GREEN SYNTHESIS OF SILVER/ZINC OXIDE NANOCOMPOSITES USING LEAF EXTRACT OF *DESMOSTACHYA BIPINNATA* (KUSH) AND STUDY OF ITS PHOTOCATALYTIC ACTIVITIES

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ABSTRACT

Removing organic pollutants including textile waste solution and dyes has become an essential requirement for maintaining a secure and healthy environment. Therefore, the present study deals with the synthesis of ZnO and Ag/ZnO nanocomposites using the leaf extract of *Desmostachya bipinnata*. The leaf extract of *Desmostachya bipinnata* contained a different bio-active compound that serves the dual purpose of acting as stabilizing and reducing agents. Out of various methods, green synthesis has been extensively employed for its simplicity, low-cost, and eco-friendly nature. The characterization of the as-synthesized ZnO and Ag/ZnO nanocomposties were subjected to ultra violet visible (UV-vis) spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, X-ray diffraction (XRD). XRD pattern revealed the crystalline nature of nanoparticles. The crystallite size of Ag/ZnO was found to be 12-14 nm, according to Debye Scherer formula. The synthesized NPs and NCs were used for the catalytic photodegradation of Methylene blue (MB). The MB was degraded at room temperature under exposure to UV light. The degradation efficiency of ZnO, and 25 % Ag/ZnO were found to be 79 % and 85 %, respectively. Therefore, it was found that Ag/ZnO nanocomposites have potential to degrade MB as organic dye, and can be used for wastewater treatment.

Keywords: Green synthesis, Ag/ZnO, *Desmostachya bipinnata* leaf extract and Photocatalytic activity.

शोधसार

जैविक प्रदुषणहरु हटाउन तथा सुरक्षित र स्वास्थ्यपूर्ण पर्यायवरण बनाउनका लागि विशेषगरी कपडाजन्य उद्योगबाट उत्सर्जन हुने प्रदुषित, पानी र रंगका विकिरणहरूलाई हटाउन उच्च आवश्यक्ता रहेको छ। यसकारण, हाम्रो अध्ययनले *Desmostachya bipinnata* कुशको रसको प्रयोगगरी ZnO र Ag/ZnO न्यानोकम्पोजिटको उत्पादन गरेको छ। *Desmostachya bipinnata* मा विभिन्न जैविकतत्व पाइएको छ जसले न्यानोकणहरुको निर्माणमा स्थिरकरण र न्यूनीकरण प्रक्रियामा काम गर्दछ। विभिन्न उपायहरूको बीचमा हरित संश्लेषणले यसको सरलता, कम लागत, र पर्यायवरण मैत्री प्रकृतिकोलागि विस्तृत भूमिका खेलेको छ। संश्लेषण गरिएको ZnO र Ag/ZnO न्यानोकम्पोजिटको चरित्रणलाई UV-vis स्पेक्ट्रोस्कोपी, FTIR स्पेक्ट्रोस्कोपी, XRD मा गरिएको छ। XRD प्याटर्नले न्यानोकणको मणिमीय प्रकृति दर्शाएको छ। Ag/ZnO को मणिमीय आकारलाई Debye Scherer सूत्रमा 12-14 nm मा पाइयो। उत्पादित NPs र NCs लाई Methylene blue को उत्प्रेरकीय प्रकाशजन्य विच्छेदनकोलागि प्रयोग गरिएको छ। ZnO र 25 % Ag/ZnO को डिग्रेडेशन दर 79 % र 85 % पाइएको छ। यसकारण, Ag/ZnO न्यानोकम्पोजिटलाई MB जस्ता जैविक रंगलाई हटाउन र पानीको प्रशोधनकालागि प्रयोग गर्न सकिन्छ।

कुञ्जी शब्दहरू: *हरित संश्लेषण, Ag/ZnO, Desmostachya bipinnata कुशको उत्पादन,* र *फोटोक्याटालिटिक क्रियाकलाप।*

LIST OF ABBREVIATIONS AND FORMULA

$AgNO_3$:	Silver Nitrate
CB:	Conduction Band
FTIR:	Fourier Transform Infrared Spectroscopy
JCPDS:	Joint Committee on Powder Diffraction Standards
MB:	Methylene Blue
MEMS:	Micro-Electro-Mechanical-Systems
mL:	Mililitre
NCs:	Nanocomposites
nm:	Nanometer
NPs:	Nanoparticles
ppm:	Parts Per Million
UV:	Ultra Violet
VB:	Valence Band
XRD:	X-Ray Diffraction
$Zn(NO_3)_2.6H_2O$:	Zinc Nitrate Hexahydrate
ZnO:	Zinc Oxide

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CHAPTER I

INTRODUCTION

1.1 General Introduction

Nanotechnology refers to a scientific discipline that focuses on materials at the nanoscale through various physical or chemical techniques to achieve desired characteristics. Nanoparticles are typically defined as particles with an average size smaller than 100 nm (Devatha & Thalla, 2018). The term "nano" comes from ancient Greek and means dwarf (Purohit et al., 2019). However, the term "nanometer" was proposed by Nobel Prize Laureate, Richard Zsigmondy. The term "Nanotechnology" was the original idea of Richard Feynman. This idea was presented through his lecture, "There's Plenty of Room at the Bottom", in which he introduced the concept of manipulating matter at the atomic level and after 15 years of this lecture, a Japanese scientist, Norio Taniguchi was the first to use "nanotechnology" (Hulla et al., 2015).

The nanoscience is the study of nanomaterials with nanomateric dimension (10^{-9} m) (Mahmoud, 2020). Nanomaterials frequently exhibit distinct and significantly altered physical, chemical, and biological properties when compared to their larger-scale counterparts. Nanomaterials can be applied to various field of interest such as health-care, life-science, engineering, information technology, biotechnology, commercial industries, etc. (Dolez, 2015). Currently, nanotechnology is receiving particular attention in academic, where new programs are being designed to accelerate the rate of innovation.

Nanomaterials are of different types according to their size, morphology, physical and chemical properties. Nanomaterials can be classified into four different categories based on their dimensions and overall shape (Saleh, 2020) , they are:

1) Zero-dimensional nanomaterials (0D): All their dimensions in nanoscale range.
2) One-dimensional nanomaterials (1D): One dimension is out of the nanoscale range.
3) Two-dimensional nanomaterials (2D): Two dimensions are out of the nanoscale range.
4) Three-dimensional nanomaterials (3D): Three dimensions are out of the nanoscale range.

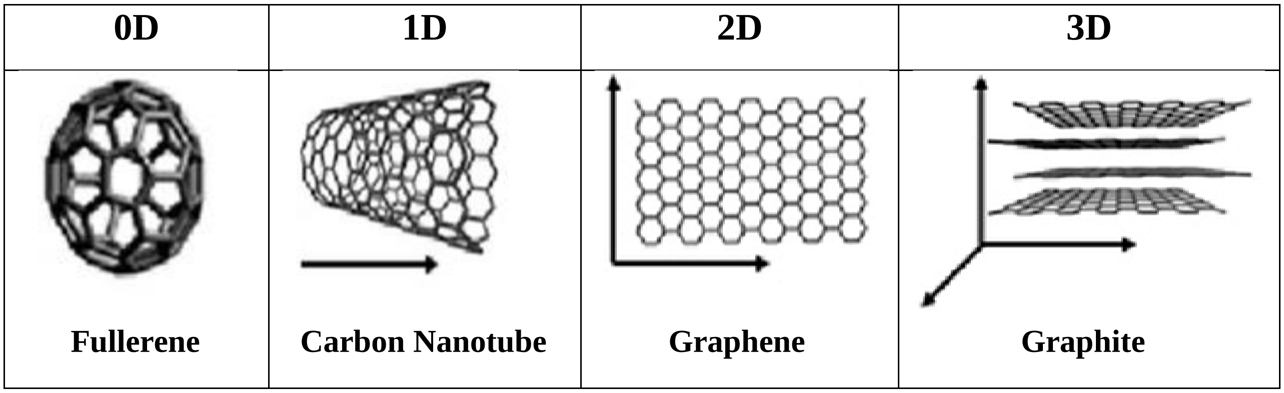
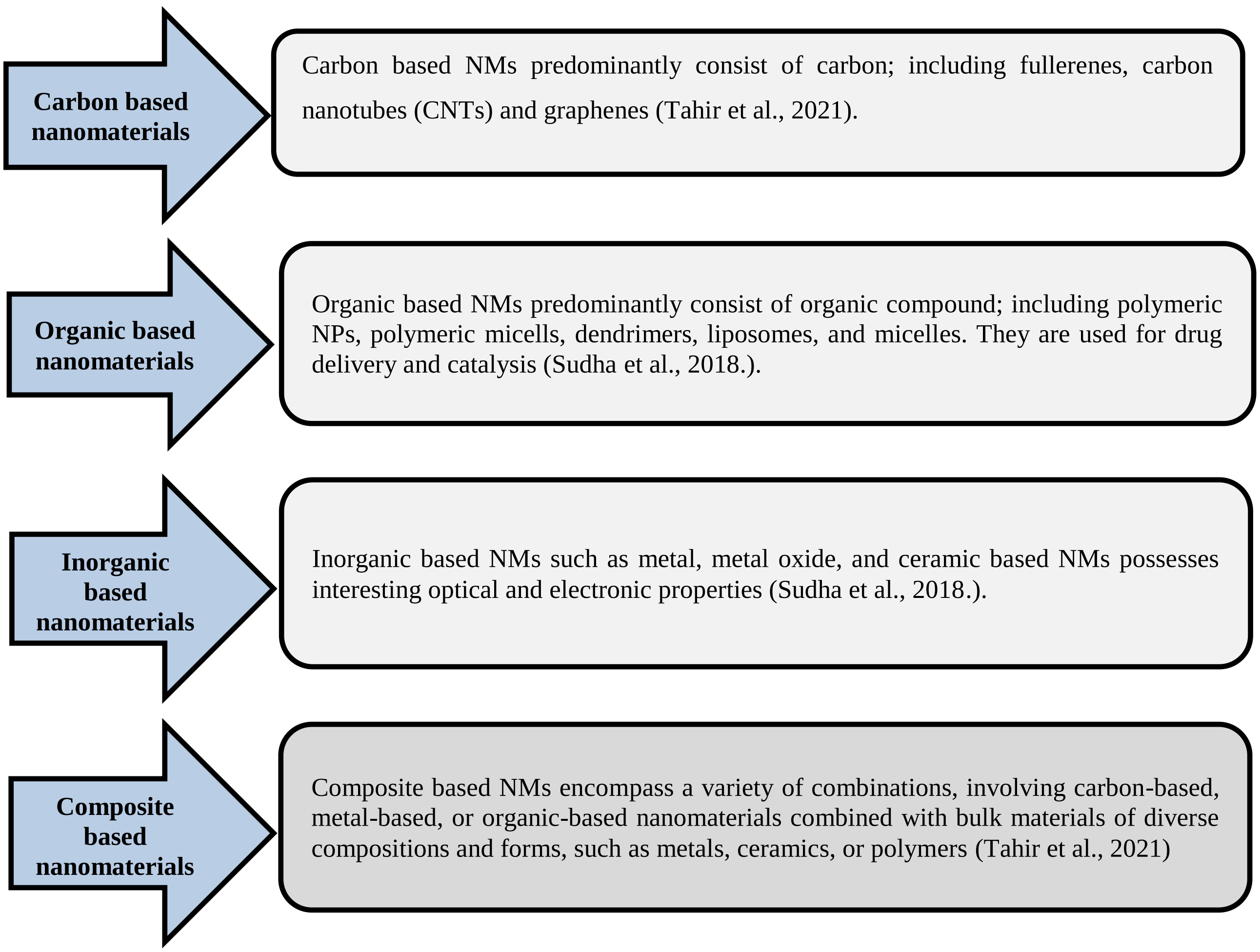


