# ADVANCES IN DYE DEGRADATION

Editors: Paulpandian Muthu Mareeswaran Jegathalaprathaban Rajesh

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# Advances in Dye Degradation (Volume 1)

Edited by

# **Paulpandian Muthu Mareeswaran**

Department of Chemistry College of Engineering Anna University, Chennai - 600025 Tamil Nadu, India

&

# Jegathalaprathaban Rajesh

Department of Chemistry Saveetha School of Engineering Saveetha Institute of Medical and Technical Sciences Saveetha University Chennai – 602 105 Tamil Nadu, India

#### **Advances in Dye Degradation**

#### (Volume 1)

Editors: Paulpandian Muthu Mareeswaran and Jegathalaprathaban Rajesh

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# FOREWORD

Synthetic dyes are a major part of dyes utilized by the textile industry. Organic dyes are the new chemicals that are extensively used in the textile industry nowadays. Most of these dyes are toxic and potentially carcinogenic in nature and pose major threats and create environmental problems. While dyeing, not all the dyes are adsorbed by the fabric materials and a considerable part is left as textile effluent. This effluent will mix with water bodies and create environmental hazards. Therefore, the treatment of textile effluents, especially dye removal, is an imperative step for environmental remediation. The removed dye is also a secondary pollutant to the environmental remediation. The conversion of carcinogenic dye molecules to an environmentally benign molecule is called dye degradation. From the industrial perspective, the process of conversion should be not only efficient but also cost-effective.

The editors have chosen the interesting aspects of dye degradation. The book "Advances in Dye Degradation" highlights the recent advantages of dye degradation pathways. However, without a basic understanding, the advanced perspective will have no meaning. Hence, Volume I of this book series deals with the fundamental aspects. In this work, the authors have discussed the nature of dye molecules, adverse effects, environmental problems caused by the dye molecules and their remediation process. The toxicity of the dyes is discussed in detail, and the application of nanotechnology, electrochemical and biological processes is also discussed. Since the advanced oxidation process is one of the important methods of the dye degradation process, the basic mechanism of photocatalytic dye degradation is also discussed in detail. This book deals with the basics of most of the relevant topics in the field of dye degradation, and it will certainly be useful for students as well as researchers.

C. Stella Department of Oceanography and Coastal Area Studies Thondi Campus Alagappa University Karaikudi – 630 003 India

# PREFACE

Dye degradation is an important step for the sustainable ecofriendly atmosphere in the dyeing and textile industry. The untreated textile effluent provides severe adverse effects on the ecosystem. This book deals with the mitigation of textile effluents. Chapter 1 is a fundamental chapter that describes the nature of dyes, classification, adverse effects and methods of removal of dyes from a bird's eye view. The aim of the first chapter is to give a basic idea of dyes and understand the necessity of textile effluent mitigation with respect to the ecosystem. This chapter also gives an overview of the most prevalent methods used for textile effluent mitigation and degradation processes.

Chapter 2 deals with the toxicity measurements of textile effluents. This chapter deals with the regulation of dyes, and analysis of dyes by means of various methods. This chapter provides a view of toxicological dosages like LD50 and LC50 values of dyes. Also, it provides details about the dose effect and dose response. This chapter also explains how the toxicity is evaluated using live animals. Chapter 3 deals with the microbial degradation of dyes as an overview. This chapter portrays microorganisms as an effective tool to mitigate textile effluents. The biological methods for decolorization and degradation of textile effluent are very successful and have various advantages over traditional procedures. Biological methods for removing toxic textile dyes are both environmentally friendly and cost-effective.

Chapter 4 pitches a platform about the utility of nanotechnology in dye degradation methods. Materials are always efficient, reusable and cost-effective over any other methods used in all the fields of technology. Nanoscience and technology provide effective solutions for various problems, and also have a deep impact in the field of dye degradation and textile effluent mitigation. The nanotechnology itself provides various methods for dye mitigation. This chapter provides an overview of the utilization of nanotechnology for textile effluent problems.

Chapter 5 deals with the electrochemical degradation of synthetic textile dyes from aqueous solution. Electrochemical methods are one of the effective methods for the treatment of effluent water. The CV, UV–Vis and chemical oxygen demand (COD) studies are the important parameters for degradation efficiency. This chapter deals with the process of applying electrochemical methods to textile companies for the mitigation of textile effluents. Chapter 6 also deals with the electrochemical processes of dye mitigation. This chapter concentrates on anodic oxidation processes. The mechanisms of electrochemical oxidation in anodic oxidation processes are explained in detail. The various anodic electrodes towards dye mitigation and degradation, their mechanism of action and efficiencies are reviewed.

Chapter 7 explains in detail the Z-scheme, which is a fundamental phenomenon for the photocatalytic degradation of dyes using various photoactive materials. Therefore, this Z-scheme has an impact on environmental remediation. The photocatalysts designed using Z-scheme have several advantages over the traditional photocatalytic processes, like efficient charge separation and electron transfer. Hence, it renders an efficient redox mechanism for the catalytic materials.

Chapter 8 deals with several photocatalytic materials for dye degradation, like metal oxides, metal sulfides, and metal ferrites. It also discusses with strategies to improve photocatalysts, such as doping the materials. This book strives to give collectively about the nature of dyes, the adverse effects of dyes and the basics of degradation methods. The editors aim to format the first volume of this series to pitch a basic idea of dye degradation. The upcoming volumes will develop the basic ideas into target-oriented dye mitigation for a better environment.

#### Paulpandian Muthu Mareeswaran

Department of Chemistry College of Engineering Anna University, Chennai - 600025 Tamil Nadu, India

&

#### Jegathalaprathaban Rajesh

Department of Chemistry Saveetha School of Engineering Saveetha Institute of Medical and Technical Sciences Saveetha University Chennai – 602 105 Tamil Nadu, India

# **List of Contributors**

Arumugam Girija	Department of Chemistry, Velumanoharan Arts & Science College for Women, Ramanathapuram-623504, Tamil Nadu, India	
Bosco Christin Maria Arputham Ashwin	Department of Chemistry, Pioneer Kumarasamy College, Nagarcoil- 629003, Tamil Nadu, India	
C. Christopher	Department of Chemistry, St.Xavier's College, Palayamkottai, Tirunelveli-627 002, India	
Dhanesh Tiwary	Department of Chemistry, Indian Institute of Technology (Banaras Hindu University), Varanasi, 221005, India	
G. Ravi	Department of Physics, Alagappa University, Karaikkudi, Tamil Nadu, India	
Jegathalaprathaban Rajesh	Department of Chemistry, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu, 602 105, India	
Jeyaraj Dhaveethu Raja	Department of Chemistry, The American College, Tallakkulam, Madurai, 625 002, Tamil Nadu, India	
K. Govindan	Environmental System Laboratory, Department of Civil Engineering, Kyung Hee University (Global Campus), Giheung-Gu, Yongin-Si, Gyeonggi-Do, 16705, Republic of Korea	
Karupannan Aravindh	Research Centre, SSN College of Engineering, Kalavakkam, Chennai- 603110, Tamil Nadu, India	
Kiruthiga Kandhasamy	Department of Chemistry, Vellammal College of Engineering and Technology, Madurai-625 009, Tamil Nadu, India	
Murugesan Sankarganesh	Department of Chemistry, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu, 602 105, India	
Nagaraj Revathi	Department of Chemistry, Ramco Institute of Technology, Rajapalayam, Virudhunagar, 626 117, Tamil Nadu, India	
Paulpandian Muthu Mareeswaran	Department of Chemistry, College of Engineering, Anna University, Chennai-600025, Tamil Nadu, India	
Perumalsamy Ramasamy	Research Centre, SSN College of Engineering, Kalavakkam, Chennai- 603110, Tamil Nadu, India	
Poovan Shanmugavelan	Department of Chemistry, School of Sciences, Tamil Nadu Open University, Saidapet, Chennai-600 015, Tamil Nadu, India	
R. Jagatheesan	Department of Chemistry, Vivekanandha College of Arts and Sciences for Women (Autonomous), Elayampalayam, Tiruchengode, Tamil Nadu- 637 205, India	
R. Yuvakkumar	Department of Physics, Alagappa University, Karaikkudi, Tamil Nadu, India	

