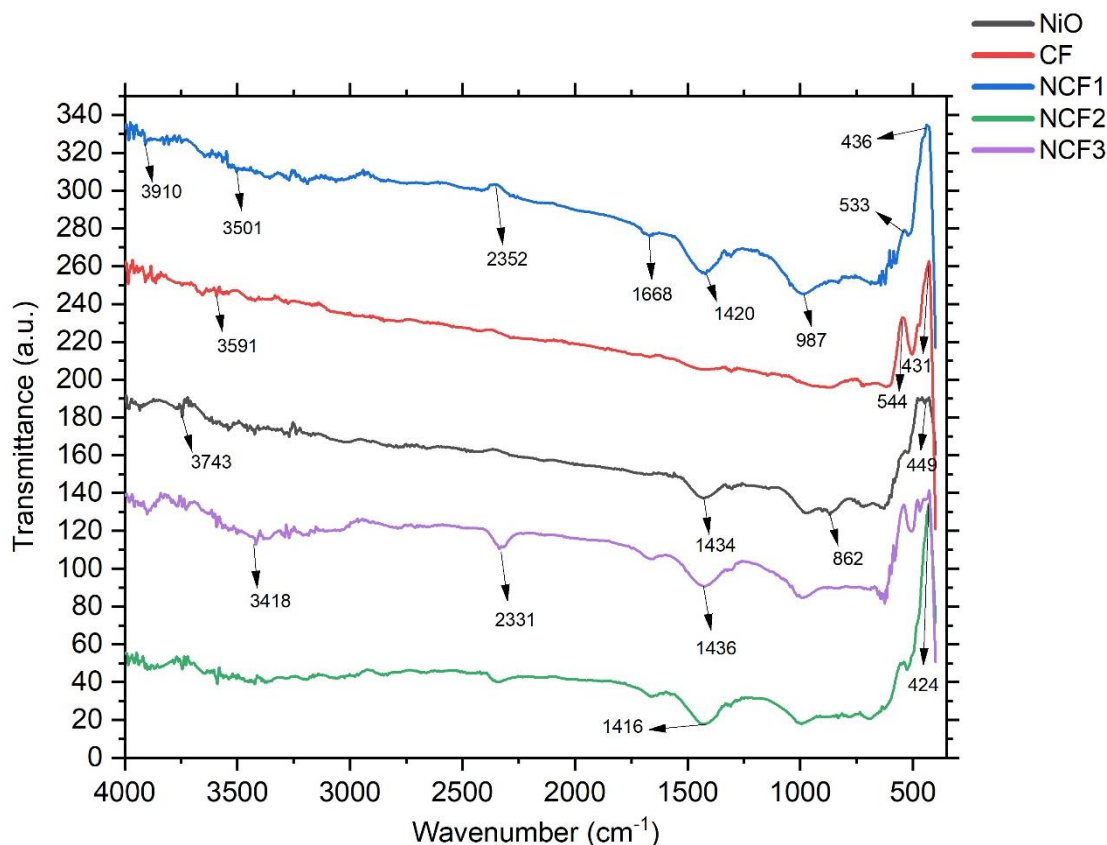


Fig 5.8: FTIR spectrum of pure and binary composite samples

5.5 FESEM WITH EDAX ANALYSIS:

The FESEM micrograph of the NiO/CoFe₂O₄ binary composite is taken under various magnification in order to justify the polycrystalline property of the sample as shown in the Figure (5.9). The sample indicated the presence of the most of the crystalline structure in range of 480 nm – 0.5 μ m. The micrograph also indicated the presence of a few crystalline structures under 100 nm range. This verifies the polycrystallinity in the structure of the binary composite. High intensity energy dispersive x-rays were used to pass through the binary composite and analyse the spectra in order to establish its chemical composition as mentioned in figure (5.13). The EDAX spectrum revealed the presence of predominant oxygen molecules over 51.0 keV, it also specified the presence of other elements like Fe, Co and Ni in the range of 3 keV, 3.2 keV, 3.1 keV. Thus, all the elements present in NiO/CoFe₂O₄ composite is identified. The table (5.2) indicates the fraction of each element contained in the sample where oxygen predominates in terms of percentage composition as high as 70.89 wt.% in the overall composition of the composite.

