PHOTOCATALYTIC DEGRADATION OF MIXED DYE SOLUTION

PROJECT REPORT

SUBMITTED BY

KARTHIKA SUBRAMANIYAN

(AB23PHY008)

Under the guidance of

Dr. MINU PIUS

Department of Physics

St. Teresa's College (Autonomous), Ernakulam

Kochi – 682011

Submitted to

Mahatma Gandhi University, Kottayam

In partial fulfilment of the requirement for the Award of

BACHELOR'S DEGREE OF SCIENCE IN PHYSICS

ST. TERESA'S COLLEGE (AUTONOMOUS)

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CERTIFICATE

This is to certify that the project report entitled **"PHOTOCATALYTIC DEGRADATION OF MIXED DYE USING ZINC FERRITE/SILVER/SILVER CHLORIDE "** is an authentic work done by **KARTHIKA SUBRAMANIYAN (AB23PHY008),** St Teresa's College, Ernakulam, under my supervision at Department of Physics & Centre for Research, St. Teresa's College, Ernakulam, for the partial requirements for the award of Degree of Bachelor of Science in Physics during the academic year 2025-26. The work presented in this dissertation has not been submitted for any other degree in this or any other university.

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B.Sc. PHYSICS PROJECT REPORT

NAME : **KARTHIKA SUBRAMANIYAN**
REGISTER NUMBER : **AB23PHY027**
YEAR OF WORK : **2025-26**

This is to certify that this project work entitled, **"PHOTOCATALYTIC DEGRADATION OF MIXED DYE USING ZINC FERRITE /SILVER/SILVER CHLORIDE "** is an authentic work done by **KARTHIKA SUBRAMANIYAN.**

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DATE: 16/04/2026

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2.

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DECLARATION

I am **KARTHIKA SUBRAMANIYAN, final** year B.Sc. Physics students, Department of Physics, St. Teresa's College (Autonomous), Ernakulam, do hereby declare that the project work entitled **"PHOTOCATALYTIC DEGRADATION OF MIXED DYE SOLUTION"**, has been originally carried out under the guidance and supervision of **Dr.MINU PIUS**, Department Of Physics, Teresa's College (Autonomous), Ernakulam, in partial fulfilment for the award of the degree of Bachelor of Physics. I further declare that this project is not partially or wholly submitted for any other purpose and the data included in the project is collected from various sources and are true to be the best of my knowledge.

PLACE: ERNAKULAM

DATE:

ACKNOWLEDGEMENT

First and foremost, I thank Almighty God for granting me the strength, clarity, and determination needed to bring this work to completion.

I am deeply grateful to **Dr. MINU PIUS**, Department of Physics, **St. Teresa's College (Autonomous)**, for her patient guidance, thoughtful feedback, and steady encouragement throughout the course of this project. Her emphasis on conceptual understanding and disciplined thinking has greatly influenced both my approach to this study and my growth as a student of physics.

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Finally, I express my heartfelt thanks to all the teaching and non-teaching staff of the Department of Physics for their cooperation and assistance at various stages of this project.

ABSTRACT

In this project, the photocatalytic degradation of Methylene Blue, Methyl Orange and Rhodamine B using Zinc ferrite/Silver/Silver chloride was studied at different sunlight exposure times.

The sample solution was placed in the dark condition first and the equilibrium adsorption-desorption time was found. After the adsorption studies, it was exposed to sunlight for photocatalytic degradation and at different intervals of time the sample solution was taken out and its absorbance studies were done using the spectrophotometer.

Using the data received from the spectrophotometer, we could analyze the sample solution, the degradation was analyzed in terms of degradation efficiency. In mixed dye system, the maximum degradation efficiencies achieved within 90 minutes were approximately 70% for Methylene Blue (MB), 40% for Methyl Orange (MO) and 10% for Rhodamine B (RhB). In contrast, the individual dye study of Methylene Blue under identical conditions showed a significantly higher degradation efficiency of about 98%, demonstrating the enhanced performance of the nanocomposite in single-dye systems compared to mixed dye environments.

PHOTOCATALYTIC DEGRADATION
OF
MIXED DYE SOLUTION

CONTENTS

LIST OF ABBREVIATIONS

AOP	Advanced Oxidation Process
COD	Chemical Oxygen Demand
CR	Congo Red
Cs/Vs	Mass of catalyst/ volume of solution
eV	Electron volt
Fe^{3+}	Ferric ion
Lux	Unit of luminance
MB	Methylene Blue
MO	Methyl Orange
RhB	Rhodamine B
pH	Potential of hydrogen
ppm	Parts per million
TOC	Total organic carbon
UV-Vis	Ultraviolet-Visible spectroscopy
$ZnFe_2O_4$	Zinc ferrite
$ZnFe_2O_4$/Ag/AgCl	Zinc ferrite/Silver/Silver Chloride
$Zn_xFe_{3-x}O_4$	Non-stochiometric Zinc Ferrite
$Zn(NO_3)_2$	Zinc nitrate
$Fe(NO_3)_3$	Iron (III) nitrate
•OH	Hydroxyl radical
A_0	Initial absorbance
A_t	Absorbance at time t
$C_{14}H_{14}N_3NaO_3S$	Molecular formula of Methyl Orange
$C_{16}H_{18}N_3ClS$	Molecular formula for Methylene Blue
$C_{28}H_{31}N_2O_3Cl$	Molecular formula for Rhodamine B

CHAPTER 1
INTRODUCTION

1.1 WATER POLLUTION

Water pollution also known as aquatic pollution is the contamination of marine systems and freshwater through human activities with a negative impact in their uses. Water bodies include groundwater, ponds, lakes, rivers, aquifers, reservoirs and ocean. Industrial discharge, agricultural runoff, domestic sewage, plastic and solid waste dumping and oil spills are few of the exemplary examples of human caused water pollution. All these human activities reduce the access to essential services such as potable water and irrigation. It compromises the water quality and generates cascading effects on ecological stability and disease transmission.

1.1.1 TYPES OF WATER POLLUTION

Water pollution is generally categorized based on the source of the contaminant. While biological and physical pollutants are significant, chemical pollution poses the greatest challenge to modern remediation technologies.

- Biological Pollution: It is the contamination of water bodies by pathogenic microorganisms including bacterial strains, virus, parasites which is caused by discharge of untreated sewage and fecal matter. It poses a risk for waterborne diseases like dysentery and cholera.
- Physical Pollution: It is the contamination of water bodies by substances that alter their physical properties like odor, temperature, color and turbidity. Contributors are solid trash, microplastic and sediment runoff.
- Chemical Pollution: It is the contamination of water bodies by hazardous substances like pesticides, fertilizers and heavy metals. Contributors are industrial discharge and improper water disposal.

1.1.2 POINT SOURCE POLLUTION

Industrial effluents containing mixed dyes are typically point source discharges that requires localized high efficiency treatments. This contamination occurs when the contaminants enter from a single, identifiable location such as a discharge pipe from a textile factory or a wastewater treatment plant. There also exists another category known as 'Non-point

sources.' This occurs when rainfall or snowmelt carries diverse natural and human-made contaminants across land into water bodies, making it a widespread issue distinct from localized point sources.

1.1.3 LAKE POLLUTION

This occurs when there is the contamination of lakes by nutrients, chemicals and waste from point and non-point sources, leading to ecological degradation and health risks. This kind of pollution causes eutrophication and reduces tourism, fishing and local livelihoods.

1.1.4 TRANSBOUNDARY POLLUTION

Transboundary water pollution occurs when contaminants cross political and geographical boundaries through rivers, lakes or aquifers shared by two or more nation. This pollution creates ripples across borders making it both an environmental and diplomatic challenge. It impacts people and ecosystems like for example the Brahmaputra and Sutlej rivers between India and China carry industrial pollutants that affect both ecosystems and human health. So, in these scenarios the solutions include international agreements, joint monitoring, technology sharing and community engagement.

1.1.5 OCEAN CONTAMINATION

Ocean contamination refers to the introduction of harmful substances into marine environments. This can stem from both land and ocean-based activities. These pollutants include plastics, nutrients, hydrocarbons, heavy metals and persistent organic compounds which collectively disrupt ecological balance and pose risks to human health. Primary sources include agricultural runoff, oil spilling, offshore drilling operations, atmospheric deposition and solid waste settlement. This can lead to eutrophication, seafood health hazards and climate interactions because the ocean cannot further act as a carbon sink. UN Ocean Decade initiatives promote cooperative management of marine pollution. Rectifications involved are technological interventions and scientific monitoring.

1.1.6 TEXTILE EFFLUENTS

Textile effluents are the liquid wastes released during dyeing, bleaching and finishing fabrics. They carry a cocktail of synthetic dyes, detergents, heavy metals and oils. If these are untreated and enters the waterbodies, they strip oxygen from water affecting the aquatic life drastically. The bright colors that make your clothes appealing can turn rivers dark thereby disrupting photosynthesis. Exposure to such toxic dyes and metals can cause skin

irritation, respiratory problems and eve long-term health hazards. Untreated water can turn the soil saline and infertile threatening food security.