Sai Sathish Ramamurthy	STAR Laboratory, Central Research Instruments Facility (CRIF), Department of Chemistry, Sri Sathya Sai Institute of Higher Learning, Prasanthi Nilayam, Puttaparthi, Anantapur, Andhra Pradesh, India
Seemesh Bhaskar	STAR Laboratory, Central Research Instruments Facility (CRIF), Department of Chemistry, Sri Sathya Sai Institute of Higher Learning, Prasanthi Nilayam, Puttaparthi, Anantapur, Andhra Pradesh, India Department of Chemistry, Indian Institute of Technology (IIT) Bombay, Powai, Mumbai-400076, Maharashtra, India
Sheeba Daniel	Department of Chemistry, Holy Cross College (Autonomous), Nagercoil-629 004, Tamil Nadu, India
SP. Keerthana	Department of Physics, Alagappa University, Karaikkudi, Tamil Nadu, India
Suresh Kumar Pandey	Department of Chemistry, Indian Institute of Technology (Banaras Hindu University), Varanasi, 221005, India
Venkatesan Sethuraman	Research and Development, New Energy Storage Technology, Lithium- Ion Battery Division, Amara Raja Batteries Ltd, Karakambadi, Andhra Pradesh-517 520, India

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**CHAPTER 1** 

## **Dye Degradation - Basics and Necessity**

Kiruthiga Kandhasamy<sup>1</sup>, Sheeba Daniel<sup>2</sup>, Poovan Shanmugavelan<sup>3</sup> and Paulpandian Muthu Mareeswaran<sup>4,\*</sup>

<sup>1</sup> Department of Chemistry, Vellammal College of Engineering and Technology, Madurai-625 009, Tamil Nadu, India

<sup>2</sup> Department of Chemistry, Holy Cross College (Autonomous), Nagercoil-629 004, Tamil Nadu, India

<sup>3</sup> Department of Chemistry, School of Sciences, Tamil Nadu Open University, Saidapet, Chennai-600 015, Tamil Nadu, India

<sup>4</sup> Department of Chemistry, College of Engineering, Anna University, Chennai-600025, Tamil Nadu, India

**Abstract:** Without colour, life is incomplete. Dye refers to the compounds that give goods their colour. Even though natural dyes have been used for generations, their limitations have led to the development of synthetic dyes. By addressing the history and significance of natural dyes, the limitations of natural dyes, the introduction of synthetic dyes, the negative effects of synthetic dyes, and an overview of several techniques used for the treatment of disposed dyes in the environment, this chapter serves as a foundation for the discussion of the entire upcoming book. The goal of this chapter is to provide a brief overview of the need for and the concept of dye degradation.

Keywords: Colour index, Degradation, Natural dyes, Oxidation, Synthetic dyes.

#### **INTRODUCTION**

Dyes are coloured substances that adhere to the substrate and give items their colour. Otto N. Witt developed a dyeing theory in 1876 that was based on functional groups like auxochrome and chromophore. According to his idea, certain auxochromic groups, which are responsible for dyeing properties, and certain unsaturated chromophoric groups, which are responsible for colour, are present in all coloured organic compounds (also known as chromogens) [1]. Dyes absorb visible wavelength ranges of radiation, and the appearance of colour depends on the wavelength ranges that are both absorbed and reflected. The term "visible" was created since the human eye can perceive light between 380 nm

<sup>\*</sup> Corresponding author Paulpandian Muthu Mareeswaran: Department of Chemistry, College of Engineering, Anna University, Chennai-600025, Tamil Nadu, India; E-mail: muthumareeswaran@gmail.com

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(violet) and 700 nm (red) [2]. Two distinct indices are used to represent the commercial dyes. First The first is called a "colour index generic number" (CIGN), and it is used by businesses. The second one is the colour index constitution number (CICN), which has to do with the chemical makeup of the dye and is primarily employed by producers and academics [3].

Based on the chemical components found in the compounds, which determine the colour of dye, dyes are divided into numerous categories (Fig. 1) [4].



Fig. (1). Classification of dyes.

#### NATURAL DYES

Natural dyes and synthetic dyes are the two main classifications, which are based on their manufacturing techniques. Animals and other plant components, including the root, bark, leaf, flower, fruit, and seed, are used to make natural colours [5]. All civilizations have a very long history associated with the dyeing industry. Chinese dyeing techniques have been used for 5000 years [6]. The Ajantha cave paintings from the Ellora caves in India date to between 600 and 1000 CE, and those at Sittanavasal belong to the seventh century [7, 8]. These paintings are painted with vegetable oil colours, which have been around for more than a thousand years, on lime plaster. In order to categorize natural dyes, several criteria are taken into consideration, including their chemical makeup (anthracenes, carotenoids, xanthophylls, flavonoids, betacyanins, tannis, indigo, and chlorophyll dyes), their sources (animal and plant sources), their application techniques (direct dyes, acidic dyes, and basic dyes), and their colour [4]. The portions of plants from which the colour is derived are used to further categorize

#### Dye Degradation

them. Based on colour, one of the most common classes is made. They are listed in the Colour Index based on their uses and chemical makeup of natural dyes. According on the application category they fall under, natural dyes have their own area in the Colour Index. Most red colour dyes are made from plant bark [9]. The predominant colour that can be derived from most plant parts is yellow.

The plant *lindigofera* tinctoria is the source of the significant blue dye known as indigo (Fig. 2). 6000 years ago, this dye was used for the first time in Peru [10]. Indigo uprising occurred in Bengal, a region of the Indian Subcontinent, as a result of the extensive cultivation of this dye during the colonial era [11]. *Lichens*, which are fungi-algae composite creatures, are some of the organisms used to colour clothes. They come in a variety of colours, including orange, red, pink, and yellow. Because these creatures must be grown in an environment free of pollution and impurities, industrial-scale production is not feasible [12].



Fig. (2). Examples of natural colourants.

