PROJECT REPORT

On

SYNTHESIS OF ZINC OXIDE / BIOCHAR COMPOSITE ITS CHARACTERIZATION AND APPLICATION

Submitted by

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In partial fulfillment for the award of the

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<u>**B.Sc. CHEMISTRY PROJECT REPORT**</u>

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This is to certify that the project "**SYNTHESIS OF ZINC OXIDE / BIOCHAR COMPOSITE ITS CHARACTERIZATION AND APPLICATION**" is the work done by **PURNIMA MALLIK**

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CERTIFICATE

This is to certify that the project work entitled "**SYNTHESIS OF ZINC OXIDE / BIOCHAR COMPOSITE ITS CHARACTERIZATION AND APPLICATION**"is the work done by **PURNIMA MALLIK** under my guidance in the partial fulfilment of the award of the Degree of Bachelor of Science in Chemistry at St. Teresa's College (Autonomous), Ernakulam affiliated to Mahatma Gandhi University, Kottayam.

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<u>**DECLARATION**</u>

I hereby declare that the project work entitled **"SYNTHESIS OF ZINC OXIDE / BIOCHAR COMPOSITE ITS CHARACTERIZATION AND APPLICATION"** submitted to Department of Chemistry and Centre for Research, St. Teresa's College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, is a record of an original work done by me under the guidance of **Mr. Linto Anto, ASSISTANT PROFESSOR,** Department of Chemistry and Centre for Research, St. Teresa's College (Autonomous), Ernakulam and this project work is submitted in the partial fulfilment of the requirements for the award of the Degree of Bachelor of Science in Chemistry.

PURNIMA MALLIK

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PURNIMA MALLIK

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Chapter 1

Introduction

Environmental pollution has become one of the most serious global challenges due to rapid industrialization, urbanization, and population growth. As a result, the living standards have improved. But it also led to severe environmental challenges. The release of various toxic chemicals from the industrial sector is a serious threat to the environment and human health. Among various types of pollution, water pollution is of major concern because clean water is essential for human health, agriculture, and ecosystem sustainability. Industrial wastes are discharged into water bodies which contain heavy metals, synthetic organic dyes, and toxic chemicals causing severe environmental and health problems. The textile, paper, leather, pharmaceutical, cosmetic, and dyeing industries extensively use synthetic dyes, which are highly stable but non-biodegradable due to their complex molecular structures and resistant to conventional treatment methods. Many of these dyes are also Toxic, mutagenic, and carcinogenic, making their removal from waste water crucial.[1]

Even small concentrations of dyes in water can cause visible coloration, reduce light penetration, disrupt aquatic life and carcinogenic risks to humans. It is found that the annual production of dye is more than 700,000 tons and approximately 10-15 % of organic dyes are discharged into waste water and as an important source to pollute the environment. High concentration of dyes found in water bodies alters ecosystem by means of absorbing and reflecting solar radiation that make difficult the process of photosynthesis by plant species inhabiting in water bodies [2,3]. Methylene blue, methyl orange, rhodamine B, and Congo red, etc., are some of the synthetic, water-soluble, organic dyes frequently used in textile, printing, and paper industries. These dyes cause skin irritation, respiratory problems, eye damage, etc. Out of various organic dyes, methylene blue is a cationic dye and a heterocyclic aromatic compound, extensively used in textile industries as synthetic coloring agent for cotton, wool, and silk. It is considered as a potential carcinogen and environmental pollutant [3,4].

Methylene blue is widely used in industries like paper, paint, textiles, pharmaceuticals, cosmetics, and food. The waste water from these sectors merges with drinking water and heavily contaminates the water, which is dangerous to humans and animals. Exposure to Methylene blue causes severe health problems like nausea, vomiting, eye irritation, profuse sweating etc. The high demand of colored dyes particularly in textile industry, has significantly increase the production volumes of dyes, results severe environmental damages therefore several treatments such as coagulation, adsorption on activated carbon, reverse osmosis, ultra-filtration are the traditional methods used to remove dyes. Nonetheless these techniques have several drawbacks such as high cost, generation of secondary pollutants, incomplete removal of dye etc. Thus, researchers developed several methods to remove Methylene blue from water, among others photocatalytic degradation is the most widely used method due to their cost-effectiveness, ease of use, moderate reaction conditions and environmentally friendly nature. It involves various metal oxide-based semiconductors as photocatalyst for waste water treatment. Several advantages of photocatalysis are high efficiency, utilization of clean solar energy, minimal generation of hazardous waste, making it applicable in diverse fields. Primary features of photocatalyst include their extensive active surface area, effective charge separation, ability to absorb visible light, and enduring photostability. TiO_2 , CeO_2, ZnO, SnO_2, CuO etc are commonly used metal oxide-based semiconductors. Zinc oxide (ZnO) has gained more attention due to its cost-effectiveness, high stability, direct energy, band gap etc. Although ZnO nanoparticles are used in photo catalytic applications, they exhibit certain limitations compared to bulk materials, such as solubility in acidic conditions, photoleaching in basic solutions under UV light, rapid electron-hole recombination, and insufficient absorption of visible light energy, all of which lowers photocatalytic efficiency. In addition, ZnO exhibits limited activity under visible light i.e; ZnO alone has limited dye adsorption capacity, which limits effective interaction between the catalyst and dye molecules. ZnO's performance is improved through elemental doping, metal deposition, and heterostructure construction in order to perform in visible light regions[5].