1.2 DYE POLLUTION AND ITS HAZARDS

Dyes are the substances that are used to impart color to textiles, fabrics, leather or other materials. These are such colored substances that bond chemically to any substrate to which they are being applied. Any fabric's attraction is its color. Utilization of manufactured colors for texture coloring has formed into a multibillion-dollar industry. These compounds pose multifaceted health risks due to their complex aromatic structures and resistance to biodegradation. The visibility of dye contamination even at low concentrations disrupts aquatic photosynthesis. Azo dyes which account for 60-70% of industrial colorants can undergo reductive cleavage which demonstrates mutagenic and carcinogenic potential. The recalcitrant nature of these compounds means they persist in water bodies and can bioaccumulate through tropic levels.

Chronic exposure can lead to allergic reactions, dermatological conditions, and potential carcinomic effects in human beings. Studies on fish species have documented hepatotoxicity, nephrotoxicity and histopathological changes in gill and liver tissues following dye exposure. Avian and mammalian species dependent on aquatic food sources face secondary exposure risks, though research in this area remains limited compared to direct aquatic toxicity studies.

1.3 WATER PURIFICATION USING NANO PARTICLES

Nanoparticle based water treatment has emerged as a promising approach due to the unique properties that materials exhibit at the nanoscale. Nanoparticles range from 1-100 nm in size possess higher surface areas relative to their volume compared to bulk materials. This increased surface area provides more active sites where contaminants can interact with the catalyst which allows effective pollutant removal using smaller amounts of material. The small particle size changes the electronic structure through quantum effects which can improve photocatalytic performance. Nanostructured surfaces also scatter and absorb more light.

1.3.1 ADSORPTION

It is a surface-based phenomenon where dissolved contaminants accumulate on the surface of a solid material called an adsorbent. This process is different from absorption, where substances penetrate into the bulk of a material. Whilst treating water, adsorption occurs by attracting pollutant molecules from aqueous phase onto the adsorbent surface through various intermolecular forces. Adsorption has various types namely Physisorption (Physical adsorption), wherein adsorbed molecules can be released back into the solution under certain conditions. There is chemisorption (Chemical adsorption) where there is stronger chemical bonds between the pollutant and the adsorbent surface, making it more permanent. Several factors affect adsorption quality which are temperature, solution's pH, surface area and contact time.

Examples include activated carbon, metal oxide nanoparticles, clay minerals, biochar and agricultural waste products are gaining attention.

1.3.2 MEMBRANE FILTRATION

This works by pushing water through a barrier with tiny holes that trap contaminants while letting clean water pass through. The concept is applying pressure to force water through the membrane, add anything bigger than the pores gets left behind. Membrane filtration if of four types namely microfiltration which can catch bacteria and protozoa. Ultrafiltration can trap viruses and colloids. Nanofiltration goes even finer and can removed small organic compounds. Reverse osmosis can produce nearly pure water by filtering out almost everything.

1.3.3 COAGULATION

It is a method we use to remove color, organic matter and turbidity. This is a critical water treatment process where to raw water we add positively charged chemicals like ferric chloride to neutralize the negative electrical charges on fine particles. Here, the particles will aggregate to form "flocs". After coagulation comes flocculation and sedimentation. In coagulation we add chemicals known as coagulants; these are added rapidly into water to ensure even distribution. There are several conditions like temperature, pH and types of contaminants present that are to be considered carefully for specific water sources.

However, the dyes we are dealing with like methylene blue, methyl orange and rhodamine B are more stable and therefore remains undissolved in water and does not respond well to coagulation so they pass through the treatment process largely untouched.

1.3.4 BIOLOGICAL TREATMENT METHODS

This is an eco-friendly alternative to chemical treatments wherein it degrades, transforms and removes organic pollutants from water, waste or soil by using microorganisms usually bacteria. It is a natural and sustainable method to efficiently treat domestic and industrial waste by breaking down containments and common techniques include aerobic and anaerobic treatments. These methods work well for biodegradable organic compounds like proteins, sugars and simple organic acids. Yet, this method cannot be used to breakdown synthetic dyes because of their complex aromatic structures and synthetic bonds make them recalcitrant to it. Sometimes degradation do occur but it is slow and incomplete and creates a product more toxic than the original dye.

1.3.5 LIMITATIONS OF CONVENTIONAL METHODS

All these methods have certain limitations. For instance, coagulation creates large volumes of chemical sludge which needs proper disposal and generates further waste problems. It does not work for water soluble dyes. In membrane filtration we are only physically separating them but not destroying them; this again creates secondary waste problems like coagulation. Biological treatment takes a long time, incomplete and the process has a lot of conditions that it must fulfil in order to work properly. As mentioned before certain azo dyes are potentially carcinogenic.

1.4 ADVANCED OXIDATION PROCESS

Advanced oxidation processes (AOPs) were first proposed for potable water treatment in the 1980s, which are defined as the oxidation processes involving the generation of hydroxyl radicals $OH\cdot$ sufficient quantity to effect water purification. Different from common oxidants such as chlorine and ozone that have a dual role of decontamination and disinfection, AOPs are applied primarily for destruction of organic or inorganic contaminants in water and wastewater. When AOPs are applied for wastewater treatment, these radicals, as a powerful oxidizing agent, are expected to sufficiently destruct wastewater pollutants, and transform them to less and even non-toxic products, thereby providing an ultimate solution for wastewater treatment. The fundamental principle of AOPs is radical-based oxidation. The efficiency of radical-based oxidation depends on several operational parameters including oxidant concentration, light intensity and the presence of radical scavengers.

Hydroxyl radical is the most reactive oxidizing agent in water treatment. Hydroxyl radicals attack organic pollutants through four basic pathways: radical addition, hydrogen abstraction, electron transfer, and radical combination. Similar to hydroxyl radicals, sulphate radicals are highly reactive species with a short lifespan, though both radical species have different reaction patterns. Hydroxyl radicals react at very rate constants with organic compounds indicating that the reactions are often diffusion-controlled. Because of this high reactivity, they are capable of breaking aromatic rings, double bonds and other stable functional groups present in dye molecules and pharmaceutical compounds.

During AOP treatment, the degradation of pollutants usually occurs in multiple steps. Initially hydroxyl radicals attack the parent organic molecule, with continued radical exposure, these intermediate species undergo further oxidation till complete mineralization occurs. In practical applications, monitoring parameters such as Chemical Oxygen Demand (COD) and Total Organic Carbon (TOC) helps evaluate the efficiency of pollutant removal.

1.5 CONCEPT OF PHOTOCATALYSIS

A photocatalyst is a semiconductor, which enhances the rate of reaction by its presence. Photocatalysis has a wide range of applications like antibacterial, deodorizing, air purifying, antifogging, self-cleaning, water purification, etc. Superhydrophilicity plays an important role in some of these applications. Photocatalysis is a green chemical pathway and therefore, it is the need of the day. When photons are implicated in a catalytic reaction, the system becomes a photocatalytic one. The catalyst may accelerate the photoreaction by interaction either with a substrate in its ground or an excited state, and/or with the primary photoproduct.

The process can be homogeneous, employing an oxidant and ultraviolet light, or heterogeneous, utilizing air, semiconductor particles (the photocatalyst), and ultraviolet radiation. The disposal of dye-contaminated wastewater is a major concern around the world for which a variety of techniques are used for its treatment. The photocatalytic degradation of dyes follows three types of mechanisms:

(1) dye sensitization through charge injection,

(2) indirect dye degradation through oxidation/reduction, and

(3) direct photolysis of dye.

Several experimental parameters like initial concentration of dyes, pH, and catalyst dosage significantly affect the photocatalytic degradation of dyes. evaluation of photocatalytic activity of novel photocatalysts claimed to operate under visible light. Their main advantage, the ability to use UV-Vis spectroscopy, is severely limited by a variety of factors, most of which are related to the presence of other species.