#### **ADVANTAGES OF NATURAL DYES**

Natural dyes are made from natural materials and are therefore not bad for the environment. Natural colours are biodegradable and renewable [2]. They can be used without risk, and there are no disposal issues. The plants used to make colours are frequently also used as medicines. Despite being used for generations, many of their therapeutic benefits have only recently come to light [12]. The dye made from henna, walnut, and alkanet, which is high in napthoquinone, also has antibacterial, antifungal, and anti-inflammatory properties. The use of natural

# **Toxicity Analysis of Dyes**

Arumugam Girija<sup>1,\*</sup> and Paulpandian Muthu Mareeswaran<sup>2</sup>

<sup>1</sup> Department of Chemistry, Velumanoharan Arts & Science College for Women, Ramanathapuram-623540, Tamil Nadu, India

<sup>2</sup> Department of Chemistry, College of Engineering, Anna University, Chennai-600025, Tamil Nadu, India

Abstract: In the textile sector, synthetic dyes are crucial. However, dyes pose a serious threat to all organisms because of their toxicity. Environmental concerns have grown over the non-selective and excessive usage of these dyestuffs. These colours have the potential to be harmful in terms of behaviour, biology, chemistry, physicality, and radiation. The toxicity of the dyes can be classified as acute (short-term effect) or chronic (long-term damage). In order to establish criteria for the regulation of dyes when they come into contact with humans and other living things, toxicity analyses of dyes are therefore required. In a toxicology study, the reaction of an organism to a specific dye at different concentrations is compared to the reaction of the same organisms not exposed to the dye. The toxic effects of an experimental substance are revealed by toxicity testing on numerous biological systems. The producers utilize this evaluation to determine the dye's toxicity and whether it has carcinogenic or non-carcinogenic effects.

**Keywords:** Acute toxicity, Chronic toxicity, Dose descriptors, Dose-response relationship, Synthetic dyes, Sub-acute toxicity.

#### **INTRODUCTION**

Dyes are sophisticated organic compounds that are used to colour products in a variety of industries, including the textile, printing, rubber, cosmetics, plastics, and leather industries. Dyes can be derived from synthetic or natural sources. Natural dyes are made without the use of chemicals from naturally existing substances such as plants (such as indigo), insects (such as cochineal beetles), animals (such as shellfish), and minerals (such as ferrous sulphate). Synthetic dyes, such as Congo red and methyl orange are synthesised in laboratories. Due to factors including inexpensive production, simple availability, straightforward application, greater colour stability, resistance to light, pH variations, oxidation,

<sup>\*</sup> Corresponding author Arumugam Girija: Department of Chemistry, Velumanoharan Arts & Science College for Women, Ramanathapuram-623540, Tamil Nadu, India; E-mail: girijabalaji99@gmail.com

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*etc.*, synthetic dyes have attracted a lot of attention and have practically supplanted natural dyes. Both humans and the environment are harmed by synthetic dyes. Lead, mercury, chromium, copper, toluene, benzene, and others are some of the compounds that are present in synthetic dyes [1 - 5]. The human body may suffer from serious consequences if exposed to high concentrations of certain drugs.

They will result in alterations in immunoglobulin levels, allergic dermatoses, and respiratory illnesses. Certain azo dyes may be mutagenic. Even the chemicals used to make colours have been shown to be poisonous, cancer-causing, or occasionally explosive [5 - 8]. For instance, aniline, which is used to make azo colours, is toxic and extremely combustible. Additionally, the chemicals used in the dying process are dangerous. For instance, dioxin may be carcinogenic and affect hormones. Heavy metals including chromium, copper, and lead are inherently carcinogenic. Table 1 lists some of the chemicals used in the dying industry along with their average usage per month [9, 10].

S. No.	Chemicals Used	Quantity Kg/month
1.	Acetic acid	1611
2.	Ammonium sulphate	858
3.	PV acetate	954
4.	Wetting agent	125
5.	Caustic Soda	6212
6.	Softener	856
7.	Organic Resin	5115
8.	Formic acid	1227
9.	Soap	154
10.	Hydro sulphites	6563
11.	Hydrogen Peroxide	1038
12.	Levelling & Dispersing Agents	547
13.	Oxalic acid	471
14.	Polyethylene emulsion	117
15.	Sulphuric acid	678

#### Toxicity Analysis of Dyes

Azo dyes are used extensively in the textile, pharmaceutical, printing, food, and other sectors, making up close to 70% of all synthetic colours. Numerous businesses release toxic effluents that contain azo dyes, which have a negative impact on ecosystem health, soil fertility, aquatic life, and resource availability. They are poisonous to aquatic creatures (fish, algae, bacteria, *etc.*), much like they are to animals (lethal impact, genotoxicity, mutagenicity, and carcinogenicity). They are not easily degradable in the environment, and typical wastewater treatment methods do not eliminate them from sewage. It has long been known that benzidine-based dyes can cause tumours in a variety of laboratory animals and can cause cancer of the urinary bladder in humans [11].

#### TOXICITY

The capacity of a material to harm living things is referred to as its toxicity. Even if a harmful agent is given in extremely small levels, the cells will still suffer long-term damage. Only when the less harmful drug is consumed in high quantities will it have an impact on the organism. As a result, it is important to define a substance's toxicity in terms of the amount of the substance administered or absorbed, the mode of administration (such as injection, ingestion, or inhalation), the time distribution (such as a single dose or repeated doses), the type and severity of the injury, and the amount of time needed to cause the injury [12].

#### **Toxic Effects**

Acute impacts and chronic effects are two different types of toxic reactions that might take place after repeated exposures over an extended period. The level of severity may also vary, and they may only affect a few or all body parts. Acute effects occur quickly following a single encounter; examples include food poisoning, inhaling chlorine spill fumes, *etc.* Examples of immediate consequences include sweating, nausea, paralysis, and death. After repeated exposure over an extended period, chronic impacts may develop; for instance, smoking cigarettes, consuming foods with low amounts of pollutants, inhaling polluted air, *etc.* Chronic impacts include cancer, organ damage, problems getting pregnant, and nervous system impairment, for instance. Chronic impacts may have cancer-causing or non-cancerous effects [13, 14].

#### **Toxicological Field Studies**

The nature of the poisonous substance, the nature of the environmental pollution it causes, and the nature of the species present in the specific environment are all taken into consideration during the toxicological testing on species. Testing wild species kept in cages in field circumstances can potentially result in several issues.

## **Utility of Nanotechnology in Dye Degradation**

Seemesh Bhaskar<sup>1,2</sup> and Sai Sathish Ramamurthy<sup>1,\*</sup>

<sup>1</sup> STAR Laboratory, Central Research Instruments Facility (CRIF), Department of Chemistry, Sri Sathya Sai Institute of Higher Learning, Prasanthi Nilayam, Puttaparthi, Anantapur, Andhra Pradesh, India

<sup>2</sup> Department of Chemistry, Indian Institute of Technology (IIT) Bombay, Powai, Mumbai-400076, Maharashtra, India

Abstract: Plasmonic nanoparticles and low-dimensional graphene-based derivatives are increasingly used for decolourization and degradation of harmful organic pollutants. However, the utility of their hybrid compositions synthesized via low-cost routes is rarely discussed. Our research examines the efficiency of surfactant-free nanomaterials and their composites with graphene oxide towards the degradation of four important textile and laser dyes, namely: Rhodamine B (RB), Methylene blue (MB), Sulforhodamine 101 hydrate (SR) and Fluorescein (FS). The surfactant-free metal-graphene oxide nanocomposites are engineered in two different techniques: (i) laser ablation mediated synthesis (LAMS) and (ii) multifunctional soret nanoassemblies (MSNAs). On account of the hybridized plasmonic effects from the large charge density oscillations in plasmonic nanoparticles and  $\pi$ -plasmons of graphene oxide, intriguing results are obtained and discussed in this chapter. The synergistic interplay and electron relay between the  $\pi$ -plasmons of graphene oxide and that of organic dyes ( $\pi$ - $\pi$  stacking), in the vicinity of the plasmonic nanocomposites, significantly enhances the performance of the engineered nanomaterials toward dye degradation. The dye-degradation of xenobiotic pollutants demonstrated here opens a new door for the development of a broad spectrum of low-cost surfactant-free nanocomposites for environmental remediation. This study presents a futuristic insight to explore the synergy of low-dimensional and plasmonic nanomaterials constituting elements from different parts of the periodic table to accomplish dye degradation and related applications.