Figure.1.1: *Example of 0D, 1D, 2D and 3D carbon nanostructure* (Bergmann & Machado, 2015)

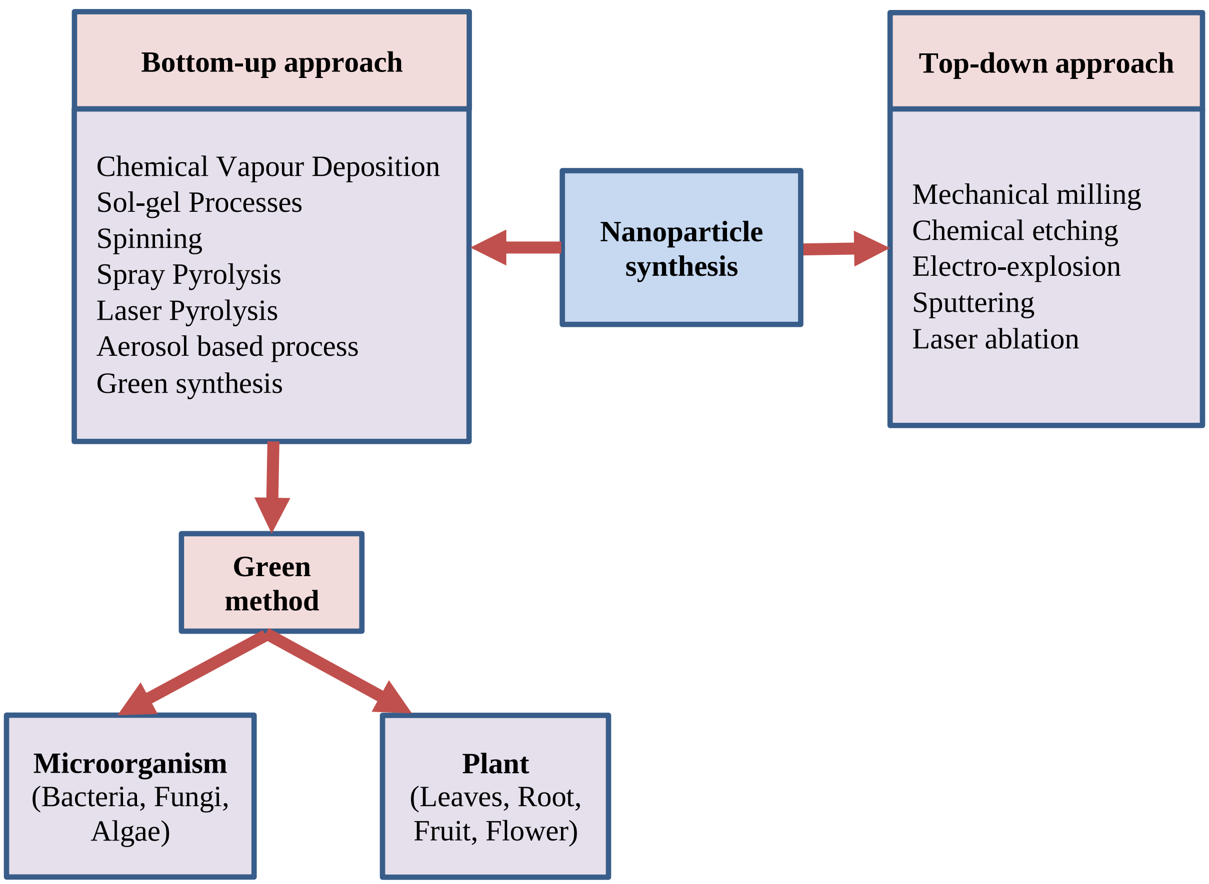
Similarly, based on composition of nanomaterials they are classified into four types as shown in scheme 1.1.



Scheme 1.1: *Classification of nanomaterials based on composition*

The synthesis of nanoparticles in general can be broadly divided into two main approaches: Bottom-Up approach and Top-Down approach.

The "Top-Down" approach involves the production of nanoparticles through size reduction using different physical and chemical techniques. In contrast, the "Bottom-Up" approach involves the creation of nanoparticles from smaller entities, such as atoms and molecules, with reduction or oxidation reactions playing a key role. Biological methods for nanoparticle synthesis, microorganism as well as plant extracts are widely used (Hussain et al., 2016). The choice of synthesis method greatly influences the properties of the resulting nanomaterials. The nanoparticle synthesis approach is represented in scheme 1.2.



Scheme 1.2: *Nanoparticle synthesis approach*

The top-down approach is often used in various industries including nanotechnology, microelectronics, and material science. It tends to produce nanoparticles with unpredictable sizes and irregular edge structures. On the other hand, the bottom-up synthesis approach is a cost-effective technique that offers the ability to create nanoparticles and nanocomposites with well-defined structures and narrow size distributions. Despite the availability of multiple procedures for synthesizing different types of nanomaterial, there is a growing need for eco-friendly synthesis routes that are cost-effective and capable of improving production rates to meet long-term application demand (Mahmoud, 2020). Bottom up approach is easiest and cost-effective in comparison to the top-down approach.

Nanoparticles produced through the utilization of natural resources or eco-friendly processes, involving plant extracts, microorganisms, or other sustainable approaches referred to as products of green synthesis and such synthesized nanoparticles are called biological nanoparticles (Narayanan & Sakthivel, 2010). Plants are regarded as highly desirable for nanoparticle synthesis because plant extract serving as reducing and capping agent. Nowadays, biological nanoparticles were found to be more pharmacologically active than those manufactured by physical and chemical methods. In addition, "green synthesis" is required to avoid the production of unwanted or harmful by-products through the build-up of reliable, sustainable, and eco-friendly synthesis procedures (Ayaz et al., 2014).

Zinc oxide (ZnO) offers unique advantages including a direct band gap, simple crystallization, easy customization of its structure and higher exciton binding energy (Janaki et al., 2015). Zinc oxide nanoparticles stands out among metal oxide nanoparticles due to its distinctive combination of both semiconducting and piezoelectric characteristics (Chikkanna et al., 2019). Researchers have shown a keen interest in modifying and coating semiconductor nanoparticles such as ZnO via metallic particles like Ag, Cu, Au, TiO_2, etc, to develop the nanocomposite having novel and enhanced physicochemical properties than that of the individual constituents.

A nanocomposite refers to a material composed of two or more nanomaterials, with atleast one of them having dimensions at the nanometer scale (Ishida et al., 2000). As compared to metallic nanomaterial, nanocomposites have more reactive sites and are more stable, efficient, and efficacious (Reshmy et al., 2021). Multiple techniques including co-precipitation, sol-gel, microwave synthesis, hydrothermal synthesis, chemical-vapour deposition, spray pyrolysis, ballmilling, biological synthesis, etc. for the synthesis of nanocomposite (Rane et al., 2018). These methods suffer from drawbacks such as high expenses, potential toxicity, and limited availability. Consequently, the most promising alternative approach for synthesis of various metal and semiconductor nanocomposites lies in environmentally friendly method i.e., “green” method. Ag/ZnO nanocomposites have been successfully fabricated through the utilization of leaf extracts such as *Trigonella foenum-graecum* (Noohpisheh et al., 2020), *Azadirachta indica* (Slathia et al., 2021), *Thymus vulgaris* (Zare et al., 2019), *Excoecaria agallocha* (Khan et al., 2020), *Aloe barbadensis miller* and *Ocimum tenuiflorum* (S. Sharma et al., 2021), *Camellia sinensis* (Basnet et al., 2019), *Murraya koenigii* and *Zingiber officinate* extract (Arumai Selvan et al., 2021), and so on. The synthesis of Ag/ZnO nanocomposites is of great interest due their versatility in various applications. These nanocomposites have greatly used as highly efficient catalysts, non-electrical devices, bio-medical, photocatalytic degradation of pollutant, sensor, solar cells, etc. (Zare et al., 2019). Among them, the photocatalytic activity of Ag/ZnO nanocomposites is focused in this study for exploring its mechanism in detail.

Desmostachya bipinnata, known as “kush” in Sanskrit, is a grass belonging to the Poaceae Family. Its cultural significance is profound, as it has been utilized in numerous Vedic rituals throughout history (Khyade & Sarwade et al., 2018). In Hunduism, kush grass is considerd as scared grass. This grass is believed to possess purifying properties and is used to create a scared space for conducting rituals (Ayaz Ahmed et al., 2014).

Taxonomic classification of *Desmostachya bipinnata*

Kingdom: Plantae

Division: Magnoliophyta

Class: Lilopsida

Order: Poales

Genus: *Desmostachya*

Species: *bipinnata*

Nepali name: Kush

Figure 1.2: *Desmostachya bipinnata*

Desmostachya bipinnata is a perennial grass that forms dense tufts or clumps. It can grow up to 1-2 meters in height, with some specimens reacting even higher. The leaves are long, narrow, and linear ranging from 20 to 60 cm in length. Various parts of *D.bipinnata* are employed to address various ailments. Leaves are applied to heal wounds and urinary tract disorders and roots are used to treat piles, cholera, wounds, dysentery (Vivekanandarajah et al., 2021). Based on these reports, it is anticipated to have efficient phytochemicals in *D. bipinnata* to synthesize nanoparticles as well as nanocomposites.

The significant release of toxic synthetic dyes from the industries is main source of environmental contamination. Synthetic dyes are used in various industries such as the textile industry, the leather tanning industry and the paper industry. The widespread utilization of synthetic dyes in these industries has led to serious consequences, including extensive environmental pollution and water contamination (Abdel Messih et al., 2019). Moreover, this contamination has been associated with various vital diseases, such as cancer, cardiovascular, and cerebrovascular diseases (Ji et al., 2019). Methylene blue (MB) is a commonly employed dye in various industries, including agriculture, textiles, papermaking, cosmetics and pharmaceuticals. Unfortunately, the discharge of methylene blue into water sources contributes to water pollution. The chemical structure of Methylene blue is shown in fig.1.3.

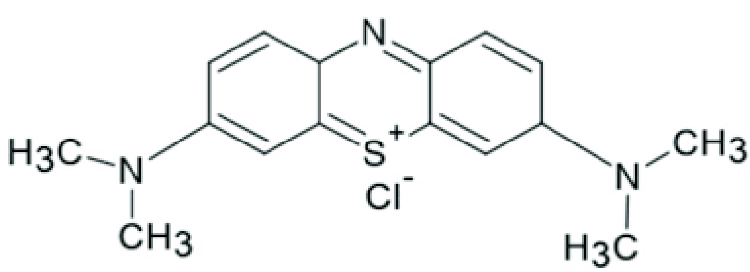


Figure 1.3: *Chemical structure of Methylene blue*

Traditional approaches for treating waste water contaminated with methylene blue involve method such as adsorption, reverse osmosis or chemical coagulations (Whang et al., 2012). However, these approaches need additional treatments, which can result in relatively high treatment cost.