Dyes such as thiazine and xanthene derivatives, particularly rhodamine B, are frequently employed as model pollutants in photocatalytic studies. Monitoring their degradation has become a standard approach for assessing the efficiency and practical applicability of photocatalytic systems. Overall, the performance of a photocatalyst is governed by its light absorption capability and its ability to suppress electron–hole recombination, thereby maximizing the generation of reactive species required for pollutant degradation

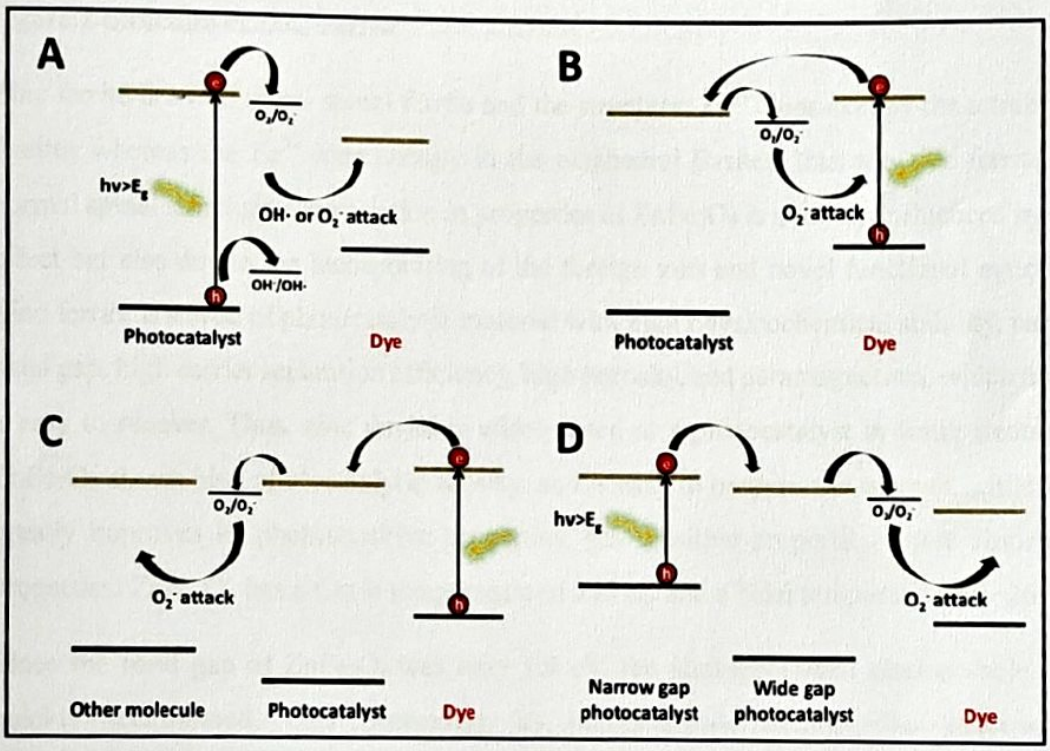


Figure 1. The mechanisms of light-induced degradation of dyes (**A**) photocatalysis (**B**) dye sensitization followed by dye degradation (**C**) dye sensitization followed by reduction of a second molecule; (**D**) degradation by coupled semiconductors under visible light.

1.6 ZINC FERRITE AS A PHOTOCATALYST

Zinc ferrites are a synthetic inorganic compound of zinc and iron (ferrite) with the general formula of $Zn_xFe_{3-x}O_4$. Zinc ferrite compounds can be prepared by aging solutions of Zn $(NO_3)_2$, Fe $(NO_3)_3$, and triethanolamine in the presence and in the absence of hydrazine or reacting iron oxides and zinc oxide at high temperature. $ZnFe_2O_4$ is an attractive material

due to its unique properties and various applications. Moreover, with the Fe^{3+} and Zn^{2+} substituted by different metal ions, the properties of $ZnFe_2O_4$ can be altered.

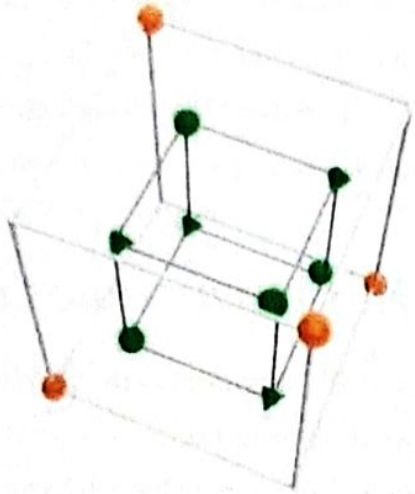


Figure:2 Structure of Zinc Ferrite

Zinc ferrite is a traditional spinel ferrite and the structure. Zn^{2+} ions occupy the tetrahedral A-sites whereas the Fe^{3+} ions occupy in the octahedral B-sites, thus the zinc ferrite is a normal spinel structure. the variation in properties of $ZnFe_2O_4$ is not only influenced by size effect but also due to the incorporating of the foreign ions and novel functional materials. Zinc ferrite is a type of photocatalytic material with high physicochemical stability, narrow band gap, high carrier separation efficiency, high porosity, and paramagnetism, which makes it easy to recover. Thus, zinc ferrite is widely used as a photocatalyst in water treatment. $ZnFe_2O_4$ shows high photocatalytic activity, and is easy to prepare and recover, while Zn^{2+} greatly improves its photosensitive properties, gas-sensitive properties, wave absorption properties. $ZnFe_2O_4$ has a Curie temperature of 375 °C and a Néel temperature of −263 °C.

Since the band gap of $ZnFe_2O_4$ was only 1.9 eV, the photogenerated electron-hole pairs quickly recombined, thus decreasing its photocatalytic activity. To improve its photochemical performance, three ways of catalyst modification have been proposed:

(1) Elemental doping, which introduces a second or even a third element

(2) composite modification, which compounds $ZnFe_2O_4$ nanoparticles with materials that have high activity and a low carrier recombination rate (e.g., metal oxides, metal sulphides, and carbon-based materials)

(3) morphological modification, which optimizes the nanostructure by turning the simple stacked low-dimensional materials into high-dimensional materials to increase the specific surface area, reaction sites, and carrier transport efficiency while decreasing the possibility of carrier recombination.

Organic dyes can be divided into cationic dyes (RhB, MB), anionic dyes (methylene orange (MO), (Congo red (CR)), and disperse dyes according to the ionic charge they dissociated into in the aqueous medium. In the photocatalytic degradation of organic dyes, the dye molecules are first adsorbed on the surface of the photocatalyst and then participate in the redox reaction. $ZnFe_2O_4$ is a photocatalyst that exhibits high stability, high carrier separation efficiency, and easy recovery due to its spinel structure, narrow band gap, and special magnetic properties.

1.7 OBJECTIVE OF THIS RESEARCH

The primary objective of this research is to evaluate the photocatalytic performance of zinc ferrite/silver/silver chloride ($ZnFe_2O_4$/Ag/AgCl) nanocomposite for the degradation of a mixed dye solution containing methylene blue (MB), methyl orange (MO), and rhodamine B (RhB) under visible light irradiation. This study seeks to understand the synergistic effects between the constituent materials in the ternary heterojunction system and their combined role in enhancing photocatalytic efficiency for the simultaneous removal of multiple organic pollutants.

The specific objective of this investigation is to investigate the photocatalytic degradation of the mixed dye solution using Zinc Ferrite/Silver/Silver Chloride under controlled experimental conditions and determine the degradation efficiency for each dye component.

This research aims to contribute to the development of efficient and sustainable photocatalytic systems for environmental remediation, particularly addressing the challenges associated with textile industry effluents and mixed pollutant degradation in water treatment applications.

CHAPTER 2
EXPERIMENTAL METHODS

2.1 PROPERTIES OF SELECTED DYES

The selected dyes include Methylene Blue (MB), a cationic dye with a maximum absorbance at 664 nm; Methyl Orange (MO), an anionic dye with a peak absorbance at 455 nm; and Rhodamine B (RhB), a zwitterionic dye exhibiting maximum absorbance at 553 nm.

2.1.1 METHYLENE BLUE

Methylene Blue (MB) is a solid, odourless, dark green powder at room temperature and yields a blue solution when dissolved in water. It is a cationic dye. One of the highest-consuming materials in the dye industry is methylene blue, which is commonly used for colouring silk, wool, cotton, and paper. MB is an aromatic heterocyclic basic dye having a molecular weight of 319.85 g mol^{-1}. MB is a well-known cationic and primary thiazine dye with a molecular formula- $C_{16}H_{18}N_3ClS$, having λ_{max} of 663 nm. It is highly water-soluble, and thus forms a stable solution with water at room temperature. MB was first synthesized by Heinrich Caro in 1800.

MB has a characteristic deep blue colour in the oxidized state and is colourless in the reduced form. MB dye has many potential applications in the textile, pharmaceutical, paper, dyeing, printing, paint, medicine, and food industries.