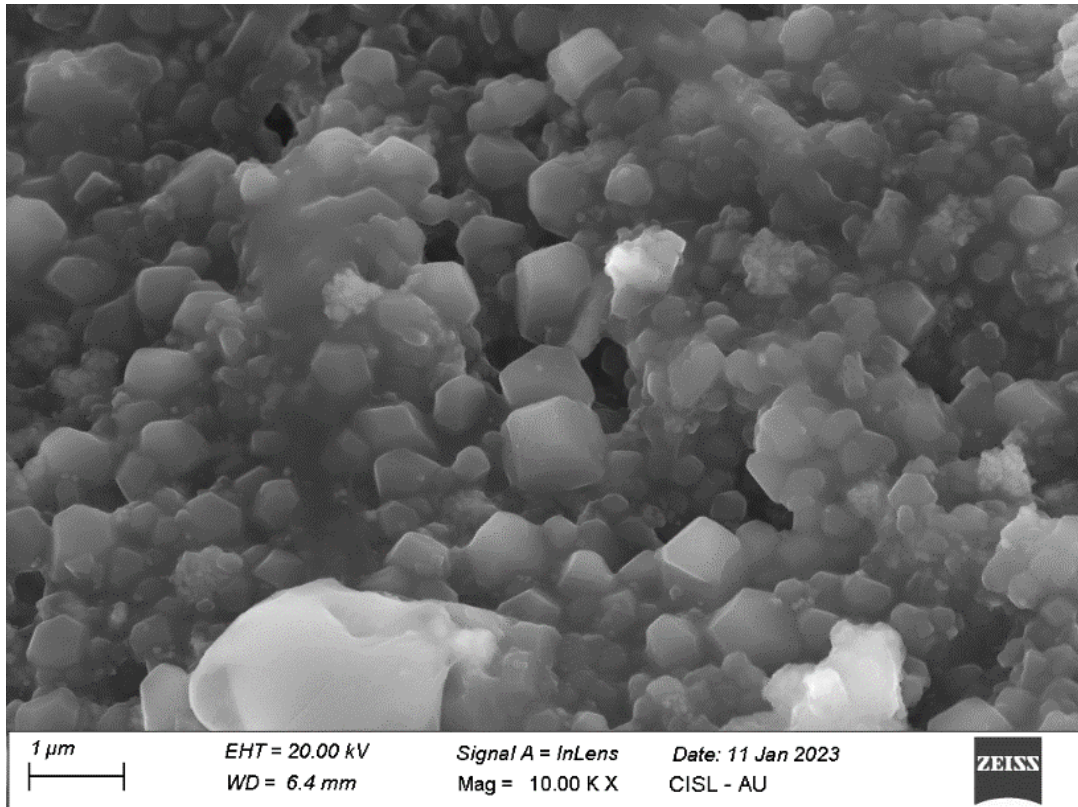


Fig 5.9: FESEM micrograph of binary composite in 1 μ m magnification

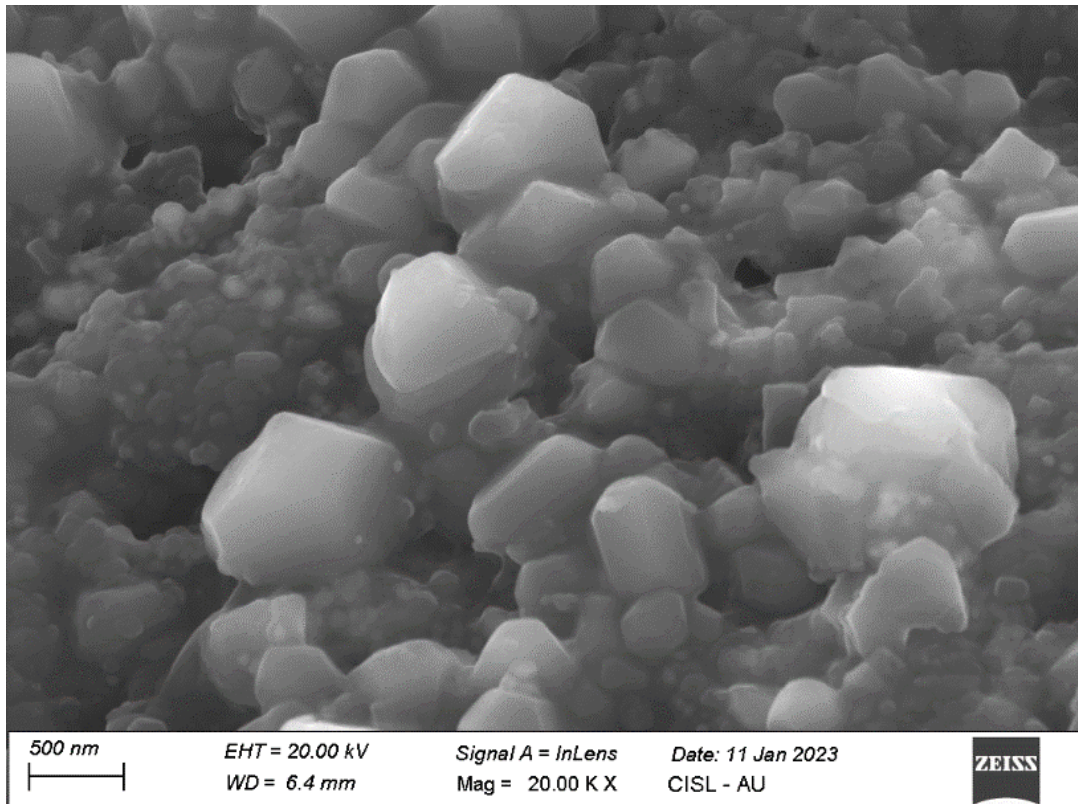


Fig 5.10: FESEM micrograph of binary composite in 500 nm magnification

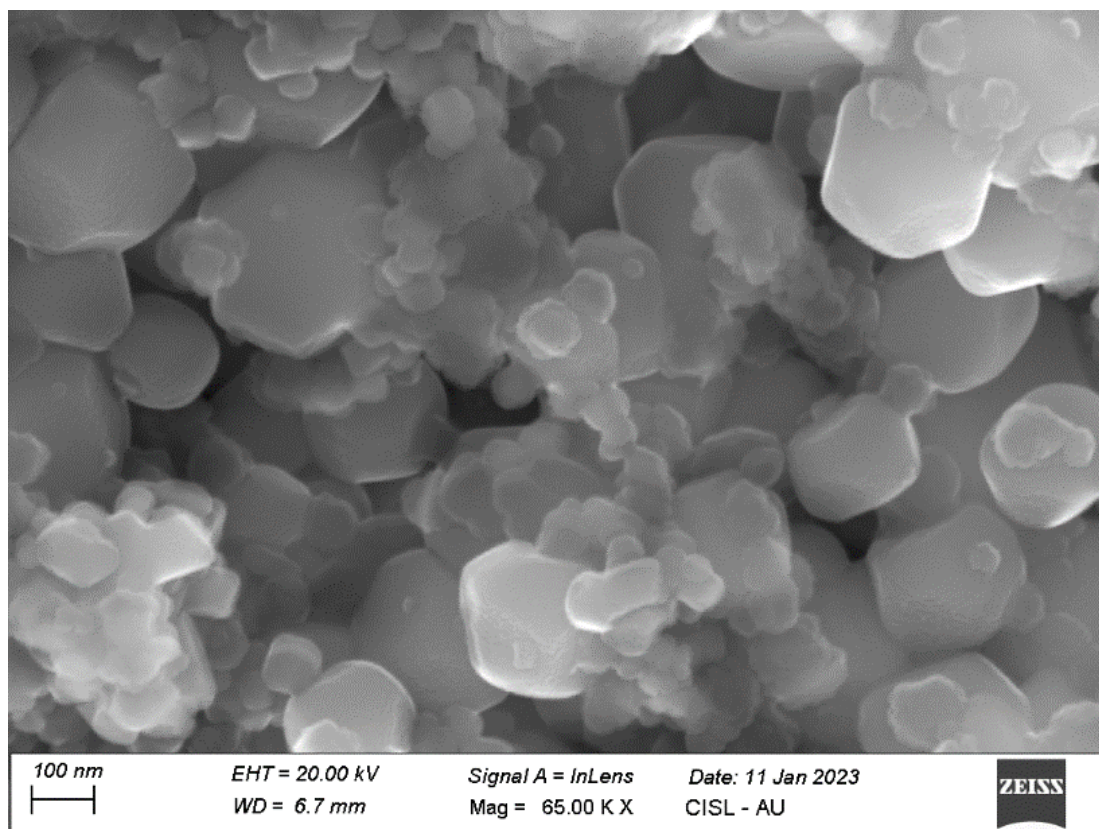


Fig 5.11: FESEM micrograph of binary composite in 100 nm magnification

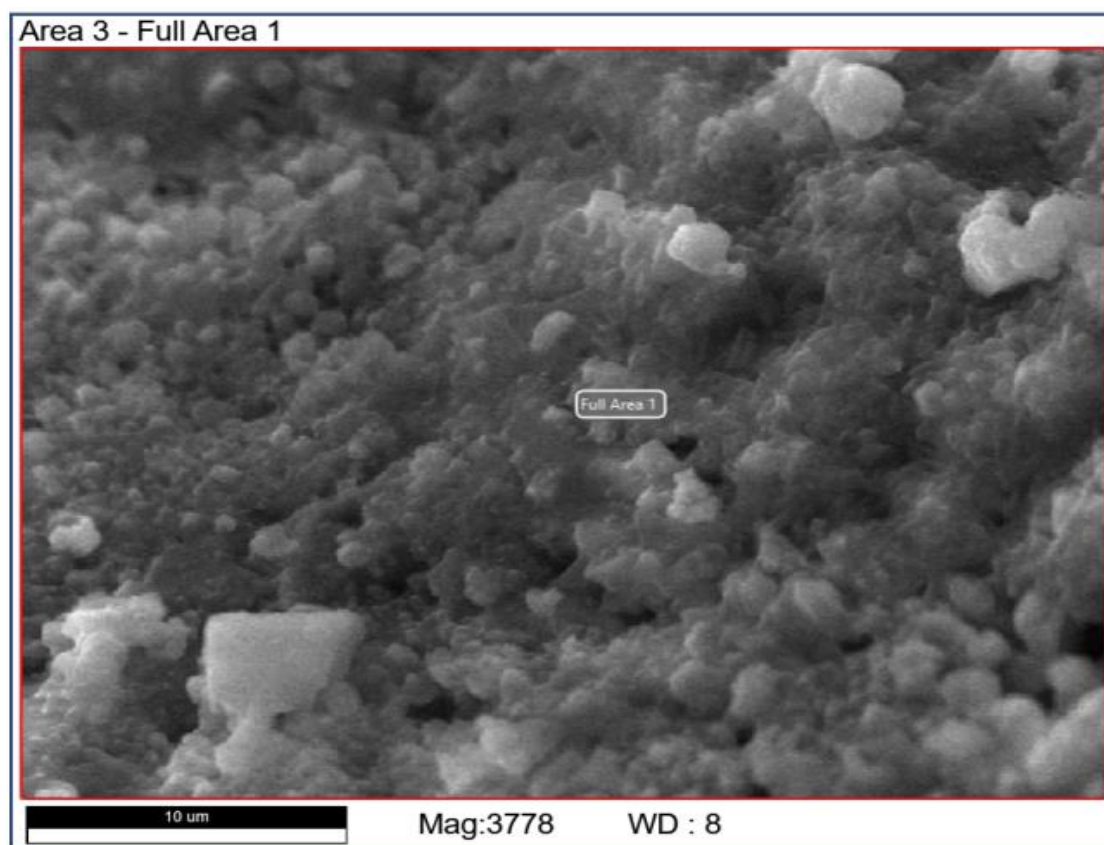
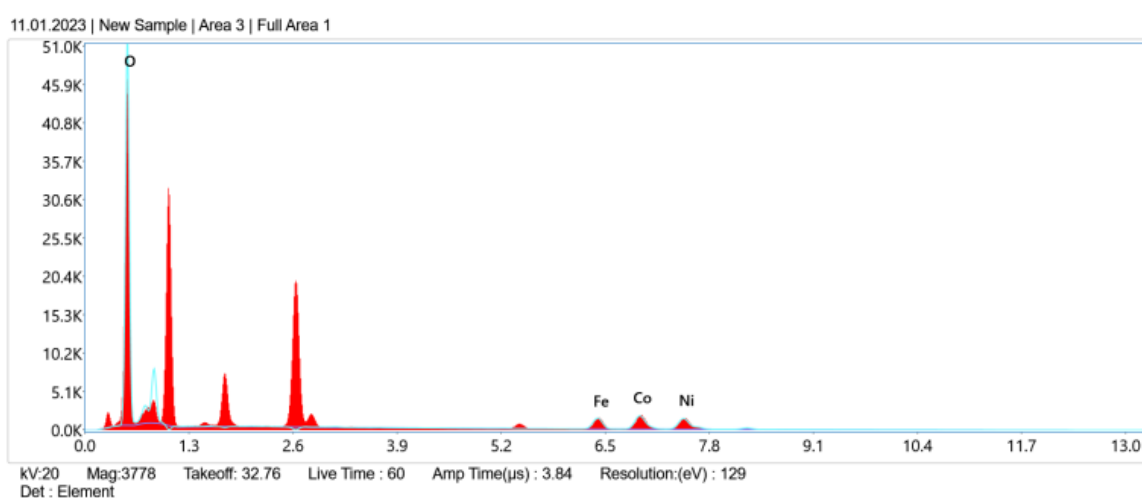


Fig 5.12: Full energy dispersive EDX at 10 μ m

Table 5.2: Elemental Quantification of the binary composite from EDAX

Element	Weight %	MDL	Atomic %	Error %	Net Int.	R	A	F
O	70.89	0.1	89.84	7.75	5126.69	0.8976	0.3359	1.0000
Fe	6.77	0.18	2.46	3.83	349.63	0.9597	0.9819	1.1691
Co	11.30	0.27	3.89	3.14	438.02	0.9625	0.9848	1.0646
Ni	11.04	0.28	3.81	3.40	368.10	0.9652	0.9785	1.0507

**Fig 5.13: EDAX Spectrum illustrating the chemical composition of the binary composite**

5.6 EPR SPECTROSCOPY:

Electron spin coupling (S-S) and spin-orbital angular momentum coupling are the causes of magnetic characteristics in materials (L-S coupling). A potent method for analysing paramagnetic species, such as organic radicals, inorganic radicals, and triplet states, is electron paramagnetic resonance spectroscopy (EPR). Nuclear magnetic resonance spectroscopy (NMR), which is more widely used, and EPR share many of the same fundamental concepts. However, EPR concentrates on the interaction of an external magnetic field with the unpaired electrons in a molecule rather than the nuclei of individual atoms. In cubic ferrimagnetic spinels, super exchange interactions between metal ions at the A and B sub lattices provide magnetic order. The A-B sub lattice exchange contact is predicted to be more