Another environmentally friendly approach for pollutant removal is adsorption, a process favored for its cost-effectiveness and ease of operation. Various adsorbents, including silica gel, alumina, and natural clays are used, they are often expensive and hard to find in large quantities and making them hard to use in many areas. This high cost and limited availability highlight the need for more sustainable alternatives. Biochar (BC), a carbon rich material produced through the pyrolysis of biomass under low oxygen conditions. It exhibits excellent adsorption capacity due to its mesoporous structure and large surface area, making it suitable for pollutant removal. However, adsorption alone may only transfer pollutants from one phase to another rather than fully mineralizing them. To overcome this, combining adsorption with advanced oxidation processes (AOPs) has been proposed. Biochar properties can also be enhanced through chemical, physical, and magnetic modifications, or by integrating nanoparticles. These modifications improve the effectiveness of biochar in pollutant removal. Thus, biochar acts as a substrate for photocatalyst, where enhancing their degradation efficiency. Adsorption and photo-catalytic degradation have emerged as promising alternatives due to their simplicity, effectiveness, and environmental compatibility. Various metal oxide-coated biochar nanocomposites have been prepared using different biomass sources. Combining ZnO nanoparticles with biochar results in a ZnO/BC composite which integrates the photocatalytic activity of ZnO with the adsorption capacity of biochar. The composite offers several advantages such as increased adsorption capacity, high surface area, numerous active sites, well-established structures, improved charge separation, and better dispersion of ZnO nanoparticles on the biochar surface. Biochar acts as a support matrix for ZnO, improving stability and photocatalytic activity. This ZnO/BC composite effectively degrades methylene blue under visible light irradiation [1,5].

Hence, In this project, the main aim is to synthesize an environmentally friendly ZnO/BC composite. By utilizing Aloe barbadensis miller extract for the initial preparation of ZnO nanoparticles and further the synthesized nanoparticles were integrated into a biochar matrix through a sucrose-sulphuric acid carbonization method to produce a stable ZnO/BC composite. UV- Visible spectroscopy, FTIR and X-ray diffraction were used to characterize the physical and chemical properties of ZnO/BC composite and further the photocatalytic efficiency of the composite was analyzed through the degradation of methylene blue under sunlight irradiation.

1.1 ZINC OXIDE NANOPARTICLES (ZnO Nps)

Among various kinds of nanoparticles, metal-based nanoparticles are important because they are less harmful to living organisms and the environment. Metal oxide nanoparticles are also crucial in different areas, especially in bio nanomedicine and agriculture, as well as in managing various infectious diseases. Among the metal oxide nanoparticles, Zinc Oxide nanoparticles (ZnO Nps) are commonly used because they are less toxic and have a range of chemical, physical, and biological properties. They also possess a high binding energy (60MeV), strong chemical stability, excellent photostability, and a wide bandgap (3.37ev). Compared to numerous other nanoparticle types, zinc oxide has been deemed non-toxic by the Food and Drug Administration (FDA), with many applications in food and cosmetics. Recently, ZnO nanoparticles have attracted extensive attention due to their large specific surface area and high active sites. Meanwhile, ZnO is a multifunctional material, which means that it has many areas of application. The ZnO NPs have great benefits of cheap production, V-blocking, and a white appearance. The process of synthesis of nanoparticles is a significant factor that influences their properties and applications. Various methods are used to synthesize ZnO nanoparticles, including physical, chemical, and green methods. Chemical and physical methods include precipitation, micro emulsion, chemical reduction, sol-gel and hydrothermal techniques, and pulsed laser deposition. Synthesized ZnO nanoparticles with different sizes and morphologies can play an excellent role in different fields. However, physical and chemical methods are costly and produce secondary waste that pollutes the environment. Green synthesis is gaining increasing popularity owing to its lower environmental impact [6].