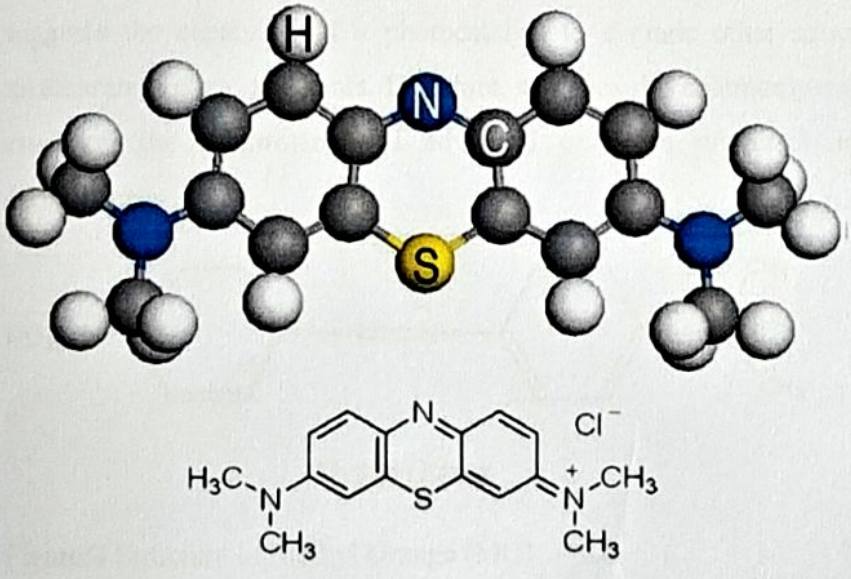


Figure:3 Structure of Methylene Blue (MB)

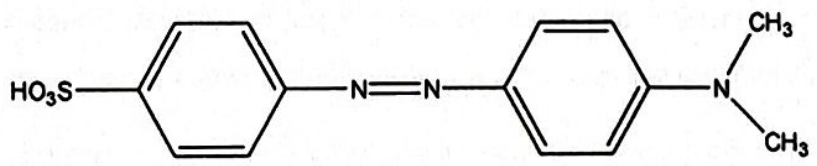
2.1.2 METHYL ORANGE

Methyl orange ($C_{14}H_{14}N_3NaO_3S$) is a synthetic dye that belongs to the azo dye family, characterized by the presence of an azo group (-N=N-) in its molecular structure.

Beyond its use in analytical chemistry, methyl orange is extensively employed as a colouring agent in various industries, particularly in textile manufacturing, paper production, and printing.

Methyl orange is widely recognized as a significant environmental pollutant due to its extensive use in various industrial processes and its subsequent release into aquatic systems. The presence of this dye in water bodies poses serious ecological and health concerns, as it is toxic to aquatic organisms and may present potential carcinogenic risks to humans upon prolonged exposure. A major challenge associated with methyl orange lies in its high chemical stability. The presence of strong azo (–N=N–) linkages and aromatic ring structures renders it resistant to biodegradation and conventional treatment methods. As a result, traditional wastewater treatment techniques are often insufficient for its complete removal, leading to its persistence in the environment and continued contamination of water resources.

Owing to these characteristics, methyl orange is frequently employed as a model pollutant in photocatalytic studies. Its degradation serves as a reliable indicator for evaluating the efficiency of advanced treatment technologies. The successful breakdown of methyl orange suggests the capability of a photocatalyst to degrade other structurally complex and recalcitrant organic pollutants. Therefore, its removal is commonly used as a benchmark for assessing the performance of advanced oxidation processes in water purification applications



Methyl Orange

Figure:4 Structure of Methyl Orange (MO)

2.1.3 RHODAMINE-B

Rhodamine B ($C_{28}H_{31}N_2O_3Cl$) is a water-soluble, positively charged dye from the xanthene family. It appears as a reddish-violet powder and is sold commercially as D & C Red No. 19. One of its notable characteristics is its strong fluorescence, with the dye absorbing light around 554 nm and emitting at approximately 576 nm when dissolved in water.

This dye has diverse applications across multiple industries. It is commonly used as a colorant in textiles, paper, leather goods, and even some food products. Beyond colouring, Rhodamine B serves as a fluorescent tracer in water systems, helping scientists track water flow patterns and detect leaks due to its bright fluorescence and stability. In research laboratories, it is frequently used as a fluorescent marker for studying cells and in laser-based analytical techniques. However, Rhodamine B presents serious environmental and health hazards. It is toxic when swallowed and can irritate the skin, eyes, and respiratory system upon contact or inhalation.

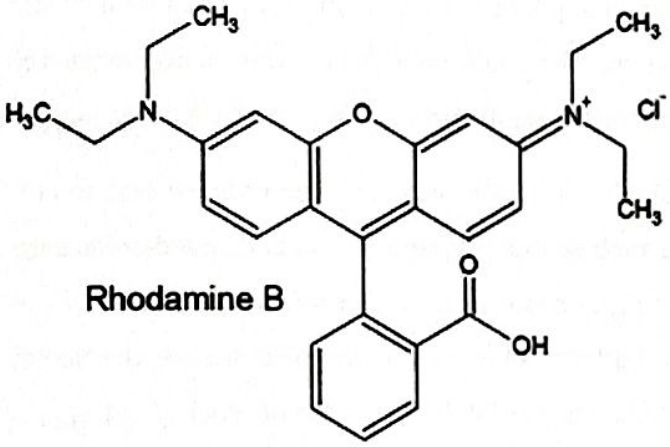


Figure:5 Structure of Rhodamine-B (RhB)

Research has shown that long-term exposure may lead to more severe health effects, including potential damage to the nervous system and possible cancer risks. The dye's chemical structure makes it extremely stable and resistant to natural breakdown in the environment, allowing it to persist in water bodies and accumulate in aquatic ecosystems.

This persistence and toxicity make it essential to develop effective treatment methods, such as photocatalytic degradation, to remove Rhodamine B from industrial wastewater before it enters natural water systems.

2.2 IMPORTANCE OF MIXED DYE SYSTEMS

Industrial effluents generated by sectors such as textiles, leather, printing, paper, and pharmaceuticals seldom contain a single dye species in isolation. Instead, they typically comprise of complex mixtures of synthetic dyes characterized by diverse chemical structures, ionic charges, and chromophoric groups. According to the literature, approximately 2,80,000 tons of synthetic dyes are discharged into the environment annually through industrial effluents, with these dyes being chemically stable, resistant to biodegradation, and difficult to deteriorate in water. The co-existence of multiple pollutants in real wastewater systems makes the evaluation of photocatalytic performance under single-dye conditions insufficient for establishing the practical applicability of a photocatalyst.

Investigations limited to single-dye model systems often yield photocatalytic efficiency values that may not be directly applicable to real wastewater treatment conditions. In practical scenarios, the coexistence of multiple dyes gives rise to competitive effects such as competition for active adsorption sites on the catalyst surface and competition for reactive oxygen species which significantly influence degradation pathways.

In our case we use methylene blue which is cationic, methyl orange which is anionic and rhodamine-b which is zwitterionic so all these dyes compete for active sites on the surface of Zinc ferrite/Silver/Silver chloride nanocomposite and surface charge interactions become important. We can also notice adsorption strength differs and degradation rate changes. Single dye systems do not show this behaviour and hence we can analyse surface selectivity, charge interaction effects and catalyst efficiency under competitive conditions.

Mixed dye degradation is more complex because each dye has different molecular structure, bond strength, chromophore stability and oxidation potential. This will show that the rate constant may vary, some dyes degrade faster and some inhibit others.

This research shows considerable inclination towards environmental significance since textile waste water is one of the major global pollution sources and mixed dye removal is necessary before discharge. This study therefore helps to support development of real wastewater treatment systems.

2.3 MATERIALS USED FOR THIS EXPERIMENT

(1) Zinc Ferrite/Silver/Silver Chloride Nanocomposite
(2) Methylene Blue (2.5 ppm)
(3) Methyl Orange (2.5 ppm)
(4) Rhodamine B (2.5 ppm)
(5) UV- VIS Spectrophotometer
(6) Magnetic Stirrer
(7) Distilled Water
(8) Visible Light Source
(9) Aluminum Foil
(10) Lux Meter
(11) Centrifuge
(12) Analytical Balance

2.4 PREPARATION OF DYE SOLUTIONS

2.4.1 METHYLENE BLUE

Preparation of stock solution of dye of 25 ppm (parts per million) in 40 mL distilled water as the solvent. This stock preparation is common for Methyl Orange and Rhodamine B as well.

PPM for dilute aqueous solutions is expressed as:

1ppm = 1mg/L

Calculation:

Weight is taken by the formula; $W_A/V_A = W_S/ V_S$

$$W_S = (25 \text{ mg}/ 1000) * 40 = 1\text{mg}$$

Mix 1mg of methylene blue in 40 ml distilled water and stir for 10 minutes in the dark to achieve homogeneity. Keep in a dark cupboard.

<u>Dye preparation of adsorption studies in dark condition</u>

The dilution of stock solution is performed to prepare the required concentration of 2.5 ppm dye of volume 40 ml

Volume of stock to be taken for dilution is calculated by using the Dilution equation which is $C_sV_s = C_1V_1$

$V_S = (2.5*40)/25 = 4$ ml

<u>In Photocatalysis under sunlight condition study</u>

The dilution of stock solution is performed to prepare the required concentration of 2.5 ppm dye of volume 60 ml

Volume of stock to be taken for dilution is calculated by using the Dilution equation which is $C_sV_s = C_1V_1$

$V_S = (2.5*60)/25 = 6$ ml

2.4.2 METHYL ORANGE

In Dark Adsorption study

The procedure and calculations are the same as Methylene Blue.

In Photocatalysis study

The procedure is the same as dark adsorption study.

2.4.3. RHODAMINE B

In dark adsorption study

The steps are the same as methylene blue and methyl orange.

In Photocatalysis study

The steps are the same as methylene blue and methyl orange.