**Keywords:** Dye degradation, Environmental remediation, Electron relay, Laser ablation, Low-cost surfactant-free, Plasmonic nanocomposites, Soret nano-assemblies,  $\pi$ -plasmons.

<sup>\*</sup> **Corresponding author Sai Sathish Ramamurthy:** STAR Laboratory, Central Research Instruments Facility (CRIF), Department of Chemistry, Sri Sathya Sai Institute of Higher Learning, Prasanthi Nilayam, Puttaparthi, Anantapur, Andhra Pradesh, India; E-mail: rsaisathish@sssihl.edu.in

#### **INTRODUCTION**

The rapid increase in the growth and expansion of industries globally has not only benefitted mankind in terms of technological revolution but has also impacted the environment deleteriously [1 - 5]. Air, water and soil pollution is a worldwide issue that endangers the health and livelihood of terrestrial as well as aquatic life systems. Unfortunately, the release of non-treated and/or incompletely treated waste has resulted in hazardous effects on living organisms. Unjustifiable exploitation and overwhelming toxification of water bodies across the world are currently the 'hot issues' regarding water management [3 - 6]. In order to address the ever-increasing demand for pure water sources (due to population growth), methodologies, such as prevention and revalorization (recycle and reuse), need to be employed [1]. Industrial sectors relevant to dye applications are known among the highly polluting sectors in terms of the composition of the effluents and the magnitude of contamination. Generally derived from coal tar and petroleum intermediates, commercially obtainable synthetic dyes are more than 100 000 in number with a yearly production of more than  $7 \times 10^5$  tons making India the second largest exporter of dyes after China [1 - 5]. While the World Bank attributes 17-20% of the water pollution to the textile finishing and dyeing industries, earlier reports underline that around 15% of the non-decomposable textile dyes are discharged into natural water bodies [4 - 6].

By and large, textile, food, leather, paper, pharmaceutical, cosmetics, paint, and printing industries massively use organic dyes and pigments in their manufacturing process. Basically, these industries use large amounts of water in fixing, dyeing, and washing procedures [6 - 10]. The emerging issue in this regard is the continuous disposal of dyes along with industrial wastes into the open environment. On account of their high solubility in water and difficulty in removal of such dyes via simple routes, they enter the surface and groundwater bodies, perpetually causing serious risks. Besides, from the chemistry point of view, these dyes are impervious to degradation by heat, light, or natural cleansing agents in environmental water. The industrial dyes and the related by-products are carcinogenic and mutagenic, thereby leading to detrimental effects on the ecosystem, including plants, mammals and birds [1 - 5]. Additionally, such effluents result in corrosion and blockages in pipes, uncontrolled eutrophication, bioaccumulation leading to allergies, diarrhoea, immune suppression, haemorrhage, and liver and kidney malfunctioning, dermatitis, DNA damage, neuro-muscular and central nervous system disorder to name a few [1 - 10]. Although there are numerous techniques reported for treating wastewater, novel routes for environmental remediation are on the rise as the existing ones are not adequate to terminate the damage.

#### Utility of Nanotechnology

Different strategies are being developed for treating the wastewater, and effective and efficient treatment techniques for dye degradation before their discharge into water bodies are the need of the hour.

In the past decade, numerous nanomaterials have been extensively explored for dye degradation applications in treating pollution caused by the release of industrial effluents, particularly xenobiotics [11 - 13]. With the growing demand for noble (Ag, Au, Pd) nanomaterials for different applications, the use of capping agents to achieve accurate control over the size and shape of nanoparticles (NPs) has substantially increased [14 - 18]. Although such nanomaterials are being explored extensively with dielectric and graphene-based hybrids in multidisciplinary applications, the capping agents strongly bind to the metal surface and conceal the intrinsic catalytic activity of the metal [19 - 21]. This chapter is directed towards the use of capping agent free catalysts and to highlight their efficiency in xenobiotic dye degradation. Generally studied dyes are chosen as model substrates for decolorization reactions. The added advantage with this dye decolorization is that the reaction can be easily monitored using a spectrophotometer. By and large, the chapter highlights the role of surfactant-free metal-graphene oxide nanocomposites that are engineered in two different techniques: (i) laser ablation mediated synthesis (LAMS) [22] and (ii) multifunctional soret nano-assemblies (MSNAs) [23 - 26] towards dye degradation and decolorization [27 - 30].

Graphene is a one-atom thick single layer of graphite. It is comprised of sp<sup>2</sup> hybridized carbon atoms with a hexagonal framework [31]. Single layer graphene has unique characteristics which can be briefly listed as (i) a high theoretical specific surface area of 2600 m<sup>2</sup>/g, (ii) excellent electrical conductivity of 9.6 x  $10^5$  S/cm, (iii) great thermal conductance of ~5000 W/Mk and (iv) it supports interesting transport phenomena such as quantum hall effect [32, 33]. Numerous applications are associated with graphene's distinctive properties on account of its multifunctional capabilities [34, 35]. Consequently, graphene is a principal substitute for other forms of carbon as a good catalytic support material. This is on account of its high surface area concomitant with superior conducting properties [36, 37]. This has assured its application in field emission, storage devices, biosensors, super-capacitors, opto-electronics, membrane materials, and electrochemistry [38, 39]. Practical applications of single-layered graphene have become inadequate on account of the cumbersome procedures involved in its development, management/handling and large-scale production [40, 41]. Present synthesis of graphene suffers from scalability, multilayer formation or usage of toxic chemicals during production. In this context, hydrogen exfoliation of graphite oxide offers a compromised approach to acquire enormous quantities of few-layered graphene sheets (BET surface area  $\sim 430 \text{ m}^2/\text{g}$ ) with notable electrical

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**CHAPTER 4** 

# Treatment of Textile Dye Effluent by Electrochemical Method

Venkatesan Sethuraman<sup>1</sup>, Karupannan Aravindh<sup>2</sup>, Perumalsamy Ramasamy<sup>2</sup>, Bosco Christin Maria Arputham Ashwin<sup>3</sup> and Paulpandian Muthu Mareeswaran<sup>4,\*</sup>

<sup>1</sup> Research and Development, New Energy Storage Technology, Lithium-Ion Battery Division, Amara Raja Batteries Ltd, Karakambadi, Andhra Pradesh-517 520, India

<sup>2</sup> Research Centre, SSN College of Engineering, Kalavakkam, Chennai-603110, Tamil Nadu, India

<sup>3</sup> Department of Chemistry, Pioneer Kumarasamy College, Nagarcoil-629003, Tamil Nadu, India

<sup>4</sup> Department of Chemistry, College of Engineering, Anna University, Chennai-600025, Tamil Nadu, India

**Abstract:** This chapter discusses the electrochemical aqueous solution-based breakdown of synthetic textile colours. Several dyeing and finishing industries produce a significant amount of dye wastewater. For the treatment of effluent water, the electrochemical technique is being studied. The discharge of textile wastewater likewise rises as there are more textile industries. So, in recent years, the electrochemical degradation of industrial effluents has gained popularity. Conductivity, pH, process detention times, total suspended solids (TSS), heavy metals, emulsified oils, bacteria, and other pollutants from water are operating factors in electrochemical treatment. Utilizing cyclic voltammetry (CV), reactive synthetic textile dyes' electrochemical behaviour has been reviewed. Studies on chemical oxygen demand (COD), UV-Vis, and CV are chosen to assess the effectiveness of degradation. There are numerous additional businesses that require electrochemical technologies for purifying effluent water. Metal recovery, tanneries, electroplating, dairies, textile processing, oil and oil in water emulsion, and other businesses are among them.