In this study, we approach the green synthesis of ZnO nanoparticles. Compared to other conventional chemical or physical synthesis, green synthesis is chosen for several key reasons such as eco-friendly and safe, cost-effective and simple, energy efficient and have high biocompatibility etc. Green methods have a wide range of raw materials, including plants, bacteria, algae, and even enzymes. Plant parts such as root, stem, leaf, fruits and even seed can be used for ZnO nanoparticles synthesis, but the plant extract used to green synthesis the ZnO Nps is a more widely used process [7]. ZnO nanoparticles prepared from plant extracts are more stable than those prepared from other raw materials because there are natural metabolites including flavonoids, polyphenols, and terpenoids, etc., which acts as eco-friendly, efficient capping and stabilizing agents, replacing the need for toxic chemical agents. The process of ZnO nanoparticles prepared from plant extracts is a safe, cheap, and simple process that is highly efficient and environmentally responsible. A variety of plants have been applied for the synthesis of ZnO nanoparticles such as mint leaves, Phoenix dactylifera, Peganum harmala seed extract etc [8]. We use Aloe barbadensis miller (Aloe Vera) extract for creating ZnO nanoparticles. Aloe vera leaf extract acts as a reliable reducing and capping agent for producing zinc oxide nanoparticles in green chemistry.

Aloe Barbadensis Miller (Aloe Vera) leaves have many beneficial properties. Properties such as the increasing effectiveness in antibiotics, the antifungal properties in agriculture, and also inhibiting cancer cells. It is cultivated mainly for agricultural and medicinal uses. All parts of the plant can be used for various applications, making it a multifunctional plant. A high number of phenolic compounds are also found in Aloe Vera, which are important in cancer treatment. All parts of aloe Vera, such as leaves, roots, and stem, have so many properties therefore, they have been used in agricultural, cosmetic, and medicinal areas. Because of this rich chemical profile, Aloe vera serves as an excellent biological agent for the green synthesis of ZnO nanoparticles [7].

1.1.1 PROPERTIES AND APPLICATIONS OF ZnO NANOPARTICLES

Zinc oxide is usually a semiconductor with a wide band gap (3.37eV) at room temperature. It has been widely used for its catalytic, electrical, opto electronic, and photochemical properties. ZnO nanostructures are highly beneficial for catalytic reactions due to their high catalytic activity and vast surface area. Zinc oxide has a crystalline structure. The three structures of ZnO are wurtzite, zinc blend, and rock salt. The rock salt structure forms under high-pressure conditions, while the zinc blende structure appears when ZnO grows on a substrate with a proper cubic lattice. The wurtzite structure is hexagonally close-packed, consisting of Zinc (Zn) and oxygen (O) atoms arranged at tetrahedral sites in an alternating manner.

Due to the properties such as high thermal conductivity, high refractive index, binding energy, UV protection and antibacterial capabilities of Zinc

Oxide, it is widely applied in various products and materials, including medicine, cosmetics, solar cells, rubber and concrete, foods. ZnO nanoparticles are broadly used in sensors, piezoelectric devices, solar energy cells, electrical instruments, optical instruments, and the elimination of environmental contaminants. ZnO is incorporated into cosmetic lotions for its ability to block and absorb harmful UV radiation. It is also used as an astringent for wounds healing, anti-hemorrhoids, eczema, and excoriation in human medicine. Currently, ZnO NPs are also applied as peripheral antimicrobial agents in food packages, textile stuff, mouthwash, ointments, and lotions for microbial growth inhibition. All the properties and its application can be modified by changing the size and morphology. It also offers promising applications in veterinary science, due to their wound healing, antibacterial, antineoplastic, and antigenic properties. Additionally, ZnO has photocatalytic properties that used in food packaging and the agriculture industry for its antifungal effects [7,9] . Table 1 shows the general properties of ZnO.

Table 1: Properties of ZnO

Chemical Structure	ZnO
Molecular Weight	81.389 g/mol
Appearance	White Solid
Odour	Odourless
Crystal Structure	Wurtzite
Coordination Geometry	Tetrahedral
Band Gap	3.37ev
Density	5.606 g/cm³

1.2 BIOCHAR (BC)

Biochar is a stable type of organic carbon. It forms when biomass is heated to temperatures usually between 300 and 1000°C, with little to no oxygen present. You can use various types of biomasses to produce biochar, including agricultural leftovers, wood chips, manure, and municipal solid waste. Different thermal processes can create biochar, but slow pyrolysis is the most common method since it works under moderate conditions and maximizes biochar production. Biochar is a source of renewable energy having the potential to generate heat, electricity, and liquid biofuels. Biochar is referred to as black carbon which is produced by thermal or chemical transformation of the original biomass material. The physical and chemical properties of biochar can vary greatly based on the feedstock and thermochemical conversion methods used for production. Therefore, the effective use of biochar in different field applications is closely linked to the production methods and the making of the source material. In some cases, the material properties of biochar may be similar to those of charcoal as an energy carrier. However, many types of biochar do not burn easily. Charcoals are usually not made to solve soil or environmental problems. A key feature of biochars, like charcoal, is the presence of specific organic carbon forms known as fused aromatic ring structures. These structures form during pyrolysis and are essential for biochar properties in mineralization and adsorption, among others. As a result, biochar is usually high in carbon and often has more phosphorus, calcium, magnesium, and sometimes nitrogen. The chemical properties of the organic carbon structure in biochar are quite different from the material it was made from, and it is low in oxygen and hydrogen. As biochar is created for purposes other than burning and collecting thermal energy, it can be made from various biogenic feedstock materials. Not all types of biochar are suitable for combustion. It may only overlap with charcoal under specific conditions [10] .