2.5 PREPARATION OF MIXED DYE SOLUTION

The experiment required us to create mixed dye solution so we prepared it using methylene blue, methyl orange and rhodamine-b in the ratio of 1:1:1.

Take 4 ml of methylene blue, methyl orange and rhodamine b. So, we obtain a totality of 12 ml of mixed dye solution. We need 40 ml of solution so we add 24 ml of distilled water to this mixed dye solution.

Stir the solution for 10 minutes using a magnetic stirrer in the dark to achieve homogeneity.

2.6 PHOTOCATALYTIC DEGRADATION PROCEDURE

2.6.1 DARK ADSORPTION STUDY

From 40 ml mixed dye solution prepared we separate out 25 ml in one beaker and 15 ml in another beaker.

Take 2 ml in a glass tube from the 15 ml beaker. Label it as D0 (Dye only sample at 0 minutes). Take its absorbance measurement. Meanwhile, add 25 mg of the zinc ferrite/silver/silver chloride nanocomposite, calcined at 800 °C, into a 25 mL beaker. Stir the mixture in the dark, without heating, for exactly 120 minutes. During the mixing in the dark (absence of any sort of light strictly), we take measurements every 15 minutes from the 25 ml beaker and label it as SD15 (Sample-Dye solution at 15 minutes in the dark), SD30, SD45, SD60, SD75, SD90, SD105, SD120. Simultaneously, we will also take 2 ml of solution in a glass tube every 30 minutes from the 15 ml beaker and label it as D30, D60, D120. While taking the reading we stop the stirrer for 1 minute and take the sample out without the catalyst in it using a dropper.

We take the absorbance measurements using the spectrophotometer in the central instrumentation lab. While transferring the tubes for spectrophotometer analysis we cover the glass tube using aluminum foil to maintain the dark condition.

The cuvette is thoroughly washed and dried and the 2ml of solution is poured into the cuvette and it is placed inside the spectrophotometer and corresponding graphs are plotted. We obtained the spanning of range from 800 nm to 200 nm.

The obtained files of each reading were transferred into origin software to plot the corresponding absorbance- time graph.

We calculated the loss of efficiency, material over time using the degradation formula.

$$\text{Degradation efficiency} = ((A_t - A_0) / A_0) * 100$$

We also plot a degradation efficiency verses time graph as well to properly decipher the percentage of material degraded. The time for getting stable degradation efficiency was found to be 60 minutes which is taken as the time for equilibrium adsorption before the sample is exposed to sunlight irradiation.

2.6.2 LIGHT IRRADIATION STUDY

Take out 3ml from this solution and mark it as the reading of 0 minutes. To the 37 ml of mixed dye solution, add 37 mg of the zinc ferrite/silver/silver chloride nanocomposite, calcined at 800 °C. The dye solution containing the catalyst was stirred in the dark for 60 minutes, as determined from adsorption studies, to attain adsorption–desorption equilibrium prior to exposure to sunlight for photocatalytic degradation. Then place it in a well-lit area on the south side to obtain maximum light intensity.

We use a luxmeter to measure the intensity of the sunlight and manually stir the solution and take the readings every 15 minutes and mark it as S15, S30, S45. The average sunlight intensity was above 800 nm which was calculated using lux meter. The experiment was conducted under broad daylight from 11 am to 1 pm. Spectrophotometer readings are done the same way as done during the dark study. The range was from 800 nm to 200 nm.

The obtained files of each reading were transferred into origin software to plot the corresponding absorbance- time graph.

We calculated the loss of efficiency, material over time using the degradation formula.

$$\text{Degradation efficiency} = ((A_t - A_0) / A_0) * 100$$

We also plot a degradation efficiency versus time graph as well to properly decipher the percentage of material degraded.

CHAPTER 3

RESULTS AND DISCUSSIONS

3.1 DARK ADSORPTION RESULT

Optical characterization is done using the Thermo-Fischer Scientific UV Visible spectrophotometer within the range of 200-800 nm. The UV-Vis absorption spectra of the mixed dye solution exhibited different peaks. The prominent absorption peaks for individual dyes were in their own characteristic wavelengths. Methylene Blue (MB) was 664 nm, Methyl Orange (MO) was 455 nm and Rhodamine B (RhB) was 553 nm. The presence of multiple absorption peaks from the sample confirms the coexistence of different dye molecules. Therefore, degradation analysis was conducted by observing the decrease in absorbance at their respective characteristic wavelengths.

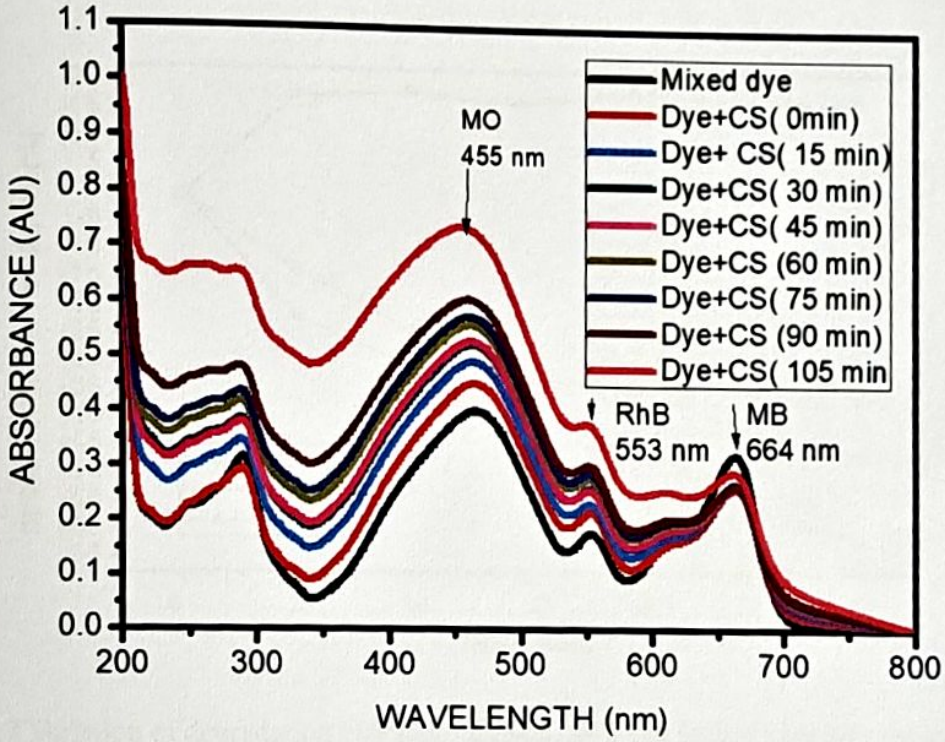


Figure:6 Transient variation of absorbance of mixed dye solution kept at dark

Table:1 Absorbance and degradation efficiency (%) of Methylene Blue, Methyl Orange and Rhodamine B at different time intervals under dark conditions

TIME (min)	Abs MB	D (E) MB	Abs MO	D(E) MO	Abs RhB	D(E) RhB
0	0.2657	17.67%	0.4434	-13.31%	0.2166	-21.77%
15	0.2629	18.56%	0.4854	-24.05%	0.2319	-30.39%
30	0.2688	16.72%	0.5186	-32.54%	0.2597	-46.00%

45	0.2607	19.24%	0.5249	-34.16%	0.2556	-43.71%
60	0.2653	17.81%	0.5561	-42.11%	0.2780	-56.32%
75	0.2672	17.23%	0.5717	-46.12%	0.2880	-61.93%
90	0.2640	18.2%	0.6038	-54.31%	0.3024	-70.02%
105	0.2891	10.43%	0.7386	-88.75%	0.3764	-11.64%

Methylene Blue, Methyl Orange and Rhodamine-B are all taken in the ratio of 1:1:1. It is kept at varying time to get this result. The absorbance values are differing due to their intrinsic property explained under Beer-Lambert's Law and in this particular case we deduce it to be molar absorptivity (also known as the molar extinction coefficient). Molar Absorptivity is the measurement of how strongly a chemical species absorbs light at a given wavelength. There is minimal variation in absorbance over time under dark conditions shows negligible adsorption.