**Keywords:** Cyclic voltammetry, Chemical oxygen demand, Dyes, Electrochemical degradation, Effluents.

#### **INTRODUCTION**

The accumulation of persistent organic pollutants (POPs) in water has grown to be a significant environmental issue during the past two decades, due to modernisation [1 - 3]. Even in smaller quantities (*i.e.*, nanogram to milligram), the

<sup>\*</sup> Corresponding author Paulpandian Muthu Mareeswaran: Department of Chemistry, College of Engineering, Anna University, Chennai-600025, Tamil Nadu, India; E-mail: muthumareeswaran@gmail.com

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presence of these compounds creates undesirable effects including toxicity, carcinogenicity, mutagenicity, etc. [4 - 6]. Even after treatment, these compounds continue to produce toxicity because they are resistant to traditional wastewater treatment techniques [7]. In order to recover and utilize significant water resources, new and effective POP removal technologies are needed. Electrochemical advanced oxidation processes (EAOPs) have been highlighted as one of the potential subcategories of alternate water treatment technologies to address this environmental problem. Because they could handle extremely refractory organic pollutants including drugs [8], insecticides [9], azo dyes [10], and even carboxylic acids [11], EAOPs have gained growing attention. In addition to effectively removing POPs, the EAOPs also have a number of environmental benefits, including (i) mild operating conditions at ambient temperature and pressure; (ii) compact reactors with smaller physical footprints that require less land space; (iii) no additional need for auxiliary chemicals, such as transportation and storage; (iv) do not produce secondary waste streams that need further treatment; and (v) could be easily combined [12]. With all these crucial features, EAOPs are low-carbon, ecologically beneficial technology. Due to its adaptability and simplicity in scaling, anodic oxidation (AO) or electrochemical oxidation (EO) is the EAOP that has received the most attention [13]. Only a few studies have been focused on synthetic water matrices and actual wastewater effluents. while the majority of experimental investigations on EO and other EAOPs in the literature deal with the oxidation of POPs in synthetic wastewater. In order to remediate synthetic water matrices and actual wastewater effluents at the laboratory and pilot plant sizes, the current book chapter tries to explore the possible applicability and limitations of EO methods. For a better grasp of how EO technology works and its potential ramifications, its fundamentals are briefly reviewed. The foundation for POP remediation in the environment is also examined, as is the possibility of combining it with other pre- or post-treatment water technologies.

Depending on the electrode material or electrocatalyst utilized in electrooxidation (EO), electrogenerated ROS [Eq. (1,2)], either physiosorbed or chemisorbed •OH, oxidize organic molecules in the anode area. As a result, some anode materials interact strongly with the produced radical, encouraging oxidation to superoxide (M=O) or chemisorbed oxygen [14]. As "active anodes," these substances (such as platinum, mixed metal oxides based on Ru and Ir, and graphite-carbon electrodes) have low oxygen evolution overpotentials and can only achieve a soft degradation of organic pollutants (electrochemical conversion) with a very low mineralization degree [15]. Other anodes (so-called "nonactive"), such as doped PbO<sub>2</sub>, SnO<sub>2</sub>, boron-doped diamond, and sub-stoichiometric titanium oxide (Ti<sub>x</sub>O<sub>2x-1</sub>), have a higher oxygen evolution overpotential and weakly interact with the produced radicals, allowing them to freely react with organic molecules until

#### Treatment of Textile Dye

mineralization (electrochemical combustion) [16]. During anodic oxidation processes, a large number of extra oxidants, including ozone, hydrogen peroxide, and peroxosalts, are produced on the anode surface. They interact with organics in large quantities and are effectively in charge of these activities.

$$M_{(s)} + H_2O \rightarrow M(^{\circ}OH) + OH^- + e^-$$
 (1)

$$M(^{\bullet}OH) \rightarrow M=O+H^{+}+e^{-}$$
<sup>(2)</sup>

Reactive chlorine species (RCS) (Cl<sub>2</sub>, HClO, and/or ClO, which are predominant at pH 3.0, 3.0 - 6.5, and > 8.0, respectively) Fig. (1) are produced in bulk solution *via* reactions (Eq. (3-5)) when treating solutions containing high concentrations of Cl ions (*i.e.*, reverse osmosis concentrates) along with reactive oxygen species (ROS) [17]. The RCSs are moderately powerful oxidants that can break down various kinds of organic pollutants. However, this approach usually results in the formation of dangerous by-products such haloacetic acids and trihalomethanes as well as other refractory organochlorinated intermediates that are challenging to mineralize [18].



Fig. (1). Main methods used for the removal of organic dyes from wastewaters.

# Effect of Electrode Materials in Decolorization of Dyestuffs from Wastewater

R. Jagatheesan<sup>1,\*</sup>, C. Christopher<sup>2</sup> and K. Govindan<sup>3</sup>

<sup>1</sup> Department of Chemistry, Vivekanandha College of Arts and Sciences for Women (Autonomous), Elayampalayam, Tiruchengode, Tamil Nadu-637 205, India

<sup>2</sup> Department of Chemistry, St. Xavier's College, Palayamkottai, Tirunelveli-627 002, India

<sup>3</sup> Environmental System Laboratory, Department of Civil Engineering, Kyung Hee University (Global Campus), Giheung-Gu, Yongin-Si, Gyeonggi-Do-16705, Republic of Korea

Abstract: The wastewater produced by the textile industry is replete with numerous contaminants that are known to be hazardous to aquatic and terrestrial living systems. Particularly dangerous contaminants in the textile sector that defy traditional degrading techniques include synthetic dyestuffs. In order to protect the environment, this chapter reviews current advancements in the electrochemical treatment of wastewater containing synthetic organic dyes by anodic oxidation. The mechanisms of electrochemical oxidation in anodic oxidation processes are thoroughly described. The electrochemical degradation of wastewater has been studied using a wide variety of electrodes. As a result, this paper attempts to summarize and discuss the most significant and recent studies on the use of anodes for the removal of organic synthetic dyestuffs that are currently available in the literature.

Keywords: Anodic oxidation, Decolorization, Dyestuffs, Organic dyes.