Biochar is a solid material created when biomass breaks down through heat and chemical processes. As biochar is cheap, eco-friendly, and can serve many purposes like soil improvement, waste management, reducing greenhouse gases, and generating energy, several studies have explored new uses for biomass. The main component of biochar is carbon, but it also includes hydrogen (H), oxygen (O), ash, and small amounts of nitrogen (N) and sulfur (S). The makeup of biochar changes based on the raw biomass used to make it and the details of the carbonization process. Due to its large surface area, porous structure, surface functional groups, and high mineral content, biochar has been used to absorb water and air pollutants, to act as a catalyst for removing tar or producing biodiesel, and as a soil enhancer. Recently, there have also been reports on using biochar cells and super capacitors [10,11] .

1.2.1 PROPERTIES AND APPLICATIONS OF BIOCHAR

Biochar is environmental friendly and a very versatile material and is significantly used in many applications. Biochar produced through different conversion technologies usually have different properties due to their operations and processing methods. The high surface area and cation exchange capacity of biochar is determined to a large extent by its source materials and post production techniques. A high surface area is important for the sorption of both organic and inorganic contaminants in Biochar. Biochar is also used to improve the soil fertility and maximize the crop yields. Biochar effects soil properties, nutritional qualities and agricultural output. The Biochar produced at different temperatures show different properties and functions. Due to the biochar's low cost, eco-friendly nature and multiple applications it helps to reduce pollutants and improve soil quality and to boost crop productivity and improve soil fertility, we can apply biochar to the soil. Biochar is a solid product rich in carbon. It comes from the biomass of agricultural crop residues, waste, and wood. This process occurs through pyrolysis in an oxygen-poor environment. Carbon sequestration using biochar is important for its potential to recycle waste, retain nutrients in the soil, and reduce greenhouse gas emissions.

Biochar is a stable nutrient in soil and plays a crucial role in adsorption and mineralization. As a carbon rich substance, it functions as a soil supplement in agricultural fields and diminishes the probability of environmental degradation and pollution. As a means of biochar production woody feedstock oak sawdust are mainly used. Biochar can also replace some of the soil's water properties. Most of the carbon can be removed through the short-lived carbon cycle after biochar is added to the soil. Biochar has various physical and chemical influence its effectiveness. Biochar is mainly affected by feedstock conditions and process of pyrolysis that changes from the difference of physical and chemical properties. Biochar contains a high percentage of oxygen containing carbon groups inert carbon components and a high surface area. The structure and properties of biochar impact its applications. Biochar can also be used to produce biofuels. Biochar improves soil properties by increasing water retention, permeability, and soil fertility. Its high charge density carries large amounts of nutrients, changing the soil properties, and boosting crop output. It can retain water and nutrients in the top layer of soil for a long time, preventing nutrient loss from the crop root zone and directly improving crop yield. Biochar has been used to support sustainability in the energy, environmental, and agricultural sectors. One emerging technology, biochar production, can boost national food security and sequester carbon to help slow climate change. Biochar is also used to support the ZVI (zerovalent iron) particles, which are both environmentally and economically beneficial. Biochar can be made from locally available materials like wood products, agricultural waste, and manure which are inexpensive [10,11,12] .

1.3 ZnO/BC NANOCOMPOSITE

A ZnO/BC composite is a functional hybrid material formed by incorporating zinc oxide nanoparticles into the porous matrix of biochar, which is a carbon-rich material derived from the pyrolysis of biomass. It is designed to combine the high surface area and adsorption capacity of biochar with the photocatalytic and antibacterial properties of ZnO. This composite is sustainable, cost-effective, and environmentally friendly. The synthesized ZnO/BC composite has the characteristics of both biochar and ZnO nanoparticles. The process to create biochar-metal oxide nanocomposites starts with treating feedstock using different metal salt solutions, including $ZnCl_2$, $PbCl_2$, $CaCl_2$, $FeCl_3$, $CuCl_2$ as reported by various researchers. Additionally, metal oxides can easily be attached to pure biochar surfaces through reduction and precipitation processes [6].

ZnO-coated biochar nanocomposites bring the advantages of increased surface area, heightened reactive sites, improved dispersion, robust electron transfer between ZnO and biochar, and amplified photocatalytic activity. The fusion of ZnO nanoparticles and biochar holds substantial promise in the photocatalytic degradation of organic pollutants [5].