Photocatalytic activity is the remarkable capability of a substance to accelerate chemical reaction through the absorption of light energy. When a photocatalyst material is exposed to light, it can produce electron-hole pairs that trigger redox reactions with molecules that have been absorbed on its surface or in the surroundings. This fascinating characteristic of the material finds practical applications in various areas. For instance, it can be applied to effectively remove contaminants from water, boost the efficiency of solar cells, and facilitate the conversion of carbondioxide into fuels, and enable the production of hydrogen from water (Liu et al., 2019). Therefore, photocatalysis has gained increasing attention over the years as a promising approach for degrading dyes and organic compounds in wastewater.

The semiconducting materials such as TiO_2, ZnO, SnO_2 (Abdel Messih et al., 2019), WO_3, ZrO_2 (Whang et al., 2012) and so on have been extensively employed as photocatalyst for water treatment. But, literature with detailed mechanism of photocatalyst is still lacking. Hence, this study is focused on exploring the photocatalytic property of biosynthesized ZnO nanoparticles and Ag/ZnO nanocomposites. Biosynthesis was carried out by using *D.bipinnata* leaf extract which presumed to function as both a reducing and capping agent (Singh et al., 2014).

1.2 Statement of Problem

The rise of industrialization and advancement of nanotechnology causes severe hazardous problems to environment and living creatures. Organic pollutants from industries are released and mixed with different resources such as river, soil, etc. which are affecting the environment seriously, particularly in the form of water pollution. This type of pollution poses not only a threat to human health but also endangers entire ecosystem. Currently, people all over the world are facing the problem of water pollution, hence it is realized that the most important task of researchers is to produce eco-friendly materials and processes to overcome such problem. Different treatments were developed for water treatment such as filtration, oxidation, adsorption, and photocatalytic techniques. Among these, photocatalysis has captured significant interest as a promising approach for water treatment. In the context of synthesis of nanomaterials, commonly employed traditional methods constitute of many disadvantages such as environmental pollution, large energy consumption and potential health problems.

The biosynthesis route is regarded as the most promising route since last few decades. Literatures having detailed mechanism of biosynthesis seemed insufficient. Furthermore, the biosynthesis of Ag/ZnO nanocomposite using *D. bipinnata* leaf extract has not been found elsewhere. Additionally, the photocatalytic activity of as-synthesized nanocomposite will be the promising material for removing such hazardous organic pollutants from water resources.

1.3. Objectives

1.3.1. General Objective

The general objective of this study is to carry out the green synthesis of silver/zinc oxide nanocomposites using leaf extract of *Desmostachya bipinnata* (kush) and study of its photocatalytic activities.

1.3.2 Specific Objectives

The specific objectives of the study can be figured out as follows;

- Synthesis of Ag/ZnO nanocomposite using *Desmostachya bipinnata* leaf extract.
- Study of the phytochemicals present in *D.bipinnata* leaf extract.
- Characterization of as synthesized Ag/ZnO nanocomposite by using UV-visible spectroscopy, X-Ray Diffraction and FTIR analysis technique.
- Study of photocatalytic activities of synthesized Ag/ZnO nanocomposite and optimization towards a Methylene blue.

CHAPTER II

LITERATURE REVIEW

2.1 Nanomaterial

Nanomaterials play a vital role in both nanoscience and nanotechnology. They serve as the building blocks for creating functional materials, devices, and system with all three external dimensions at the nanoscale. At this scale, they exhibit emergent properties, including optical, magnetic, electrical, and other characteristics that make nanomaterials highly desirable for a wide range of applications. Nanocarbons like fullerenes, carbon nanotubes, and graphene are example of nanomaterial with extraordinary properties such as exceptional strength and electrical conductivity. Nanostructured materials also exhibit distinctive physical and chemical properties, including optical absorption and fluorescence, melting points, catalytic activity, magnetism, and electrical and thermal conductivity (Kumar N et al., 2016). Nanomaterial opens up new possibilities for applications in various fields such as medicine, nutrition and energy, development of electrochemical sensors and biosensors (Parveen et al., 2016; Luo et al., 2006). Nanomaterials display different mechanical properties like superplaticity, exceptional hardness, toughness, strength and also exhibit higher catalytic activity towards chemical reaction (Asha & Narain, 2020). These properties differ significantly from larger materials. Some of the basic properties and applications are presented in table no.2.1.

Table No.2.1: *Properties of nanomaterial and ensuring applications* (Herrera et al., 2000)

Property	Applications
High surface area-to-volume ratio	Application in catalysis, gas sensor and solar veils
Increasing hardness with decreasing grain size	Development in hard coatings and protective layers
Increasing resistivity with decreasing grain size	Application in electronics, passive components, and sensors
Improved atomic transport kinetics	Utilization in batteries and hydrogen storage
Improved reliability and fatigue resistance	Application in electronic components and MEMS
Low percolation threshold	Usage in the creation of conductive materials and sensitive sensors

2.1.1 Properties of ZnO

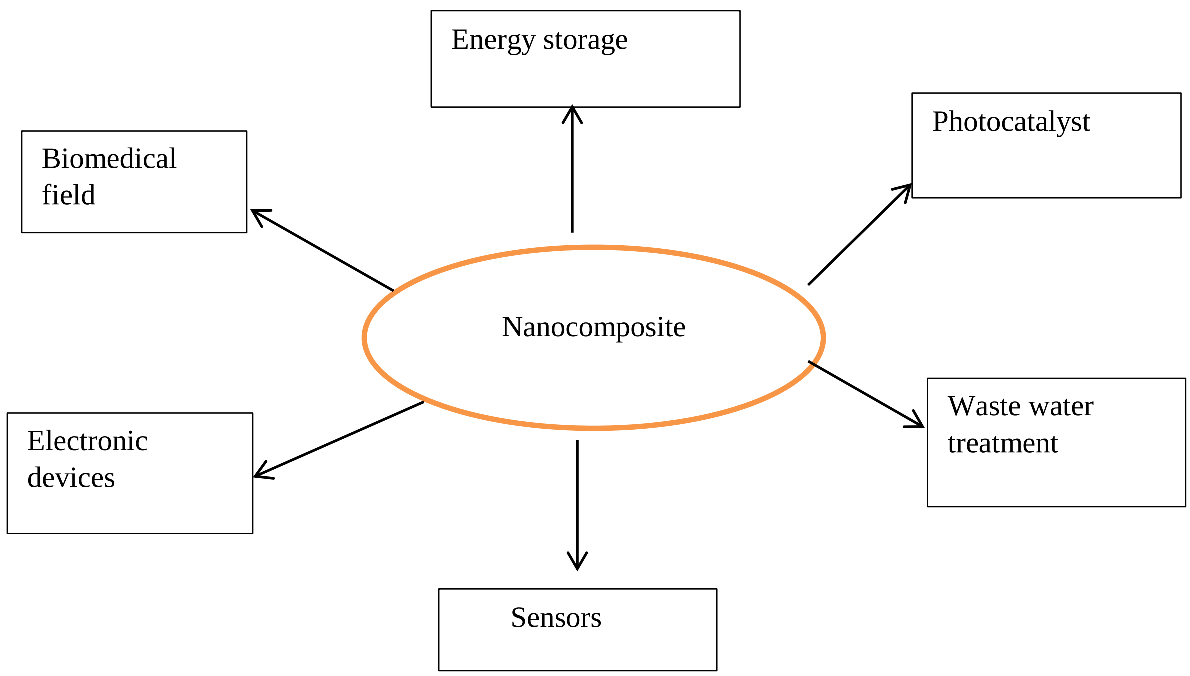
Zinc oxide (ZnO) is found to be one of the most common metallic oxide semiconducting nanomaterials for its characteristic property such as direct band gap (3.37 eV), electron mobility and higher exciton binding energy (Hong et al., 2009). Kahouli et al., (2015) synthesized zinc oxide nanoparticles which show diverse applications such as an ultraviolet (UV) light emitter, piezoelectric devices, chemical and gas sensors, transistors, solar cells, and catalysts. Because of its optoelectronic properties, zinc oxide nanoparticles have opened up new route for innovative advancements across a broad spectrum of industries and technologies.

In addition, it is cheap and environmentally friendly and also potentially useful in photocatalyst, cancer treatment, antibacterial activity (Singhal et al., 2012). Similarly, Chikkanna et al., (2019) synthesized zinc oxide nanoparticles by utilizing agro waste material like sheep and goat faecal matter as a reducing agent and the characterization of synthesized ZnO nanoparticles was done by using UV-visible spectroscopy, FTIR, XRD, and SEM analysis. By using agar- well plate method ZnO NPs were subjected to good antibacterial activity against both *Bacillus subtilis* and *Salmonella typhimurium*. Minimum inhibitory concentration (MIC) against both organisms was tested; they reported that at 10 µg/mL nanoparcticles exhibits minimum activity.

Despite its potential, ZnO faces certain challenges when used as a photocatalyst. These challenges encompass a high recombination rate of electrons and holes once they are generated, as well as the requirement for ultraviolet light with energy levels exceeding its band gap for effective activation. Addressing these issues was crucial for ZnO materials to emerge as practical photocatalysts. To overcome such challenges, coupling of ZnO nanoparticles with Ag helps to improve photocatalytic effectiveness (Türkyılmaz et al., 2017). Ag/ZnO is one of the metal-semiconductor nanocomposites that demonstrated higher photocatalytic performance, which was synthesized through biomolecule assisted hydrothermal method (Gao et al., 2011). Chennimalai et al., (2021) synthesized Ag/ZnO and Au/ZnO nanocomposites using *Opuntia ficus* fruit extract which show significant biological activity for biomedical application. According to the research done by Khan et al., (2020), Ag-ZnO nanocomposite created from *Excoecaria agallocha* leaf extract were shown to possess good photocatalytic performance. The green synthesized ZnO NPs and Ag-ZnO NCs were tested for photocatalytic degradation of methylene blue and the effective degradation of MB was found to be 56.44 % for ZnO NPs and 98.44 % for Ag-ZnO NCs within 100 min under solar irradiation light.

2.1.2 Application of nanocomposites

Nanocomposites have gathered significant interest due to their unique properties and potential applications. Nanocomposites finds applications across various fields including biosensing, photovoltaics, energy storage, optics, and catalysis (Sharwani et al., 2022). Additionally, nanocmposites are increasingly significant in environmental remediation, particularly in water purification. The high surface area and reactivity of nanocomposites make them effective in capturing and degrading contaminants (Kusdianto et al., 2021). Therefore, a composite is one of the best option in which two or more materials are combined to obtain enhanced functionalities. Some of the significant applications of nanocomposites are presented diagrammatically in scheme 2.1.



Scheme 2.1: *Applications of nanocomposites*

2.2 Method of Synthesis

A number of methods have been devoted for the fabrication nanomaterial such as sol-gel method, co-precipitation method, hydrothermal method, green synthesis method and so on. Some important methods of synthesis are described below:

2.2.1 Sol-gel method

The sol-gel method is a conventional and industrial method for the synthesis of nanoparticles with different chemical composition. Basically, gel is formed from the precursor's solution. The solvent in the gel is then removed from the gel structure and the remaining gel is dried. Dried gels in various ways are used in industries such as surface coating, building insulation, and the production of special clothing (Bokov et al., 2021). Karunakaran et al., (2011) synthesized ZnO and Ag-ZnO by using sol-gel method which shows larger photo-catalytic and bactericidal activities.