powerful than the A-A and B-B exchange contacts by Neels' molecular field model. ESR spectroscopy was used to investigate the effects of paramagnetic species and magnetic dipole interactions in polycrystalline ferrites. The electron spin resonance (ESR) spectra of the NiO/CoFe₂O₄ optimised sample NCF1 is displayed in figure (5.14) at room temperature. Magnetic dipole interactions between particles and super exchange interactions between magnetic ions via oxygen ions affect line width, or spectral shape.

The line width is wider when dipole interactions predominate, as they do in the NCF1 sample, whereas the line width is less when super exchange interactions predominate. As Ni concentration increases, the line shape of the spectrum narrows (i.e., line width decreases), demonstrating a dominant super exchange interaction. It has been shown that when Ni concentration increases and the lattice parameter decreases, super exchange interactions between magnetic ions on octahedral and tetrahedral sites increase. Since Ni²⁺ ions occupy the octahedral sites of the spinel structure, the concentration of Fe³⁺ ions at the tetrahedral sites increases as the Ni content increases, according to the hypothesised cation distribution. In NiO substituted CoFe₂O₄, A-O-B super exchange connections are increasingly common as the number of Fe³⁺ ions at tetrahedral sites increases. Using the following equation, the "g" factor of the sample was determined from the EPR signal (Gao et al., 2010):

$$g = \frac{h\nu}{\beta H_r}$$

, where ν is the spectrometer's frequency, equivalent to 9451.271×10^6 Hz, h is Planck's constant, equal to 6.626×10^{-34} Js⁻¹, β is the Bohr magneton, equal to 9.2740×10^{-24} JT⁻¹, and H_r is the resonance field, equal to 365.7×10^{-3} T. The calculated g-factor value for NCF1 binary composite is 1.85 which implies that the signal is considered to be created by small clusters or pairs of Fe³⁺ ions in NiO/CoFe₂O₄ samples which thereby influenced the degradation pathway of pantoprazole sodium.