#### **INTRODUCTION**

It is inevitable that dyes will play a role, ideally in sectors like the textile, food, paper, and plastic industries. The toxins produced by these sectors provide a variety of life-threatening problems for the environment, which in turn severely disrupts aquatic life [1 - 4]. The water clarity, aesthetic appeal, and gas solubility are all significantly impacted by dye concentrations, even at very low levels [5]. Because of this, dyes are carcinogenic, poisonous, and teratogenic, and the process of photochemical and biochemical degradation is highly difficult for complete mineralization as secondary contaminants are produced. Additionally, this contaminated wastewater is not recommended for human consumption or

<sup>\*</sup> **Corresponding author R. Jagatheesan:** Department of Chemistry, Vivekanandha College of Arts and Sciences for Women (Autonomous), Elayampalayam, Tiruchengode, Tamil Nadu-637 205, India; E-mail: jagan3311d@gmail.com

#### Effect of Electrode Materials

even for domestic use [6]. Critical studies have so far reported on a variety of techniques, such as physio-chemical, chemical, and biological oxidation, for removing colours from dye effluents [7 - 10]. The method is costly, less successful in removing colour, and less favourable to a variety of dye-polluted waterways. Anodic electrochemical oxidation has received a lot of attention in recent years due to its low cost, lack of sludge creation, simple operating procedure, and ecologically friendly process. Additionally, the organic matter is intended to be mineralized into  $CO_2$  or changed into biocompatible chemicals by this process [11]. Thus, because the electrode is so important to the electrochemical oxidation process, novel anode materials are increasingly being preferred to boost current efficiency [12]. To lay the groundwork for the electrochemical treatment of dyestuffs, we outlined in this chapter potential dye degradation procedures employing different anodic electrode materials.

#### **Oxidation Mechanisms**

Electrochemical cell types (divided/undivided), electrolyte medium, electrode materials, and power supply are the fundamental requirements for bulk electrolysis. Typically, no separator is used in the undivided batch electrochemical redox tests (Scheme 1). However, divided cells have a membrane-based divider that separates the anodic and cathodic sections, and reactions happen in several compartments (Scheme 2). A semi-porous membrane divides the cathode and anode chambers into divided cells. Sintered glass, porous porcelain, polytetrafluoroethylene, and polypropylene are examples of typical membrane materials. The function of divided cell is to allow ion diffusion while limiting the flow of products and reactants. Galvanostatic conditions in an undivided cell setup using direct or indirect electrochemical electron transfer reactions are taken into consideration for the large-scale setup in the dye degradation process (Table 1) [13].



Scheme (1). Schematic electrolysis in an undivided cell.

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Scheme (2). Schematic electrolysis in a divided cell.





\* Strong oxidizing agents or metallic redox couples can act as oxidation mediators.

Metallic Redox Couple- Ag (II), Co(III), Ce (IV), and Fe (III)

$$M^{z+} \rightarrow M^{(z+1)+} + e^{-} \tag{1}$$

# **Z-scheme:** A Photocatalysis for the Remediation of Environmental Pollutants

#### Suresh Kumar Pandey<sup>1</sup> and Dhanesh Tiwary<sup>1,\*</sup>

<sup>1</sup> Department of Chemistry, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India

Abstract: Modern artificial heterostructures control redox reactions at the catalyst's active sites by effectively separating charges and transporting excitons with the help of light sources. Regarding environmental remediation, the Z-scheme—particularly in the degradation and mineralization of organic pollutants—plays a crucial role. Appropriately designed photocatalysts with Z-scheme have several benefits over conventional photocatalytic processes, including improved charge separation and effective redox process management in response to visible light. It provides the way for the creation of newer and more effective photocatalysts because it is said to make reduction and oxidation processes easier than with the constituent single precursor. In contrast to other heterostructure schemes like the Type-I and Type-II schemes, heterostructures with the Z-scheme mechanism attracted a lot of attention.

**Keywords:** Conduction band, Degradation, Excitons, Electron mediator, Holes, Heterojunction, Photocatalyst, Pollutants, Redox process, Semiconductor, Solar light, Valence band, Z-scheme.

#### **INTRODUCTION**

The intoxication of water poses a severe threat to life on Earth as a result of the regular growth in population and social processes. Numerous significant issues, including skin conditions, diarrhoea, dengue, malaria, and more, are brought on by contaminated water. Water is necessary for life, yet it has since turned into a deadly liquid. According to a WHO report, poor water quality is to blame for 80% of diseases in the entire world. Effluents from the food, leather, textile, and chemical industries are just a few examples of the many sources of water contamination [1 - 3]. Thus, it is essential to create efficient methods for eliminating organic material from water streams. Although there are several established classical methods for treating water, including filtration, adsorption,

<sup>\*</sup> **Corresponding author Dhanesh Tiwary:** Department of Chemistry, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India; E-mail: jagan3311d@gmail.com

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and biological treatment, they all have significant shortcomings [4, 5]. One of the continually developing green technologies, photocatalysis can address both energy and environmental issues at once [6]. The photocatalysis method can also be used to reduce  $CO_2$  and produce fuels, such as hydrogen from water splitting, in addition to degrading contaminants [7 - 17].

Semiconductor photocatalysts are responsible for all these reactions. In 1972, Fujishima used TiO<sub>2</sub> to create H<sub>2</sub> from water, which is when he first coined the phrase "photocatalysis" [18]. Since then, semiconductor photocatalysis has become the method of choice among chemists due to its special ability to directly employ solar light and the reaction's ability to take place at room temperature [19]. During photocatalysis, holes are left in the semiconductor's valence band (VB) as the electrons that were previously present are driven to their conduction band (CB) by absorbing sufficiently energetic photons. The photocatalysts must have the correct energy levels of their CB and VB as well as the desired reduction and oxidation potential to generate superoxide ( $O_2^{-}$ ) (EO<sub>2</sub>/ $O_{2-}$  = -0.33 eV) and hydroxyl (OH<sup> $\cdot$ </sup>) (EOH-/OH• =2.30 eV) radicals, which are required to start the photocatalytic degradation process [20 - 22]. However, it is exceedingly difficult for a single semiconductor (type I) to have both oxidation and reduction potentials to simultaneously straddle the redox process, since, the bandgap of the semiconductor is so wide that the light response will be reduced. Additionally, individual semiconductors experience a high rate of photo-generated charge carrier recombination, which ultimately reduces the photocatalytic activity. Numerous studies have been conducted on the photocatalytic activity of certain typical semiconductor metal oxide photocatalysts, including TiO<sub>2</sub>, ZnO, WO<sub>3</sub>, and Ta<sub>2</sub>O<sub>5</sub> [23 - 26]. However, the high recombination rate of photo-generated excitons ( $e/h^+$ ), poor utilization of visible light, and low quantum yield further place limitations on the photocatalytic approaches using metal oxides. Scientists therefore made numerous modifications to the semiconductor (SC) in order to enhance its photocatalytic performance. The three basic components of the photocatalytic process are; (1) broad light absorption; (2) effective charge separation and their quick migration at the surface of the photocatalysts; and (3) surface catalytic redox process [27].

Despite the enormous advancements made in the science of photocatalysis over the past few decades, it still has a low photocatalytic efficiency due to the rapid recombination of photo-generated excitons which is reducing the semiconductor efficiency to utilize solar energy [28 - 31]. Therefore, for the scientists to construct this doping, loading of metal, and heterojunction, we need to develop advanced photocatalytic techniques to defeat the photoconversion process. Due to its capacity for spatial charge separation between excitons, a heterojunction type II photocatalyst is created when two semiconductors come together [29 - 32]. This

#### Z-scheme: A Photocatalysis

catalyst exhibits efficient photocatalytic activity. The band alignment and schematic pathway of charge flow in typical type II photocatalysts are shown in Fig. (1). This states the migration of photo-generated electrons from the conduction band (CB) of SC-II to the CB of SC-I and simultaneously photo-generated holes travel from the valence band (VB) of SC-II to the VB of SC-II [33 - 35]. Fig. (1) shows that the CB of SC-I and VB of SC-II have accumulated electrons and holes, respectively.



