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Photocatalytic activity of nanoparticles synthesized by green process

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Abstract

This study explores the green synthesis of zinc oxide nanoparticles (ZnO NPs) using an extract from the Sonchus wightianus plant, and evaluates their photocatalytic performance in degrading organic pollutants. The photocatalytic activity of the synthesized ZnO was tested against two model dyes: Methylene Blue and Rose Bengal. The results revealed good degradation efficiency, particularly for Rose Bengal, suggesting that the nature of the dye significantly influences the photocatalytic response. Factors such as dye structure, charge, and interaction with the ZnO surface played a key role. This study confirms the potential of Sonchus wightianus as a natural and efficient reducing and stabilizing agent for green nanoparticle synthesis. Furthermore, it demonstrates the applicability of the resulting ZnO NPs in the field of environmental remediation, especially in the photocatalytic degradation of hazardous organic dyes.

Keys words : Green synthesis; zinc oxide; nanoparticles; Sonchus wightianus; photocatalytic degradation; dyes

Résumé

Cette étude explore la synthèse verte de nanoparticules d'oxyde de zinc (ZnO NP) à partir d'un extrait de la plante Sonchus wightianus, et évalue leur performance photocatalytique dans la dégradation des polluants organiques. L'activité photocatalytique du ZnO synthétisé a été testée sur deux colorants modèles : le bleu de méthylène et le rose Bengale. Les résultats ont révélé une bonne efficacité de dégradation, en particulier pour le rose Bengale, suggérant que la nature du colorant influence considérablement la réponse photocatalytique. Des facteurs tels que la structure du colorant, sa charge et son interaction avec la surface du ZnO ont joué un rôle clé. Cette étude confirme le potentiel du Sonchus wightianus en tant qu'agent réducteur et stabilisateur naturel et efficace pour la synthèse verte de nanoparticules. En outre, elle démontre l'applicabilité des NP de ZnO obtenues dans le domaine de la remédiation environnementale, en particulier dans la dégradation photocatalytique des colorants organiques dangereux.

Mots clés : Synthèse verte ; oxyde de zinc ; nanoparticules ; Sonchus wightianus ; dégradation photocatalytique ; colorants.

الملخص

تستكشف هذه الدراسة التوليف الأخضر لجزيئات أكسيد الزنك النانوية (ZnO NPs) باستخدام مستخلص من نبات Sonchus wightianus ، وتقيّم أداءها التحفيزي الضوئي في تحليل الملوثات العضوية. تم اختبار النشاط التحفيزي الضوئي لجزيئات ZnO المؤلفة ضد صبغتين نموذجيتين: الميثيلين الأزرق و وردي البنغال. أظهرت النتائج كفاءة تحليل جيدة، خاصة لصبغة الروز بنغال، مما يشير إلى أن طبيعة الصبغة تؤثر بشكل كبير على الاستجابة التحفيزية الضوئية. لعبت عوامل مثل بنية الصبغة، شحنتها، وتفاعلها مع سطح ZnO دورًا رئيسيًا. تؤكد هذه الدراسة إمكانية استخدام Sonchus wightianus كعامل مختزل ومثبت طبيعي وفعال لتوليف الجزيئات النانوية الخضراء. علاوة على ذلك، تُظهر قابلية تطبيق جزيئات ZnO الناتجة في مجال المعالجة البيئية، خاصة في التحليل التحفيزي الضوئي للأصباغ العضوية الخطرة.

الكلمات المفتاحية: التوليف الأخضر؛ أكسيد الزنك؛ الجزيئات النانوية ؛ Sonchus wightianus ؛التحليل التحفيزي الضوئي؛ الأصباغ.

الاهداء

إلى مَن غرَسا في قَلبي حُبَّ العِلمِ وَالعَمَلِ،
إلى مَن سَانَدَاني فِي كُلِّ لحظَةٍ، بِصَبرِهِما، بِدُعَائِهِما، بِقَلبَيهِما اللَّذَيْنِ لَمْ يَعْرِفَا إِلَّا العَطَاء...
إلى أُمِّي وَأبِي، تَاجِ رَأسِي وَنَبْضِ قَلبِي، أُهدِي هذا العَمَلَ بِكُلِّ فَخْرٍ وَامْتِنَان.

إلى إِخْوَتِي الأعِزَّاء، الذِينَ كَانُوا سَنَدِي فِي الصِّعَاب، وَفَرَحِي فِي النَّجَاحَات، لَكُمْ مِنِّي كُلُّ الحُبِّ وَالتَّقْدِير.

إلى أصْدِقَائِي الذِينَ شَارَكُونِي هذِهِ الرِّحْلَة، لَحَظَاتِهَا الحُلْوَةِ وَالمُرَّةِ، وَتَرَكُوا فِي قَلْبِي بَصْمَةً لَا تُنْسَى.

إلى كُلِّ مَنْ أَحَبَّنِي وَدَعَمَنِي بِكَلِمَةٍ طَيِّبَةٍ، بِدُعَاءٍ صَادِقٍ، بِابْتِسَامَةٍ مُحَفِّزَةٍ، إِلَى كُلِّ الأحِبَّةِ الذِينَ كَانُوا جُزْءًا مِن رِحْلَتِي...

أُهْدِيكُم ثَمَرَةَ جُهْدِي، وَنَجَاحِي الذِي مَا كَانَ لِيَرَى النُّورَ بدنوكم جَمِيعًا.

شكر وعرفان

الحمد للهِ الذي بنعمته تتمُّ الصالحات، وبفضله تيسَّرت الصعوبات، الحمد لله الذي وهبنا القوة والصبر لإتمام هذا العمل العلمي، وجعل لنا في كل خطوة عونًا وسدادًا.

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كما لا يفوتني أن أتقدم بالشكر لإدارة وطاقم المخبر على ما وفروه لنا من إمكانيات وتجهيزات وتجارب علمية، وعلى تعاونهم المستمر الذي كان له الدور الكبير في الجانب التطبيقي من هذا البحث.

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Abbreviations list

Chapter 1

Symbol	Description
C6-C2-C6	Basic structure of flavonoids
VOC	Volatile Organic Compounds
ROS	Reactive Oxygen Species
H_2O	Water

Chapter 2

Symbol	Description
ZnO	Zinc Oxide
TiO_2	Titanium Dioxide
SiO_2	Silicon Dioxide
Al_2O_3	Aluminum Oxide
SnO_2	Tin Dioxide
In_2O_3	Indium Oxide
Fe_2O_3	Iron Oxide
WO_3	Tungsten Trioxide
NPs	Nanoparticles

List Abbreviations

e^-	Electron
h^+	Hole
e^-/h^+ pair	Electron–hole pair
ROS	Reactive Oxygen Species
$\cdot OH$	Hydroxyl radical
O_2^-	Superoxide anion
CO_2	Carbon Dioxide
H_2O	Water
HOMO	Highest Occupied Molecular Orbital
LUMO	Lowest Unoccupied Molecular Orbital
Band gap	Energy gap between valence and conduction bands
Valence band	Highest energy band filled with electrons
Conduction band	Lowest unoccupied band that conducts electricity
Ev	Electron volt
D_0, D_1, D_2	0D, 1D, 2D Nanostructures
PLA / PLGA	Biodegradable polymers
CNT	Carbon Nanotubes
CMC	Critical Micelle Concentration

List Abbreviations

a.u.	Arbitrary Units
%	Percent
H	Efficiency
MOFs / COFs	Metal-Organic / Covalent Organic Frameworks
SACs	Single-Atom Catalysts
HEAs	High Entropy Alloys
LSPR	Localized Surface Plasmon Resonance
$g\text{-}C_3N_4$	Graphitic Carbon Nitride
CdS / CdSe	Cadmium Sulfide / Cadmium Selenide
LDPE	Low-Density Polyethylene

chapter 3

Symbol	Description
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
UV-Vis	Ultraviolet–Visible Spectroscopy
DRIFTS	Diffuse Reflectance Infrared Fourier Transform Spectroscopy

List Abbreviations

HPLC	High-Performance Liquid Chromatography
CE	Capillary Electrophoresis
λ / Λ	Wavelength
kV / mA	Kilovolt / Milliampere
Θ / B / FWHM	Bragg angle / Full Width at Half Maximum
Å	Angstrom
RGO	Reduced Graphene Oxide
CuO / NO	Copper Oxide / Nitric Oxide
O–H / C=O / N–H	Chemical bonds seen in IR spectra
FESEM / FIB	Field Emission SEM / Focused Ion Beam

chapter 4

Symbol	Description
ZnO	Zinc Oxide
$C_4H_6O_4Zn$	Zinc Acetate
NaOH	Sodium Hydroxide
MB	Methylene Blue
RB	Rose Bengal

List Abbreviations

a.u.	Arbitrary Units
Nm	Nanometer
Rpm	Revolutions per minute
Mg	Milligram
mL	Milliliter
L	Liter
°C	Degree Celsius
UV-Vis	Ultraviolet–Visible Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-ray Diffraction
pH	Acidity/basicity scale
Θ	Bragg angle
Λ	Wavelength
Cu Kα	Copper X-ray wavelength (1.5406 Å)
K	Scherrer constant
FWHM	Full Width at Half Maximum
CO_2	Carbon Dioxide
H_2O	Water
O_2	Oxygen

List Abbreviations

$\cdot OH$	Hydroxyl radical
O_2^-	Superoxide anion
e^- / h^+	Electron / hole pair
Abs	Absorbance
Λmax	Maximum absorbance wavelength
Φ	Efficiency / Quantum Yield

General introduction

In recent decades, the expansion of industrial activities has led to increasingly complex environmental issues, most notably the contamination of water resources with organic pollutants. Among these, synthetic dyes used extensively in industries such as textiles, pharmaceuticals, and cosmetics are particularly problematic due to their chemical stability and resistance to biodegradation. These dyes accumulate in aquatic environments, reducing light penetration, disrupting ecosystems, and posing potential risks to human health. As a result, advanced treatment techniques have drawn attention—chief among them heterogeneous photocatalysis, which utilizes semiconductor materials like ZnO or TiO_2 activated by light to degrade complex organic molecules [1]

Zinc oxide (ZnO) has shown remarkable efficiency as a photocatalyst due to its wide band gap, strong light absorption, environmental stability, ease of synthesis, and low cost. ZnO is gaining recognition for its high performance in degrading organic dyes such as methylene blue and rose bengal. However, conventional synthesis methods for ZnO often rely on hazardous chemical reagents and organic solvents, raising concerns about environmental safety and sustainability [1]

This has led to a growing interest in green synthesis of nanoparticles, which employs natural resources such as plant extracts as reducing and stabilizing agents. Bioactive compounds found in plants (such as polyphenols and flavonoids) facilitate the formation of stable nanoparticles without the need for toxic chemicals. Studies have shown that the composition of plant extracts significantly influences the size, shape, and stability of the synthesized nanoparticles, which in turn affects their photocatalytic performance [2]

In this context, the present study aims to synthesize ZnO nanoparticles using an extract of Sonchus wightianus, a plant known for its high polyphenol content. The synthesized nanoparticles are then tested for their efficiency in degrading two common organic dyes (methylene blue and rose Bengal) under natural sunlight, simulating real-world environmental conditions and offering an affordable and eco-friendly wastewater treatment solution.

The research focuses on four main objectives: (1) synthesizing ZnO nanoparticles using the green route with Sonchus wightianus extract, (2) characterizing the obtained nanoparticles using advanced analytical techniques and (3) evaluating their photocatalytic activity against the selected dyes. Key characterization techniques include UV-Visible spectroscopy (UV-Vis), Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD), all essential tools for analyzing nanoparticle composition, and structure.

This memory is divided into two main parts: a theoretical section and a practical section. The theoretical part comprises three chapters. The first chapter discusses polyphenolic compounds and the use of plants as raw materials, including a description of the geographic distribution of Sonchus wightianus. The second chapter covers nanomaterials and photocatalysis, detailing synthesis methods (physical, chemical, and green), their advantages and disadvantages, and the importance of sustainability. The third chapter presents the main nanoparticle characterization techniques. The practical section is presented in the fourth chapter, which outlines the steps of ZnO green synthesis, the photocatalytic experiments, the results obtained, and their interpretation. The work concludes with a general conclusion.

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Theoretical

part

Chapter I :

Plants and polyphenolic compounds

1. Introduction

The concept of green manufacturing has evolved from a reactive approach focused on end-of-pipe pollution control to a proactive, holistic strategy that integrates environmental considerations throughout the product life cycle. Initially, efforts centered on mitigating environmental damage post-production. Over time, the emphasis shifted towards minimizing resource consumption, reducing waste, and adopting sustainable practices such as the 4Rs—reduce, reuse, recycle, and remanufacture. This progression reflects a paradigm shift towards sustainable development, aiming to harmonize economic growth with environmental stewardship[1].

Essential for advancing bio-based materials, polyphenolic compounds, particularly the versatile flavonoids, are key to plant physiology and ecological interactions. These metabolites ensure plant adaptation and survival through UV protection, potent antioxidant activity, and defense against herbivores and pathogens, alongside roles in signaling and symbiosis. This highlights their significant potential for sustainable materials and broad industrial/pharmaceutical applications [2] In nature, these compounds actively contribute to plant defense, symbiosis, stress adaptation, and allelopathy, highlighting their role in environmental equilibrium[3]

2 Plant

2.2 The use of plants as raw materials in industry

The transition to a sustainable chemical industry is increasingly driven by the utilization of renewable carbon sources, particularly plant-derived biomass. This biomass provides access to diverse molecular structures—especially platform molecules—that can be efficiently converted into key industrial chemicals using advanced technologies like continuous flow chemistry. Such processes not only enhance reaction control and scalability but also support environmental goals by reducing greenhouse gas emissions and the dependency on non-renewable fossil carbon sources [4].

Building upon this foundation, recent advances in polymer science focus on designing entirely new materials that exploit the intrinsic structural complexity of these renewable feedstocks. Rather than merely replicating petrochemical-based polymers, researchers are leveraging the chemical richness of plant-based compounds to create performance-advantaged polymers with superior strength, flexibility, or thermal stability. These innovations highlight the potential of bio-derived inputs not just as replacements, but as sources of next-generation materials with tailored functionalities for diverse industrial applications[5].

Indeed, a significant aspect of this green chemistry approach involves the resourceful utilization of agricultural residues and non-edible plant waste as renewable sources for high-value chemicals. Through catalytic and enzymatic processes, these often-overlooked by-products can yield bioactive compounds such as phenolics, terpenes, and lactones, finding applications in cosmetics and pharmaceuticals. This strategy not only promotes substantial waste reduction but also strengthens circular bioeconomy models by adding significant value to biomass otherwise considered refuse[6].

The comprehensive valorization of biomass waste is thus pivotal for achieving broader sustainable development goals. By transforming diverse agricultural and industrial residues into valuable products—including biofuels, biochemicals (as highlighted above), and bioplastics—it is possible to markedly reduce environmental pollution and our collective dependence on fossil resources. Advanced conversion technologies, encompassing both thermochemical and biochemical processes, facilitate the efficient utilization of these varied biomass feedstocks. This holistic approach not only addresses critical waste management challenges but also makes a substantial contribution to the circular economy by creating renewable energy sources and sustainable materials, positioning such strategies as essential for transitioning towards a low-carbon and resource-efficient global future[7]

2.2 Introduction to the region

The Oued Righ region, located in the northeastern part of the vast Algerian Sahara Desert, occupies a broad rectangular depression approximately 160 km long and 30–40 km wide. It is flanked by the Eastern Arak plateau and the Jannoud hills and has been known by various historical names such as "Zab al-Saghir," "Righ," and "Bilal Righ"[8]

2.2 Climate of the Oued Righ Region

The Oued Righ region's harsh desert climate, with its extreme heat and scant rainfall, dictates its agricultural practices, rendering the sandy soils entirely dependent on irrigation.