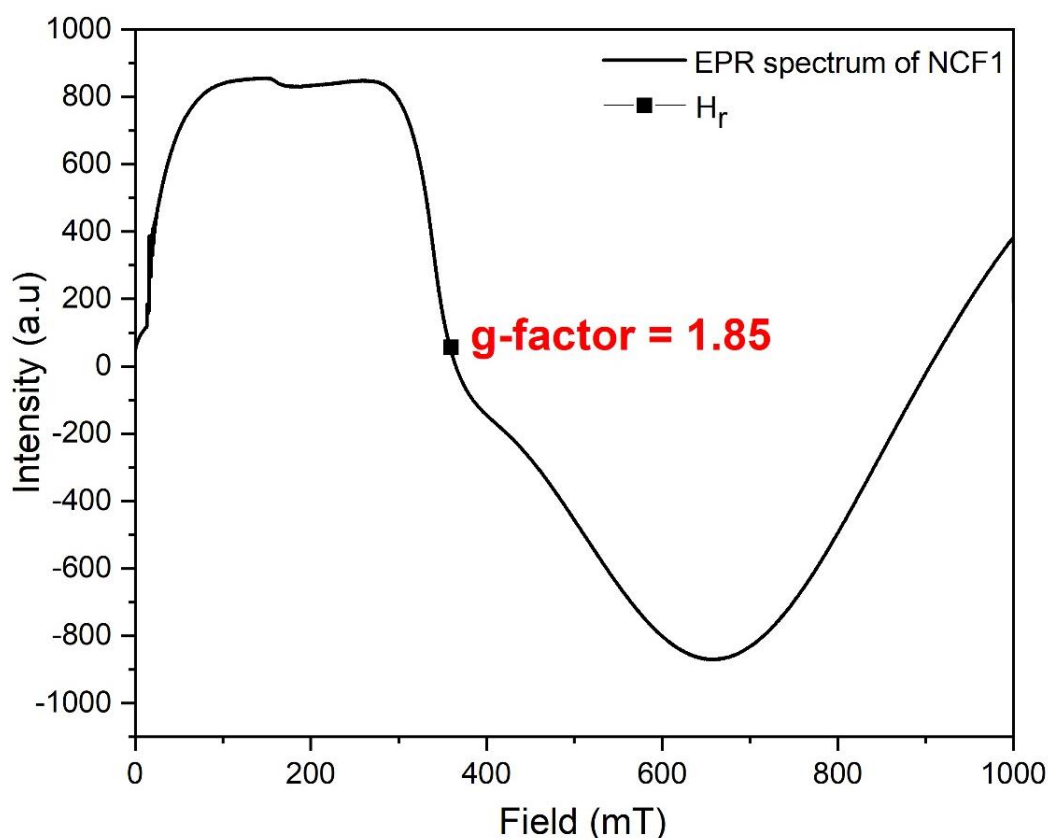


Fig 5.14: Room temperature EPR spectroscopy of the optimized binary composite NCF1

5.7 PHOTOCATALYTIC DEGRADATION KINETICS:

Photodegradation is the breakdown of a pollutant molecule, brought on by the absorption of photons, particularly those with wavelengths present in sunlight, such as infrared radiation, visible light, and ultraviolet light. However, various types of electromagnetic radiation can result in photodegradation. In order to assess the photocatalytic activity of the developed binary composite photocatalyst, the sample was collected and centrifuged at regular intervals of 15 minutes. By analysing the pantoprazole absorption at 212 nm with a UV-vis spectrophotometer (JASCO V-630-C220761148), it was possible to calculate the pantoprazole sodium degradation percentage using the following equation.

$$\text{Degradation \%} = \frac{C_0 - C}{C_0} \times 100 \quad (1)$$

Where C is its concentration after different intervals of time and C_0 is the initial concentration of pantoprazole. The degradation performance gradually increased

when increasing the catalyst dosage from 300-500 mg, further increasing the dosage resulted in slight reduction in degradation performance. Similarly, by increasing the concentration of the pollutant from 15-50 ppm, the degradation performance is found to be reduced. The maximum degradation of 86.24% is achieved for the optimal conditions (catalyst dosage - 500 mg, pollutant concentration – 15 ppm, pH – 12, time = 150 min). The COD removal was observed as high as $89 \pm 3\%$ within 120 min. No significant changes in COD removal were observed when pure 15 ppm of pantoprazole was irradiated under visible light without catalyst for the same duration.

Table 5.3: COD Measurements for various samples

S.No	Samples	% COD removal
1	DW	0
2	15 PPM (without catalyst)	0
3	15 min	$30 \pm 7 \%$
4	30 min	$36 \pm 9\%$
5	45 min	$40 \pm 4 \%$
6	60 min	$54 \pm 6\%$
7	75 min	$63 \pm 8\%$
8	90 min	$76 \pm 5\%$
9	105 min	$82 \pm 2 \%$
10	120 min	$89 \pm 3 \%$

In general, the Langmuir-Hinshelwood model is utilized to describe the kinetic parameters of photodegradation processes that occur at a solid-liquid interface (Gao et al., 2010; Joshi et al., 2014). Based on the initial concentration, the fundamental equation of the pantoprazole photodegradation rates are computed as mentioned in the table (5.4).

$$\ln C = -kt + \ln C_0 \quad (2)$$

Where k is the photodegradation rate constant, and C_0 and C are the pantoprazole concentrations at the initial and current time, respectively. Equation (3) was obtained after derivation of Equation (2)

$$\ln \frac{C_0}{C} = kt \quad (3)$$

Where k is the rate constant of the pseudo-first order reaction, C_0 and C are the corresponding concentrations at $t = \text{initial}$ and $t = t$, respectively. The straight line of the plot $\ln (C_0/C)$ against time established that the photocatalytic degradation followed the pseudo-first order as shown in figure (5.22). The resulted k values were found to be $1.573 \times 10^{-2} \text{ min}^{-1}$, $0.368 \times 10^{-2} \text{ min}^{-1}$, $0.778 \times 10^{-2} \text{ min}^{-1}$, $1.435 \times 10^{-2} \text{ min}^{-1}$ and $0.180 \times 10^{-2} \text{ min}^{-1}$ for NiO/CoFe₂O₄ - 0.5, NiO/CoFe₂O₄ – 0.3, NiO/CoFe₂O₄ – 0.7, NiO/CoFe₂O₄ - Scavenger and NiO/CoFe₂O₄ - PPM, as computed in the table (5.4). Regarding to these results, the highest reaction rate was achieved by NiO/CoFe₂O₄ - 0.5 ($1.57 \times 10^{-2} \text{ min}^{-1}$), which was consistent with the performance results.

Table 5.4: Kinetic study and degradation of pantoprazole using NiO/CoFe₂O₄

S. No	Photocatalyst	Photodegradation (%)	Time (min)	Rate constant (k) ($\times 10^{-2} \text{ min}^{-1}$)	R ²
1.	NiO/CoFe ₂ O ₄ - 0.5	86.24	150	1.573	0.9804
2.	NiO/CoFe ₂ O ₄ – 0.7	72.84	150	0.778	0.8342
3.	NiO/CoFe ₂ O ₄ Scavenger	62.49	75	1.435	0.7284
4.	NiO/CoFe ₂ O ₄ – 0.3	38.17	150	0.368	0.9781
5.	NiO/CoFe ₂ O ₄ -PPM	28	150	0.180	0.8545

The absorbance values obtained from the UV-vis absorbance spectroscopy were plotted against the wavelength for the various samples ranging from 15 min to 150 min, in order to estimate the degradation parameters as mentioned in the figure (5.15).