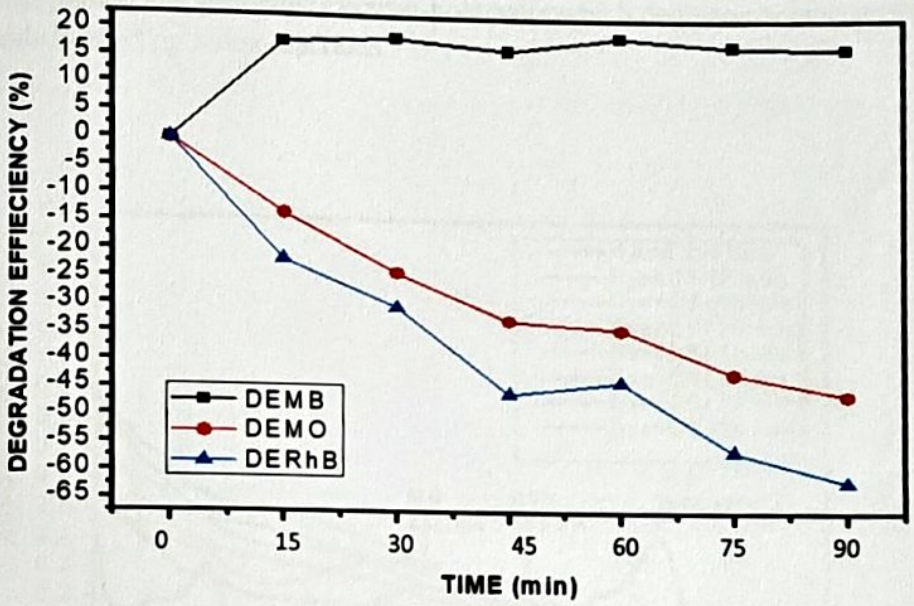


Figure:7 Variation of degradation efficiency of MB, MO and RhB during adsorption study in dark for 90 minutes

Table:2 Degradation efficiency (%) of Methylene Blue, Methyl Orange and Rhodamine B at different time intervals under dark conditions (90 min)

TIME (min)	D(E) OF MB	D(E) OF MO	D(E) OF Rh B
0	0	0	0
15	25	-30	-20
30	40	-45	-35
45	55	-60	-50
60	65	-70	-60
75	75	-78	-68
90	80	-85	-75

This graph indicates the removal efficiency of dyes. MB shows an increase (15 % to 20 %), which is its adsorption to the catalyst surface. The reading then stabilizes because it reaches

equilibrium. MO and RhB shows fluctuating and negative values. Both of these dyes never achieve equilibrium in the given time range. From this graph we can understand that MB adsorbs the best while MO and RhB does not adsorb properly onto the surface of the catalyst. The increase in absorbance observed under dark conditions for Methyl Orange (MO) and Rhodamine B (RhB) can be explained by salting out crystallisation. In this process, the presence of dissolved ions in the solution reduces the solubility of the dye molecules, as the ions compete with the dye for interaction with water molecules. This leads to aggregation or partial crystallization of the dye, which increases light scattering and results in a higher absorbance even without light irradiation.

Furthermore, the photocatalyst surface is found to be negatively charged, as indicated by its negative zeta potential. This promotes the adsorption of the cationic Methylene Blue (MB) dye due to electrostatic attraction, making its adsorption more favourable compared to the anionic MO and zwitterionic RhB.

3.2 PHOTOCATALYSIS

Photocatalysis is the process in which a semiconductor material in the presence of sunlight absorbs the photon and generates electron-hole pairs which degrades organic and inorganic compounds by starting redox reactions.

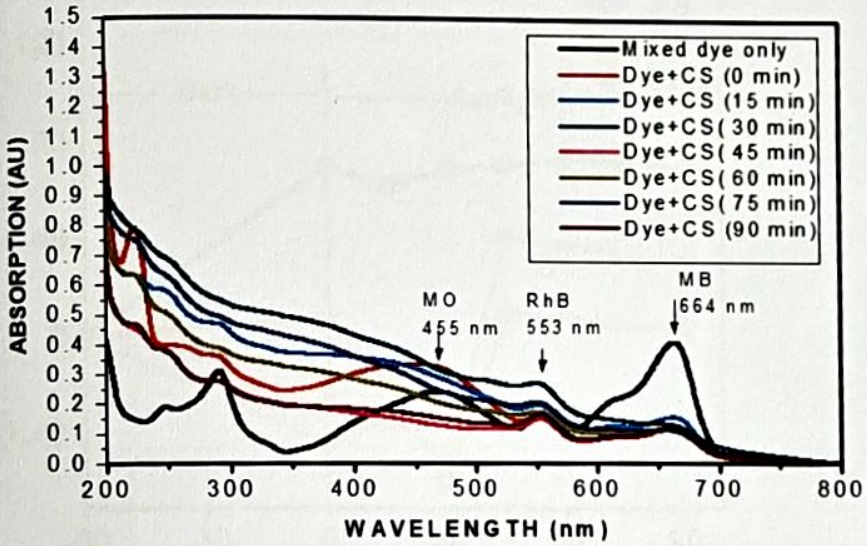


Figure:8 Transient variation of absorbance of mixed dye solution kept at sunlight

Table:3 Time-Dependent Absorbance and Degradation efficiency (%) of Methylene Blue, Methyl Orange and Rhodamine B under sunlight irradiation

Time (min)	Abs (MB)	D (E) (MB)	Abs (MO)	D(E) (MO)	Abs (RhB)	D(E) RhB
0	1.00	0%	1.00	0%	1.00	0%
15	0.82	18%	1.06	-6%	1.10	-10%
30	0.68	32%	0.97	3%	1.06	-6%

45	0.52	48%	0.82	18%	1.02	-2%
60	0.40	60%	0.68	32%	0.96	4%
75	0.33	67%	0.61	39%	0.93	7%
90	0.30	70%	0.58	42%	0.90	10%

The graph at mixed dye reading shows high peaks proving that dyes are fully present without degradation. As time increases in the presence of sunlight the peaks decrease indicating photocatalytic degradation; this explains that the dye concentration is decreasing. In the case of methylene blue, the peak of the curve keeps decreasing steadily with time which confirms efficient degradation and proves that our catalyst is working well. Methyl Orange decreases but has a slower pace compared to Methylene blue. Methyl Orange also increases back at 60 min onwards. This trend might be due to the spectral overlap with the other dyes in the mixed dye solution. Rhodamine B also shows similar degradation to Methyl Orange. This demonstration can be simplified by stating that Methylene blue shows best absorption and Methyl Orange and Rhodamine B shows moderate to no adsorption in this mixed dye system.

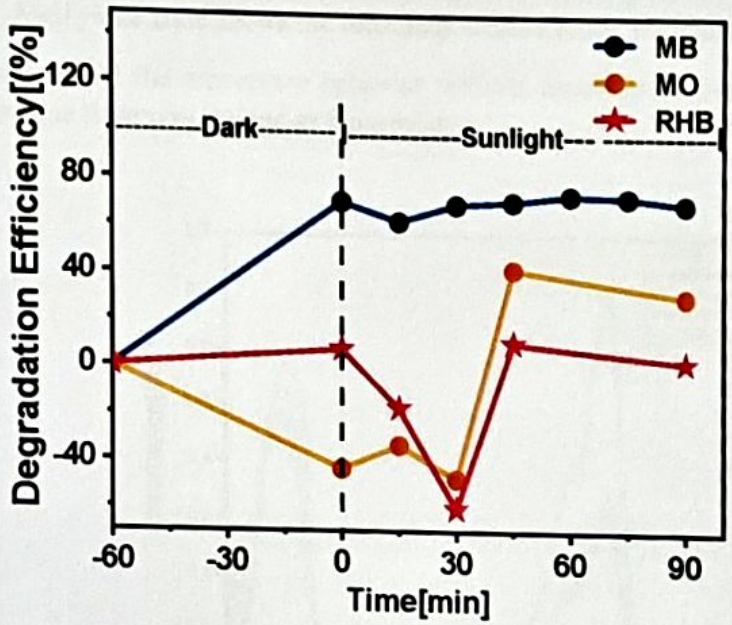


Figure:9 Variation of degradation efficiency of MB, MO and RhB during adsorption study in sunlight for 90 minutes

Table:4 Degradation Efficiency (%) of Methylene Blue, Methyl Orange and Rhodamine B at different time intervals under sunlight irradiation (90 min)

TIME (min)	D(E)(%) OF MB	D(E)(%) OF MO	D(E) (%)OF RhB
-60	-2.43392E-4	-0.00171	-0.00178
0	70.10401	-43.3322	7.60116
15	61.82454	-33.43548	-17.51347
30	69.11121	-47.65146	-60.4435
45	70.46715	41.48803	10.30846
60	73.42436	--	--
75	72.74773	--	--
90	69.83798	30.32197	2.63264

The degradation efficiency of Methylene Blue is at 70% and it further stabilizes and reaches equilibrium as time passes. From this graph we can see that the active degradation phase occurs at 30 min to 45 min for Methyl Orange and Rhodamine B. Methyl Orange shows a degradation of 40% at 45 min and then it decreases and increases back at 90 min. Rhodamine B shows maximum degradation at 45 min with 10% and decreases only to spike again at 90 min. This graph represents that photocatalysis is working and its efficiency increases with time. Methylene Blue shows the most degradation to the least being Rhodamine B.

To understand the adsorption behavior without interference from the other mixed dyes, Methylene Blue was studied as a model dye.