Fig. (1). A schematic representation of Type-II photocatalytic system.

As a result, effective spatial charge separation can be established to increase the photocatalytic activity of the type II photocatalytic system. Although type II systems achieve efficient charge separation, there are still some significant drawbacks, including lower redox ability and difficulty in charge migration (electrons and holes) due to electrostatic repulsion between electrons (e) in the CB of SC-I and SC-II as well as holes  $(h^+)$  in the VB of both semiconductors [36, 37]. The direct Z-scheme photocatalysis hence gained attention in 2013 when Yu et al. explained the remarkable photocatalytic performance of formaldehyde by g- $C_3N_4/TiO_3$  heterojunction photocatalysts [38, 39]. Direct Z-scheme and type II systems have been found to be fundamentally comparable, with their main difference being the direction of charge migration. The photo-generated electron in the CB of SC-I recombines with the holes existing in the VB of semiconductor SC-II in the direct Z-scheme, in contrast to type II, by the action of electrostatic attraction. Thus, direct Z-scheme photocatalysts have much better redox ability than type II photocatalytic systems because the electrons with high reduction potential and holes with strong oxidation potential remain intact in the CB of SC-

### **CHAPTER 7**

# A Review of Various Materials under Different Conditions for Efficient Photocatalytic Dye Degradation

SP. Keerthana<sup>1</sup>, R. Yuvakkumar<sup>1,\*</sup> and G. Ravi<sup>1</sup>

<sup>1</sup> Department of Physics, Alagappa University, Karaikudi, Tamil Nadu, India

**Abstract:** Large amounts of more toxic dye water have been released into the environment recently as a result of the expansion of the textile industry. There are numerous approaches that have been found and applied to lessen the water's toxicity. One of the processes that operate when there is light illumination is photocatalysis. The electrons in the valence band absorb light illumination when exposed to it, excite the conduction band, and create a hole in the valence band. The dye compounds will be lessened by the recombination of these created electron-hole pairs. Materials for effective photocatalysis are being researched. Many factors affect the photocatalytic performance, including narrow bandgap, high surface area, and good recombination rate. TiO<sub>2</sub> is a semiconducting material, however, due to its higher bandgap values, it has a lower potential when exposed to light. This article provides a brief overview of several materials that can be affected by a variety of factors, such as doping, surfactant addition, and composites made of carbon-based materials. It also compares how well each material performs in terms of lowering hazardous pollutants and provides an illustration of the mechanism.

**Keywords:** Bandgap, Composite, Doping, Methylene blue, Malachite green, Photocatalysts, Reusage, Rhodamine B, Surfactant,  $TiO_2$ , Wastewater management.

#### INTRODUCTION

All living things on Earth depend primarily on water. It is the best place to go for daily requirements. 75% of the surface of the planet is covered by water sources, but just 2.5% of those are freshwater sources. 2.5% of the water source is insufficient for the entire population in our world which is expanding at an incredibly rapid rate. For humans and all other living things in the environment, it would be disastrous if 2.5% of the water were polluted [1]. The water has been

<sup>\*</sup> Corresponding author R. Yuvakkumar: Department of Physics, Alagappa University, Karaikudi, Tamil Nadu, India; E-mail: yuvakkumarr@alagappauniversity.ac.in

#### A Review of Various Materials

contaminated by both humans and businesses. The increased expansion of industrialisation has been a major factor in the release of highly hazardous, contaminated wastewater. On the other hand, companies utilize a lot of water, and without any pretreatment, they completely release that water into the neighbouring water resources. When a material causes a change in the behaviour of water that is detrimental, this change is referred to as water pollution [2].

The wastewater in this now-industrialized area is primarily composed of organic salts, carcinogens, poisons, and untreated dyes. The primary sources of pollution today are the food, pharmaceutical, leather and tannery, and textile industries. One of the main contributors to the societal exchange of highly polluted wastewater is the textile industry. To control the water shortage, this needs to be eliminated quickly [3]. All varieties of organic, non-organic, and processed pharmaceutical and dye chemicals can be found in wastewater as pollutants. The main elements that have been found in the wastewater are textile dyes. There are 100,000 different dyes, and  $7 \times 10^5$  tonnes of dyes produced each year. Dyes are stable materials that will form a solid bond with the host material, and they are extremely difficult to remove [4]. Due to their complex structural makeup, the dye strains are challenging to remove. The benefit of dye products is their rich colorization. The substances that have auxochromes and chromophores, which are responsible for colour and colour intensity, are called dyes. Auxochrome will determine how intense the light is, and the chromophore will determine how colourful it is. The dyes were divided into natural, synthetic, azo, diazo, anthraquinone, direct, dispersion, vat, sulphur, solvent, reactive, etc. based on the source, chromophore type, and substrate [5]. The major issue right now is that these organic materials are thrown into water resources and the ground, which is highly bad for the ecology. These dangerous substances will undoubtedly have negative consequences on both aquatic life and people. The Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) will change as a result of the organic substances being deposited into water sources. Aquatic animals will suffer from negative impacts from them, which will cause their demise [6]. The dissolved solids, BOD, COD, colours, and hazardous substances make up the textile wastewater. The percentages of BOD, COD, pH, TOC, and temperature are included in the standard value for water quality checks. There are no acceptable water quality guidelines from before the 20<sup>th</sup> century. The parameters are introduced by the rise in effluents. Researchers have developed a variety of physical, chemical, and biological strategies to address these issues. In the past, physical processes like reverse osmosis, ion exchange, nanofiltration, coagulation, adsorption, and radiation were used. Following biological techniques like enzyme degradation and microbial adsorption. It was pursued to use chemical processes such as oxidation, ozonation, improved oxidation processes, electrochemical destruction, and the Fenton reaction, as well as ultraviolet light. Physical measures were no longer used because of their lengthy processes and ineffectiveness in eliminating novel dyes with aggressive behaviours. The best technique for efficiently degrading harmful contaminants was the Advanced Oxidation Process (AOP) [7]. Here, we discuss several AOPs and materials with a range of characteristics to effectively remove pollutants.

#### **ADVANCED OXIDATION PROCESSES (AOPS)**

AOPs are effective chemical processes that completely purify the water for reuse. Complete compound mineralization will result from this method, which is suited for both harmful contaminants and chemically stable substances. Fig. (1) displays the various AOP processes. AOPs can be classified as homogeneous or heterogeneous techniques. In addition, there are two categories for homogenous methods: with and without energy. The processes  $O_3/UV$ ,  $H_2O_2/UV$ ,  $O_3/H_2O_2/UV$ , electrochemical oxidation, electro-Fenton, and photo-Fenton all required energy. Heterogeneous photocatalysis, photocatalytic ozonation, and catalytic ozonation make up the heterogeneous process. Photocatalysis has the potential to be one of these processes that reduce contaminants from wastewater the most effectively [8].



Fig. (1). Shows different types of AOP process.

When a semiconducting photocatalyst is made, photocatalysis is the process of producing electron-hole pairs while the catalyst is exposed to light. The mechanism of photocatalysis is depicted in Fig. (2). The term "photocatalyst" is also used to describe the semiconductors involved in photocatalysis. The phrase "photocatalyst" comes from the words "photon" and "catalyst," and it means that the substance will alter the rate of reaction in relation to light.