Consequently, the vegetation is dominated by drought and salt-tolerant species like Tamarix, which are adapted to survive these conditions and are essential for stabilizing the soil[8]

2.3 Invasive weeds

Within this unique flora, some plants are considered harmful weeds, posing a threat to agricultural productivity by competing for water and nutrients. Notable among these are Najm (Cyperus), reeds, sammar, maleeh, sweid, and ghdam, with dis (Imperata cylindrica) being particularly aggressive and problematic, sometimes forcing farmers to abandon their land. However, the recent introduction of chemical herbicides has offered a means to mitigate their impact[8]

2.4 Beneficial herbs

Conversely, Oued Righ is also rich in various beneficial wild herbs that are crucial for local livelihoods, particularly as livestock fodder during the spring some of these ,such as harmel (Peganum), shih (Artemisia), qartifa, hmayda (Rumex), wild fennel, capers, mallow, and luway, grow naturally. Among these beneficial plants, "Ghareem" (Sonchus wightianus.) holds particular significance within the Oued Righ region due to its notable presence and raditional recognition**.** It is this distinct species, Sonchus wightianus, that has been specifically selected for investigation in our current experimental work, aiming to explore its potential in green synthesis applications. Other plants like alfalfa, oats, and sorghum are cultivated, and many of these beneficial herbs are dried and stored for winter fodder[8]

2.5 Description of Sonchus wightianus.

The plant species selected for this investigation was Sonchus wightianus DC., which is known locally in our region as 'Al-Ghoraym'. Morphologically, it is a robust perennial herb characterized by its growth from a creeping, subterranean rhizome that facilitates its persistence and vegetative propagation. The plant features an erect, hollow (fistular) stem that can attain a height of up to 1.5 meters, typically branched in its distal portion and often covered with glandular hairs. The foliage is distinguished by its deeply pinnatifid, oblong-lanceolate leaves, with margins that are either sharply dentate or spinous. These leaves are differentiated into a basal rosette and cauline leaves arranged along the stem. The inflorescence is a terminal corymbose panicle bearing multiple yellow-flowered capitula. Following anthesis, the plant produces small, compressed, and prominently ribbed achenes that turn brown upon maturity[9]

Figure1. 1 ***Sonchus wightianus plant 'Al-GhREEM'***

2.6 Geographical Distribution of Sonchus wightianus

Sonchus wightianus possesses a wide native distribution, primarily across various regions of Asia, including Pakistan, India, Afghanistan, and extending to China and Malaysia. In the context of our study, this species is particularly well-established and abundant in the Saharan regions of Algeria. The plant material for this research was specifically collected from its thriving populations in the Oued Righ region, where it is a prominent element of the local flora[10] The following table (1) provides a clear and systematic overview of the plant's scientific classification.

Table I. 1 the plant scientific classification [11]

Taxonomic Rank	Scientific Name
Kingdom	Plantae
Order	Asterales
Family	Asteraceae (or Compositae)
Genus	*Sonchus*
Species	*Sonchus wightianus* DC

2.7 Phytochemical Composition of Sonchus wightianus

Phytochemical screening of Sonchus wightianus reveals a rich and diverse chemical profile, underscoring its potential as a source of bioactive compounds. Qualitative analysis of various solvent extracts confirmed the presence of several major classes of secondary metabolites, including alkaloids, flavonoids, tannins, and general phenolic compounds. Furthermore, the investigation also detected the presence of saponins, terpenoids, steroids, and glycosides. This complex phytochemical composition, particularly the abundance of flavonoids and phenolic compounds, provides a strong scientific basis for the plant's traditional medicinal uses and is consistent with the significant antioxidant and antimicrobial properties observed in related studies[12]Several specific chemical compounds have been isolated from Sonchus wightianus, as detailed in Table(2).

Table I. 2 Isolated Chemical Compounds from Sonchus wightianus[13]

Compound Name	Plant Part	Remarks/Activity
β-sitosterol	Whole plant	Plant sterol
β-sitosterol glycoside	Whole plant	Glycosidic form of β-sitosterol
1-Hexacosanol	Whole plant	Long-chain fatty alcohol
Hexadecanoic methyl ester	Whole plant	Fatty acid methyl ester
Lupenone	Leaves	Major constituent (~38%)
Leupeyl acetate	Roots	Abundant (~33%)
24-Noursa-3,12-diene	Stems	Abundant (~27%)
Sesquiterpenoid guaianes	Whole plant	New structures (1–7) including glycosides
Cytochalasin	Whole plant	New compound; potent immunosuppressant
Known guaianes	Whole plant	Previously characterized guaianes
Known cytochalasins	Whole plant	Previously reported cytochalasins

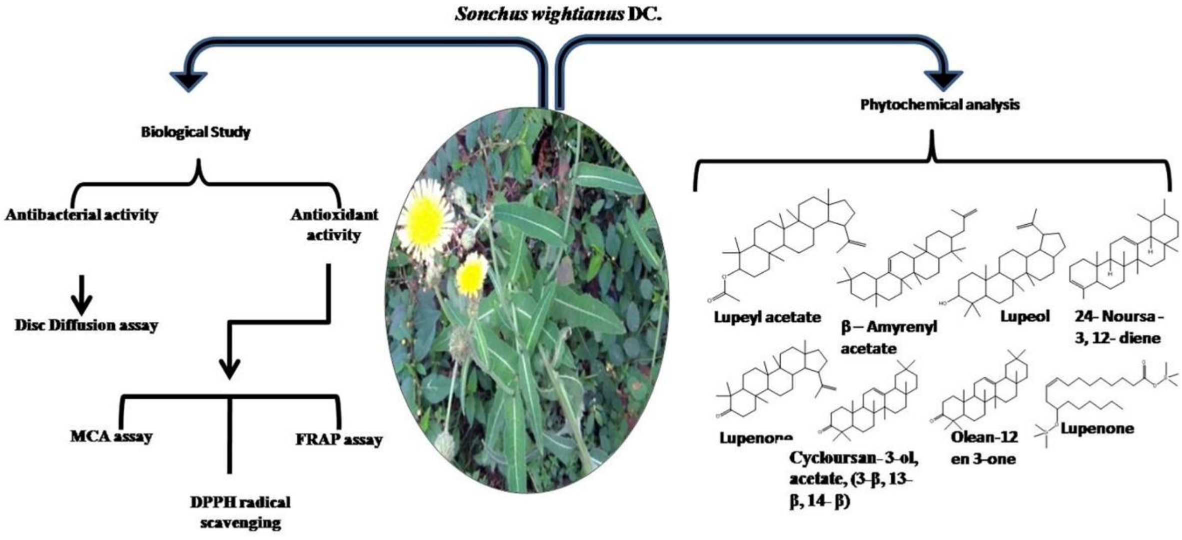

Figure1. 2 Biological activities and phytochemical constituents identified from Sonchus wightianus DC[14]

3 Polyphenolic Compounds

3.1 Definition of polyphenolic compounds

Polyphenolic compounds are natural substances primarily found in plants, characterized by the presence of more than one phenolic group (–OH) attached to an aromatic structure. These compounds fulfill a crucial role in protecting plants from environmental stressors such as UV radiation and microbial attacks. Their potent antioxidant properties have garnered significant scientific interest, highlighting their importance in the prevention of various human diseases[15]

3.2 Types of polyphenolic compounds

The diverse world of polyphenols can be broadly categorized into several major types, each with unique structural features and biological activities. Flavonoids represent the largest and most extensively studied group, characterized by their C6-C3-C6 skeleton which consists of two aromatic rings linked by a three-carbon bridge.This broad category is further subdivided into several subclasses, including flavonols like quercetin, flavones such as luteolin, flavanones (e.g., naringenin), isoflavones (e.g., genistein), anthocyanins which are responsible for the vibrant red and blue pigments in many plants, and flavanols such as catechins.These compounds are widely recognized for their powerful antioxidant, anti-inflammatory, and cardioprotective properties[16]

Another significant class of non-flavonoid polyphenols is phenolic acids are a major group of non-flavonoid polyphenols, consist primarily of hydroxybenzoic acids (e.g., gallic acid, protocatechuic acid) and hydroxycinnamic acids (e.g., caffeic acid, ferulic acid, chlorogenic acid). They are characterized by a benzene ring with a carboxyl group and hydroxyl substituents. Found widely in plants, these acids can exist in free or bound forms and contribute significantly to the antioxidant capacity and health-promoting properties of many foods[17]

Flavonoids represent the largest and most diverse class of plant polyphenols, characterized by their fundamental C6-C3-C6 skeletal structure, which consists of two aromatic rings linked by a three-carbon bridge. This vast family is further classified into several major subclasses, including flavonols like quercetin, flavones such as luteolin, and the anthocyanins responsible for the red, blue, and purple pigmentation in many fruits and flowers. Abundantly found in foods like onions, apples, berries, and tea, flavonoids are renowned for their wide spectrum of biological activities, particularly their potent antioxidant, anti-inflammatory, and cardioprotective effects[18]

Lignans are phytoestrogens formed by the dimerization of two phenylpropanoid units, often substituted cinnamic alcohols. They are widely distributed in the plant kingdom, with particul arly high concentrations found in seeds such as flaxseed and sesame seed, as well as in whole grains and vegetables. Prominent lignans include secoisolariciresinol, matairesinol, and pinoresinol, which are often converted by gut microbiota into enterolignans like enterodiol and enterolactone[19]

Finally, Tannins are high molecular weight polyphenols, broadly classified into hydrolyzable tannins (esters of gallic or ellagic acid) and condensed tannins (proanthocyanidins, which are polymers of flavan-3-ols). Known for their astringent properties due to protein binding, tannins also contribute to plant defense and possess notable antioxidant, antimicrobial, and anti-inflammatory activities[20]A summary of these key types of polyphenolic compounds, along with representative examples and general structural features, is presented in Table(3).

Table I .3 Types of Polyphenolic Compounds[21]

Main Type	General Chemical Structure	Key Examples
Flavonoids	C6-C3-C6 carbon Skeleton	Quercetin, Kaempferol Catechins
Phenolic Acids	Derivatives of benzene Acids	Gallic acid, Caffeic acid
Lignans	Dimeric phenolic Compounds	Secoisolariciresinol, Matairesinol
Tannins	Polymers of flavanols or phenolic acids	Proanthocyanidins, Ellagitannins

3.3 Properties of Polyphenolic Compounds

The diverse biological activities of polyphenolic compounds stem from several key properties. Perhaps most notably, these compounds exhibit strong antioxidant activity by effectively scavenging reactive oxygen species (ROS) such as superoxide anions, hydroxyl radicals, and hydrogen peroxide. Through this crucial mechanism, they protect vital cellular components like lipids, proteins, and DNA from oxidative damage, which is a primary contributor to aging and various degenerative diseases, including cancer and cardiovascular disorders[22]

Polyphenols exert potent anti-inflammatory effects by modulating critical signaling pathways, such as **NF-κB** and **MAPK**. This, in turn, suppresses the expression of key pro-inflammatory enzymes, including **COX-2** and **iNOS**, and downregulates the production of cytokines like **TNF-α, IL-1β, and IL-6**. This multi-targeted mechanism underlies their therapeutic potential in managing chronic inflammatory conditions[23]

Furthermore, polyphenols demonstrate potent, broad-spectrum antimicrobial activity against a wide range of pathogens, including bacteria, fungi, and viruses. Their multifaceted mechanisms of action include the disruption of microbial cell integrity, the inhibition of essential enzymes and nucleic acid synthesis, and the interference with key virulence factors such as biofilm formation and quorum sensing. These properties establish polyphenols as valuable candidates for natural food preservatives and as a foundation for developing novel antimicrobial therapies[24]

The chemopreventive potential of polyphenolic compounds is another area of active research.They contribute to cancer prevention through multiple mechanisms, including the inhibition of tumor cell proliferation, induction of apoptosis (programmed cell death) in cancerous cells, suppression of angiogenesis (the formation of new blood vessels that supply tumors), and modulation of key molecular pathways involved in cancer development. Their ability to act at various stages of carcinogenesis underscores their significance in developing chemoprevention strategies[22]

Lastly, polyphenols exert considerable cardioprotective effects.They are known to promote cardiovascular health by improving endothelial function, reducing blood pressure, decreasing the oxidation of LDL ("bad" cholesterol), and enhancing overall lipid profiles.Coupled with their anti-inflammatory and antioxidant properties, these effects contribute to reducing the risk of atherosclerosis and other heart diseases, and regular intake of polyphenol-rich foods has been linked to a lower incidence of cardiovascular events[22]

4. Conclusion

This chapter highlights the growing importance of green manufacturing and the use of renewable plant resources, particularly in the Oued Righ region. The shift towards sustainable practices, incorporating the 4R principles (reduce, reuse, recycle, remanufacture), reflects a commitment to balancing economic growth with environmental preservation. Polyphenolic compounds, particularly flavonoids found in plants such as Sonchus wightianus, play a key role in the development of bio-based materials and offer promising applications in the chemical, pharmaceutical and cosmetic industries thanks to their properties. In the local context, the valorisation of agricultural residues and wild plants, despite the challenges posed by the desert climate and invasive weeds, reinforces circular economy models. These advances position the region as a potential player in the transition to a green and sustainable economy.

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Chapter II :

Nanomaterials and Photocatalysis

1. Introduction

The rapid emergence of nanotechnology over the past decade has introduced novel avenues across diverse scientific disciplines, including medicine and biotechnology. This advancement is largely attributed to nanomaterials, which exhibit distinct characteristics stemming from their nanoscale dimensions and significantly high surface-to-volume ratios, thereby attracting considerable research interest [1].

Among the array of nanomaterials, zinc oxide nanoparticles (ZnO NPs) have garnered substantial attention due to their unique attributes, including cost-effectiveness, biocompatibility, and versatile physicochemical properties. Consequently, plant-mediated green synthesis has become an increasingly favored approach for producing ZnO NPs, as it offers an eco-friendly alternative that allows for the modulation of nanoparticle characteristics through the selection of plant extracts and synthesis conditions[2].

Photocatalysis stands out as a highly promising application for nanomaterials, particularly in addressing environmental concerns such as water pollution. The efficacy of semiconductor photocatalysts is often challenged by factors like limited light spectrum utilization and the rapid recombination of photogenerated charge carriers, which can hinder their overall performance in degrading pollutants, necessitating strategies such as doping to improve performance [3].

This chapter will explore how the unique properties of nanomaterials, particularly zinc oxide, can be leveraged and optimized for advanced photocatalytic applications.

2. Nanomaterials

2.1 Concept of Nanotechnology

Nanotechnology is recognized as a multidisciplinary research area focused on materials with nanoscale dimensions, typically ranging from 1 to 100 nanometers. Such materials exhibit unique and improved properties that fundamentally distinguish them from their bulk counterparts, primarily owing to the significantly high surface area to volume ratio inherent in these fine particles [4].

The ability to control particle size, reducing it below a specific critical threshold contingent upon the material's nature and processing conditions, results in the emergence of novel and compelling properties. Quantum effects are particularly prominent at this scale, paving the way for innovative applications previously unattainable with conventional bulk materials [5].

A core tenet of nanotechnology is the strategic employment of nanoparticles to bestow new or significantly enhanced functionalities upon diverse materials. For instance, nanoparticles possessing photocatalytic capabilities can be incorporated into matrices such as paints or construction materials, thereby imparting advanced characteristics like self-cleaning, antimicrobial activity, or the purification of air from contaminants [6].

Nanostructured materials, including various metal oxides, exhibit tunable physicochemical properties, allowing for their synthesis with specific sizes and geometries tailored to the intended application. The efficacy and applicability of these materials are substantially influenced by the synthetic route, the precursors employed, and the cationic distribution within the crystal lattice, all of which impact surface characteristics and, consequently, their functional activity across diverse fields [7].

Table II. 1 Principles and Advantages of Nanotechnology[8]

Principle	Advantage
The ability to manipulate individual atoms and molecular structures; at the nanoscale, the physical and chemical properties of matter differ from those at the macro scale.	The possibility to build any material from atomic components and reconstruct all materials.
Nanotechnology is based on new physical, chemical, electrical, and mechanical properties that differ at the nanoscale compared to the bulk material.	Unique material properties are exploited in numerous innovations.

Nanotechnology relies on the engineering design of molecules and materials and the ability to control the arrangement of atoms.	Production of new materials with physical and chemical properties different from traditional materials.
Nanotechnology is based on fabricating materials from atomic foundations.	Improved material and device performance, enabling ultra-small and fast technologies.
Nanotechnology emphasizes scientific creativity with practical potential for innovations and beneficial applications.	Turns scientific imagination into reality.