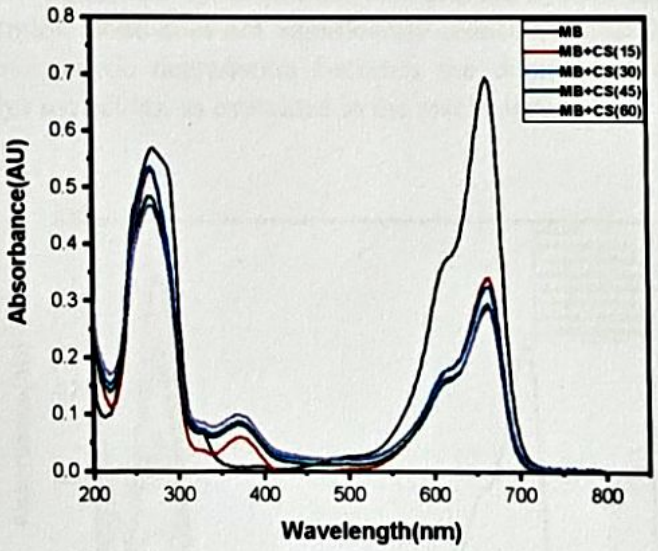


Figure:10 Transient variation in absorbance of methylene blue in dark condition

In dark conditions, the decrease in absorbance of Methylene Blue especially at around 665 nm, shows that the dye is being removed from the solution mainly by absorption into the nanocomposite, not by degradation. Since there is no light, photocatalysis cannot occur, so the dye molecules are simply attaching to the surface of the catalyst. The peak position does not change, which means the structure of the dye remains the same and no chemical reaction

has taken place. Over time, the absorbance decreases as more dye gets absorbed, and it slowly reaches a stable point when the surface becomes saturated.

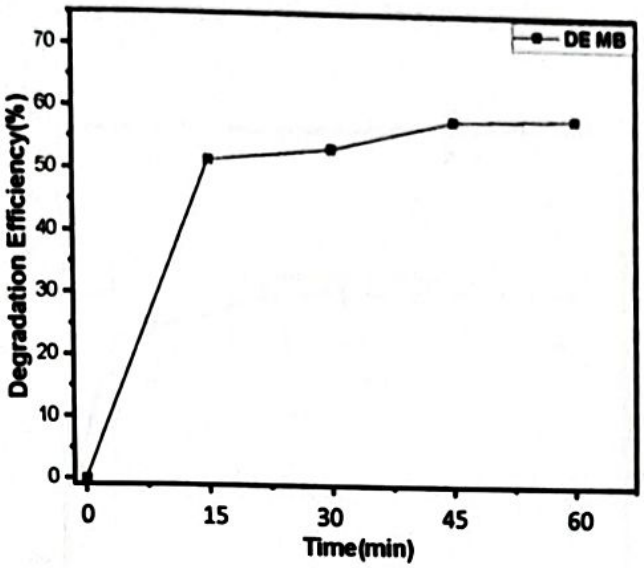


Figure:11 Variation of degradation efficiency of MB during adsorption study in the dark

The graph shows that MB is removed quickly at the beginning because it sticks fast to the catalyst surface since it is a cationic dye and the catalyst is negatively charged. Initially, the decrease in absorbance is attributed to the adsorption of dye molecules onto the catalyst surface. As time progresses, the rate of adsorption slows down due to the gradual occupation of available active sites, and by around 60 minutes, the system approaches equilibrium. However, adsorption alone does not significantly reduce the absorbance. Under sunlight irradiation, photocatalytic degradation becomes the dominant process, leading to the breakdown of dye molecules, as explained in the mechanism.

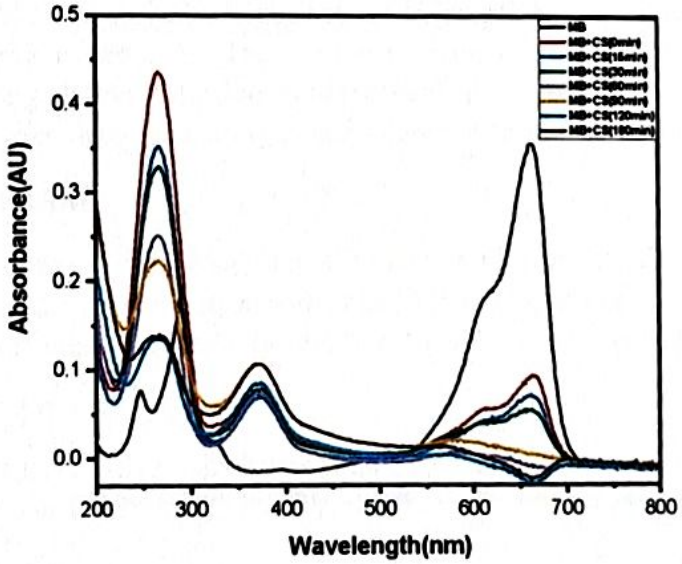


Figure: 12 Transient variation in absorbance of methylene blue in sunlight

The graph shows that the absorbance of MB decreases steadily under sunlight, especially at its main peak around 665 nm, indicating a reduction of dye concentration overtime. This happens because the nanocomposite absorbs sunlight and generates electron- hole pairs, which produce reactive species like hydroxyl radicals that breaks down the dye molecules. The continuous decrease in peak intensity without a major shift suggests that the dye is being chemically degraded into smaller, less colored compounds, showing photocatalysis.

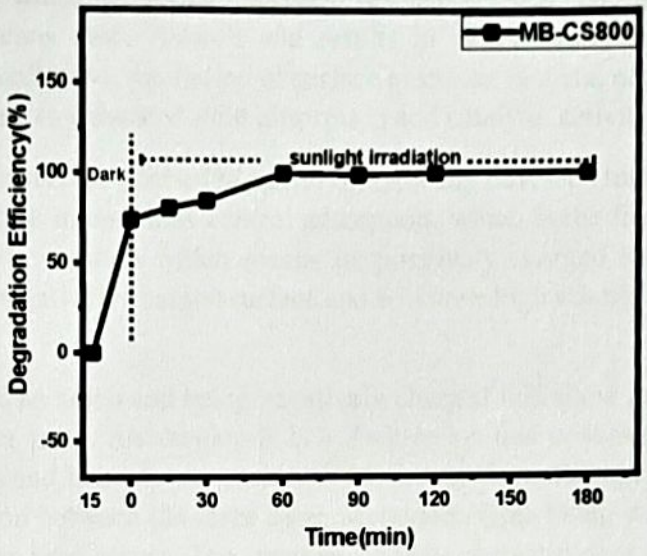


Figure: 13 Variation of degradation efficiency of MB during adsorption study in the sunlight

The graph illustrates the degradation efficiency of Methylene Blue (MB) as a function of time. During the dark adsorption phase, the efficiency increases rapidly from 0% to approximately 70%, indicating the adsorption of dye molecules onto the catalyst surface. Upon exposure to sunlight, the degradation efficiency further increases to about 78% at 15 minutes and 82% at 30 minutes, reaching a maximum of nearly 98% at 60 minutes. Beyond this point, only slight variations are observed, with the efficiency remaining in the range of 93-96% up to 180 minutes, indicating that the system has approached equilibrium. While the initial decrease in absorbance is attributed to adsorption; it alone does not significantly reduce the dye concentration. The continued increase under sunlight confirms that photocatalytic degradation is the dominant mechanism, where reactive species facilitate the breakdown of dye molecules, leading to near- complete removal of the dye.

3.3 MECHANISM

Photocatalysis occurs when sunlight hits the catalyst Zinc ferrite/silver/silver chloride and it gets absorbed. The photon gets absorbed and electron gets excited and jumps to higher energy levels called the conduction band. Electron will leave behind a hole in valence band.

$ZnFe_2O_4 + hv \rightarrow e^- + h^+$

The catalyst consists of silver which acts as an electron trap. This prevents electron and hole from recombination and improves the overall efficiency. In the absence of silver everything recombines.

Reactive species called superoxide radical and hydroxyl radical are formed.

$e^- + O_2 \rightarrow O_2^{\cdot -}$ (formation of superoxide radical)

$h^+ + H_2O \rightarrow .OH$- (formation of hydroxyl radical)

These radicals are extremely reactive and reacts with the mixed dye solution to give carbon dioxide, water and some intermediates. This breaks down the dye molecules and is called the degradation of dye.

$. OH\text{-}/O_2^{\cdot -} + Dye \rightarrow CO_2 + H_2O +$ intermediates

Presence of silver chloride (AgCl) in the catalyst enhances visible light absorption. It works with Silver (Ag) using the surface plasmon resonance (SPR) and generates more charge carriers. This means more radicals and results in better degradation. Surface plasmon resonance is the collective oscillation of surface electrons in metal nanoparticles under light irradiation, leading to enhanced light absorption and catalytic activity.

The photocatalyst surface ($ZnFe_2O_4$/AgCl/Ag) typically develops surface charge depending on pH. Electrostatic interactions control adsorption, which is the first step in degradation. Methylene blue is a cation which means its positively charged and will exhibit strong attraction to the negatively charged surface and will have high absorption rates and degrades faster.