2.2.2 Co-precipitation method

The co-precipitation method is a highly adaptable and commonly used technique utilized in the synthesis of nanoparticles and composite materials. This method involves the simultaneous precipitation which leads to the formation of solid particles that possess a wide range of applications, including catalysis, drug delivery, and advanced materials development. According to R. et al., (2021) synthesized Ag-ZnO nanocomposites by using the co-precipitation method and the synthesized nanocomposite show photocatalytic activity for the waste water treatment. Similarly, Jazi et al., (2012) synthesized Ag/ZnO nanocomposites via a chemical co-precipitation method. By using co-precipitation method, Subhan et al., (2014) synthesized Ag/ZnO nanocomposite, which shows appreciable photocatalytic activity for dye degradation and also Ag-ZnO nanocomposites was found to be effective antimicrobial agent.

2.2.3 Hydrothermal method

Hydrothermal synthesis is a widely employed method for preparation of nanomaterials. The term "hydrothermal process" is defined as performing chemical reactions in solvents contained in sealed vessels in which the temperature of solvents can be brought to around their critical point via heating concurrently with autogeneous pressures. This process is referred to as “hydrothermal” when water is used as the solvent (J. Li et al., 2016). There are various advantage of hydrothermal synthesis method over others such as use of simple equipment, catalyst-free growth, low-cost, large area uniform production, environmental friendliness and less hazardous (Aneesh et al., 2007). Hydrothermal synthesis can generate nanomaterials which are not stable at elevated temperature. Nanomaterials with high vapour pressures can be produced by the hydrothermal method with minimum loss of materials (Gan et al., 2020). According to the research done by Zare et al., (2019), ZnO-Ag nanocomposite were created from *Thymus vulgaris* leaf extract by using single step bio-hydrothermal method.

2.2.4 Green synthesis method

The conventional methods for the production of NPs are expensive, toxic, not eco-friendly. To overcome these problems, researchers have found the precise green routes, i.e., green synthesis simply means synthesis of metal nanoparticles using plant or plant parts or using their extract, an alternative to chemical and physical method. The “green chemistry” focuses on using environmentally friendly substances and reducing the use of hazardous chemicals during the synthesis and other chemical processes (Bamal et al., 2021). For example, Gawade et al., (2017), created ZnO nanoparticles by using the leaves extract of *Calotropis procera* which acts as a reducing and stabilizing agent. The green synthesis of ZnO NPs from the leaves of *Calotropis procera* plant appears to be a promising, low cost and ecofriendly method without using any toxic chemicals. Furthermore, the green synthesized ZnO NPs were tested for the photocatalytic degradation of methyl orange (MO) and the degradation efficiency of MO was found to be 81% within 100 min under UV light. According to the research done by Alharthi et al., (2020), nanocomposite (Ag-ZnO) were created from potato waste and used as photocatalytic activities.

Similarly, (Sorbiun et al., 2018), successfully synthesized Ag, ZnO, and bimetallic Ag/ZnO alloy nanoparticles via green method using aqueous extract of oak fruit hull (Jaft). The extract played the role stabilizing and reducing agent. The characterization of synthesized Ag, ZnO and Ag/ZnO nanoparticles was done by using FTIR, XRD and FESEM. For Ag/ZnO samples, the average particle size is 19.2 nm. In addition, the degradation of basic violet 3 was examined for photocatalytic activity. The photocatalytic activity for Ag/ZnO nanocomposites took only 30 minute to decolorized 79 % against basic violet 3 while for ZnO it took more than 90 minute to decolorized similar amount. The advantages of this biosynthesis method include use of nonhazardous, simple, low cost and environment friendly materials. Therefore, green synthesis method can be the best alternative approach.

2.3 Photocatalysis

Environmental contamination and wastewater treatment are critical global challenges. Traditional methods like adsorption, reverse osmosis, and chemical coagulations, however, these approaches have limitations in terms of efficiency since they often require additional treatments and cost-intensive (Whang et al., 2012). This has led to the search for alternative, cost-effective, and efficient approaches to seize this issue. In recent year, photocatalytic materials have gained more importance due to their potential application on water purification and environmental remediation (Liu et al., 2019). Therefore, low-cost and highly efficient photocatalysis has been attracting more attention since it has a great potential in solving environmental problem.

Semiconductor-mediated photocatalysis is an attractive route for reducing environmental pollution because it efficiently degrades various contaminants under light irradiation, converting them into harmless byproducts such as carbon dioxide, water, and mineral acids (Ahmad et al., 2013). Metal oxides, including TiO_2, ZnO, SnO_2, and others, have been widely used for photocatalysis due to their desirable characteristics such as high reactivity, affordability, excellent stability, non-toxicity, and chemical inertness (Ansari et al., 2013). The enhancement of photocatalytic activity in ZnO modified with silver (Ag) occurs because this modification increases the efficiency of separating photogenerated electrons and holes in ZnO. So, the improved photocatalytic activity of ZnO with Ag is attributed to a significant reduction in the number of surface defect sites on ZnO after the addition of Ag (Divband et al., 2013).

Various recent research studies indicate that the introduction of noble metal nanoparticles, such as Pd, Pt, Au, and Ag, onto the ZnO surface can significantly boost photocatalytic activity, particularly in degrading organic dyes (Abdel et al., 2019). According to the research done by Sun et al., (2012), Ag/ZnO nanocomposites were created via a facile microwave method. Also, the degradation of MB was examined for photocatalytic activity. As compared to ZnO, Ag/ZnO nanocomposite showed enhanced photocatalytic activities. Basnet et al., (2019) biosynthesized Zinc oxide NPs and Silver-Zinc oxide NCs using the leaves extract of tea plant (*Camellia* *sinensis*) and the characterization of synthesized nanomaterials was done by using powder X-ray diffraction, High resolution transmission electron spectroscopy, Energy dispersive X-ray spectroscopy, FTIR, UV-visible spectroscopy and X-ray photoelectron spectroscopy (XPS). The results indicated a substantial enhancement in the catalytic and photocatalytic behavior of ZnO NPs when combined with Ag, particularly for degrading the contaminants in water bodies under visible light irradiation.

As per the study, the synthesis of Ag/ZnO nanocomposite using *Desmostachya bipinnata* leaf extract has not been reported yet. According to a study conducted by (Guntur et al., 2018), it was demonstrated the leaf extract of *Desmostachya bipinnata* contained various phytochemicals including alkaloids, carbohydrates, flavonoids, saponins, tannis and protein. These diverse phytochemicals make *Desmostachya bipinnata* leaf extracts a highly potential candidate for nanoparticle synthesis. This study aims to produce Ag/ZnO nanocomposites through an eco-friendly and sustainable approach using *Desmostachya bipinnata* leaf extract as a natural reducing agent to enhance the photocatalytic activity.

CHAPTER III

MATERIALS AND METHODOLOGY

3.1 Materials

3.1.1 Sample collection and study area

Desmostachya bipinnata leaves were gathered from Tulsipur municipality ward No 16 (28.1545° N and 82.3235° E), Dang, Nepal in Feburary, 2022 which is shown in fig.3.1. All the experimental works have been performed in the research laboratory of Amrit Campus, Tribhuvan University Kathmandu. The leaf of *Desmostchya bipinnata* has been used for this study.

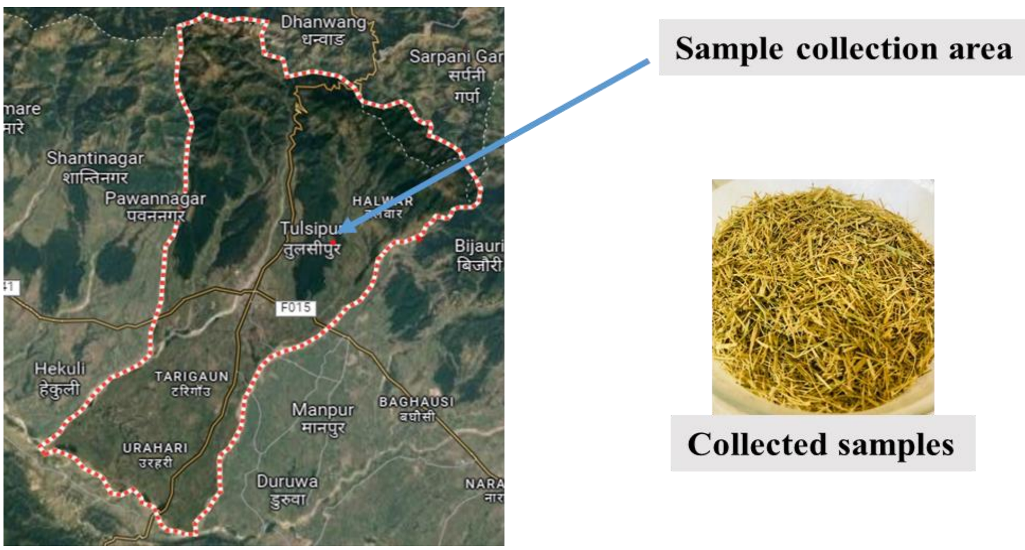


Figure 3.1: *Sample collection area and collected samples*

3.1.2 Chemicals

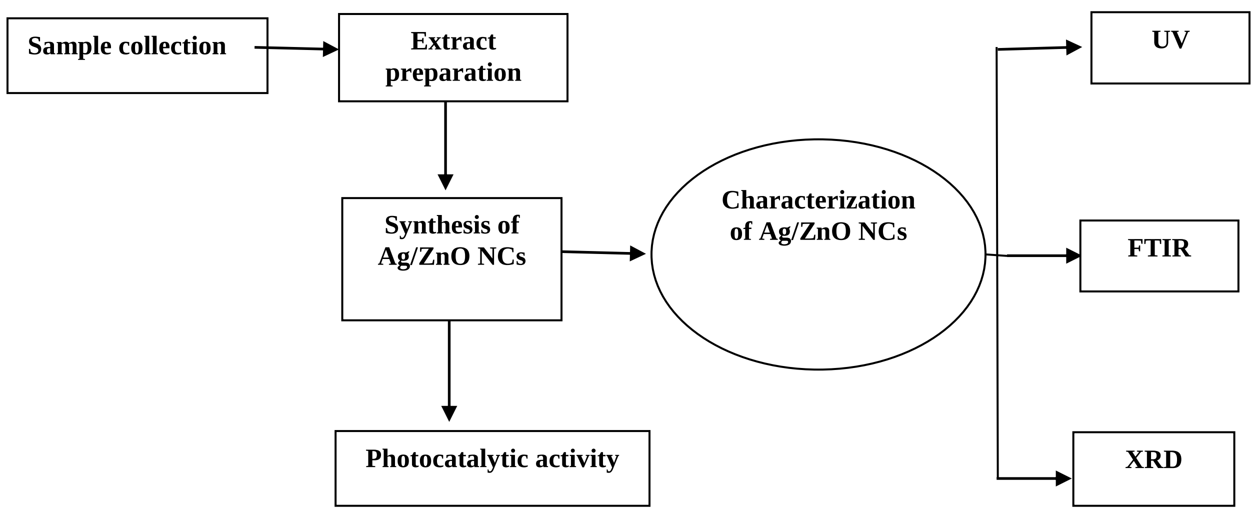
All the required chemicals used for this study were of analytical grade and were used from the department of chemistry, Amrit Campus, Tribhuvan University Laboratory.

a) Silver nitrate (99.8 %, Qualigens)
b) Zinc nitrate hexahydrate (96-103 %, Fisher Scientific)
c) Sodium hydroxide pellets (97 %, LOBA CHEMIE PVT.LTD)
d) Methylene blue (70 %, s.d. fine-chem limited)

3.1.3 Equipment

The instruments like Magnetic stirrer, Whatmann filter paper, Sonicator, Centrifuging apparatus, Muffle furnace, UV lamp, Auto deluxe digital pH meter (Labtronics-10, India), UV-vis spectrophotometer (Labtronics-LT2802), Fourier Transform Infrared Spectroscopy (PerkinElmer 10.6.2), X-Ray Diffraction (Rigaku diffractometer), Dryer, Other glasswares.