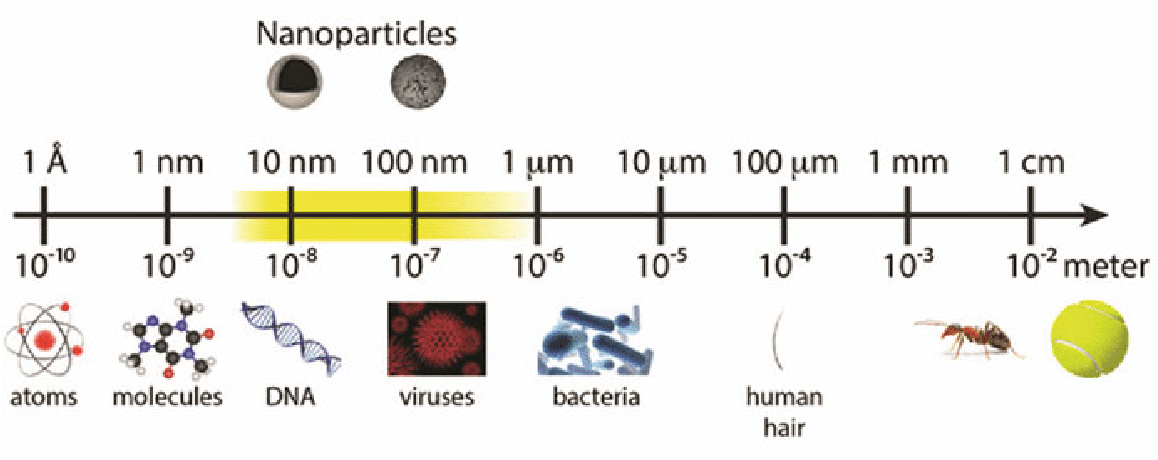

Figure 2. 1 Comparison of different sizes of materials with nanoscale dimension. [9]

2.2 Definition and concept of nanomaterials

Nanomaterials are materials that have at least one external dimension within the range of 1 to 100 nanometers, or contain internal or surface structures within this range. These materials exhibit new physical, chemical, and biological properties as a result of their nanoscale dimensions, which differ significantly from the properties of conventional bulk materials. For example, the large ratio of surface area to volume, along with the emergence of quantum effects at this scale, enhances their optical, magnetic, and electrical properties [10].

According to international standards and regulatory definitions, nanomaterials can be classified as natural, incidental, bio-inspired, or manufactured, depending on their source and production method. Natural nanomaterials form through natural processes such as volcanic eruptions or weathering, whereas intentionally designed nanomaterials are manufactured for specific applications. These definitions are important for regulating potential risks associated with nanomaterials to the environment and human health [11].

The international standard ISO/TS 80004 defines nanomaterials as "materials having specific external nanoscale dimensions, or internal or surface nanoscale structures," within the size range of 1 to 100 nanometers. Additionally, the European Commission classifies nanomaterials as any natural, incidental, or manufactured materials containing single, aggregated, or agglomerated particles, where 50% or more of these particles fall within the 1 to 100 nanometer range [11].

2.3 Properties of ZnO Nanoparticles

Zinc oxide nanoparticles (ZnO NPs) are materials of significant interest due to their versatile and controllable physicochemical properties. Structurally, ZnO typically exhibits a hexagonal Wurtzite crystal structure, which is its most energetically stable configuration. The surface characteristics of these nanoparticles, such as the presence of hydroxyl groups or ionic vacancies, play a pivotal role in determining their activity, and these characteristics can be enhanced through specific surface treatments without necessarily altering the bulk internal structure of the particle [12].

The morphological (shape) and size characteristics of ZnO nanoparticles are significantly influenced by the synthesis conditions, allowing for the attainment of diverse forms ranging from spherical particles, nanocones, and flakes to flower-like structures. The specific surface area plays an important role, as it can vary considerably with differences in preparation factors, such as the concentration of precursors in green synthesis methods. Optical properties, such as the band gap energy, are also affected by these factors, leading to variations in their light absorption range and their ability to respond to different spectra [13]. ZnO nanomaterials are distinguished by unique electronic and optical properties, including a wide and direct band gap (approximately 3.37 eV) and a large exciton binding energy at room temperature, leading to strong luminescence. They also possess good optical transparency, high electron mobility, and distinct piezoelectric and spontaneous polarization properties that influence defect formation and chemical reactivity, making them essential materials in many advanced technological applications [14].

When ZnO nanoparticles are prepared via biological routes, active compounds present in natural extracts, such as flavonoids and phenolic acids, play a dual role as reducing and stabilizing agents. This approach influences the final crystallite size, optical properties like the band gap value, and the ability to exhibit a blue shift in the absorption spectrum, which positively impacts their photocatalytic activity under visible light and their effectiveness as antibacterial agents [15].

The green synthesis of ZnO nanoparticles involves complex mechanisms, including nucleation and growth phases. Reaction conditions, such as temperature, reaction time, precursor concentration, and the nature of the reaction medium, directly control the final properties of the nanoparticles, including their size, shape, and stability, and consequently, their effectiveness in various applications like photocatalysis and antimicrobial activity [16].

The photocatalytic efficacy of ZnO nanoparticles depends on their ability to efficiently generate reactive oxygen species (ROS) upon appropriate light irradiation. Structural defects, such as oxygen and zinc vacancies, play a significant role in this process, as they can act as charge traps, thereby enhancing the separation of photogenerated electron-hole pairs and prolonging their lifetime. Despite their high activity, the stability of ZnO in certain environments, such as acidic solutions or under prolonged UV irradiation in alkaline solutions, presents a challenge that must be considered when designing practical applications [17].

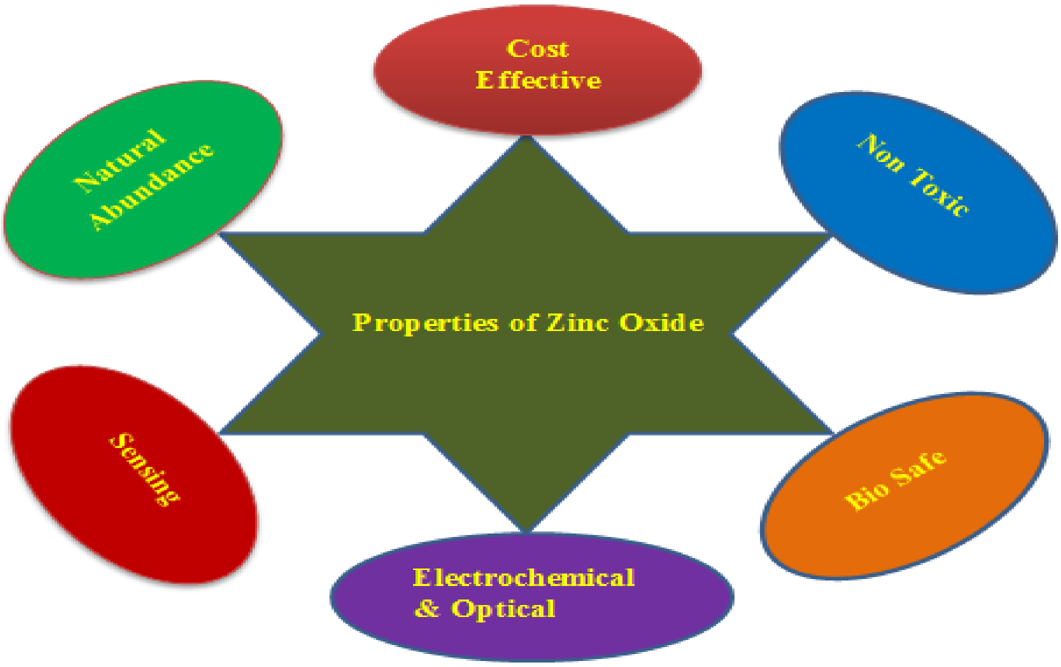

Figure 2. 2 Some Appealing Properties of Zinc Oxide Nanoparticles[18]

Table II. 2 Characteristics of ZnO nanoparticles[19]

Characteristic	Value
Purity	+99%
Color	Milky white
Size	35–45 nm
True density	5.606 g/cm³
Specific surface area (SSA)	~65 m²/g
Thermal conductivity	19 W/m·°C
Specific heat	544kg·°C

2.4 Classification of Nanoparticles

Nanomaterials are primarily classified based on their dimensions into four different categories, defined as materials where at least one dimension is within the nanoscale, i.e., smaller than 100 nanometers. These categories include zero-dimensional (0D) nanoparticles, where all three dimensions are in the nanoscale range, such as quantum dots and fullerenes; one-dimensional (1D) nanoparticles, which have one dimension outside the nanoscale, like nanotubes, nanofibers, and nanorods; two-dimensional (2D) nanoparticles, with two dimensions outside the nanoscale, such as nanosheets and thin films; and finally, three-dimensional (3D) nanomaterials or bulk nanomaterials, which are not confined to the nanoscale in any dimension and include nanopowders and arrays of nanotubes [20].

Nanoparticles can generally be categorized into three main classes based on their chemical composition: organic nanoparticles, inorganic nanoparticles, and carbon-based nanoparticles. Organic nanoparticles, such as liposomes, micelles, and dendrimers, are primarily composed of organic molecules and are often characterized by their biodegradability and sensitivity to external factors like heat and light. Inorganic nanoparticles, on the other hand, are those that do not primarily contain carbon and include metallic nanoparticles (e.g., gold and silver) and metal oxide nanoparticles (e.g., zinc oxide and titanium oxide); this class is distinguished by unique properties dependent on quantum size effects and large surface areas [21].

Nanoparticles represent a broad class of materials that include metallic nanoparticles such as gold, silver, and copper, which are noted for their antibacterial and antiviral properties, and carbon-based nanoparticles like fullerenes, carbon nanotubes, graphene, and carbon nanofibers, which exhibit distinct mechanical, chemical, and physical characteristics. This group also encompasses metal oxide-based nanoparticles, which are synthesized from metals converted to their corresponding oxides, and these particles often display enhanced qualities compared to their metallic counterparts [22].

2.5 Applications of nanoparticles

Nanoparticles exhibit tremendous potential in the biomedical field, where their applications extend to include the development of advanced drug and gene delivery systems, precise detection of proteins, and contributing to the treatment of complex diseases such as diabetes through improved insulin delivery, and in tissue engineering for repairing and regenerating damaged organs. They also play a role in exploring DNA structure, tumor eradication, and developing innovative cancer therapies that target affected cells with high precision [23].

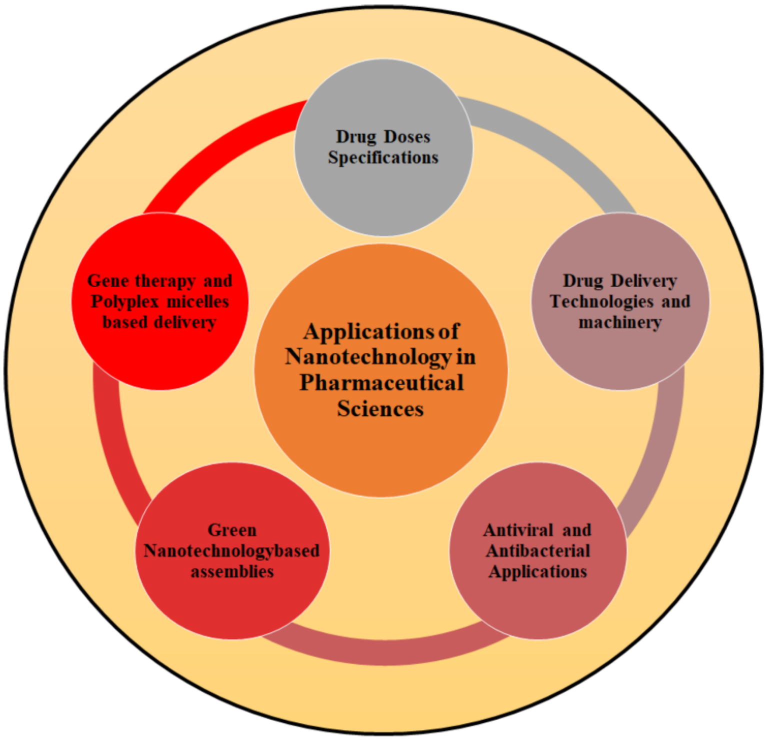

Figure 2. 3 Applications of nanotechnology in pharmaceutical sciences **[24]**

In the context of environmental protection and clean energy generation, bionanoparticles (BNPs) are utilized in the bioremediation of diverse pollutants, improving water treatment quality, as well as contributing to the production of new forms of renewable energy. For instance, bacteria-derived magnetic nanoparticles serve as promising alternatives to traditional drug carriers due to their unique properties, such as a narrow nanoscale size distribution and paramagnetism, which enhances the efficiency of drug delivery, for example, with Doxorubicin[25].

The applications of nanoparticles extend to the electronics sector, where they are used in developing nanoelectronic components such as transistors made from silicon nanowires or carbon nanotubes, and quantum dots that exhibit unique quantum confinement effects. They are also incorporated into the manufacturing of high-definition displays, and utilized in molecular electronics, spintronics, with their potential in building quantum computers being explored. In the field of nanophotonics, plasmonic nanoparticles are employed to enhance solar cell efficiency and in the development of innovative optical communication devices [26].

Nanoparticles find diverse applications across multiple industries. In the field of cosmetics, particles such as titanium dioxide and zinc oxide are incorporated into sunscreens for their properties in absorbing and reflecting ultraviolet radiation. In the construction sector, the addition of nanosilica particles contributes to improving the mechanical properties and durability of concrete, while titanium dioxide nanoparticles are used in self-cleaning and anti-pollution coatings. As for the food industry, they are utilized in developing smart packaging that extends product shelf life and are incorporated into the manufacturing of antimicrobial films[27].

Nanoparticles are revolutionizing the solar energy sector through their use in nanowire-based solar cells that improve light absorption and charge transport, as well as in solar thermal collectors where nanofluids are employed to enhance radiation absorption. In the field of fuel cells, carbon nanotubes are integrated into membranes and catalysts to improve performance and reduce costs. Furthermore, nanocomposite materials are used in the manufacturing of wind turbine blades to increase their strength and durability while reducing weight, thereby contributing to the efficiency of energy generation [28].

Figure 2. 4 **Applications of nanamaterials** ***[29]***

2.6 Methods of nanomaterial synthesis[29]

A variety of methodologies exist for the synthesis of nanomaterials, each differing significantly in its fundamental principles, resource requirements, and associated impacts. These methods can be broadly categorized into physical, chemical, and increasingly important green (biological) approaches, with the latter representing a significant shift towards more environmentally conscious practices.

2.6.1 Physical Methods

Physical Physical methods for synthesizing nanoparticles, often referred to as the top-down approach, rely on reducing the size of larger, bulk materials to the nanoscale. These methods utilize high energy such as radiation, condensation, or thermal energy. Prominent methodologies include high-energy ball milling, laser ablation, electrospraying, inert gas condensation, physical vapor deposition, laser pyrolysis, flash spray pyrolysis, and melt mixing. High-energy ball milling (HEBM) is a mechano-chemical process where moving balls are used to break the chemical bonds of the milled substances through their kinetic energy, leading to structural changes and chemical reactions at room temperature. This method is characterized by its ability to generate very high local temperatures and pressures. Advantages of ball milling include its economy compared to some other methods and the ability to produce small amounts of nanoparticles in a relatively short time. However, a disadvantage is that the electrostatic forces generated by surfactants, which prevent agglomeration, can significantly enhance the particle size. Inert gas condensation uses inert gases like helium or argon with liquid nitrogen as a substrate holder for preparing nanoparticles and is considered very effective for synthesizing silver and platinum nanoparticles, with the advantage of depositing nanoparticles in a silicon shell, which prevents their oxidation and maintains their aggregation. Physical vapor deposition (PVD) is a collective synthesis process based on vacuum deposition technology, involving the vaporization of metal particles from a solid state, then transportation of the vaporized particle, and finally nucleation and formation of thin films of nanoparticles. PVD techniques include sputtering and electron beam evaporation (EBE). Sputtering, especially using magnetron, is characterized by higher deposition rates and prevents target overheating and damage, while electron beam evaporation is suitable for producing thin films and nanoparticles and has been used in developing 2D and 3D metal patterning. Laser ablation (LA) and pulsed laser deposition (PLD) involve evaporating particles from a solid source using a high-power laser beam. This method is applicable for producing polymeric materials and allows control over the size and shape of nanoparticles by optimizing argon gas pressure and the number of pulses, although a disadvantage is that particle shape can change with pressure. Other physical methods include vacuum arc (VA), laser pyrolysis, electrospraying technique, and finally, the melt mixing technique [30].