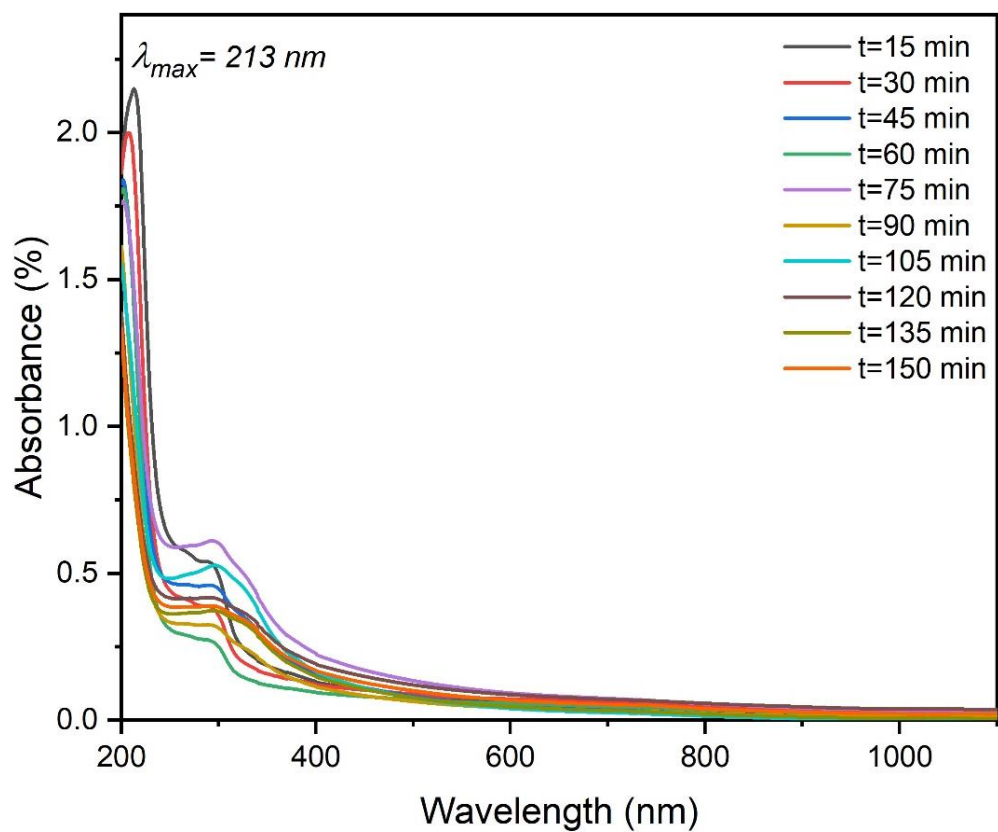


Fig 5.15: UV-vis Absorbance plot of photocatalytic reaction – 300 mg

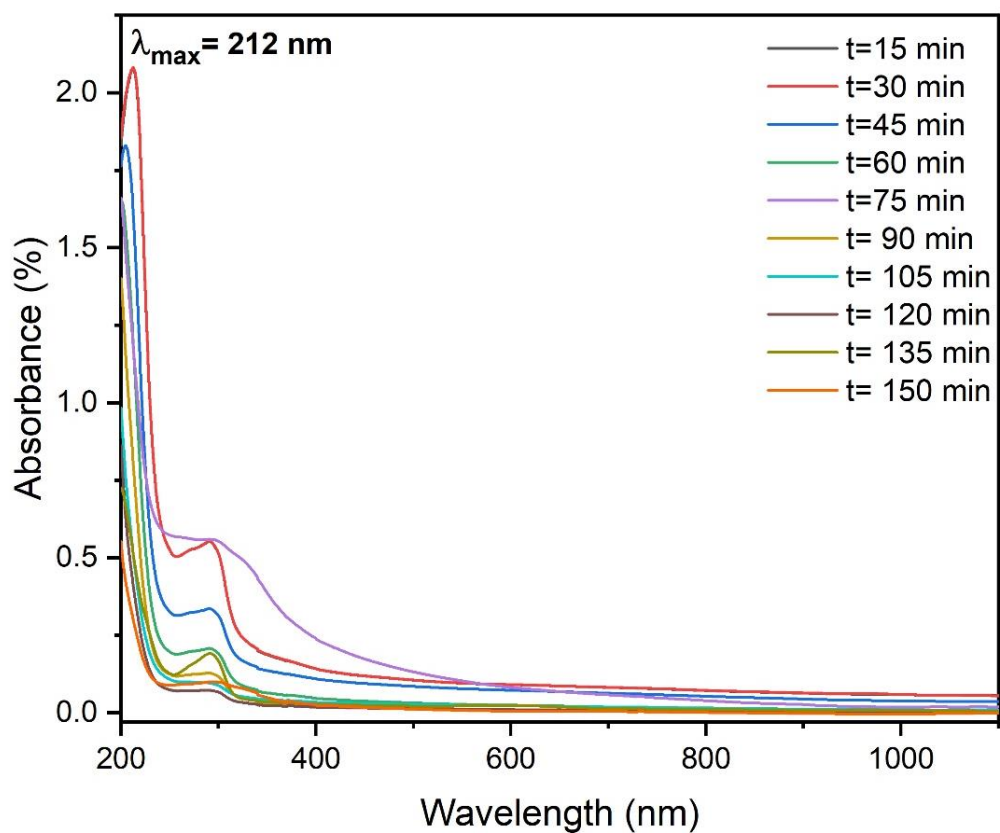


Fig 5.16: UV-vis Absorbance plot of photocatalytic reaction – 500 mg

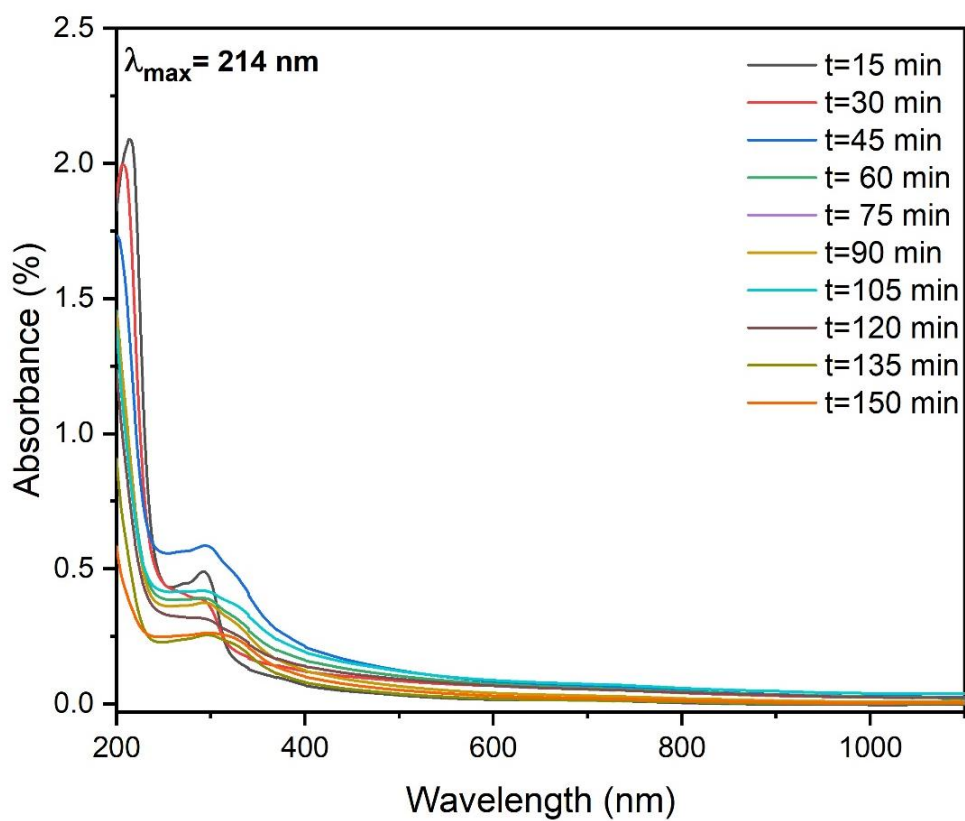


Fig 5.17: UV-vis Absorbance plot of photocatalytic reaction – 700 mg