The whole experiment was carried out based on the following framework.



Scheme 3.1: *Systematic representation of research framework*

3.2 Preparation Methods

3.2.1 Preparation of *Desmostachya bipinnata* leaves extract

Firstly, the leaves of *Desmostachya bipinnata* were cleaned with distilled water and then dried in shade for 10 days. Well dried and clean leaf of *Desmostachya bipinnata* powdered by grinder. 12.5 g powdered was mixed with 250 mL of distilled water then, heated at constant temperature of 60 ˚C for half an hour and mixture was let down to cool. Finally, leaf extract was obtained after filtering by Whatmann filter paper no. 41 to remove the residual solids. Schematically, the method of extraction is shown in fig.3.2.

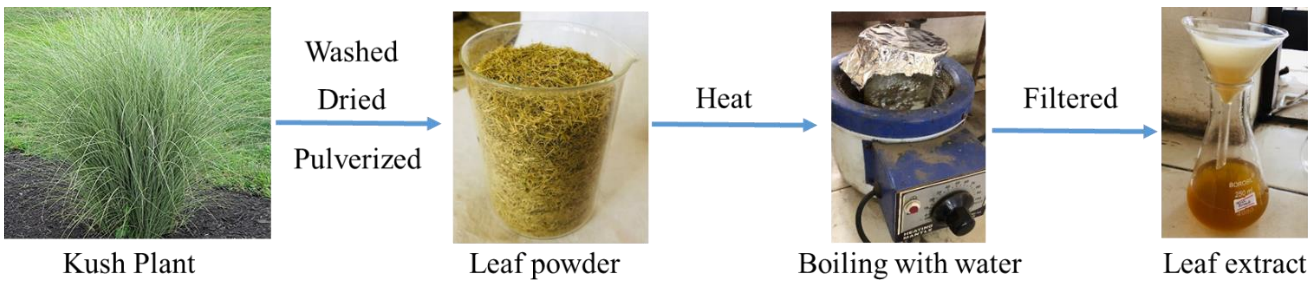


Figure 3.2: *Schematic representations of the leaf extract preparation.*

3.2.2 Phytochemical analysis

Phytochemical analysis stands for identification of various bio-active compounds present in *Desmostachya bipinnata* which were analyzed by observing the colour using specific reagent for specific bio-active compound. To investigate the phytochemical constituents present in leaf extract standard protocol was followed (supplementary Table-S_1, page No; 47).

3.3 Reagent Preparation

3.3.1 Preparation of $AgNO_3$ solution

Silver nitrate solution of 0.1 M was prepared by dissolving 4.24 g of silver nitrate in 250 mL of volumetric flask.

3.3.2 Preparation of $Zn(NO_3)_2.6H_2O$ solution

Zinc nitrate solution of 0.1 M was prepared by dissolving 7.43 g of zinc nitrate hexahydrate with 250 mL distilled water in volumetric flask.

3.3.2 Preparation of NaOH solution

Sodium hydroxide solution of 2 M was prepared by dissolving 8 g of NaOH in 100 mL of volumetric flask.

3.3.3 Preparation of methylene blue solution

100 ppm MB stock solution was prepared by dissolving 0.1 g of MB in 100 mL of volumetric flask. 5 mL of 100 ppm MB was pipetted out and diluted to 5 ppm in 100 mL of volumetric flak to carry out photocatalytic activities

3.4 Synthesis of Zinc oxide nanoparticles

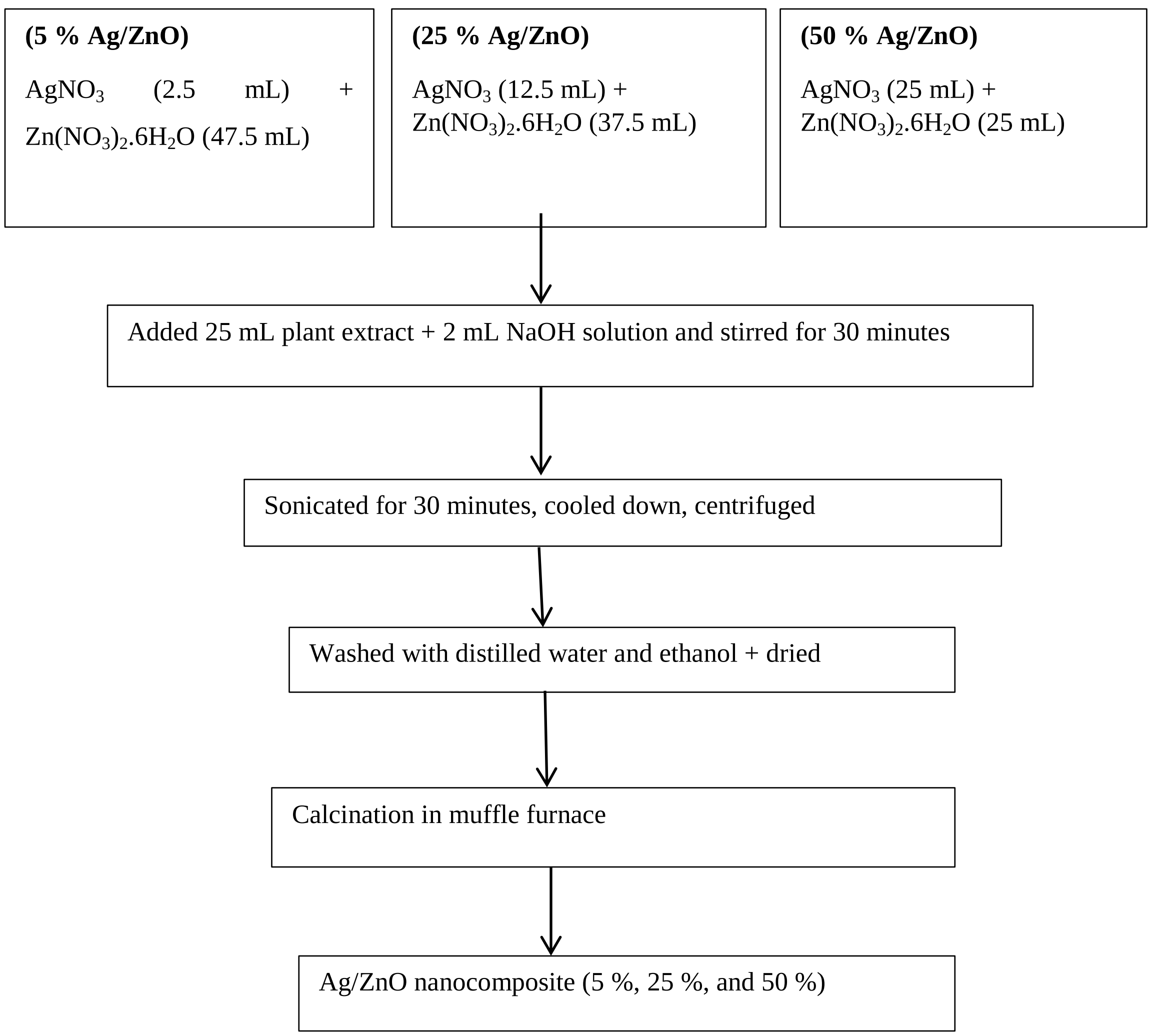
The synthesis of Zinc oxide nanoparticles (ZnO NPs) was carried out using a 0.1 M solution of zinc nitrate hexahydrate and 25 mL of freshly prepared aqueous extract from *Desmostachya bipinnata* in 250 mL beaker. 2 mL of 2 M NaOH was also added to this mixture so as to adjust the pH at 8. The solution was stirred on magnetic stirrer at room temperature for 30 minutes and subsequently the solution was sonicated for another 30 minutes. After cooling, the solution was centrifuged to separate the precipitate. The precipitate was then washed repeatedly with distilled water and ethanol. After that, it was dried in hot air oven for 12 hrs at 70 ˚C. Finally, it was calcined in muffle furnace at 300 ˚C for 3 hours resulting in the collection of pure white ZnO NPs. Synthesis process was schematically represented as in fig.3.3.

Figure 3.3: *Schematic representations for the green synthesis of ZnO NPs.*

3.5 Synthesis of Silver/Zinc oxide (Ag/ZnO) nanocomposites

For the synthesis of Ag/ZnO (5 %) nanocomposite, 2.5 mL of $AgNO_3$ (0.1 M) and 47.5 mL of zinc nitrate hexahydrate (0.1 M) solution was taken in 250 mL beaker. 25 mL of freshly prepared aqueous extract of *D.bipinnata* and 2 mL of NaOH (2 M) was added for adjusting the alkaline pH and then solution was stirred on magnetic stirrer at room temperature for 30 minutes. Subsequently, the mixture was sonicated for 30 minutes. The solution was let down to cool. After that, the mixture was centrifuged, filtered, and washed with distilled water and ethanol, thus obtained precipitate was dried and calcinated in muffle furnace at 300˚C for 3 hours. Finally, sample was collected for further characterization and analysis.

For the synthesis of Ag/ZnO (25 %) nanocomposite, 12.5 mL of silver nitrate (0.1 M) and 37.5mL of zinc nitrate hexahydrate (0.1 M) solution was taken respectively. Similarly, for the synthesis of Ag/ZnO (50 %) nanocomposite, 25 mL of silver nitrate (0.1 M) and 25 mL of zinc nitrate hexahydrate (0.1 M) solution was taken respectively and 25 mL of freshly prepared extract was added. Same procedure was followed for synthesis of both 25 % and 50 % Ag/ZnO nanocomposites as described for 5 % nanocomposite which was outlined in scheme 3.3.



Scheme 3.4: *Flow sheet diagram of synthesis of Ag/ZnO (5 %, 25 %, and 50 %) nanocomposites*

3.6 Physicochemical Characterization

Structural and morphological characterizations were carried out using different instruments, such as UV-vis spectroscopy, FTIR, XRD, and Photocatalytic efficacy of as-synthesized composite materials was performed using Methylene Blue solution as a model compound. The phytochemical test was based on the chemical test method where visual changes were noted.