2.6.2 Chemical Methods

Chemical methods for synthesizing nanoparticles, often referred to as the bottom-up approach, rely on assembling basic units of nanoparticles (such as atoms or molecules) to form more complex structures, where a specific chemical and physical effort is used to pack the basic units of nanoparticles into a complex structure. Commonly used chemical methods include the sol-gel method, micro-emulsion technique, hydrothermal synthesis, polyol synthesis, chemical vapor synthesis, and plasma-enhanced chemical vapor deposition. The sol-gel method combines two components: "sol," a colloidal suspension of solid particles in a liquid, and "gel," a liquid containing polymers. This process involves the formation of a "sol" in the liquid, leading to the formation of a network of discrete particles or network polymers by connecting sol particles. This method is characterized by its ability to achieve a quantum size effect in the manufactured nanoparticles, which is important for developing advanced optoelectronic devices. The micro-emulsion technique consists of three components: a polar aqueous phase, a second non-polar phase (hydrocarbon liquid or oil), and a surfactant. It is primarily used in the synthesis of inorganic nanomaterials such as metal nanoparticles and semiconducting metal sulfides, and is characterized by effective control in the synthesis of metal nanoparticles. However, a disadvantage is that controlling several parameters such as the water-to-surfactant ratio and the type of continuous phase is crucial. Hydrothermal synthesis is known for its ability to fabricate nanoparticles of metal oxides, iron oxide, and lithium iron, and offers an advantage in nanoparticle synthesis by optimizing size, morphology, composition, and surface chemistry. In polyol synthesis, metal-containing compounds are used as a reaction medium, acting as a reducing agent and complexing agent as well as dissolving stabilizing agents, and it is characterized by the ability to synthesize a wide range of metal-based nanoparticles. Chemical vapor deposition (CPD) is used for the deposition of solid films from the vapor phase under very high-temperature conditions, while plasma-enhanced chemical vapor deposition (PECVD) is suitable for thin film deposition and is distinguished from traditional chemical vapor deposition by its comparatively lower temperature requirements [31].

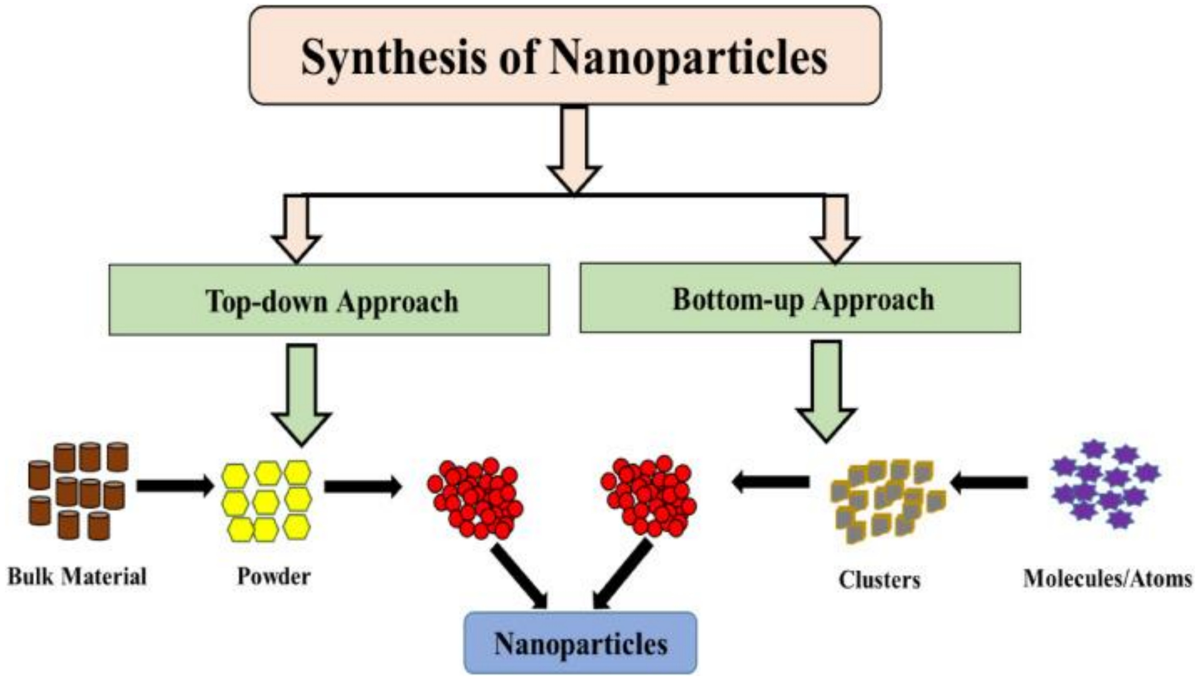

Figure 2. 5 Schematic representation of 'top-down approach' and 'bottom-up approach' for synthesis of nanoparticles.[32]

2.6.3 Green Synthesis (Biosynthesis)

Green synthesis of nanoparticles represents a sustainable and innovative approach within the field of nanotechnology, offering a viable alternative to conventional physical and chemical synthesis methods that often involve hazardous substances and energy-intensive conditions. This eco-friendly method employs biologically active agents—such as bacteria, fungi, yeast, viruses, and particularly plant extracts—as both reducing and stabilizing agents in the formation of metal and metal oxide nanoparticles. Plant-mediated synthesis, in particular, has attracted significant attention due to its simplicity, cost-effectiveness, environmental compatibility, and the avoidance of toxic chemicals.

Plants serve as rich reservoirs of phytochemicals, including phenolic compounds, flavonoids, terpenoids, alkaloids, polysaccharides, proteins, enzymes, and organic acids such as citric acid. These compounds play a crucial role in reducing metal ions to nanoparticles and simultaneously stabilize the formed particles by capping their surfaces. This dual functionality eliminates the need for additional chemical agents, making the process cleaner and safer. Furthermore, nanoparticles synthesized via plant extracts often exhibit enhanced stability and monodispersity. The plant-based approach also demonstrates a faster synthesis rate and greater scalability compared to microbial methods, largely due to the ease of handling and processing plant materials ,Among the key advantages of green synthesis is the ability to finely tune the physicochemical properties of the resulting nanoparticles—such as size, shape, and surface charge—by simply adjusting synthesis parameters, including the type and concentration of the extract, temperature, pH, and reaction duration. These controllable parameters allow for tailored nanoparticle synthesis suited to specific applications. A notable example of this is the use of Senegalia catechu leaf extract in the biosynthesis of iron oxide nanoparticles (IONPs), where phenolic compounds and flavonoids in the extract not only reduce the metal ions but also serve as effective capping agents. The resulting nanoparticles demonstrate improved stability and functional properties, opening pathways for their application in environmental remediation, particularly in the treatment of dye-contaminated wastewater Despite these promising advantages, green synthesis also presents several limitations and challenges. One of the primary concerns is the incomplete understanding of the underlying biochemical mechanisms governing the reduction and stabilization processes, especially in the context of complex plant extracts that contain a multitude of interacting compounds. Moreover, achieving consistency and reproducibility in nanoparticle synthesis remains a challenge, as variations in plant composition, extraction methods, and reaction conditions can significantly influence the characteristics of the final product. Another critical consideration is the need for comprehensive toxicological studies to evaluate the long-term safety and potential environmental impact of biosynthesized nanoparticles before their adoption in sensitive applications such as medicine or food packaging In conclusion, while green synthesis of nanoparticles offers a promising and sustainable route with clear environmental and functional advantages, further research is necessary to address existing challenges, optimize protocols, and ensure the safe and reproducible production of nanoparticles for practical applications [33, 34]

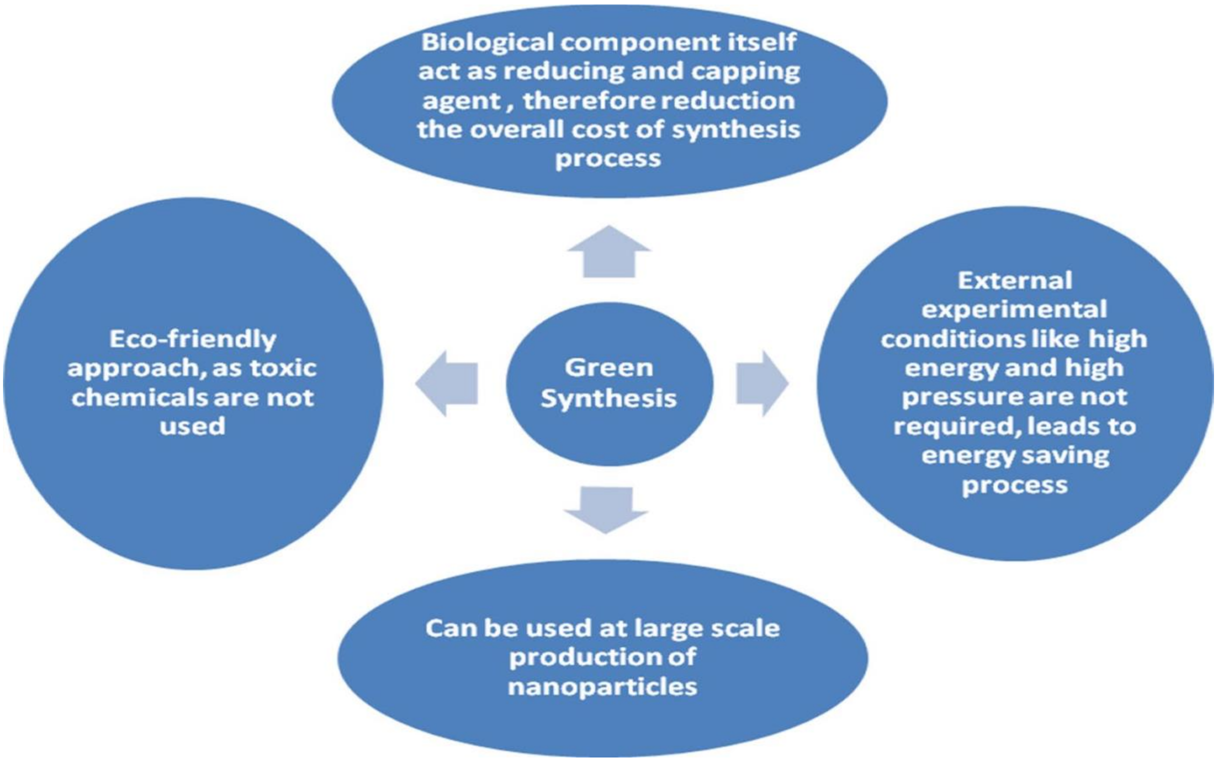

Figure 2. 6 Key merits of green synthesis methods.[35]

2.6.4 Importance of Sustainability in Chemical Processes and Nanomaterial Synthesis

The importance of sustainability in chemical processes and nanomaterial synthesis is increasingly prominent due to the growing awareness of the potential environmental and health impacts associated with conventional nanotechnology. Although nanomaterials offer unique properties beneficial across numerous fields, traditional chemical and physical synthesis methods often entail high energy consumption, the use of ecotoxic reagents, and can result in low material conversion rates. Furthermore, the very nature of nanoscale materials allows them to interact readily with biological systems and undergo physicochemical alterations when released into the environment, raising concerns about their fate and effects. These concerns have spurred the academic, industrial, and governmental sectors to actively pursue the development of sustainable synthetic routes, adopt systemic thinking, and implement standardization and regulation. This collective effort is encapsulated in the emergence of "green nanoscience," which seeks to integrate the principles of green chemistry into nanotechnology. The core objective is to foster the ecofriendly development of nanotechnology by creating safer and more sustainable products and processes, thereby minimizing ecological footprints and contributing to innovative remediation strategies for environmental restoration. However, a significant challenge lies in the qualitative nature of many "green" principles, necessitating the development and adoption of quantifiable metrics—such as atomic efficiency, waste-to-product ratio, energy efficiency, and life cycle assessments—to objectively evaluate the sustainability and potential environmental impact of nanomaterial synthesis processes [36].

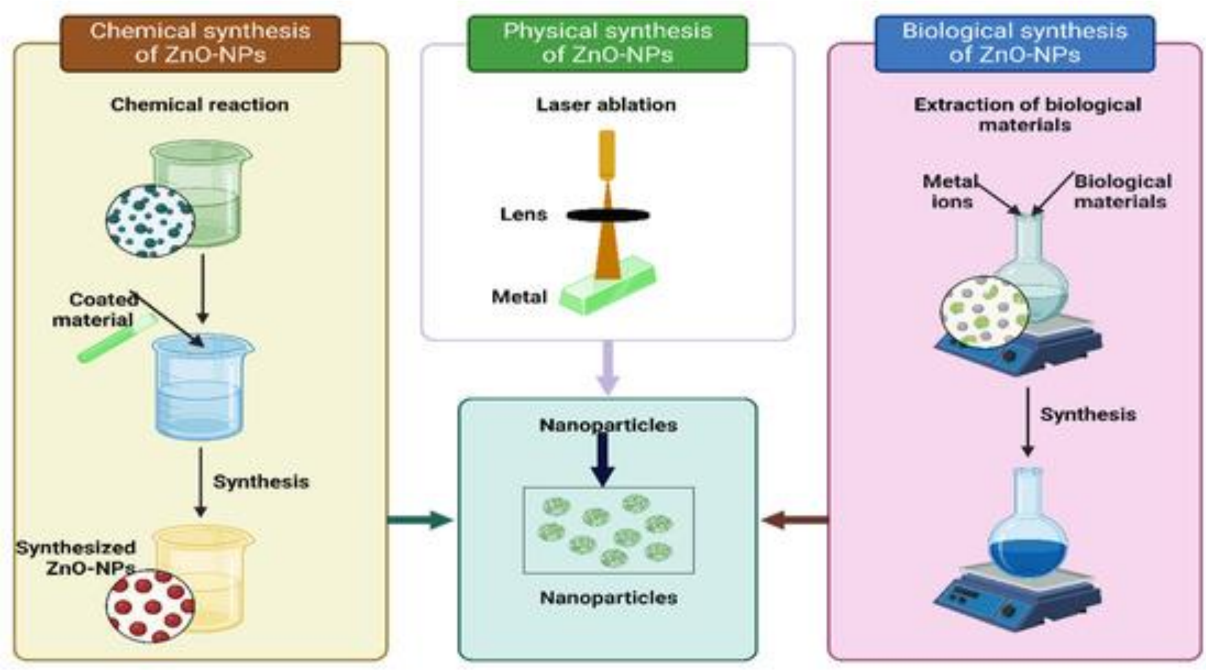

Figure 2. 7 Different methods of used for the synthesis of ZnO-NPs[37]

3. Photocatalysis

3.1 The concept of photocatalysis

The term "photocatalyst" is a combination of two words: "photo," which relates to light or photons, and "catalyst," which is a substance that alters the rate of a chemical reaction simply by being present. Therefore, photocatalysts are materials that speed up or slow down chemical reactions when exposed to light. This entire phenomenon is known as "photocatalysis." The process of photocatalysis includes reactions that utilize light and a semiconductor material to proceed. The substance that absorbs light and acts as a catalyst for chemical reactions is what we call a "photocatalyst." It's worth noting that all photocatalysts are essentially semiconductor materials. Photocatalysis can be described as a phenomenon in which a pair of electrons and holes (positive charges) is generated when a semiconductor material is exposed to light.

Photocatalytic reactions can be categorized into two main types, based on the physical state of the reacting substances:Homogeneous photocatalysis: When both the semiconductor material (the catalyst) and the reactant are in the same physical state (whether gaseous, solid, or liquid) these reactions are termed homogeneous photocatalysis.

Heterogeneous photocatalysis: However, when the semiconductor material and the reactant are in different physical states, this type of reaction is classified as heterogeneous photocatalysis.

The energy difference between what is called the "valence band" (or HOMO, which is the highest occupied molecular orbital) and the "conduction band" (or LUMO, which is the lowest unoccupied molecular orbital) is known as the "band gap"[38].

3.2 Scientific principle of Photocatalysis

Photocatalysis using semiconductor materials is based on a series of fundamental photophysical and photochemical processes. The mechanism begins with the absorption of photons whose energy equals or exceeds the semiconductor’s band gap. This results in the excitation of electrons from the valence band to the conduction band, thereby generating electron-hole pairs. These charge carriers are the driving force for all subsequent reactions. For effective photocatalytic activity, it is crucial that these excited species avoid recombination, as recombination leads to the loss of usable energy in the form of heat or light. Instead, the efficient separation and transport of charge carriers to the surface of the photocatalyst enables redox reactions with adsorbed species, initiating the desired chemical transformations[39].

The intrinsic mechanism of photocatalysis, regardless of the specific details of reactions and pathways, can be described through four main and consecutive stages. The process begins with (I) the absorption of light by the photocatalytic material, leading to the generation of electron-hole pairs. This is followed by stage (II) the efficient separation of these excited charges to prevent unproductive recombination. Then, (III) the separated electrons and holes migrate to the surface of the photocatalyst. Finally, (IV) these surface-localized charges are utilized to catalyze redox reactions with the adsorbed species [39].

Photocatalysis is defined by its ability to accelerate photo-induced chemical reactions through the presence of a semiconductor catalyst, where this process is controlled by modifying the electron density in the conduction band. This modification is effectively achieved upon the absorption of sufficient light energy to cause an electronic excitation, whereby an electron transitions from the valence band to the conduction band. As a result of this process, the semiconductor generates electron-hole pairs, which in turn create the conditions for subsequent redox reactions, often involving radical-based chemical pathways [40].