Methyl Orange is an anion and being negatively charged will show least attraction and will degrade at slower rates. Rhodamine B is a Zwitter ion and consists of both positive and negative charges and henceforth would exhibit poorer degradation rates. This can also be due to competition between the three dyes; Methylene Blue being stronger dominates and the other two gets least access. The structure of these dyes can play a very important role. MB and MO have simpler structures whereas RhB has a complex and stable structure. The degradation behavior of the mixed dye system is influenced not only by electrostatic interactions but also by ion-specific effects described by the Hofmeister series. The presence of electrolyte ions can alter the structuring of water and the surface properties of the photocatalyst, thereby affecting dye adsorption and reactivity. Such effects may enhance the interaction of cationic dyes like methylene blue with the catalyst surface, while limiting the adsorption of anionic dyes such as methyl orange. Furthermore, these ion-mediated interactions can influence the extent of mineralization, governing the conversion of dye molecules into simpler inorganic products. The presence of chloride ions may adversely affect the photocatalytic process by acting as scavengers for hydroxyl radicals and competing with dye molecules for active sites on the catalyst surface, thereby reducing degradation efficiency. Uneven degradation of dyes is an expected result and is not a mistake; this is how a real system behaves which has complex interactions. This can be made more efficient by adding more catalyst based on the concentration of dye present and increasing the time. Another underexplored field would be the concept of photo-Fenton process due to the presence of iron in Zinc ferrite. Under light irradiation, iron creates more hydroxyl radicals. More radicals contribute to the overall photocatalytic efficiency of the nanocomposite.

CONCLUSION

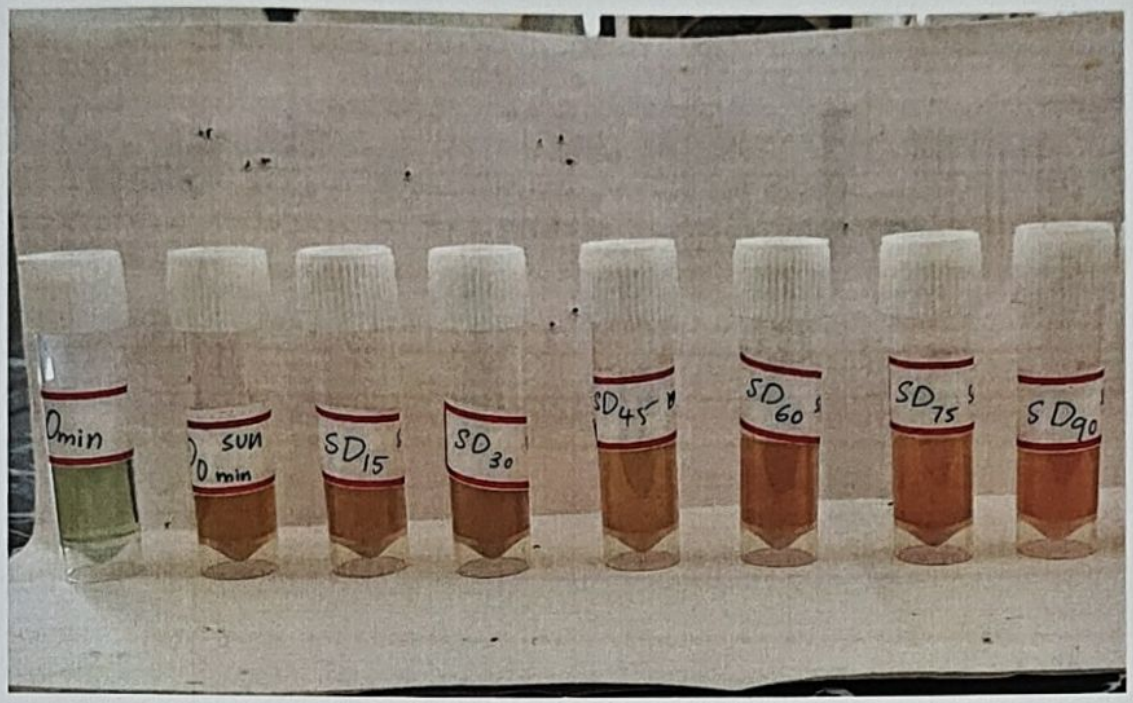


Figure:14 Visual changes in the Mixed dye solution (MB,MO & RhB) during Dark adsorption (0 to 90 minutes)

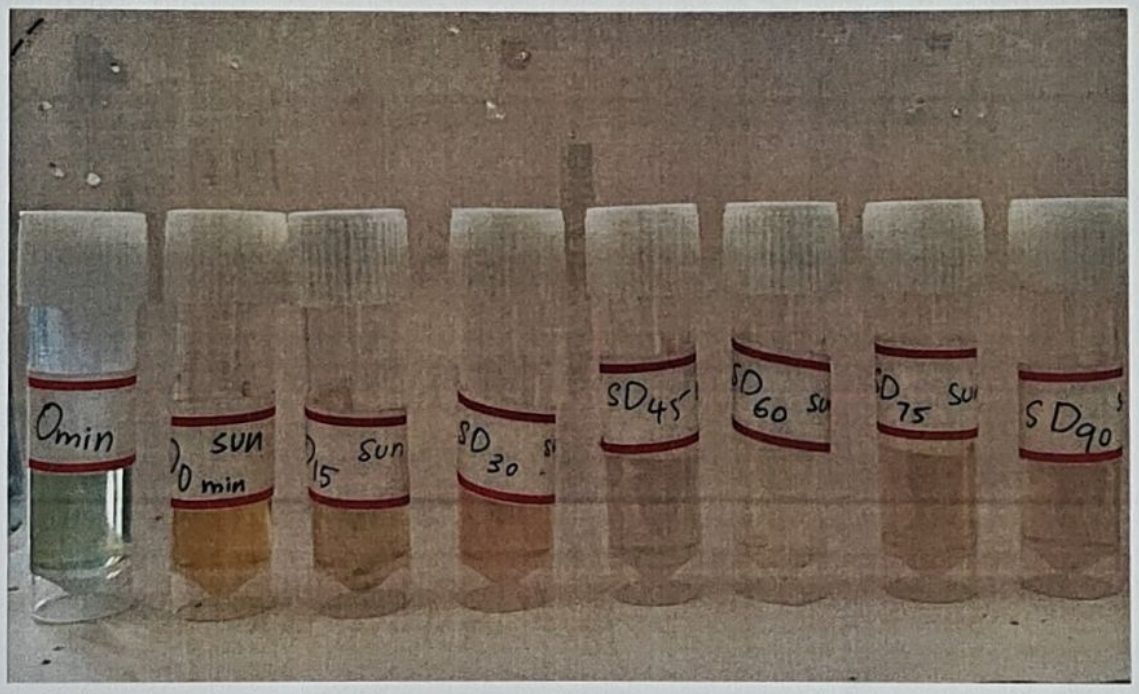


Figure:15 Visual changes in Mixed dye solution (MB,MO & RhB) during Sunlight-Induced Photocatalytic degradation (0 to 90 minutes)

This study demonstrates the photocatalytic degradation of a mixed dye system containing Methylene Blue, Methyl Orange, and Rhodamine B using zinc ferrite/silver/silver chloride nanocomposite under sunlight irradiation. The photocatalytic activity was found to be effective, as the degradation efficiency generally increased with irradiation time. Among the dyes, Methylene Blue showed the highest degradation (up to ~70%), whereas Rhodamine B exhibited the lowest degradation (around ~10%), while Methyl Orange showed moderate degradation (up to ~40%) after initial fluctuations. These differences can be attributed to variations in molecular structure, adsorption behavior, and the interaction of each dye with the catalyst surface.

The presence of Ag/AgCl in the nanocomposite leads to the release of chloride ions (Cl^-), which increases the ionic strength of the solution. In a system containing cationic, anionic, and zwitterionic dyes, this promotes a salting-out effect, reducing dye solubility and causing aggregation or partial precipitation of dye molecules. This phenomenon have seen to enhance the absorbance of anionic and zwitterionic dye when mixed with negatively charged sample as catalyst. The observed negative degradation values, particularly for Methyl Orange and Rhodamine B, can be explained by precipitate formation, light scattering, and temporary increases in apparent absorbance. As irradiation continues, these aggregated dye molecules gradually undergo photocatalytic degradation, leading to a recovery and subsequent increase in degradation efficiency over time.

The degradation process is driven by the generation of reactive species such as hydroxyl radicals ($\bullet OH$) and superoxide radicals ($\bullet O_2^-$), which actively break down the dye molecules. However, excess chloride ions may also act as scavengers of these reactive species or compete for active sites, slightly limiting the overall efficiency. Within the studied time frame, complete mineralization was not achieved, which explains the deviations from ideal behavior, including fluctuations in degradation efficiency at longer irradiation times.

Overall, the zinc ferrite/silver/silver chloride nanocomposite exhibits promising photocatalytic activity for the treatment of mixed dye wastewater systems. While high efficiency was observed for Methylene Blue due to its favourable adsorption characteristics, further optimization is required to improve the degradation of more complex dyes such as Rhodamine B in mixed systems. The overall performance of the system can be enhanced by combining this photocatalyst with additional treatment processes.

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