UV-visible spectroscopy was used to determine the formation of ZnO NPs, Ag/ZnO nanocomposites. UV- visible spectrometer (Labrotonics, model LT2802) having double beam wavelength from 200 to 800 nm was run through the sample solution (at a concentration of 1 mg/10 mL distilled water) using a cuvette and absorbance was observed. It was carried out in the Department of Chemistry, Amrit Campus, Kathmandu.

Fourier Transform Infrared Spectroscopy (FTIR, Tracer 100) was used for the identification of functional group present in the extract, which acts as the reducing and stabilizing agent in ZnO NPs and Ag/ZnO nanocomposite formation. The FTIR spectra were recorded with an FTIR spectrometer (PerkinElmer 10.6.2) at the cut off range 400-4000 cm^{-1} with scan interval 4 cm^{-1}. It was also carried out in the Department of Chemistry, Amrit Campus, Kathmandu.

The crystallinity and crystal phase of the obtained materials were probed by an X-ray diffraction (XRD) instrument (Cu Kα (λ = 1.5406 Å) radiation on a Rigaku diffractometer, JNCASR, Bengaluru, India).

3.6.1 Photodegradation of Dye

The release of different types of synthetic dyes like methylene blue (MB) into water is leading to the growing problem of water pollution. This results harmful effects on aquatic life and the entire ecosystem, which are becoming increasingly severe. To overcome this issue, this study focused on employing photocatalytic degradation techniques to refine the polluted water. For photocatalytic degradation of methylene blue (MB), 50 mL of 5 ppm MB was taken in 100 mL beaker containing 30 mg sample with constant stirring under UV light (a series of six Philips UV lamps 15 W having a center wavelength of 254 nm positioned at 20 cm a distant over the suspension surface) source. In order to ensure adsorption/desorption equilibrium, the solution was stirred for 30 min in dark, prior to the irradiation. The absorbance of the irradiated sample solution was recorded in each 5 min interval. The experimental conditions for all set of experiments are tabulated in table 3.1.

Table No.3.1: *Experimental Conditions*

Methylene Blue	**50 mL, 5 ppm**
Photocatalyst	Ag/ZnO nanocomposites and ZnO nanoparticles, 30 mg for 50 mL MB (5ppm)
Temperature	25 ˚C
pH	Neutral
Light source	UV light
Irradiation time	0-30 min

CHAPTER IV

RESULTS AND DISCUSSION

4.1. Phytochemical Analysis

Aqueous extract from leaves of *Desmostachya bipinnata* was successfully prepared out. Phytochemical screening was done to identify bioactive compounds present in leaf extract of *Desmostachya bipinnata* which could play effective role as stabilizing and reducing agents. Various phytochemicals present were as shown in the table no. 4.1.

Table No.4.1: *Phytochemical Analysis Test*

S. No.	Phytochemicals	Result
1.	Alkaloids	Present
2.	Carbohydrates	Present
3.	Flavonoids	Present
4.	Saponins	Present
5.	Tannins	Present
6.	Polyphenols	Present
7.	Proteins	Present

During this study, phytochemical analysis of the aqueous leaf extracts from *Desmostachya bipinnata* revealed the presence of bioactive compounds like alkaloids, carbohydrates, flavonoids, saponins, tannis, polyphenols and proteins. These bioactive phytoconstituents were considered to function both as reducing agent and stabilizers throughout the nanomaterial synthesis process (Guntur et al., 2018). According to the research conducted by Veera et al., (2017), it was demonstrated the leaf extract of *Desmostachya bipinnata* contained various phytochemicals including alkaloids, carbohydrates, saponins, tannins, and phenolic compounds, protein and flavonoids, which are similar with the findings presented in this thesis work.

4.2. UV- visible Spectroscopy

UV-vis spectroscopy is useful analytical tool for optical characterization of nanomaterial. Result obtained from UV-vis spectroscopic characterizations are shown in fig.4.1. It is found that ZnO nanoparticles shows broad peak at 390 nm. Nevertheless, the Ag/ZnO nanocomposites also exhibited shoulder peaks in UV-vis spectrum around 420 nm due to Ag particles attached. The peak of ZnO shows the red shift in the UV-vis spectrum of the composite which could be associated with the agglomeration of particles into relatively larger sizes or may be due to the some sort of interaction between Ag and ZnO. The incorporation of silver nanoparticles into ZnO broadens the UV band. This red shifting of the absorption band indicate for the formation of composite as reported by Jobe et al., (2022).

Basnet et al., (2019) reported that the UV absorbance peak of ZnO NPs and Ag/ZnO NCs at 352 nm and nearly (427-577) nm respectively. In comparision to ZnO, the Ag/ZnO showed a broad peak in UV region due to the interaction and transfer of electrons between the silver and ZnO (Saoud et al., 2018). Similarly, Patil et al., (2014) determined that λ_{max} of ZnO nanoparticles at 385 nm and UV spectrum of Ag/ZnO nanocomposites, specifically between 400 and 525 nm, caused by surface plasmon resonance, providing strong evidence for the interaction between Ag and ZnO as similar to our result presented here.

4.2.1. Calculation of Band Gap Energy

The band gap energy (E_g) of the ZnO NPs and Ag/ZnO NCs was obtained from the value of wavelength using the following equation (Gawade et al., 2017):

$$\mathbf{E_g = hc/\lambda\ eV} \longrightarrow \quad (1)$$

Where,

E_g is band gap energy (eV) of the material, h refers to Plank's constant (6.626 x 10^{-34} Js), C is the velocity of light (3 x 10^8 ms^{-1}) and λ is the wavelength in nm.

- **For ZnO:**

$$E_g = (6.626 \times 10^{-34})\ (3 \times 10^{8})\ /390$$

- **For Ag/ZnO:**

$$E_g = (6.626 \times 10^{-34})\ (3 \times 10^{8})\ /420$$

Thus, the band gap energy of ZnO and Ag/ZnO was found to be 3.17 eV and 2.95 eV respectively. Likewise, in our finding Jobe et al., (2022) determined optical band gap of ZnO NPs with an energy band gap of 3.5 eV and Ag/ZnO with reduced band gap 2.6-2.7 eV. The lowered band gap energy and widening of the absorption range make the composite material more efficient at capturing light.

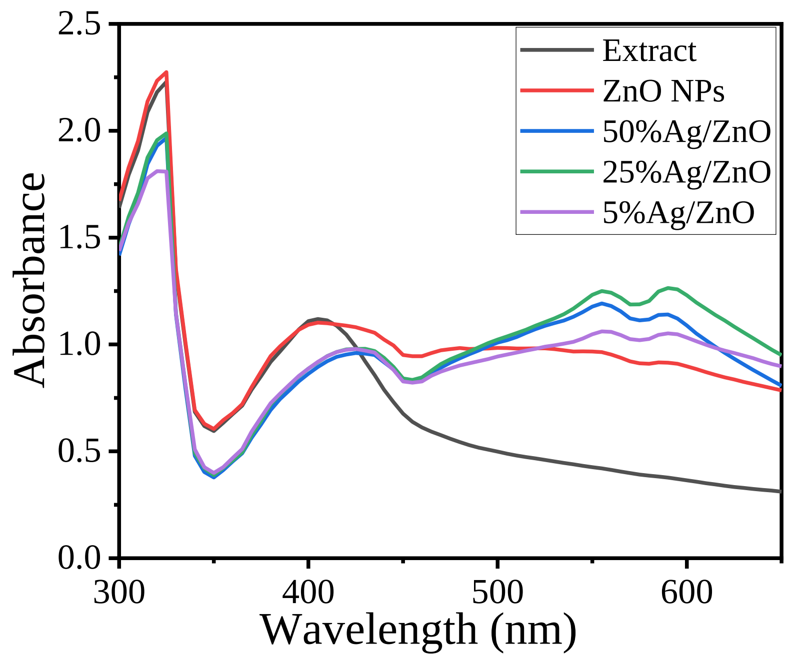


Figure 4.1: *UV-vis spectra of extract, ZnO, and Ag/ZnO (5 %, 25 %, and 50 %) nanocomposites*

Since, the leaf extract contains bioactive compounds, which act as reducing agents and the reaction occur as shown in following equation. These bioactive compounds reduce the Zn^{++} to Zn of zero valency which upon calcinations forms ZnO NPs.

$$Zn^{2+} + \text{leaf extract} \longrightarrow \text{ZnO NPs}$$

4.3. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a vibrational spectroscopic technique that can be used to identify and characterize the chemical component present in the extract. The functional groups present in as-synthesized products were investigated by using FTIR spectroscopy in the range of 500- 4000 cm^{-1}.

The FTIR spectra of ZnO NPs, Ag/ZnO NCs of varied wt % and *D.bipinnata* leaf extract were shown in fig.4.2. The extract exhibited peak at 1385 cm^{-1}, 1636 cm^{-1}, 2112 cm^{-1}, and 3339 cm^{-1}. The extract exhibited broad peak at 3339 cm^{-1} illustrates the stretching of the hydroxyl (-OH) group and the peak around 1635 cm^{-1} due to the bending of –OH group (Srivastava et al., 2013). The peak at 2112 cm^{-1} due to C≡C stretching of alkyne group (Silverstein & Bassler, 1962) while, the peak around 1385 cm^{-1} due to C-N stretching vibration of aliphatic and aromatic amines (Sorbiun et al., 2018). The ZnO exhibited peak around 1571 cm^{-1} is due to C=O stretching (Silverstein & Bassler, 1962). Peak appeared around 1051 cm^{-1} attributed for C-O stretching of ester and tertiary alcohol. The nanocomposites also exhibit the similar peaks however the peaks are of less intensity. Verma & Basheer Khan, (2021), reported that the peak at 1051 cm^{-1} attributed to the stretching of Zn-O-Zn in Ag/ZnO nanocomposite. Further, the nanoparticles exhibited the sharpest and dominated band at ~ 530 cm^{-1} attributed to stretching vibration of M-O bonds (M= Zn and Ag). According to Jobe et al., (2022), the bio-synthesized ZnO NPs and Ag/ZnO nanocomposite showed major absorption band at 3294, 2919, 1612, and 1035 cm^{-1} due to O-H, C-H, C=C, and C-O stretching respectively. Consequence of FTIR spectral results indicate the presence of variety of phytoconstituents acting as the capping agent over the surface of as-synthesized materials. Hence, the FTIR result support for formation of Ag/ZnO nanocomposites.