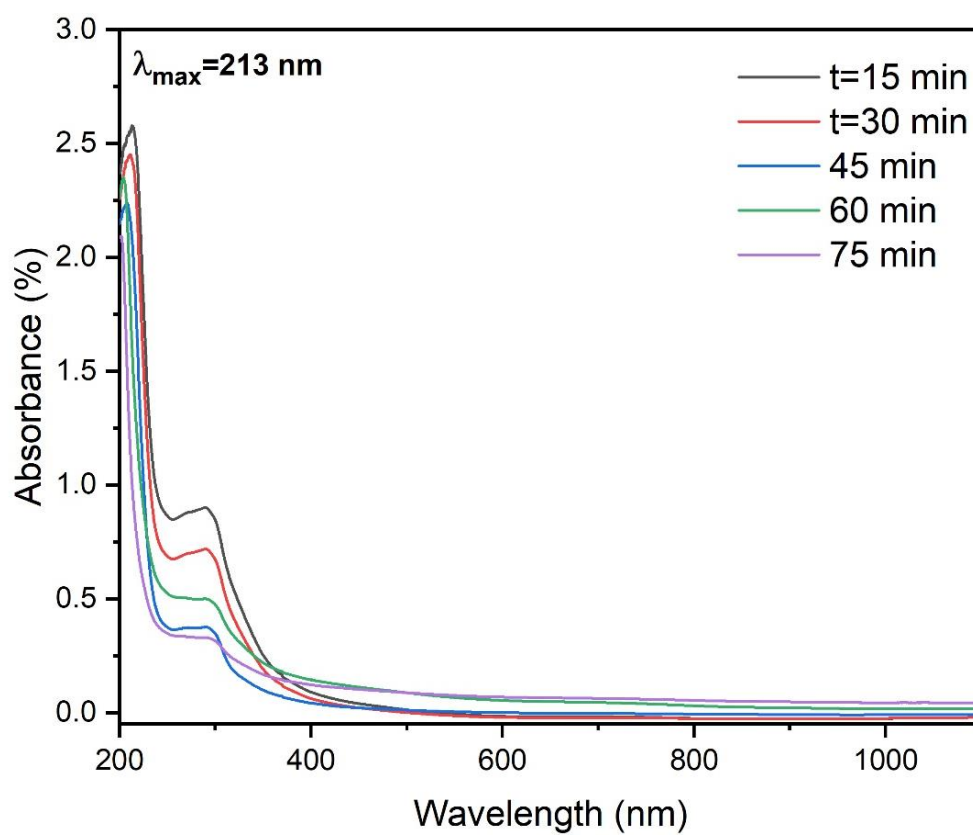


Fig 5.18: UV-vis Absorbance plot of photocatalytic reaction – scavenger

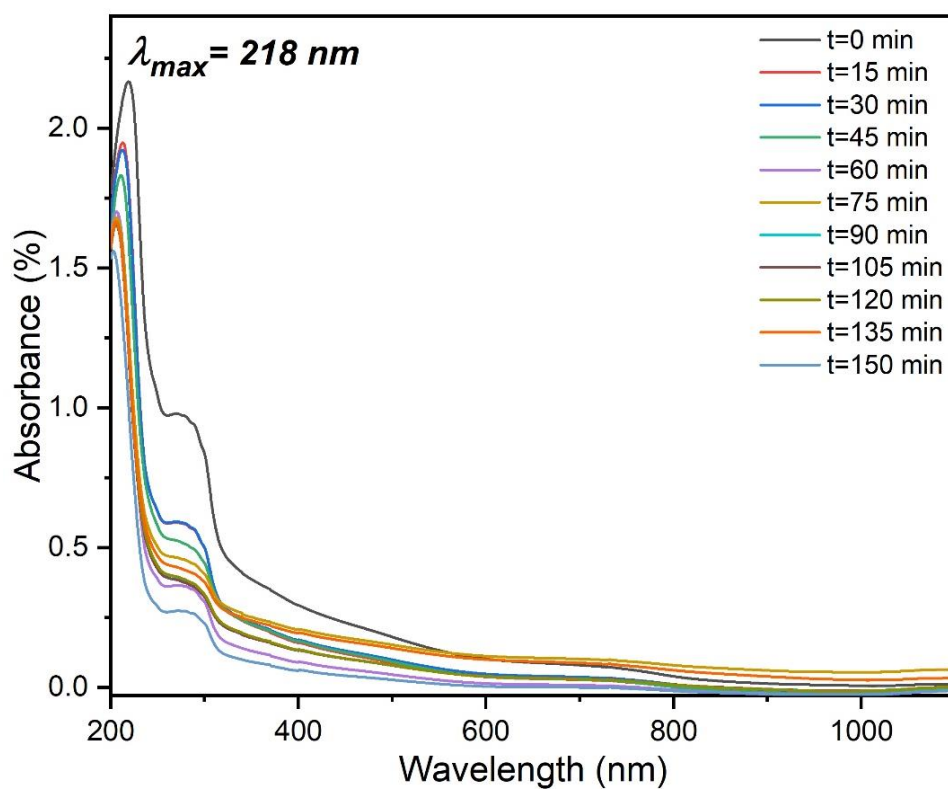


Fig 5.19: UV-vis Absorbance plot of photocatalytic reaction – 50 PPM

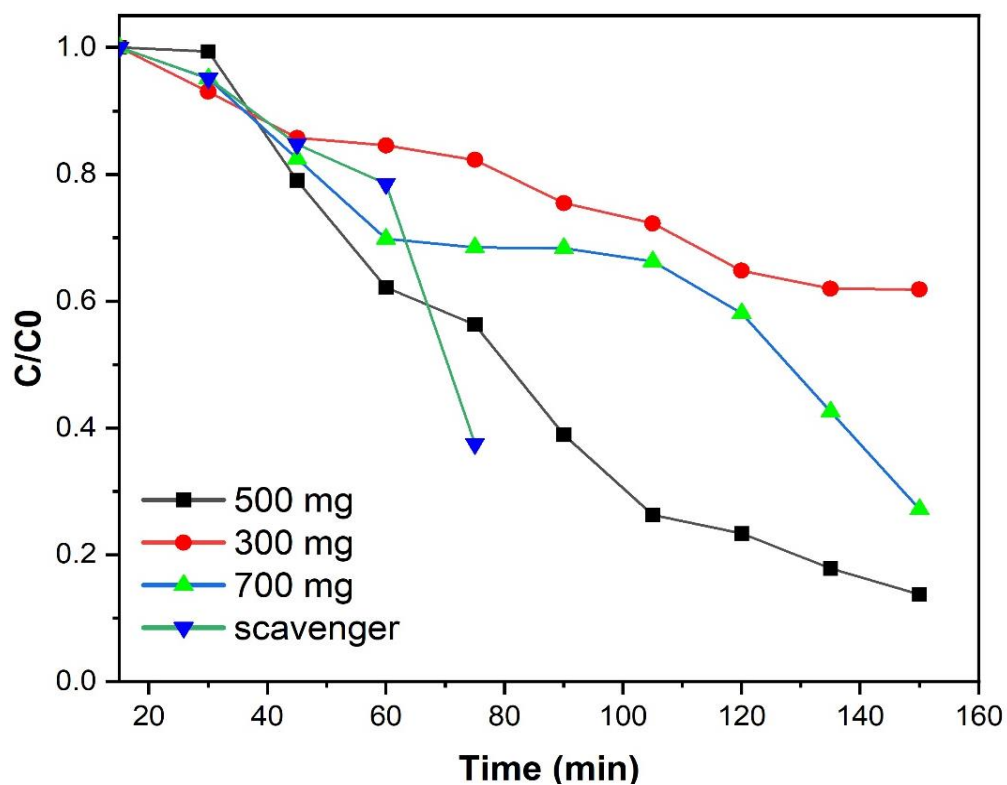