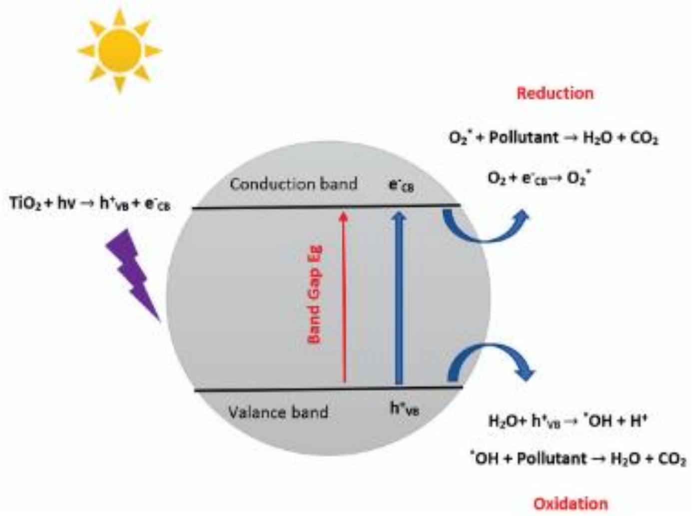

Figure 2. 8 ***Schematic illustration of the photocatalytic reaction mechanism*** **[41]**

3.3 Photocatalytic Materials

Nanomaterials possess significant potential in photocatalysis, as their superior size-dependent features – including unique structure, optical properties, and tunable electronic characteristics – endow them with remarkable catalytic performance and novel functionalities. This research area focuses on the design and development of nanomaterials for photocatalysis, emphasizing the description of synthesis methods, physicochemical properties, and the understanding of complex relationships between processing, resultant structure, and final properties of photoactive nanomaterials proposed for various applications. Among these applications, the degradation of environmental pollutants and hydrogen production are prominent, with recent studies, for example, demonstrating the efficacy of platinum nanoparticles deposited on zinc oxide nanorods (ZnO-Pt) in degrading low-density polyethylene (LDPE) microplastic fragments in water under visible light, highlighting the enhanced capability of plasmonic photocatalysts [42].

The range of materials used as photocatalysts is diverse and has significantly evolved from traditional inorganic materials to the exploration of new and advanced nanomaterials. These modern materials include graphitic carbon nitride (g-C_3N_4) as a promising metal-free semiconductor polymer, other polymers, materials with piezoelectric and ferroelectric properties that aid charge separation, Metal-Organic Frameworks (MOFs) and Covalent Organic Frameworks (COFs) known for their high porosity and tunability, Single-Atom Catalysts (SACs), and High-Entropy Alloys (HEAs) targeting maximum atomic efficiency. Furthermore, two-dimensional (2D) materials (such as few-layer BiOBr with oxygen vacancies) and materials that exploit Localized Surface Plasmon Resonance (LSPR) to enhance visible light absorption, hold a significant place among modern photocatalytic materials [43].

Semiconductors represent the primary and most common class of materials in photocatalytic applications, with their operational principle based on possessing a specific band gap energy. Upon absorption of a sufficient photon, electrons are excited from the valence band to the conduction band, generating electron-hole pairs responsible for the catalytic activity. Among semiconductors, titanium dioxide (TiO_2) and zinc oxide (ZnO) remain the most widely used and studied, owing to their effectiveness, chemical and thermal stability, low cost, and non-toxicity. Other metal oxides like WO_3, Fe_2O_3, and SnO_2, as well as metal sulfides such as CdS and CdSe, are also employed as photocatalytic materials, noting that most of these traditional wide band gap materials typically require UV radiation for activation [44].

To overcome the inherent limitations of single photocatalytic materials, such as high electron-hole recombination rates or poor visible light absorption, advanced design strategies involving the creation of composite materials or the construction of heterojunctions are employed. These strategies aim primarily to improve the separation of photogenerated charges by directing electrons and holes to different materials based on their respective energy band positions, thereby minimizing recombination. They also seek to broaden the light absorption range, particularly into the visible region, by using a narrow band gap semiconductor capable of visible light absorption coupled with another material possessing good catalytic activity. Furthermore, one component of the composite material can serve as an active site for charge accumulation and facilitate surface chemical reactions. Beyond solid-state semiconductors, molecular photocatalysts, such as polyoxometalates (POMs) and certain transition metal complexes like those of rhenium and ruthenium, are also utilized in specific applications requiring different catalytic mechanisms or reaction conditions [45].

3.4 Applications of Photocatalysis

Photocatalysis demonstrates vast potential in addressing numerous environmental and industrial challenges, owing to its capacity to harness light energy for catalyzing chemical reactions. The applications of this technology are diverse, encompassing various vital fields, thereby positioning it as a focal point of escalating research and applied interest. One of the most prominent applications of photocatalysis lies in the treatment of polluted water. This technology, particularly when employing titanium dioxide (TiO_2) as a catalyst, is utilized for the degradation of a broad spectrum of chemical and organic pollutants. These include recalcitrant compounds such as pesticides, fungicides, dyes, and nitrogenous compounds, which photocatalysis can completely oxidize into carbon dioxide, water, and simple inorganic substances. Furthermore, the efficacy of this technique extends to the elimination of pathogenic microorganisms like bacteria, viruses, and fungi present in water, thereby underscoring its significance as a promising tool for water purification and disinfection[46].

In a related context, photocatalysis finds significant application in the remediation of organic pollutants, where it serves as an effective tool for the degradation of complex and hazardous chemical compounds. For instance, studies have demonstrated the capability of photocatalytic materials, such as graphitic carbon nitride ($g\text{-}C_3N_4$) and its composites, to decompose organic pollutants like industrial dyes, including Rhodamine B (RhB), upon exposure to visible light. This process extends beyond mere decolorization, aiming to break down the molecular structure of the pollutant, transforming it into less toxic or harmless substances, thereby representing a promising strategy for treating water contaminated with industrial dyes originating from various industria lactivities[47].

Attention is increasingly directed towards photocatalysis as a sustainable technology for clean energy generation, particularly in the production of hydrogen from water using solar energy. Photocatalytic nanomaterials absorb photons to generate electron-hole pairs, which subsequently drive the oxidation and reduction reactions of water molecules, leading to their dissociation into hydrogen and oxygen gases. This pathway presents a promising solution for addressing energy and climate change challenges by providing an environmentally friendly and renewable fuel source[48].

Furthermore, photocatalysis, particularly utilizing materials based on titanium dioxide (TiO_2), sometimes integrated with other substances like silicon dioxide (SiO_2), contributes to the development of self-cleaning surfaces or in applications related to the adsorption or degradation of dyes. When thin films of these photocatalytic materials are applied to various surfaces, their exposure to light, whether solar or artificial, can lead to the oxidation and decomposition of organic pollutants, dirt, or adsorbed dyes. This characteristic makes them ideal for use in building materials, glass, and even in the development of colored optical filters or surfaces capable of removing adhered contaminants [49].

4. Conclusion

This chapter reviews recent scientific literature concerning the synthesis of nanomaterials, whether metallic or semiconducting in nature, which can be photostimulated within the UV-Vis range. It also explores their potential applications as photocatalytic materials, particularly in the realm of environmental pollutant removal on a laboratory scale.

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Chapter III: Characterization Techniques

1. Introduction:

To gain an understanding of the different properties of the nanoparticles produced, draw conclusions about the protocol used, and determine the factors that need to be improved to obtain good results, it is necessary to perform a series of analytical techniques. These techniques include X-ray diffraction (XRD), scanning electron microscopy (SEM), infrared spectroscopy, and UV-Vis spectroscopy.

Using these different techniques allows us to obtain comprehensive information on the physical, chemical and optical properties of the nanoparticles produced, which helps us to improve the methods used in their production and obtain better results [1],[2].

2. UV-Visible Spectroscopy

2.1 An Overview

UV-Visible spectroscopy is a fundamental analytical technique that explores how materials interact with light within the ultraviolet and visible portions of the electromagnetic spectrum. When a beam of light is directed through a sample—be it a solution or a solid material like a thin film—specific wavelengths are absorbed by its components. This absorption is intrinsically tied to the material's electronic structure, as the incident photons elevate electrons to higher energy levels, a concept that also underpins processes such as photocatalysis where absorbed light energy drives chemical reactions. By measuring the intensity of the transmitted or absorbed light across various wavelengths, a characteristic absorption spectrum is obtained. This spectrum can be used for qualitative identification or, more commonly, for the quantitative determination of a substance's concentration in various analytical contexts, including serving as a detection system for separation techniques like high-performance liquid chromatography [3-5]

Figure 3. 1 UV-Visible Spectrophotometer [6]

2.2 Mechanism of Operation and Metrics

The UV-Visible absorption spectrophotometry technique is used for sample analysis by relying on a device designed to analyze ultraviolet and visible light .This device operates using a double beam system (à double faisceaux) to ensure accurate results . For measurements, the sample is placed in special containers known as cuvettes, and in this case, quartz cuvettes were used .This technique allows the measurement of absorbance at specific wavelengths of light ,which aids in analyzing the sample's characteristics. The device is used to analyze the filtrate or the clarified liquid after the removal of solid particles [7]

2.3 Applications

UV-Visible Spectroscopy is widely utilized to characterize the optical properties of materials and determine their interaction with light .This technique allows for an understanding of the electronic structure of materials, which is crucial for assessing their suitability for light-dependent applications, including the estimation of band gaps .It is also effectively employed to monitor and follow the progress of degradation or other chemical processes over time by observing changes in spectral absorbance, thereby enabling the evaluation of process efficiency Furthermore, UV-Visible spectroscopy can detect specific optical phenomena, which are often indicative of the formation of particular nanostructures or the presence of certain components within materials It plays a significant role in confirming the successful synthesis or fabrication of materials [8],[9]

2.4 Factors Influencing Measurements

UV-Visible spectroscopy measurements involve recording the absorption spectrum of a solution, for instance, over a range like 190-1100 nm. A key aspect is the determination of the concentration of a target molecule by correlating its absorbance at a specific wavelength, often the wavelength of its maximum absorption peak (e.g., 664 nm for the molecule discussed in the paper), using principles such as the Beer-Lambert relationship. In processes where the target molecule undergoes changes, such as degradation, monitoring the overall UV-Vis spectrum is crucial. This includes considering the degradation pathway of the molecule and the potential formation of by-products, as these can affect the spectral readings and the interpretation of the extent of change [10]

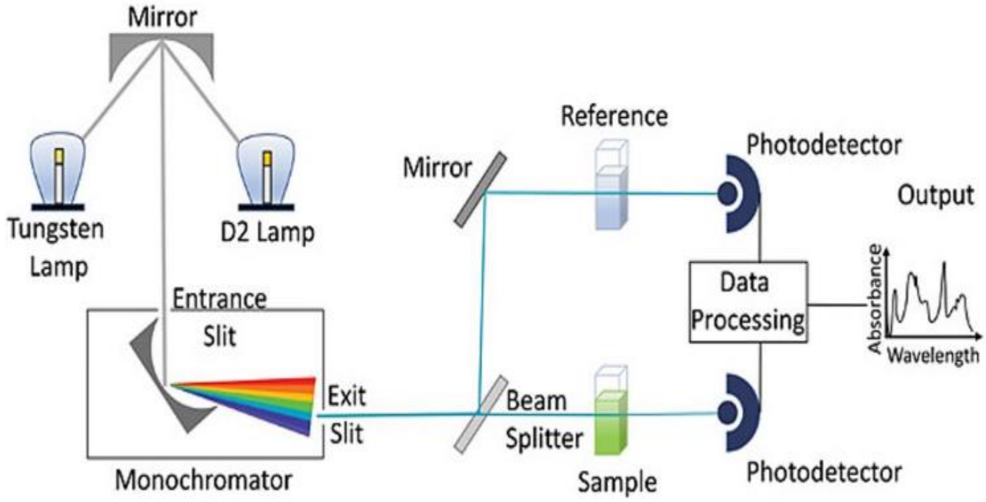

Figure 3. 2 ***Schematic setup of a UV-Vis spectrophotometer*****[11]**

3. FTIR Spectroscopy

3.1 An Overview

Fourier-Transform Infrared (FTIR) spectroscopy is a significant analytical technique employed for the identification of chemical substances and the elucidation of their molecular structure through interaction with infrared radiation, a portion of the electromagnetic spectrum. The fundamental principle of this method lies in the selective absorption of IR radiation by the chemical bonds within a sample. For absorption to occur, the chemical bonds must be IR-active, meaning they possess a dipole moment that changes during their characteristic vibrations. When the incident infrared radiation's frequency matches the natural vibrational frequency of these bonds, energy is absorbed. This process generates an absorption spectrum that is unique to each compound, often described as a molecular "fingerprint," which reveals the presence of distinctive functional groups and the overall chemical bonding. FTIR can be applied to a wide range of materials, including both organic and, in certain instances, inorganic substances, and is particularly useful for characterizing the chemical composition of materials like thin films [12], [13]

Figure 3. 3 Fourier transform infrared instrumental setup[14]

3.2 Mechanism of Operation

-infrared Fourier Transform Infrared (FTIR) spectroscopy operates on the fundamental principle of how molecules interact with infrared radiation. When IR light passes through a sample, molecules absorb this radiation at specific frequencies. These frequencies directly correspond to the natural vibrational and rotational modes of the chemical bonds within the molecule. For a molecule to absorb IR radiation and be "IR active," its dipole moment must change during the vibration. This selective absorption results in a unique spectrum for each compound, often described as a "molecular fingerprint," which allows for the identification and characterization of different substances, both organic and inorganic. Furthermore, functional groups, which are responsible for the main chemical properties of compounds, absorb IR radiation in distinct wavenumber ranges even when part of different molecules, facilitating their identification. Overall, this technique provides valuable insights into molecular structures, the nature and strength of chemical bonds, and potential intermolecular interactions. The midregion, typically spanning from 4000 to 400 cm^{-1}, is commonly utilized for these spectral analyses [15], [16]

3.3 Applications

Fourier Transform Infrared (FTIR) spectroscopy serves crucial roles in materials characterization, particularly for nanoparticles and solid surfaces. One significant application involves identifying the specific functional groups derived from plant extracts, such as hydroxyl (O-H), carbonyl (C=O), amine (N-H), and others. These groups are vital as they are accountable for the bioreduction of metal ions and the subsequent capping and stabilization of the synthesized nanoparticles via adsorption onto the particle surface .Furthermore, FTIR, especially utilizing the Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) technique, is extensively applied to investigate the surface characteristics of materials like metal oxides (e.g., TiO_2). This includes probing surface acidity and basicity through the adsorption of specific molecules (like CO_2, NO, benzene, ammonia) and identifying the nature of the adsorbed species (e.g., carbonates, bicarbonates, nitrates, nitrites, phenoxy groups) and their bonding modes to surface sites .DRIFTS is particularly effective for analyzing powdered samples directly with minimal preparation required [17],[18].