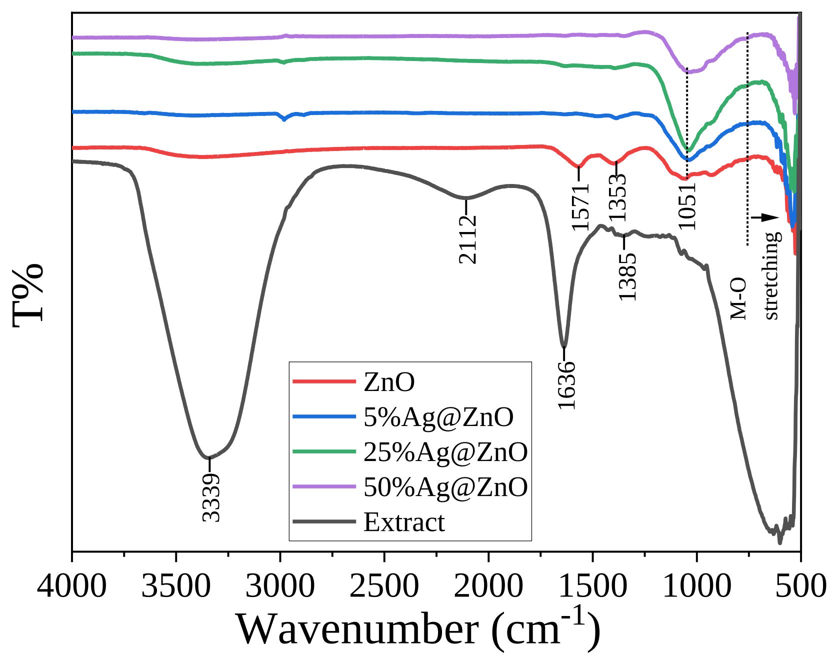


Figure 4.2: *FTIR spectra of D.bipinnata leaf extract, ZnO and Ag/ZnO NCs*

4.4. X-Ray Diffraction (XRD)

The XRD pattern of the as-synthesized nanomaterials shows that the materials are of crystalline nature. The observed diffraction peaks at 2θ value of 31.8˚, 34.49˚, 36.1˚ , 47.5˚ , 56.7˚, 62.87˚ and 68˚ corresponding to the (100), (002), (101), (102), (110), (103), and (112) plane of crystal lattice, respectively, reveal their hexagonal wurtzite structure which matched well with the reference data sheet for zinc oxide nanoparticles in Joint Committee on Power Diffraction Standards (JCPDS) card no. 05-0664 shown in fig.4.3 (Li et al., 2015; Sorbiun et al., 2018).

With the incorporation of Ag NPs, the intensity of zinc oxide nanoparticles got decreased which is clearly shown in XRD pattern of Ag/ZnO nanocomposite. The intensity of peak assign the crystallinity, the peaks of Ag/ZnO nanocomposites are of less intensity compared to that of ZnO and silver which indicate that the Ag/ZnO are of less crystalline. In Ag/ZnO nanocomposite the additional peak at 38˚ , 44.4˚, 64.26˚, and 77.30˚ correspond to (111), (200), (220), and (311) planes respectively matched with the XRD pattern of Ag NPs (Ag XRD Ref. No. 01-087-0719) in fig.4.3 (Shameli et al., 2012). The intensity of the peaks can provide important information about composition and crystalline structure of Ag/ZnO nanocomposite material. As the percentage of silver (Ag) in the composite increases, the intensity of ZnO peaks decreases, reaching its minimum at 50 % Ag/ZnO, signifying a decrease in ZnO content. Conversely, the intensity of Ag peaks rises with higher Ag percentages, reaching its maximum peak point at 50 % Ag/ZnO, indicating an increase in the presence of Ag (Sharma et al., 2018).

The average crystallite size of the green synthesized Ag/ZnO nanocomposites was calculated by using Debye-Scherer formula (Panchal et al., 2020),

$$D = \frac{K\lambda}{\beta \cos\theta} \longrightarrow \quad (2)$$

Where,

D= Crystallite size of materials

λ= Wavelength of Cu Kα radiation (0.15406 nm)

θ = Bragg's angle

β = Corrected half width of the diffraction peak (in radian)

K= Shape factor which usually equals to 0.94

Furthermore, the calculated value of crystallite size of Ag/ZnO nanocomposite was 12-14 nm.

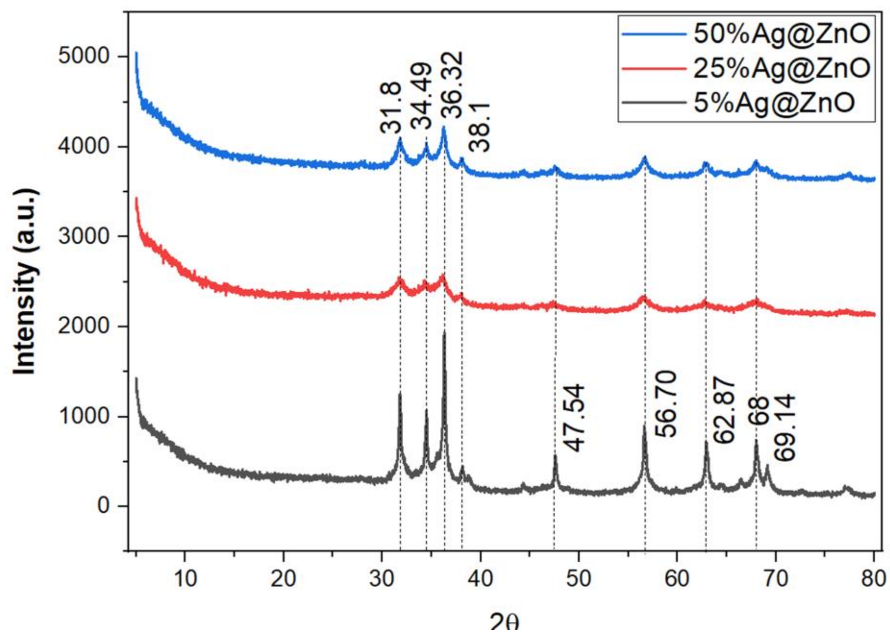


Figure 4.3: *XRD of Ag/ZnO nanocomposites with varied weight % (5 %, 25 % & 50 % Ag/ZnO)*

4.5. Photocatalytic Dye Degradation

Over the recent years, photocatalytic degradation has gained significant attention due to its potential for efficient and sustainable dye removal. The UV-vis spectra of MB solution containing ZnO, and different ratios of Ag/ZnO nanocomposites are shown in figure 4.4 (a-d). Initially, the pure MB solution exhibited a prominent peak at 665 nm. When this pure MB solution was exposed to UV light for 30 minutes in the presence of different samples i.e, ZnO or various ratio of Ag/ZnO, a progressive decrease in the UV absorbance peak was observed over successive time intervals. The result indicated that when MB was subjected to UV exposure in the presence of as-synthesized nanocomposites, degradation of MB occurred.

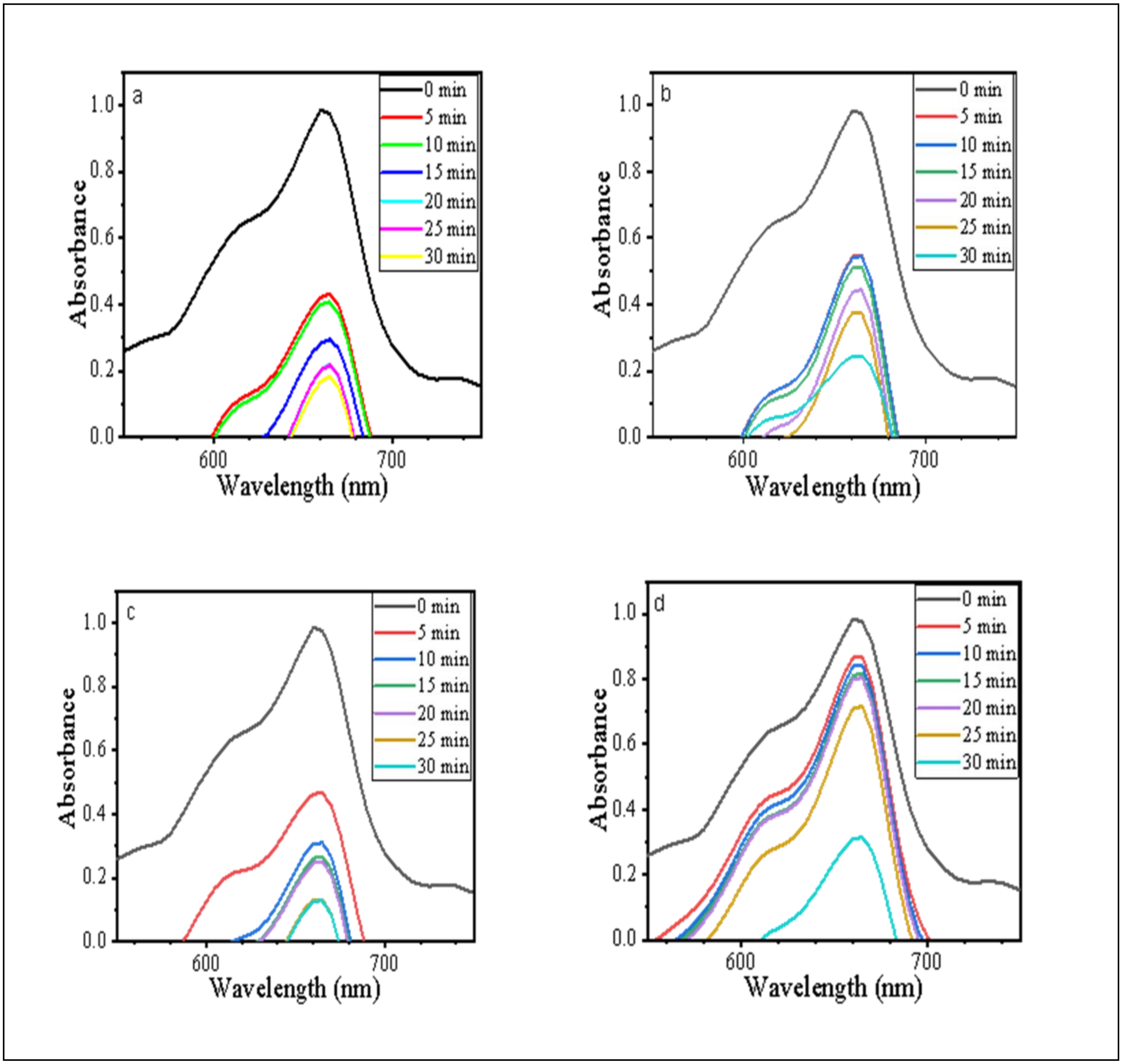


Figure 4.4: *Photocatalytic degradation of MB using (a) ZnO,(b) 50 %Ag/ZnO, (c) 25 %Ag/ZnO, (d) 5 %Ag/ZnO*

4.5.1. Degradation efficiency

The degradation efficiency of the as-synthesized catalyst towards MB is determined by using the following equation (Jothibas et al., 2019).

$$D\ \% = \frac{C_0 - C_t}{C_0} \times 100 \longrightarrow (3)$$

Where, C_o and C_t are concentration at time 0 and t.