1.3.1 PROPERTIES AND APPLICATIONS OF ZnO/BC COMPOSITE

The composite has both the properties of ZnO nanoparticles and biochar. ZnO nanoparticles have attracted the attention of researchers because of their easy processability, low toxicity, thermal stability, bio-compatibility, and environmentally friendly nature, but most notably because of their photocatalytic activity and antimicrobial activity. Biochar is a carbon-rich material, which has high adsorption capacity, chemical stability, and low cost. When we combine them, biochar prevents ZnO nanoparticle agglomeration, improves dispersion and stability, enhances adsorption of pollutants and ZnO adds its photocatalytic properties (especially under UV/visible light). Therefore, combining ZnO nanoparticles with biochar results in a ZnO/BC composite, which integrates the photocatalytic activity of ZnO with the adsorption capacity of biochar. The composite material offers several advantages such as enhanced surface area, improved charge separation, reduced recombination of electron-hole pairs, better dispersion of ZnO nanoparticles and increased dye degradation efficiency. Common applications are water and wastewater treatment, photocatalysis, antibacterial and antimicrobial applications, soil remediation, sensors and environmental monitoring, energy storage and electrochemical devices, agriculture and food safety etc. Composite shows high efficiency in dye degradation. It removes dyes through two main mechanisms, adsorption and photocatalysis. This synergistic effect significantly improves dye removal efficiency compared to pure ZnO or biochar alone [11,12,13].

1.4 CHARACTERIZATION TECHNIQUES

1.4.1 UV VISIBLE ABSORPTION SPECTROMETER

UV visible spectrometry is an analytical method that is based on Beer-Lambert law. The sample to be analyzed is passed through electromagnetic radiation in the UV-visible region (200nm- 800nm). As a result, the molecules absorb energy, causing transitions of electrons in the molecule. This results in the absorption of the spectra. The absorption spectra of a sample are indicative of its colour within the visible spectrum. This technique has several applications such as drug development, structural analysis of organic compounds, determination of molecular weight, and impurity detection. UV spectroscopy enables both qualitative and quantitative analyses to be performed efficiently [14].

1.4.2 X-RAY DIFFRACTION (XRD)

X-ray diffraction techniques are used for characterizing crystalline materials. It provides information regarding the structure and arrangement of atoms within the compound. X-ray diffraction peaks result from the constructive interference of a monochromatic X-ray beam scattered at particular angles by the lattice planes in a sample. The intensities of these peaks reflect the arrangement of atoms within the lattice. As a result, the

X-ray diffraction pattern serves as a fingerprint for the periodic atomic arrangement present in a material [15].

1.4.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Infrared (IR) spectroscopy, particularly Fourier Transform Infrared Spectroscopy (FT-IR), is a widely used technique for chemical analysis in laboratories. It provides valuable insights into molecular vibrations, enables the identification of functional groups within a molecule, making it a powerful tool for recognizing the chemical composition of a compound.

The method is based on the absorption of infrared light by a sample, resulting in a spectrum that displays absorbance or transmittance intensity against frequency (wave number). Each peak in the IR spectrum corresponds to specific molecular vibrations, allowing for the identification of functional groups. As such, the IR spectrum acts as a unique fingerprint for unknown compounds, which can be compared with standard reference spectra for structural identification. IR spectroscopy is versatile and can be used to analyze both organic compounds and inorganic materials, such as minerals and metals. Additionally, it can be applied to liquids and solids at atmospheric pressure [16].

Chapter 2

Materials and Methods

This chapter gives a brief description of the materials and experimental procedures adopted for the present investigation.

2.1 MATERIALS

- Aloe barbadensis miller (Aloe vera) extract
- Zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$)
- Sodium hydroxide ($NaOH$)
- Sucrose
- Conc. Sulfuric acid (H_2SO_4)

2.2 EXPERIMENTAL METHODS

2.2.1 SYNTHESIS OF ZnO NANOPARTICLES

The green synthesis of Zinc oxide (ZnO) nanoparticles was initiated by preparing an aqueous Aloe vera extract, where 50 g of fresh gel was boiled in 100 mL of distilled water at 80 °C for 30 minutes. The resulting solution was cooled and filtered through Whatman No.1 filter paper to obtain the clear plant extract. Separately, a precursor solution was formulated by dissolving 4.4 g of zinc acetate dihydrate in 100 mL of distilled water. The synthesis was carried out by mixing 25 mL of the Aloe vera extract with 75 mL of the zinc precursor solution under continuous stirring for two hours. During this period, the NaOH solution (prepared by dissolving 16 g of NaOH pellets in 200 mL of distilled water) was added dropwise to the mixture until the solution reached a pH of 12, resulting in the formation of a dense white precipitate. The precipitate was washed multiple times with distilled water to remove impurities and recovered through centrifugation. The obtained precipitate was dried in a hot air oven at 120 °C for 3 to 5 hours. Finally, the dried product underwent calcination in a muffle furnace at 600 °C for 1 hour to yield the stable, crystalline ZnO nanoparticles[7,8].