3.4 Factors Influencing Measurements

When employing Fourier Transform Infrared (FTIR) spectroscopy, a widely utilized characterization tool for materials such as nanoparticles or for analyzing specific components like impurities in semiconductors, several essential factors influence the accuracy and reliability of the measurements. Among these, precise control over the sample temperature is of critical importance. In certain situations, for instance, when analyzing interstitial oxygen in silicon, it becomes necessary to cool the sample to very low temperatures (e.g., 4.2 K) using specialized cooling techniques such as liquid helium, in order to resolve overlapping absorption bands and enhance their clarity. The aperture size of the instrument, through which the infrared beam passes, also plays a role in determining the analyzed area and consequently impacts the collected signal. Furthermore, the type of detector employed, such as MCT detectors, and the spectral range it can cover are key determinants for the measurement, potentially requiring the use of different detectors or even higher intensity light sources like synchrotron radiation for advanced applications. Lastly, the significance of the sample preparation method cannot be overlooked; for example, analyzing certain samples in transmission mode necessitates preparing them as thin films or precise cross-sections to ensure the radiation passes through appropriately [19], [20]

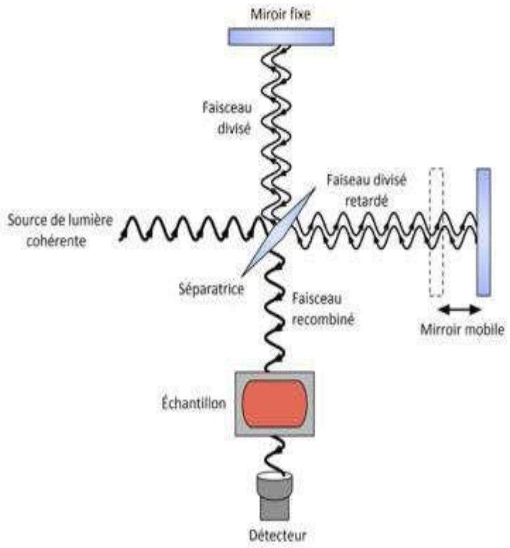

Figure 3. 4 Principle of FT-IR Spectroscopy [21]

4. X-Ray Diffraction (XRD)

4.1 An Overview

X-ray diffraction (XRD) is employed as a primary characterization technique to analyze the crystalline structure of nanomaterials such as ZnO and Pd-Au/ZnO nanoparticles. This technique provides diffraction patterns (spectra) containing peaks that appear at specific angles (2-theta values). These peaks correspond to particular crystallographic planes within the material, allowing for the identification of its crystal phase. For example, the observed XRD patterns revealed the wurtzite phase of ZnO, consistent with the standard card (JCPDS No. 36-145), and also enabled the determination of the lattice constants [22]

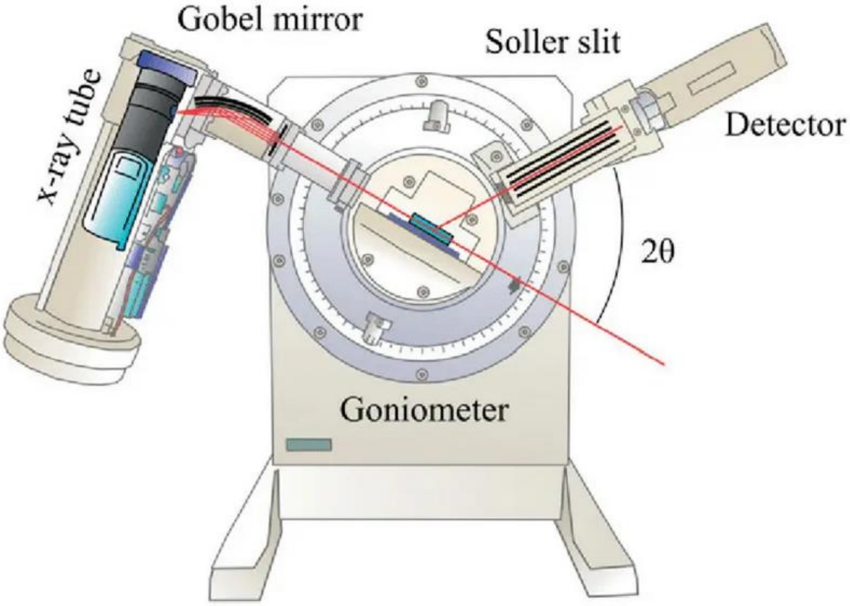

Figure 3. 5 Schematic representation of XRD technique[23]

4.2 Mechanism of Operation and Metrics

X-Ray Diffraction (XRD) is an analytical technique fundamentally based on the interaction of X-rays with matter, specifically the diffraction phenomenon that occurs when X-rays encounter a crystalline material. The mechanism involves an incident X-ray beam interacting with the electron clouds of atoms within the sample. If the material possesses a crystal structure, these scattered X-rays will interfere constructively at specific angles, governed by Bragg's Law ($n\lambda = 2d \sin\theta$), which relates the X-ray wavelength (λ), the interplanar spacing (d) of the crystal lattice, and the diffraction angle (θ). The analysis of the resulting diffraction pattern, which consists of peaks at various diffraction angles, allows for the determination of several key metrics. These include identifying the crystalline phases present (by comparison with standard data, such as JCPDS cards), calculating lattice parameters (the dimensions of the unit cell), and estimating the average crystallite size, particularly for nanomaterials, often by applying the Debye-Scherrer formula. Furthermore, XRD data can be used to evaluate stress and lattice strain within the crystalline structure, and to determine the dislocation density [24], [25]

4.3 Applications

The X-ray diffraction (XRD) technique is utilized as an essential tool for examining the structural properties of materials, enabling the determination of the crystal structure and the assessment of phase purity for the studied sample. It also allows for the identification of the present phase components by comparing the resulting diffraction patterns with standard reference data. Additionally, XRD data are used to estimate the crystallite diameter and can be employed for detailed structural analysis using methods such as Rietveld refinement [26]

4.4 Factors Affecting the Measurements

According to the methodology described in the referenced study ,X-ray diffraction (XRD) analysis is influenced by several essential factors. The wavelength (λ) of the radiation used (such as the mentioned Cu Kα with a wavelength of 1.5406 Å) and the instrument's operating conditions, including voltage (40 kV), current (40 mA), and the angular scanning range (2θ), are fundamental parameters that determine the nature of the collected data. Structural information is extracted from the resulting diffraction pattern; the position of the diffraction peaks (Bragg angle θ) is used to identify crystalline phases and their corresponding atomic planes .Furthermore, the peak width at half maximum (β or FWHM), in conjunction with the wavelength and Bragg angle, is utilized in equations like the Scherrer formula to estimate the average crystallite size (D) of the material .The intensity and shape of the peaks in the diffraction pattern can also provide indications of the sample's degree of crystallinity [27]

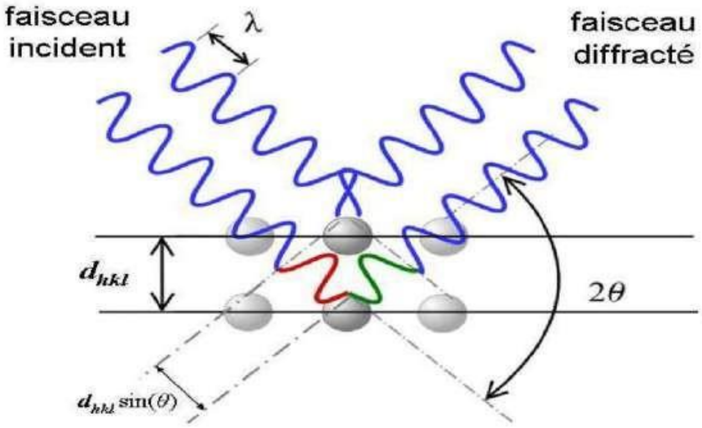

Figure 3. 6 Principle of X-ray Diffraction (XRD) [21]

5. Scanning Electron Microscopy (SEM)

5.1 An Overview

Scanning Electron Microscopy, specifically the Field Emission type (FESEM), is an essential technique used to identify and study the morphological characteristics of prepared materials. This technique allows for obtaining detailed images of the sample surface, revealing important information about the shape, size, and distribution of the constituent particles. Consequently, this type of analysis enables the understanding and precise characterization of the material's external structural features at the micro or nano level [28]

Figure 3. 7 SEM Quanta device[29]

5.2 Mechanism of Operation and Metrics

To investigate the surface characteristics and structural details of the zinc oxide nanoparticles synthesized through both chemical and green methods, a Scanning Electron Microscope (SEM) was utilized. Specifically, an FEI Quanta 250 SEM model, manufactured by Thermo Fisher Scientific, was employed for this purpose. This advanced instrument allowed for dynamic in-situ analysis of the various samples in their natural state and was also crucial for the structural and chemical analysis of metallographic specimens. The SEM boasts impressive specifications, including a magnification capability of up to 1,000,000 times and a high spatial resolution down to 3 nanometers. In the context of this particular study, various magnifications such as 10,000× and 50,000× were utilized to effectively characterize the nanoparticle morphology [30]

5.3 Applications

Scanning Electron Microscopy (SEM) is a widely utilized analytical technique for material characterization, particularly for nanoparticles. It allows for detailed examination of a material's surface, providing crucial insights into its morphology, such as particle shape, size, and distribution. SEM is also instrumental in visualizing the surface topography and identifying any agglomeration or aggregation of particles. Furthermore, when coupled with Energy Dispersive X-ray Analysis (EDX), SEM can determine the elemental composition of the scanned area, making it an invaluable tool for understanding the microstructure and overall surface characteristics of materials [31], [32]

6. Conclusion

Nanoparticles play a pivotal role in advancing the field of photocatalysis , largely due to their unique physicochemical properties such as quantum size effects and high surface-to-volume ratios, which contribute to enhanced photocatalytic performance compared to bulk materials . Understanding and characterizing these nanomaterials is crucial, and advanced analytical techniques are indispensable for this purpose. UV-Visible spectroscopy (UV-Vis) enables the study of optical properties, determination of band gap energy, and observation of quantum confinement effects .Fourier Transform Infrared spectroscopy (FTIR) provides valuable insights into surface chemistry and the functional groups present on the nanoparticles X-ray Diffraction (XRD) is essential for determining the crystal structure, phase, and crystallite size of these nanomaterials .Finally, Scanning Electron Microscopy (SEM) offers direct visualization of the morphology, size, and distribution of the nanoparticles. The integrated application of these techniques, as highlighted in the literature, facilitates a comprehensive understanding of the relationship between nanoparticle properties and their photocatalytic efficiency, both in environmental remediation applications and in advanced organic synthesis ,thereby paving the way for the design of innovative and efficient catalysts for sustainable application[33].[34]

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The applied part

Chapter IV : Synthesis, Characterization, and Application of Nanomaterials in Photocatalysis

1. Introduction

This chapter presents the methodology for the green synthesis of ZnO nanoparticles as an environmentally friendly alternative to conventional chemical methods. The synthesis was carried out using an extract from Sonchus wightianus leaves, which served as a natural reducing and stabilizing agent.

The synthesized nanoparticles were then subjected to a series of physicochemical analyses to determine their crystal structure, surface characteristics, and optical behavior, using advanced techniques such as UV-Vis spectroscopy, FTIR and XRD. The final part of the work involved evaluating the photocatalytic efficiency of the ZnO nanoparticles in degrading two organic dyes (Methylene Blue and Rose Bengal) under sunlight exposure, in order to assess their potential as effective photocatalysts for pollutant removal.

2. Materials and tools and equipment used

2.1 Materials

- Sonchus wightianus plant leaf powder
- Distilled water
- Zinc acetate ($C_4H_6O_4Zn$)
- NaOH (Sodium hydroxide)
- Methylene blue
- Rose bangle

2.2 Tools

- Test tube rack
- Filtration funnel
- Filter paper (Whatman No: 42)
- Magnetic stirrer
- Metal spatula
- Graduated cylinder (50 ml + 1000 ml capacity)
- Beaker (50 ml + 100 ml + 250 ml + 500 ml + 1000 ml capacity)
- Volumetric flask
- Crystallization dish

- Strainer / Sieve
- Cloth piece
- Aluminum foil
- Petri dishes

2.3 equipment used

- Analytical balance
- Mechanical grinder
- Drying oven
- pH meter
- Heating and stirring device
- Centrifuge
- Refrigerator
- SECOMAM UV-visible spectrophotometry, model 9600
- Fourier transform infrared spectroscopy FTIR (Thermo Fisher Scientific, model Nicolet iS5)
- X-ray diffraction using a Proto Manufacturing Company bench-top diffractometer

3. Steps for green synthesis of Nanomaterials

3.1 Preparation of the Plant

Fresh leaves of Sonchus wightianus were collected from a forest area and thoroughly cleaned with tap water multiple times until the wash water ran clear. Subsequently, the leaves were rinsed with distilled water to remove any remaining impurities. After allowing excess water to drain, the leaves were evenly spread on a large sieve (or tray) and left to air-dry at room temperature for 15 days. Once dried, the leaves were finely ground using an electric coffee grinder, and the resulting powder was stored in a tightly sealed glass container until further use

Figure 4. 1 Bringing Sonchus wightianus plant from the forest

Figure 4. 2 Drying process of the leaves of the Sonchus wightianus plant

Figure 4. 3 electric grinder

Figure 4. 4 Sonchus wightianus powder

3.2 Extraction of Phenolic Compounds

To isolate natural compounds for the first time using solvents, solid-liquid extraction is applied when the target material is in solid form. This technique is based on the principle that the solvent must penetrate the interface between the solid and liquid phases, allowing the active compounds within the solid matrix to be dissolved and transferred into the surrounding solvent.

In the present study, to extract phenolic compounds, we employed the soaking extraction method. Specifically, 50 grams of Sonchus wightianus leaf powder were weighed and immersed in 1000 milliliters of distilled water. The mixture was continuously stirred at room temperature for 24 hours using a magnetic stirrer.

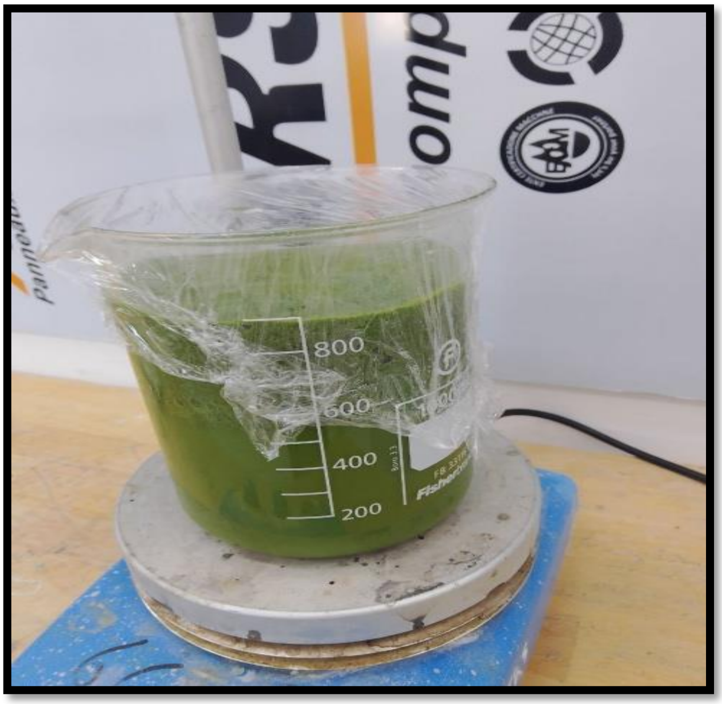

Figure 4. 5 Soaking Extraction of Phenolic Compounds

3.3 Filtration

After the 24-hour extraction period, the mixture was first filtered using a strainer to remove coarse impurities. The resulting solution was then passed through a clean, sterilized cloth to eliminate finer particulates, and the solid residue was discarded. Vacuum filtration was subsequently performed using cotton, and the process was repeated four times to enhance the clarity of the extract. Finally, filtration was completed using filter paper until the solution became entirely clear and free of any remaining impurities.

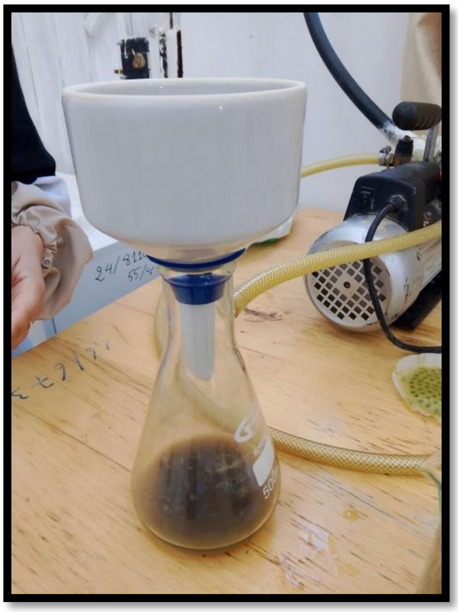

Figure 4. 6 Vacuum Filtration Using Filter Paper

3.4 Preparation of Zinc Acetate Solution

Zinc acetate solution was prepared by weighing 2.2 grams of zinc acetate and dissolving it in 50 milliliters of distilled water. The mixture was thoroughly stirred to ensure complete dissolution and obtain a homogeneous solution.

Figure 4. 7 Weighing of Zinc Acetate Powder

Figure 4. 8 Dissolution of Zinc Acetate in Distilled Water

3.5 Synthesis of Zinc Oxide Nanoparticles

We A volume of 100 milliliters of the prepared plant extract was taken, and its pH was adjusted to 10 by the gradual addition of sodium hydroxide (NaOH). Following pH adjustment, 50 milliliters of the previously prepared zinc acetate solution were added dropwise to the mixture at a temperature of 70°C, while stirring continuously using a magnetic stirrer. The reaction was maintained under these conditions for two hours.

After the reaction period, the resulting mixture was divided into equal portions and transferred into centrifuge tubes. The tubes were then centrifuged at 4500 rpm for 10 minutes, resulting in the formation of two distinct phases: a solid precipitate and a clear supernatant. The solid phase was carefully separated from the liquid, washed thoroughly with distilled water, and the washing process was repeated multiple times until the supernatant became completely clear and free of impurities.