Fig 5.20: Photocatalytic degradation of pantoprazole sodium by varying catalyst dosage

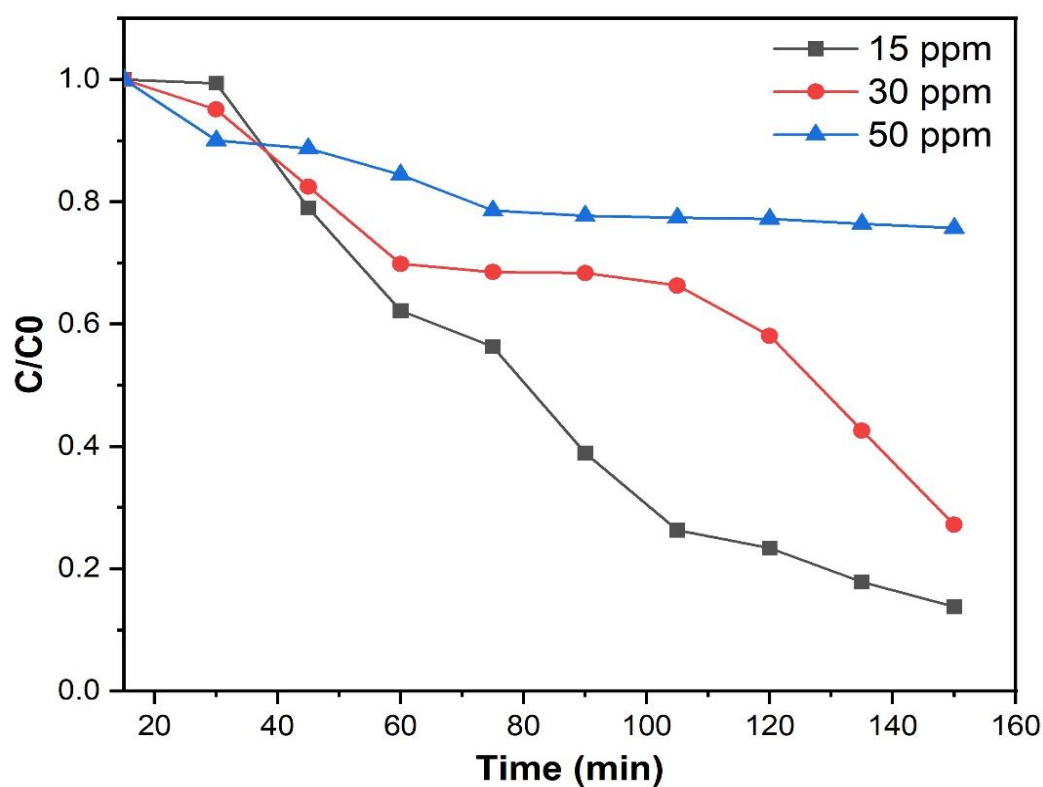


Fig 5.21: Photocatalytic degradation of pantoprazole sodium by varying pollutant concentration

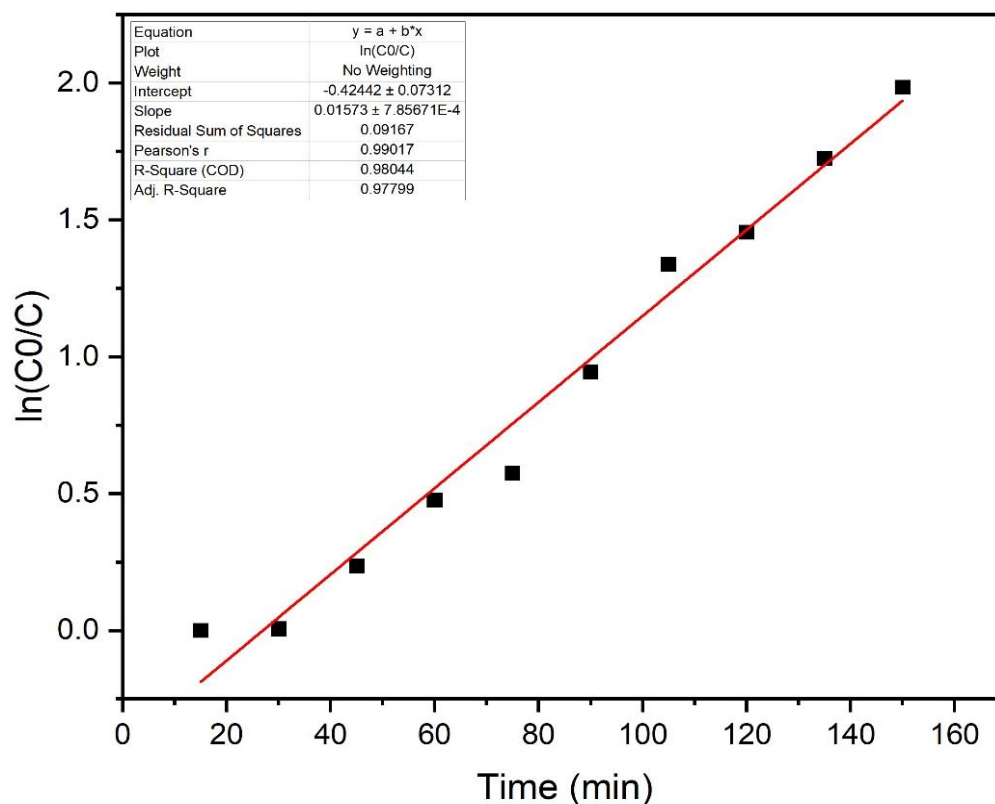


Fig 5.22: Kinetic plot of Photocatalytic degradation reaction of pantoprazole sodium.