**CHAPTER 8** 

# **Recent Techniques in Dye Degradation: A Biological Approach**

Nagaraj Revathi<sup>1</sup>, Jeyaraj Dhaveethu Raja<sup>2</sup>, Jegathalaprathaban Rajesh<sup>3</sup> and Murugesan Sankarganesh<sup>3,\*</sup>

<sup>1</sup> Department of Chemistry, Ramco Institute of Technology, Rajapalayam, Virudhunagar-626 117, Tamil Nadu, India

<sup>2</sup> Department of Chemistry, The American College, Tallakkulam, Madurai-625 002, Tamil Nadu, India

<sup>3</sup> Department of Chemistry, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu-602 105, India

Abstract: Synthetic dyes are organic compounds that are mostly employed in the manufacturing industry. A huge number of dyes are unbound and released into the environment during the dying process. The discharge of dye/effluent with a high biological oxygen demand (BOD) and chemical oxygen demand (COD) into the environment has several negative consequences for the area's flora and fauna. They are poisonous and mutagenic, and have other significant negative impacts on a variety of creatures, including unicellular and multicellular organisms. Besides the costly Physico-chemical treatment methods, biological approaches involving bacteria, fungi, algae, plants, and their enzymes have got a lot of attention in recent years for the decolorization and degradation of dyes contained in effluents due to their economic effectiveness and environmental friendliness. Microbial degradation appears to be the most promising of these technologies for resource recovery and sustainability. Microorganism and plant-derived enzymes' ability to decolorize and break down dyes has long been known, and they are shown to be the most effective molecular weapon for bioremediation. Several sophisticated approaches are currently being investigated for the effective decolorization of textile dyes as well as eco-toxic effluent, including genetic engineering, nanotechnology, mobilized cells or enzymes, biofilms, and microbial fuel cells, among others. These biological methods for decolorization and degradation of textile effluent are very successful and have various advantages over traditional procedures. Biological methods for removing toxic textile dyes are both environmentally friendly and cost-effective.

**Keywords:** Dye degradation, Dye contaminated industrial effluents treatment, Graphene oxide, Nanocomposites.

<sup>\*</sup> **Corresponding author Murugesan Sankarganesh:** Department of Chemistry, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Saveetha University, Chennai, Tamil Nadu-602 105, India; E-mail: msankarajan1990@gmail.com

#### **INTRODUCTION**

Colour has always fascinated humankind, for both aesthetic and social reasons. A dye is a colored substance that is typically used in solution and can be fixed to the fabric. It is necessary for the dye to be "fast" or chemically stable so that it does not wash out with soap and water.

The most popular industrial coloring chemical substances are dyes. Dyes are organic substances that have a distinct color. Contrary to most organic substances, dyes have color because they:

- 1. Absorb visible spectrum light (400-700 nm).
- 2. Have a chromophore (color bearing group).
- 3. Have a conjugated system.
- 4. Display electron resonance.

The color is lost when one or more of these characteristics is missing from the molecular structure. Most dyes also contain groups referred to as auxochromes (color aids), such as carboxylic acids, sulfonic acids, amino and hydroxyl groups, in addition to chromophores. Although they do not produce colour, they are frequently utilized to affect dye solubility and their presence can change the color of a colorant. The correlations between visible wavelength and colour absorbed/observed are shown in Table 1. Almost all commercial products require colour at some point in their production and there are currently more than 50000 trade names for more than 9000 different colourants. The huge quantity results from the wide variety of tints and colours needed, the chemical makeup of the materials to be coloured and the fact that colour is inversely proportional to dye's molecular structure.

S. No.	Wavelength Absorbed (nm)	Colour Absorbed	Colour Observed
1.	400-435	Violet	Yellow-Green
2.	435-480	Blue	Yellow
3.	480-490	Green-Blue	Orange
4.	490-500	Blue-Green	Red
5.	500-560	Green	Purple
6.	560-580	Yellow-Green	Violet
7.	580-595	Yellow	Blue

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S. No.	Wavelength Absorbed (nm)	Colour Absorbed	Colour Observed
8.	595-605	Orange	Green-Blue
9.	605-700	Red	Blue-Green

The textile industry in India accounts for 14% of all industrial production, 4% of the country's GDP and around 27% of all foreign exchange earnings. Up to 10,000 dyes are accessible worldwide, and they have produced more than 7,105 tonnes annually. These colours are employed not only in the textile industry but also in the paper, food and pharmaceutical sectors. The processing of 1 kg of textiles in India requires more than 100 L of water and the textile industry has significantly impacted the contamination of ground and surface water supplies over most of the nation.

Due to its extensive use of up to 8000 chemicals and large amounts of water, the textile industry has been one of the main pollutants of surface and groundwater resources. According to some sources, an average-sized textile sector uses 1.6 million liters of water per day to produce 8000 kg of cloth [1]. The World Bank estimates that the dyeing and finishing processes used on the cloth account for 17% to 20% of the water pollution caused by the textile sector [2, 3]. Thirty of the 72 hazardous compounds found in the wastewater released from the textile dyeing industry cannot be eliminated by wastewater treatment procedures [4]. The textile and dyeing industries are using chemical-based textile dyes more and more because they are more affordable and have higher temperature and light stability than natural colours.

Dye molecules are comprised often key components:

• Chromophores: They are responsible for producing the color. Fig. (1) represents various chromophores present in organic dyes.

• Auxochromes: They can not only increase the chromophore content but also make the molecule water soluble and increase its affinity for the fiber [5].

#### EFFECT OF DYES

The textile industry and its wastewater have grown proportionally along with the demand for textile products, making it one of the major contributors to the world's serious pollution issues. The colouring (textile, cosmetic and leather) industries utilize around 1,00,000 commercial dyes and dyestuff and roughly 10-15% of all dyestuff is lost to wastewater directly. The textile dye industry involves various processes like printing, dyeing, mercerizing, bleaching, scouring, desizing for the production of fabrics, which release textile effluents (Fig. 2). The effluents are

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### Paulpandian Muthu Mareeswaran

Prof. Paulpandian Muthu Mareeswaran completed Ph. D. from Madurai Kamaraj University, Madurai, Tamil Nadu, India in 2012. He worked as post-doctoral researcher at Korea Advanced Institute of Science and Technology, Daejeon, South Korea up to 2014. After finishing post-doc, he joined as DST INSPIRE Faculty in the Department of Industrial Chemistry, Alagappa University, Karaikudi, Tamil Nadu, India. He also served as guest faculty in the Department of Oceanography and Coastal Area Studies, Thondi Campus, Alagappa University, Karaikudi, Tamil Nadu, India. Currently, he is working as assistant professor in the Department of Chemistry, College of Engineering, Anna University, Chennai, Tamil Nadu, India. He published 56 papers in international journals. He has also guided three Ph. D. and 31 PG dissertations.



### Jegathalaprathaban Rajesh

Prof. Jegathalaprathaban Rajesh completed Ph.D. in Madurai Kamaraj University, Madurai, Tamil Nadu, India in 2008. After that she served as post-doctoral researcher at Complutense University, Madrid, Spain till 2009. She served as assistant professor at various institutes. Currently, she is working as associate professor at Savitha University, Chennai, Tamil Nadu, India. She has published 70 papers in international journals. She has several patents to her credit. She has guided three Ph.D. dissertations.