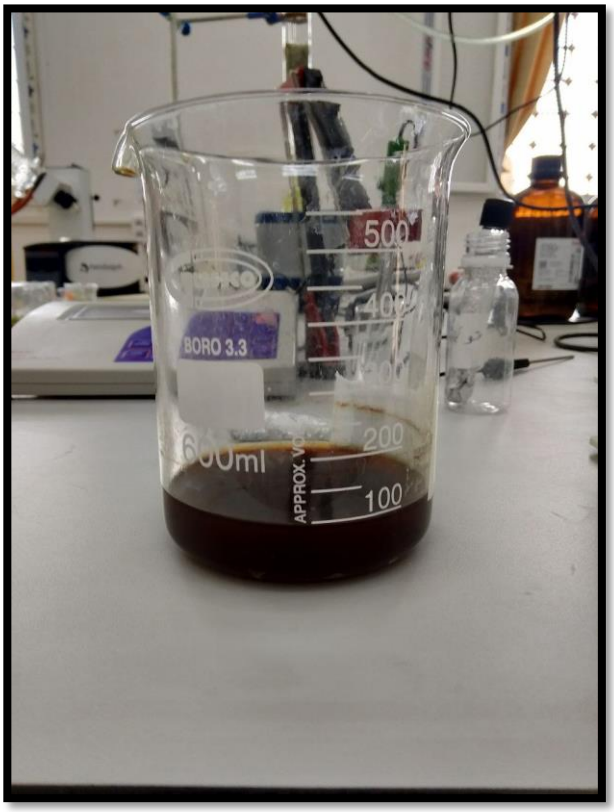

Figure 4. 9 Sonchus wightianus plant extract

Figure 4. 10 Adjustment of Extract pH

Figure 4. 11 Synthesis Reaction Setup on Magnetic Stirrer

Figure 4. 12 centrifuge

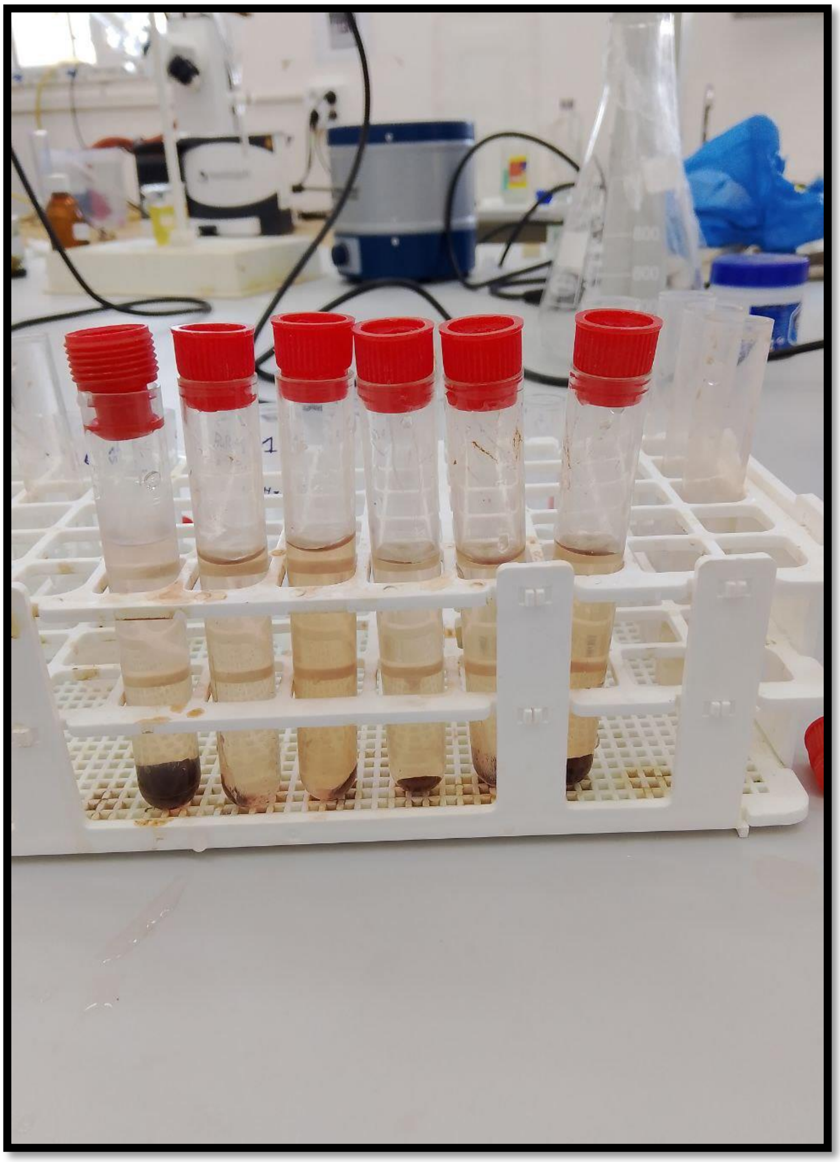

Figure 4. 13 Centrifuge tubes after phase separation into solid and liquid phase

3.6 Drying

The collected product was transferred into a clean glass container and placed in a drying oven at 100°C for 18 hours . This process resulted in the formation of a thin layer of dark green precipitate. Finally, the dried precipitate was carefully collected and stored in a tube for further use.

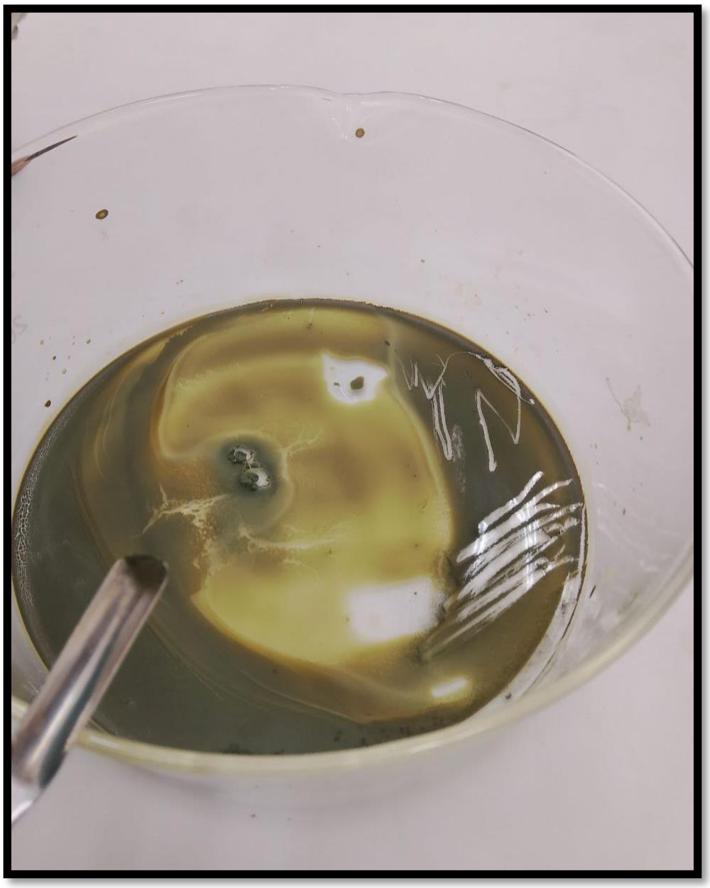

Figure 4. 14 The sample after drying

3.7 Calcination

Following the drying process, the obtained green powder underwent calcination at 500°C for two hours using a muffle furnace. This step was conducted to improve the crystallinity of the synthesized zinc oxide nanoparticles and to eliminate any residual organic compounds resulting from the green synthesis process.

Calcination is a critical step in ensuring the formation of a stable nanomaterial with enhanced physical and chemical properties, thereby improving its performance as a photocatalyst. After calcination, the sample was allowed to cool gradually to room temperature. The resulting powder was then carefully collected and stored in a tightly sealed glass container to protect it from moisture and potential contamination, in preparation for subsequent photocatalytic experiments.

Figure 4. 15 Dried sample after oven treatment

3.8 Characterization of the Synthesized Nanoparticles

Upon completing the green synthesis of zinc oxide nanoparticles using the plant extract, a series of experimental analyses were conducted to investigate their physical and chemical properties. To achieve this, four primary laboratory characterization techniques were employed

3.8.1 UV-Visible Spectroscopy (UV-Vis)

UV-visible spectroscopy analysis was performed to characterise the optical properties of biosynthesised ZnO nanoparticles and confirm their formation. The UV-visible spectrum obtained (figure 4.16), shows the absorbance of ZnO nanoparticles as a function of wavelength in the range 300 to 900 nm. A characteristic absorption peak is observed at a maximum wavelength (λ_{max}) of 267 nm, which is a key signature of ZnO nanoparticles.

The UV-visible spectrum confirms that the biosynthesised ZnO nanoparticles exhibit the expected optical properties, with an absorption peak at 267 nm attributable to the quantum confinement effect. This observation validates not only the formation of ZnO nanoparticles, but also the quality of the biosynthesis, which has resulted in nanometre-sized particles with improved optical properties [1, 2]

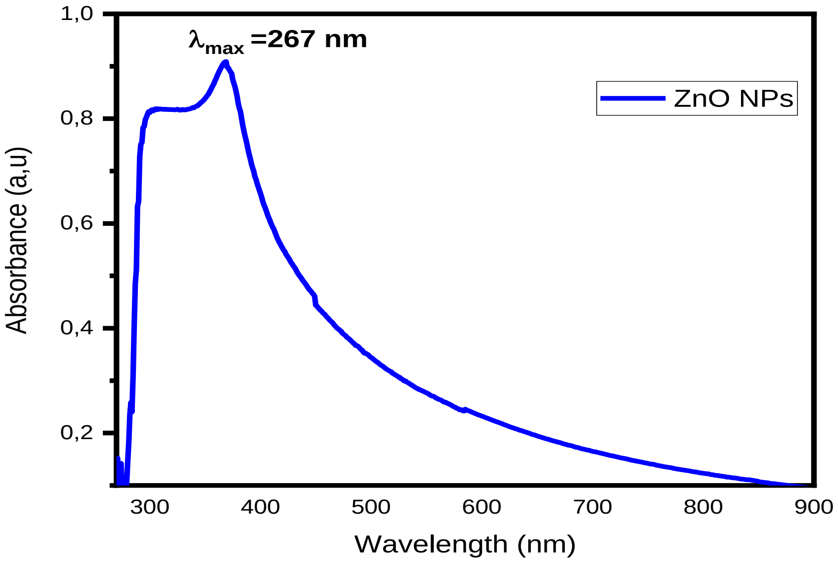

Figure 4. 16 UV-visible spectrum of synthesized ZnO

3.8.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis (figure 4.17) of ZnO nanoparticles biosynthesized using Sonchus wightianus leaf extract, performed in transmission mode in the range 400 to 4000 cm^{-1}, confirms the successful synthesis of the nanoparticles and highlights the role of the extract as a reducing and stabilising agent in a green chemistry approach, with a characteristic band at ~500 cm^{-1} attributed to Zn–O stretching vibration, absent in the spectrum of the pure extract, validating the formation of nanoparticles, while the bands common to both spectra, notably at ~3450 cm^{-1} (–OH, hydroxyl groups of phenolic compounds or adsorbed water), ~1630 cm^{-1} (C=O, carbonyl groups of flavonoids or phenolic acids), ~2920–2950 cm^{-1} (C–H, aliphatic chains) and ~1050–1400 cm^{-1} (C–O and C–N, alcohols, phenols or amines), indicate that the bioactive compounds in the extract have adsorbed onto the surface of the nanoparticles, contributing to their reduction and stabilization, preventing their aggregation and ensuring their relative purity with no detectable impurities, thus highlighting the effectiveness of this environmentally friendly method for producing functional nanomaterial [3, 4]

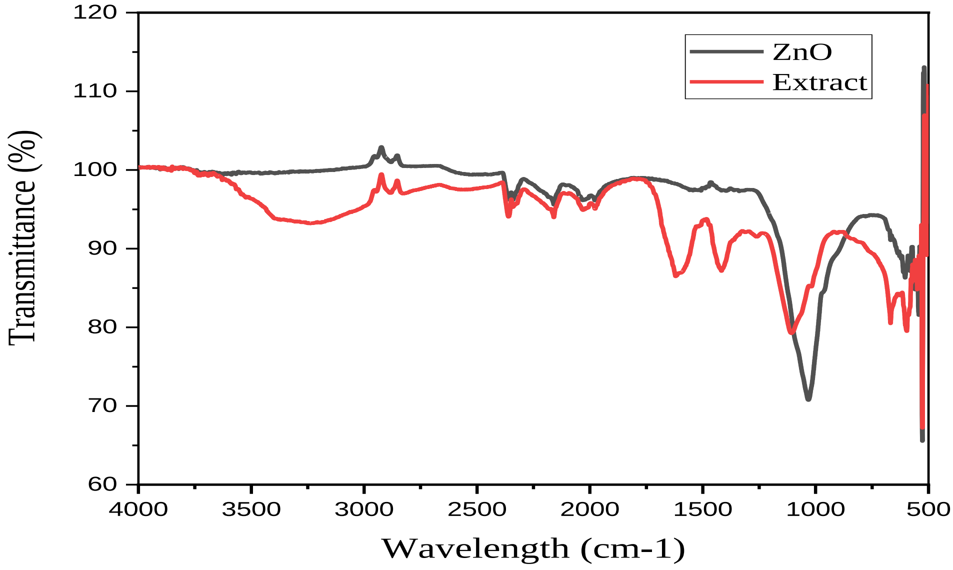

Figure 4. 17 FTIR spectrum of synthesized ZnO and extract

3.8.3 X-Ray Diffraction (XRD)

In this study, X-ray diffraction (XRD) analysis (figure 4.18) was performed to characterize the crystalline structure of biosynthesized ZnO nanoparticles and confirm their formation. The XRD diagram obtained shows distinct diffraction peaks, indicating a well-defined crystalline structure. The main peaks observed are located at 2θ angles of 31.5°, 34.4°, 36.1°, 47.4°, 56.5°, 62.6° and 67.6°. These peaks were compared to reference data from the ICDD (International Centre for Diffraction Data) database, specifically reference card 00-001-1136 for zinc oxide (ZnO, zincite).

The peaks correspond respectively to the (100), (002), (101), (102), (110), (103) and (112) planes of the hexagonal structure of ZnO, thus confirming the pure phase of ZnO with no detectable impurities in the XRD diagram[5, 6]

- **Crystallite size**

The average crystallite size was estimated using Scherrer's formula[7], given by:

$$D = \frac{k\,\lambda}{\beta\ \cos\theta} \qquad (x)$$

- Where:

D is the average size of the crystallites (in nm),

k is the Scherrer constant (usually taken as 0.9),

λ is the wavelength of the X-rays (1.5406 Å for Cu Kα),

β is the full width at half maximum (FWHM) of the diffraction peak in radians,

θ is the Bragg angle.

Using the dominant peak at 2θ = 36.1° (plane 101), the crystallite size was calculated to be approximately 21 nm. This nanometric size is consistent with the expected properties of ZnO nanoparticles, conferring specific characteristics such as a large specific surface area and improved optical and electronic properties compared to bulk material.

The XRD results demonstrate that the biosynthesis method used produced ZnO nanoparticles with a well-defined hexagonal crystal structure, consistent with the ICDD reference data. The crystallite size of 21 nm suggests good control of the synthesis process, enabling the production of nanoparticles suitable for potential applications such as photocatalysis, sensors, and optoelectronic devices. The absence of impurities and the purity of the ZnO phase highlight the effectiveness of the biosynthesis method, which could offer advantages in terms of biocompatibility and environmental friendliness compared to conventional chemical methods.

XRD analysis confirms the successful formation of pure ZnO nanoparticles with a hexagonal crystal structure and a crystallite size of approximately 21 nm, thus validating the effectiveness of the biosynthesis method used in this study.