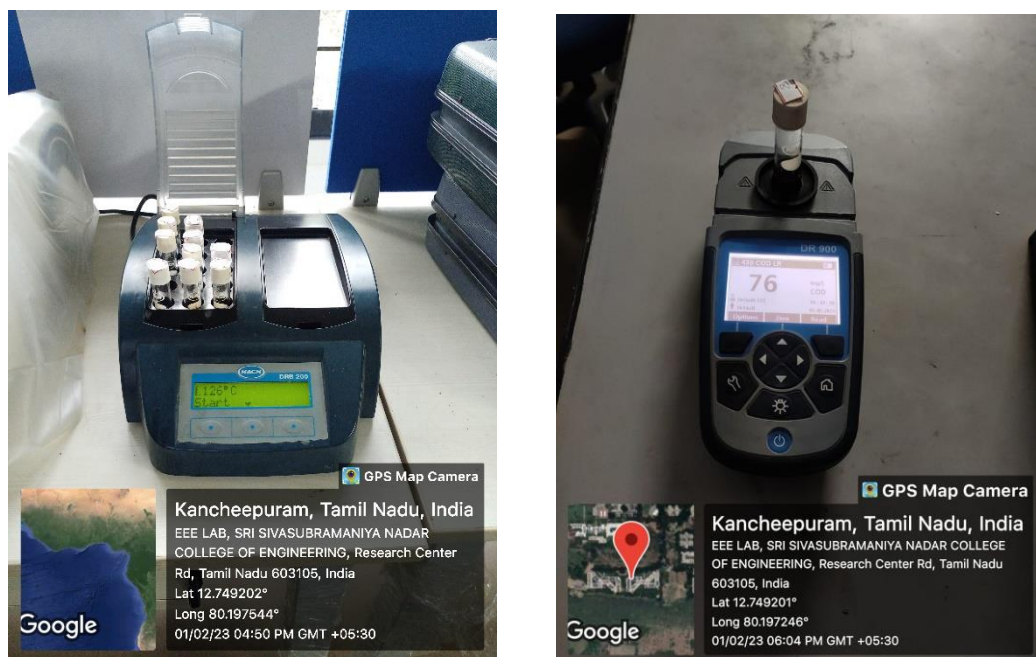


Fig 5.23: COD reactor setup and measurements of % COD removal

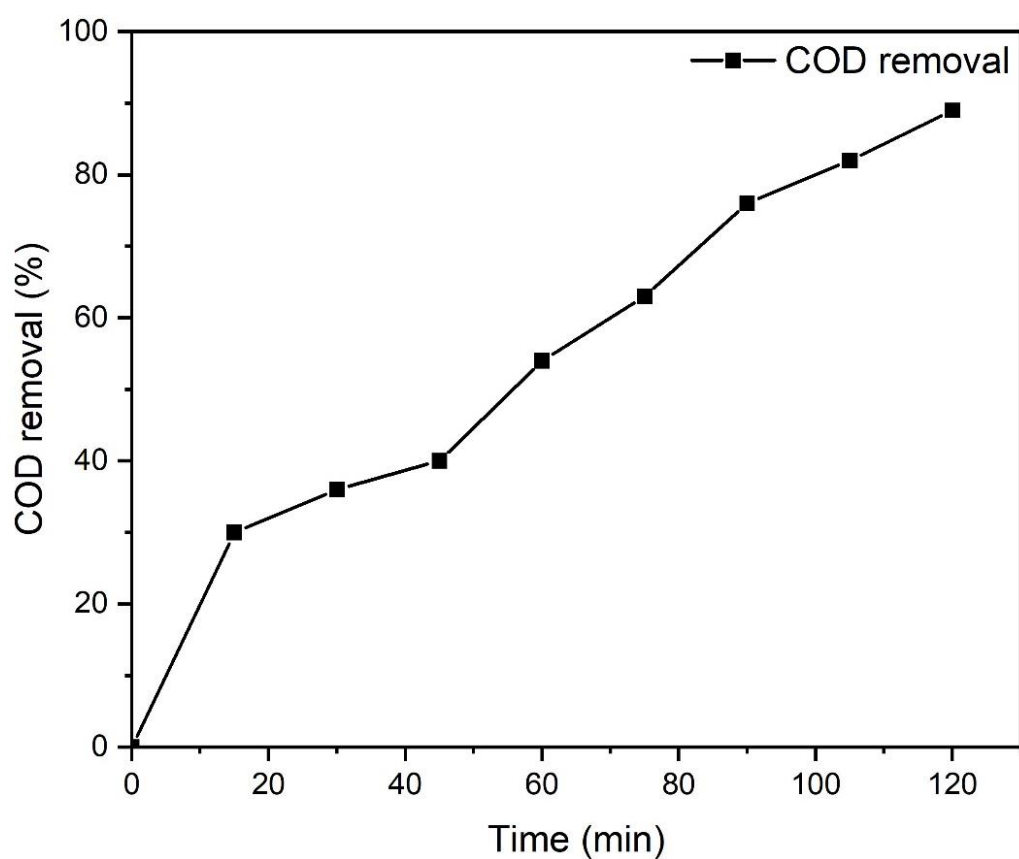


Fig 5.24: Chemical Oxygen Demand (COD) removal plot vs time

CHAPTER 6

CONCLUSION

NiO/CoFe₂O₄ composites were prepared effectively in three different ratios by varying NiO concentration as NCF1, NCF2, and NCF3. The obtained products were characterized by applying various analytical routes like XRD, UV-DRS, FTIR, and PL spectra. The band gap (E_g) is found to be enhanced with an increase in the concentration of Ni among the composite ratios. The NCF1 is observed to have the least E_g of 2.45 eV when compared to other ratios, because strong electrostatic contact results from the lower ionic radii of Ni²⁺ ions compared to Co²⁺ ions which causes the deeper Ni²⁺ states to form than Co²⁺ states thereby widening the E_g between unoccupied and occupied levels of NiO/CoFe₂O₄. The oxygen vacancies and gradual increase and decrease in emission peaks on varying the NiO concentration specified the optimal sample as NCF1 among the other ratios. The surface morphology of the NCF1 is analysed through FESEM and the composition of Ni, Fe, O, and Co was estimated using the EDAX spectrum. The binary composite sample indicated the presence of most of the crystalline structure in the range of 480 nm – 0.5 μ m. The micrograph also indicated the presence of a few crystalline structures under the 100 nm range. This verifies the polycrystallinity in the structure of the binary composite. The significance of magnetic dipole interactions in polycrystalline ferrites as well as the contribution of paramagnetic species were next investigated using ESR spectroscopy as a confirmation test. The G-factor was calculated to be 1.85 implying that the signals are caused by pair (or) small clusters of Fe³⁺ ions in NiO/CoFe₂O₄ composite which in reaction produces ROS (Reactive Oxygen Species) majorly •OH which influences the degradation pathway of Pantoprazole sodium. Fe³⁺ concentration in tetrahedral locations increased as a result of the optimised Ni concentration, leading to a dominating A-O-B lattice super exchange interaction. The maximum degradation of 86.24% is achieved for the optimal conditions (catalyst dosage - 500 mg, pollutant concentration – 15 ppm, pH – 12, time = 150 min). The COD removal was observed as high as 89 $\pm$ 3% within 120 min indicating the high photocatalytic performance and outstanding stability of the composite which is found to be a possible candidate for pharmaceutical contaminant degradation.

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