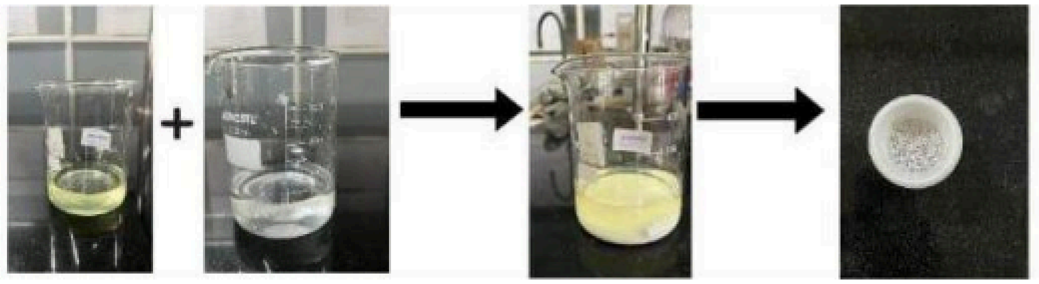

Fig 2.1 *Synthesis of Zinc Oxide nanoparticles*

2.2.2 PREPARATION OF ZnO/BC NANOCOMPOSITE

To prepare the ZnO/BC, 5.25 g of sucrose was first dissolved in 10 mL of water. To this solution, 12 drops of concentrated sulfuric acid (H_2SO_4) were added as a dehydrating agent, followed by the addition of 3 g of ZnO nanoparticles. The resulting white dispersion was stirred thoroughly to ensure the uniform distribution of the nanoparticles within the sugar matrix. This mixture was then transferred to an oven and maintained at 160°C for 16 hours, facilitating dehydration and formation of a pre-char material. Following the hydro thermal stage, the solidified product was mechanically ground into a fine powder using a mortar and pestle. The powdered material was then pyrolysed at 700°C for 2 hours, a high-temperature process that converted the organic precursor into a stable, carbon-rich biochar matrix. Once the process was complete, the composite was allowed to cool to room temperature and was once again crushed to ensure a uniform particle size. The final product obtained was a structured ZnO/BC composite ready for further characterization and application [8].

Fig 2.2 *ZnO / BC Composite*

2.3 CHARACTERIZATION METHODS

2.3.1 INFRARED-RED SPECTROMETRY (IR)

IR spectroscopy was used to identify the functional groups present in the synthesized ZnO/BC composite. The IR spectrum of synthesized particles was recorded using a Fourier Transform Infrared spectrometer (Thermo Nicolet iS50) inthe wavenumber range of 4000-400 cm^{-1}.

2.3.2 X- RAY DIFFRACTION (XRD)

XRD analysis was used to determine the crystalline structure and phase purity of the synthesized ZnO powder and the ZnO/BC composite. The X-ray pattern was recorded using Bruker D8 Advance, typically spanning a 2-θ range from 10° to 80°.

2.3.3 UV-VISIBLE SPECTROSCOPY

UV-Visible spectroscopy is an analytical technique that measures the absorption of light in the UV and visible region by a sample. The optical properties of ZnO were determined by UV- vis spectroscopy.

2.4 DYE ADSORPTION OF METHYLENE BLUE

About 0.1 g of methylene blue was taken and made up to 100 mL using distilled water. 10 mL of this solution was pipetted out into a beaker. The pipetted solution was made up to 1000 mL using distilled water. To 10 mL of the above solution, 0.01 g of activated carbon was added and kept for photocatalytic studies for 1 to 2 hours.The absorbance spectrum of the supernatant solution before and after adding activated carbon was measured using UV- visible spectrometer.

Chapter 3

Results and discussion

3.1 CHARACTERIZATION OF ZnO NANOPARTICLES AND ZnO / BC COMPOSITE

3.1.1 INFRARED SPECTROSCOPY

IR spectroscopy is an analytical technique used to identify the functional groups , metal oxygen vibration bands, and changes in metal-oxygen frequency due to modification .