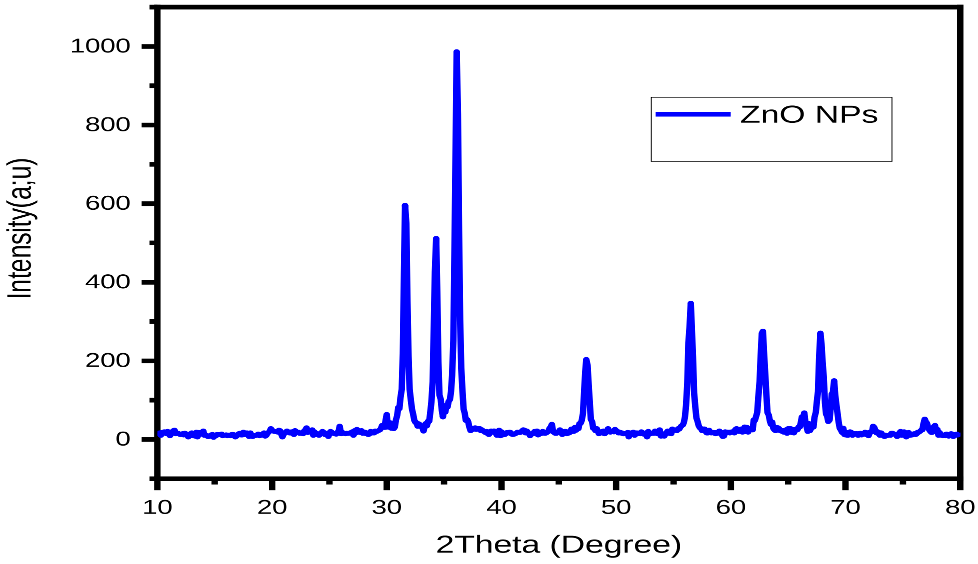

Figure 4. 18 XRD spectrum of synthesized ZnO

4 Conducting the Photocatalytic Activity Experiment

After completing the green synthesis and characterization of the nanoparticles, we proceeded to evaluate their photocatalytic efficiency in degrading organic dyes under natural sunlight exposure.

2.2 Preparation of Dye Solutions

To assess the photocatalytic performance of the synthesized nanoparticles, two synthetic dyes were selected: Methylene Blue and Rose Bengal. A solution of 250 mL was prepared for each dye according to the provided concentrations:

- **Methylene Blue**

Concentration: 6.25×10^{-5} mol/L

Volume: 250 mL = 0.250 L

Molar mass: 319.85 g/mol

Required mass:

$$m = C.V.M \qquad (x)$$

$$m = 5\ mg$$

A mass of 5 mg of Methylene Blue powder was accurately weighed and dissolved in 250 mL of distilled water.

- **Bengal rose**

Concentration: 2.36×10^{-5} mol/L

Volume: 250 mL = 0.250 L

Molar mass: 1017.64 g/mol

Required mass:

$$m = C.V.M \qquad (x)$$

$$m = 6\ mg$$

A mass of 6 mg of Bengal Rose powder was accurately weighed and dissolved in 250 mL of distilled water.

Figure 4. 19 Preparation of Methylene Blue and Rose Bengal Solutions

4.2 Sample Division and Nanoparticle Weighing

Each dye solution was divided into six 50 mL beakers, with 15 mL of solution in each. A total of 12 beakers were prepared (six for each dye).

Next, 12 equal portions of nanoparticles were weighed, each of 5 mg, totaling 60 mg. A 5 mg portion was added to each beaker to maintain consistency across all samples

Figure 4. 20 Distribution of the Rose Bengal dye solution into beakers

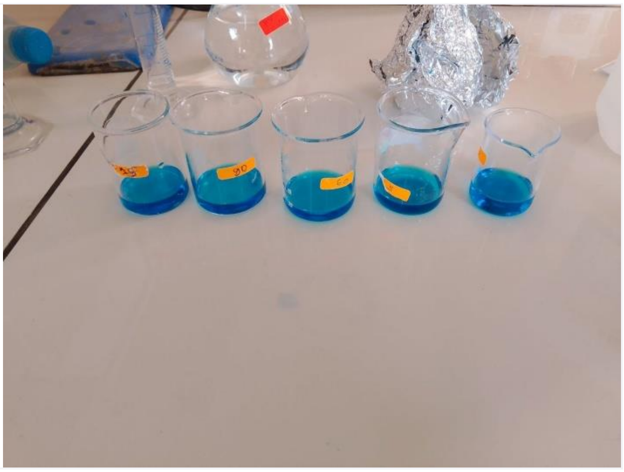

Figure 4. 21 Distribution of the Methylene Blue dye solution into beakers

4.3 Exposure to Sunlight

The prepared beakers were exposed to natural sunlight for different time intervals: 5, 15, 30, 60, 90, and 120 minutes. Each beaker was removed immediately after its assigned exposure duration

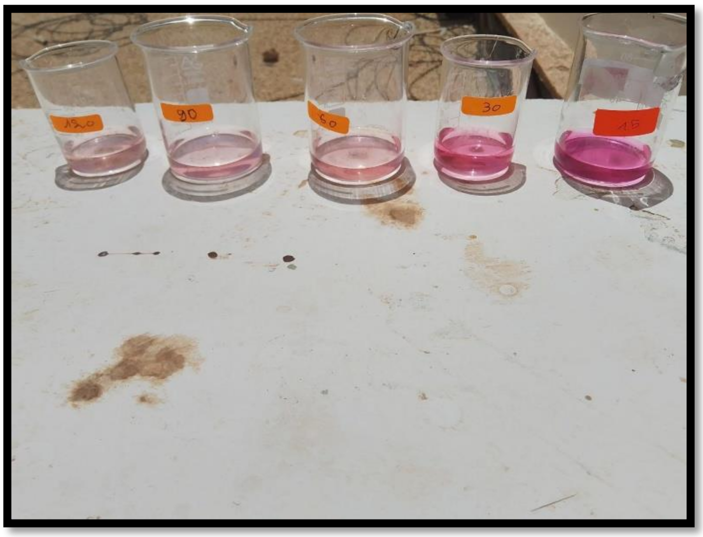

Figure 4. 22 Sunlight irradiation of Rose Bengal dye samples in the presence of ZnO nanoparticles

Figure 4. 23 Sunlight irradiation of Methylene Blue dye samples in the presence of ZnO nanoparticles

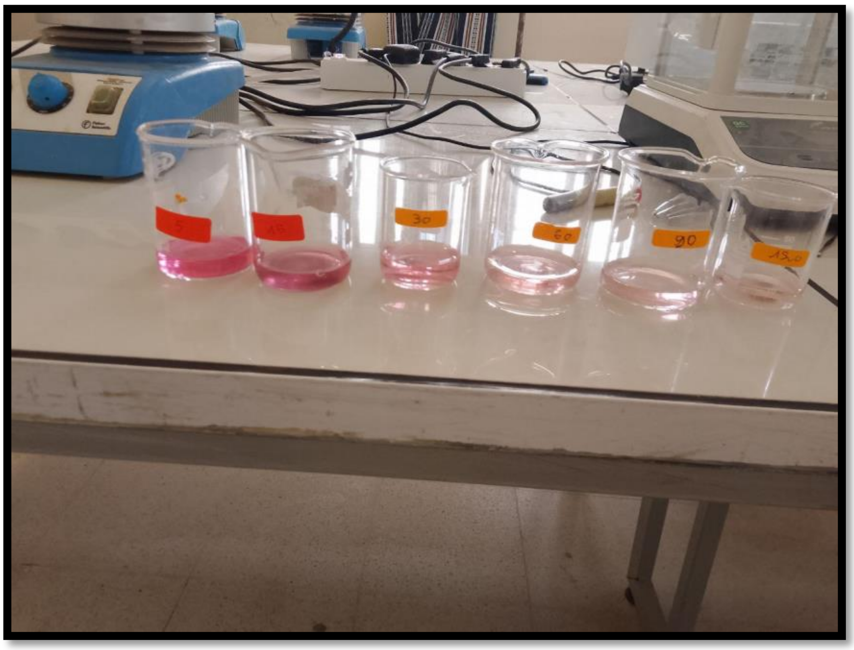

Figure 4. 24 Samples after sunlight exposure

4.4 Centrifugation

After exposure, all samples were centrifuged at 4500 rpm for 10 minutes to separate the nanoparticles from the liquid.

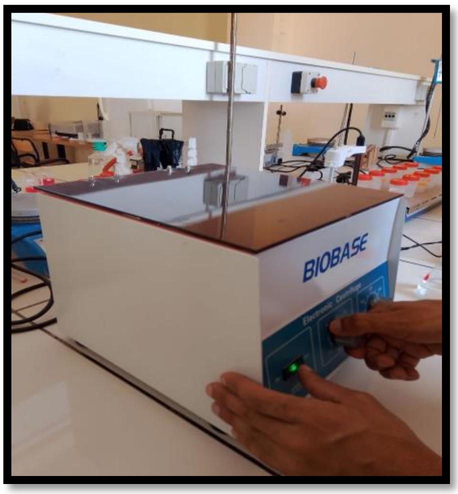

Figure 4. 25 Centrifugation After Sunlight Exposure

4.5 Photocatalytic Activity

UV-visible spectroscopy analysis was performed to evaluate the photocatalytic activity of biosynthesized ZnO nanoparticles in the degradation of two organic dyes: methylene blue (MB) and rose bengal (RB).

4.5.1 Photocatalytic degradation of EB dye:

The UV-visible spectrum of methylene blue (figure 4.26) shows a characteristic absorption peak around 664 nm, typical of this cationic dye. At the initial time (0 min), the maximum absorbance is approximately 2.5 arbitrary units (a.u.), indicating a high initial concentration of the dye. Over time, a gradual decrease in the intensity of the peak at 664 nm is observed at 5, 15, 30, 60, 90 and 120 minutes. After 120 minutes of irradiation, the absorbance is significantly reduced, approaching values close to zero, indicating almost complete degradation of the methylene blue.

This decrease in absorbance is attributed to the photocatalytic degradation of MB, facilitated by ZnO nanoparticles. Under light irradiation, ZnO nanoparticles generate electron-hole pairs. The excited electrons in the conduction band and the holes in the valence band react with adsorbed oxygen and water molecules to produce reactive species, such as hydroxyl radicals (•OH) and superoxide anions (O_2^-), which effectively break down the dye molecules. The gradual reduction in absorbance without the appearance of new peaks in the spectrum suggests that MB is degraded into colourless by-products or simple compounds (such as CO_2 and H_2O), confirming the photocatalytic efficiency of the nanoparticles [8, 9]

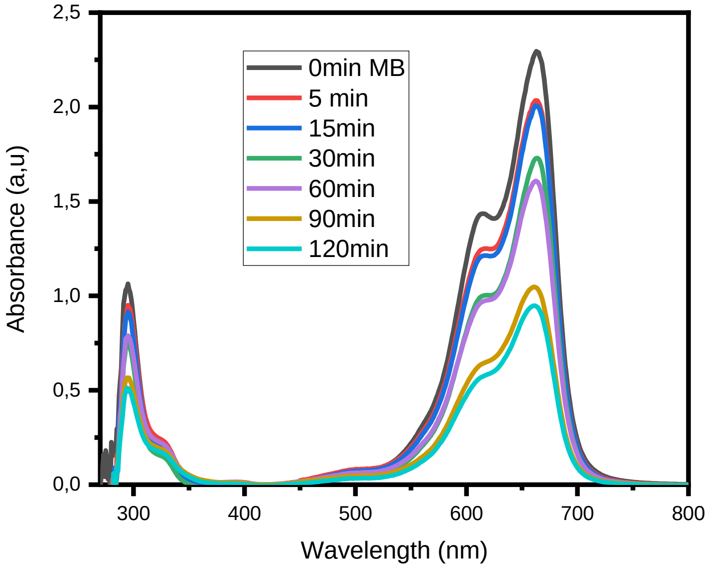

Figure 4. 26 UV–vis spectra for photocatalytic degradation of Methylene Blue dye.

4.5.2 Photocatalytic degradation of RB dye:

The UV-visible spectrum of Bengal pink (figure 4.27) shows a characteristic absorption peak at λ_{max} = 542, corresponding to the chemical structure of this anionic dye. At the initial time (0 min), the maximum absorbance is approximately 1,4 a.u. A gradual decrease in the intensity of this peak is observed at 15, 30, 60, 90 and 120 minutes. After 120 minutes, the absorbance is greatly reduced, although slightly less markedly than for MB, indicating significant massive degradation.

As with methylene blue, the decrease in absorbance at 542 nm is attributable to the photocatalytic activity of ZnO nanoparticles, which generate reactive species under light irradiation, decomposing the dye molecules. The absence of new peaks in the spectrum suggests that rose Bengal is degraded into colorless by-products, confirming the effectiveness of the photocatalytic process[10]

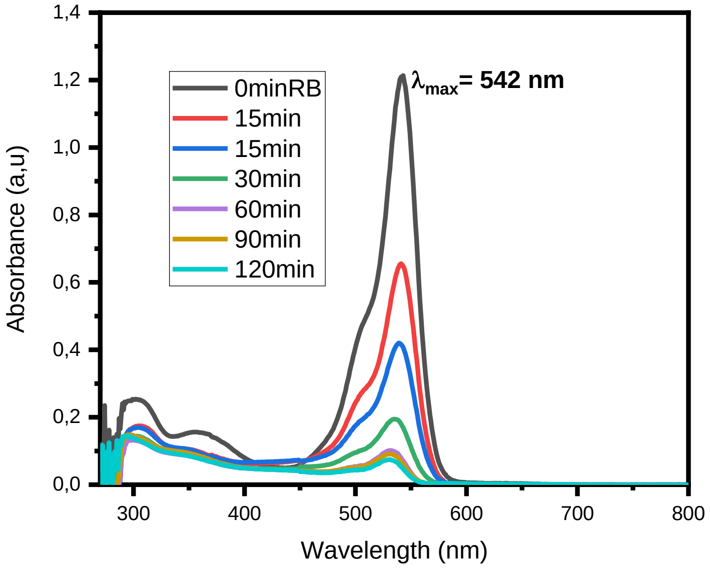

Figure 4. 27 UV–vis spectra for degradation of rose Bengal dye.

4.5.3 Compare and explain

ZnO nanoparticles show awesome photocatalytic activity for both dyes, but the degradation of rose Bengal is more effective, with almost zero residual absorbance after 120 minutes, compared to a slightly higher absorbance for methylene blue. This difference can be attributed to the anionic nature of RB, which could promote specific interactions with reactive species or active sites on the nanoparticles. In addition, the lower initial concentration of RB (indicated by lower absorbance at t = 0) could facilitate faster degradation. The spectra show no new absorption peaks, confirming that the dyes are degraded into colorless by-products, probably CO_2, H_2O or other simple compounds[11].

3. Conclusion:

In conclusion UV–vis, FTIR and XRD analyses confirms the successful formation of pure ZnO nanoparticles with a hexagonal crystal structure and a crystallite size of approximately 21 nm, thus validating the effectiveness of the biosynthesis method used in this study.

The biosynthesised ZnO nanoparticles exhibit excellent photocatalytic activity for the degradation of organic dyes, with slightly higher performance for Bengal pink than for methylene blue. These results highlight the potential of ZnO nanoparticles for applications in wastewater treatment and environmental remediat

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General Conclusion

The present memory explored the green synthesis of zinc oxide nanoparticles (ZnO NPs) using a natural extract from Sonchus wightianus, and evaluated their photocatalytic performance for the degradation of harmful organic dyes under sunlight.

The use of Sonchus wightianus as a biologically active and eco-friendly reducing agent demonstrated significant potential for the green fabrication of ZnO NPs, offering an alternative to conventional chemical synthesis methods that often involve hazardous reagents. The synthesized nanoparticles exhibited favorable physicochemical properties such as good dispersion, and a well-defined crystalline structure.

A series of advanced characterization techniques were employed to analyze the synthesized ZnO NPs. **UV-Vis spectroscopy** provided insights into their optical properties and confirmed the formation of nanoparticles; **FTIR spectroscopy** identified the functional groups and molecular interactions involved in the synthesis process; while **XRD analysis** confirmed the crystalline nature and structural arrangement of the particles. These findings confirmed the successful synthesis and structural integrity of the ZnO NPs.

Functionally, the green-synthesized nanoparticles displayed excellent photocatalytic activity in degrading dyes such as Rose Bengal and Methylene Blue under natural sunlight, achieving high degradation rates in short periods. These outcomes underline the effectiveness of this green method and its applicability for environmental remediation, especially in regions facing wastewater treatment challenges.

Beyond the environmental impact, this research contributes to the advancement of sustainable nanotechnology and highlights the potential of medicinal plants as untapped natural sources for nanomaterial production. The study opens new prospects for further exploration, particularly in optimizing synthesis parameters such as pH, temperature, and concentration, as well as in testing other plant species for similar or enhanced results.

In conclusion, the results of this memory were encouraging and support the feasibility of using Sonchus wightianus in green nanotechnology. The synthesized ZnO NPs demonstrated not only structural and chemical integrity but also practical effectiveness in photocatalytic applications. Continued research in this area is highly recommended, to broaden the scope of eco-friendly nanoparticle synthesis and reinforce the integration of sustainable practices in modern science.