Fig.4.5 shows C_t/C_o plot versus irradiation time for ZnO and Ag/ZnO nanocomposites with varied weight percentage. This fig.4.5 illustrates the degradation of MB using ZnO nanoparticles, which exhibits lower degradation compared to the 25 % Ag/ZnO nanocomposite. However, in the case of 5 % Ag/ZnO and 50 % Ag/ZnO nanocomposites they show less degradation efficiency could be due to the oxidation of silver and less visible light absorption.
In fig.4.6, the photocatalytic degradation of different nanoparticles was compared at 30 minute. The efficiencies of pure ZnO and various ratio of nanocomposites i.e, 5 % Ag/ZnO, 25 % Ag/ZnO, and 50 % Ag/ZnO were found to be 79 %, 66 %, 85 %, and 73 % respectively. The order of photocatalysis efficacy is in the order of 25 % Ag/ZnO >ZnO >50% Ag/ZnO >5 % Ag/ZnO. Interestingly, for 25 % Ag/ZnO, the photodegradation observed after 30 minute of UV-exposure was noticeably higher compared to both other Ag/ZnO nanocomposites and the pure ZnO nanoparticles. It may be because of the formation of smaller sized crystallites in 25 % Ag/ZnO nanocomposites rather than that of other nanocomposites. Literature has shown that the silver incorporated ZnO is responsible for both charge separation and the absorption of visible light, leading the process more efficient. By separating charges, the Ag NPs prevent the recombination of electrons and holes, which is a common issue during photocatalysis. This preventation is a critical factor contributing to the increased effectiveness of Ag/ZnO in breaking down dyes and organic compounds when exposed to visible light. Degradation efficiency of different nanocomposites are also presented in bar diagram in fig.4.6

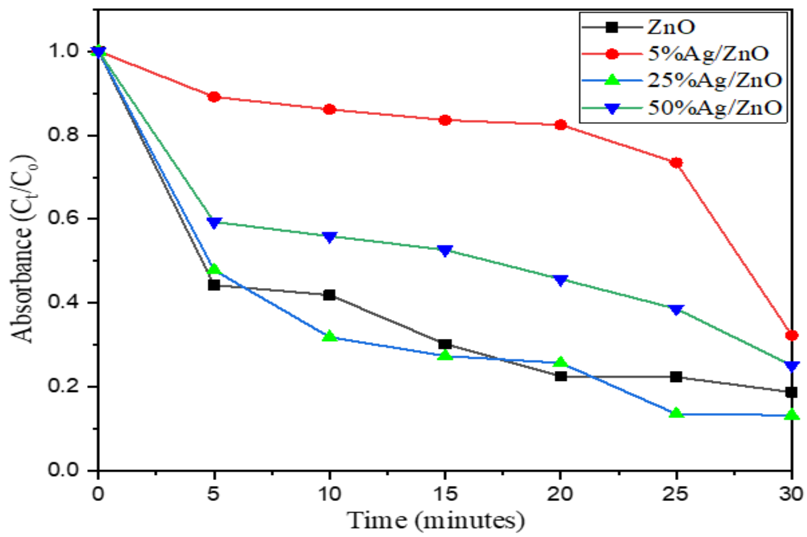


Figure 4.5: *Photocatalytic degradation of MB solution under UV light in the presence of different samples versus irradiation time*

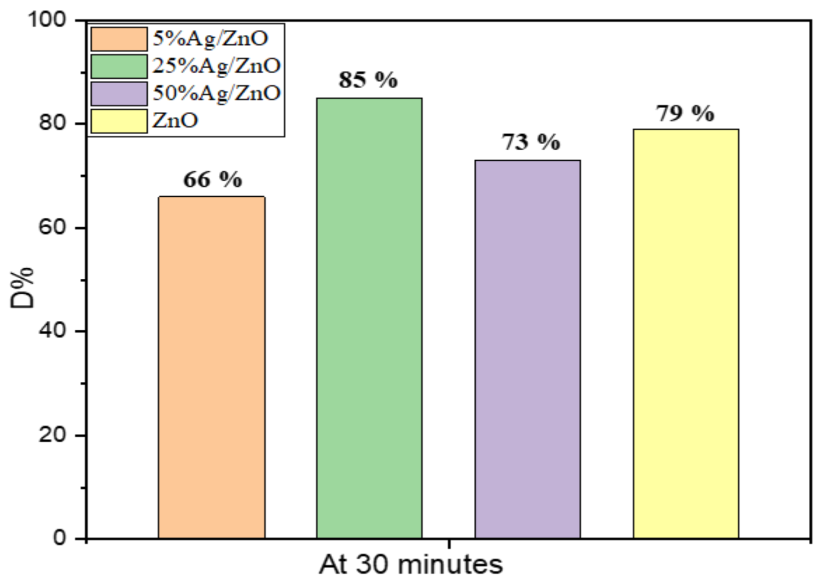


Figure 4.6: *Degradation efficiency at the time of 30 minute*

The degradation behavior is observed from the following mechanism. Schematic representation of model of degradation of MB is shown in figure 4.7. When ZnO nanoparticles are exposed to UV light with energy equal to or greater than the band energy gap, an electrons (e^-) from the valence band (VB) is excited to the conduction band (CB), resulting in the generation of a hole

(h^+) in VB. Subsequently, the photo-generated electrons in the conduction band of ZnO nanoparticles are transferred to the Fermi level of silver nanoparticles and this electron transfer process is energetically favorable and helps to suppress electron-hole recombination (Abbas et al., 2021). The electrons in the conduction band (CB) of ZnO nanoparticles react with O_2 to form superoxide radical ($^{\cdot}O_2^-$), while the holes in the valence band (VB) react with water to form hydroxyl radical ($^{\cdot}OH$). These newly created intermediates, $^{\cdot}O_2^-$ and $^{\cdot}OH$; possess high reactivity and strong oxidizing properties. They effectively oxidize the MB dye, leading to the formation of carbondioxide (CO_2), water (H_2O) and corresponding mineral acids as degradation products. The findings suggest that the presence of Ag nanoparticles anchored on the surface of ZnO can enhance the rate of formation of $^{\cdot}O_2^-$ and $^{\cdot}OH$ reactive species (Alharthi et al., 2020). Simultaneously, this synergistic effect facilitates the degradation of organic pollutants, such as MB dye.

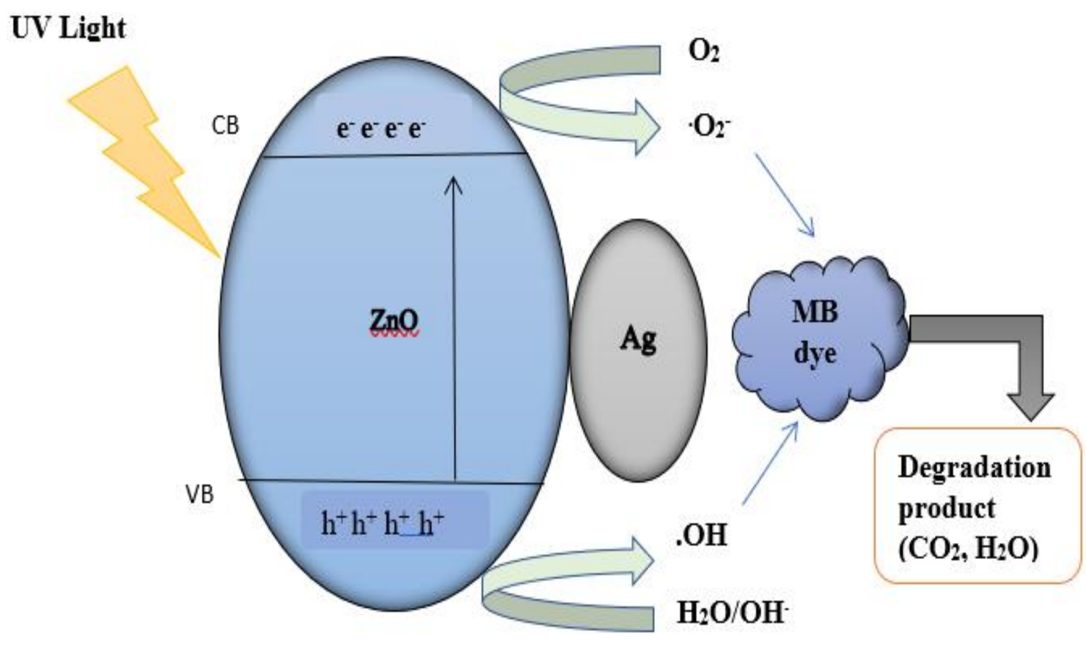


Figure 4.7: *Mechanism of photocatalytic degradation*

The possible reactions are as follows:

$$Ag/ZnO + UV\ light \longrightarrow (e^-_{CB}) + (h^+_{VB})$$

$$(e^-_{CB}) + O_2 \longrightarrow {}^{\cdot}O_2^- \text{ (reduction product)}$$

$$h^+ + OH^-/H_2O \longrightarrow {}^{\cdot}OH \text{ (oxidation product)}$$

$${}^{\cdot}\ O_2^- + MB \longrightarrow \text{Degradation product}$$

$${}^{\cdot}OH + MB \longrightarrow \text{Degradation product}$$

CHAPTER V

CONCLUSIONS

In this study, ZnO and Ag/ZnO nanocomposites were successfully synthesized using *Desmostachya bipinnata* extract as a reducing and stabilizing agent. The successful formation of ZnO NPs and Ag/ZnO NCs was confirmed from the UV-peak observed at 390 nm and 420 nm respectively. The FTIR confirmed that the aqueous extract of *Desmostachya bipinnata* contained different bio-active compounds that were responsible for stabilizing and reducing factor during the synthesis of ZnO and Ag/ZnO nanocomposites. Similarly, as-synthesized Ag/ZnO NCs was found to be of crystalline nature having crystallite size of 12-14 nm. The as-synthesized materials were used for the photocatalytic degradation of methylene blue (MB). The Ag/ZnO nanocomposite show higher photodegradation efficiency as compared to ZnO. Overall, this study demonstrated that, Ag/ZnO nanocomposites has greater potential for the degradation of methylene blue and can be used for the treatment of organic pollutants presence in waste water, when exposed to UV-vis light irradiation. As the nanocomposites other than 25 % Ag/ZnO show somewhat less degradation efficiency as expected, hence, it will be better if the degradation process is repeated.

Further Recommendation

This study involved the utilization of different concentration of zinc nitrate hexahydrate and silver nitrate solution for synthesize the Ag/ZnO nanocomposites. Many researchers have carried out different approaches for synthesis of nanocomposites. Here, is some additional recommendation for the synthesis of Ag/ZnO nanocomposites:

- Ag/ZnO nanocomposite can be more applicable for the optical, electronic and photocatalytic behavior.
- Shape and size controlled synthesis of ZnO NPs and Ag/ZnO NCs can be an interesting research topic for future.

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Supplementary

Table-S1: Phytochemical Analysis Protocol

S.N.	Experiment	Observation	Inference
1	Test for alkaloids a. Wagner's test Few drops of Wagner's reagent + few mL of extract, shake well	Reddish-brown ppt.	Presence of alkaloids
	b. Mayer's test Few mL of plant extract + 2 drops of Mayer's reagent, shake well	White creamy ppt.	Presence of alkaloids
2	Test for carbohydrates a. Molish's test 2 mL of plant extract + 2 drops of Molish's reagent shake well + few drops Conc. H_2SO_4 slowly from the side tube and allow to stand for few minutes	Violet ring at junction of two layers	Presence of carbohydrates
3	Test for flavonoids 1 mL of aqueous extract + 1 mL of 10% lead acetate solution	Yellow ppt	Presence of flavonoids
4	Test for saponins 5 mL extract + 5 mL distilled water, warmed	Appearance of stable foam	Presence of saponins
5	Test for tannins 2 mL of aqueous extract stirred with 2 mL of distilled water + few drops of $FeCl_3$	Green colored ppt.	Presence of tannins
6	Test for polyphenols 3 drops of 5 % $FeCl_3$ + 2 mL extract, shake	Black color	Presence of polyphenols
7	Test for proteins Biuret Test: 2 mL 5 % NaOH + 2 mL extract + $CuSO_4$ solution	Pink color	Presence of proteins