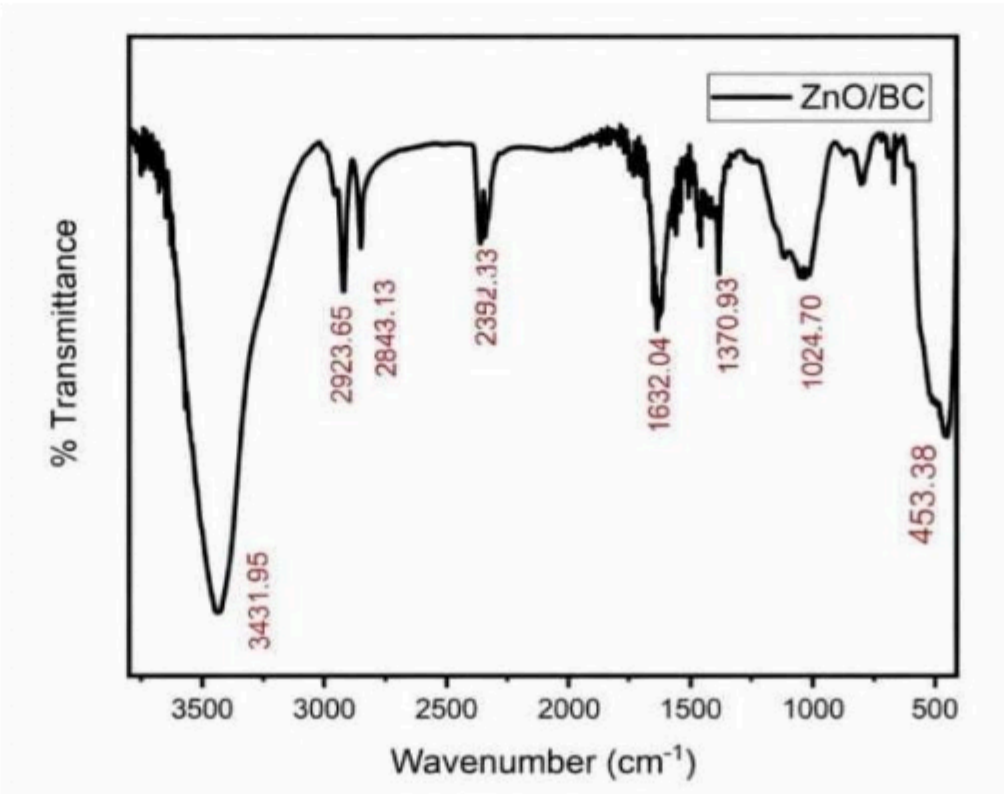

Fig 3.1 *IR spectrum of the ZnO / BC images*

The IR spectrum of the ZnO/BC Composite is presented in the fig 3.1. A broad absorption peak of 3431.95 cm^{-1} is associated with the O-H stretching vibration band of hydration on the ZnO surface. The peaks appearing at 2923.65 cm^{-1} and 2843.13 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of aliphatic C–H bonds (–CH_2 and –CH_3). A weak band found at 2352.33 cm^{-1} is linked to the stretching vibration of CO_2 or atmospheric carbon dioxide on the sample surface. This is commonly seen in FTIR measurements. The band at 1632.04 cm^{-1} is related to the bending vibration of H–O–H of physically adsorbed water and to the stretching vibration of C=C or C=O groups present in the biochar structure. The peak located at 1370.93 cm^{-1} belongs to the C–H bending vibration or C=O stretching of carboxylate or phenolic groups of biochar. The strong band seen at 1024.70 cm^{-1} comes from the C–O stretching vibration of alcohol, ether, or phenolic groups. These are common functional groups found in carbon-based materials. The absorption band at the lower wavenumber region, specifically at 453.38 cm^{-1}, relates to the Zn-O stretching vibration. This confirms that zinc oxide has formed successfully in the ZnO/BC composite. Thus, the FTIR spectrum shows that there are many oxygen-containing functional groups, like –OH and C– O, on the biochar surface. It also shows the specific Zn–O vibration. These results indicate that ZnO has been successfully added to the biochar matrix. This results in a strong interaction between ZnO nanoparticles and the functional groups of biochar. These interactions helps to improve the stability of the ZnO/BC composite.

3.1.2 UV – VISIBLE CHARACTERIZATION

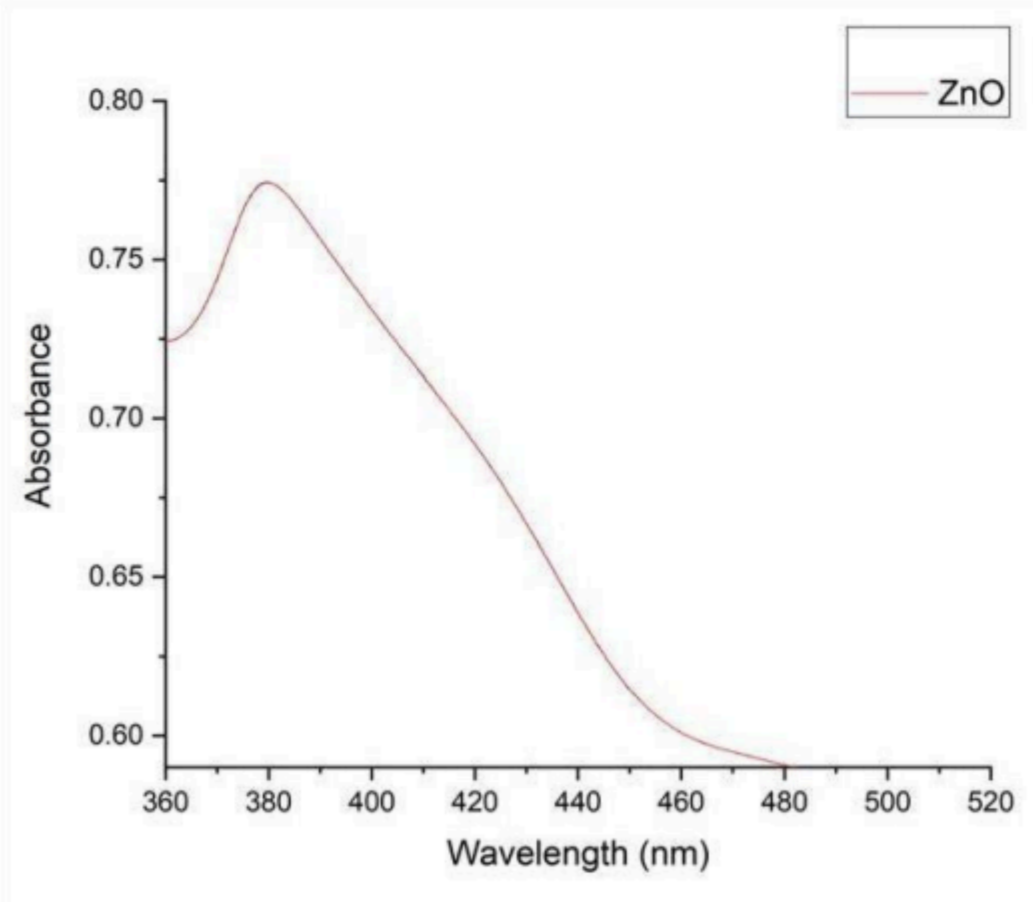

FIG 3.2 *UV-Visible spectrum of Zinc Oxide*

The UV-visible absorption spectrum of the synthesized ZnO nanoparticles is displayed in the figure 3.2, covering the wavelength range of 360 to 520 nm. A clear absorption band appears around 375 to 380 nm. A sharp absorption peak at 372 nm, which has been described as an intrinsic transition between the valence band and the conduction band of ZnO NPs.

This band is the typical absorption of ZnO nanoparticles. It relates to the band-to-band electronic transition from the valence band to the conduction band of ZnO. This confirms the successful creation of ZnO nanoparticles. The absorbance gradually decreases as the wavelength increases and becomes very low in the visible range, above approximately 450 nm. The graph confirms that ZnO absorbs strongly in the UV region (below 400 nm). This shows that the prepared ZnO nanoparticles strongly absorb UV light while remaining mostly transparent in the visible region. From the absorption edge at around 380 nm, the optical band gap is estimated to be about 3.26 eV. This value aligns well with the reported band gap of ZnO, further supporting the formation of ZnO nanoparticles with strong optical properties. ZnO/BC nanocomposite has lower bandgap energy than ZnO NPs, records a lower electron holes recombination rate, and is more useful for visible light photocatalysts. ZnO/BC nanocomposite can improve optical properties and enhance the photocatalytic degradation of MB under sunlight irradiation.

3.1.3 X-RAY DIFFRACTION (XRD) ANALYSIS

Powder X-ray diffraction is a useful method for examining both the amorphous and crystalline features of prepared materials. The XRD pattern of the pure ZnO nanoparticles (ZnO NPs) shows a series of distinct diffraction peaks (fig 3.3) . Notably, these peaks are identified at specific angles: 31.8° (100), 34.5° (002), 36.3° (101), 47.6° (102), 56.6° (110), 62.9° (103), 67.1° (200), 68° (112) and 69.1° (201). These clear peaks clearly show the presence of the wurtzite structure typical of ZnO. Importantly, the observed peaks provide strong evidence of the material's well-defined crystalline nature. These reflections align with the standard hexagonal wurtzite structure of ZnO. They confirm the successful formation of crystalline ZnO. Additionally, the lack of any secondary phases or impurity-related peaks indicates the high purity of the prepared sample. In the ZnO/BC sample, the main diffraction peaks of ZnO are still clearly visible. This confirms that the crystal structure of ZnO is maintained after it is added to the BC matrix. However, there is a slight reduction in peak intensity and some broadening of the peaks in the composite. This suggests a decrease in crystallite size or partial coverage of ZnO by the BC support. The lack of extra impurity peaks also shows that forming the composite does not introduce any new crystalline phases. The average size of the ZnO NPs within the ZnO/BC nanocomposite was determined from the maximum crystalline peak of ZnO, which corresponds to the (101) plane. To compute this size, the Debye-Scherrer formula was employed, as out-lined by the equation:

$$D = K\lambda / (\beta \cos\theta)$$

Where K is the shape factor (0.9), λ is the x-ray wavelength (λ=1.5406 Å), β is the full width at half maximum of the different peaks in radians, and θ is the Bragg angle. For the ZnO/BC nanocomposite, the average size was calculated at the 2θ position of the (101) peak of ZnO NPs. Thus the average size in nanometer (nm) was found to be 29.899, and so Debye-Scherrer equation is in XRD to estimate the size of nanoscale crystallites.

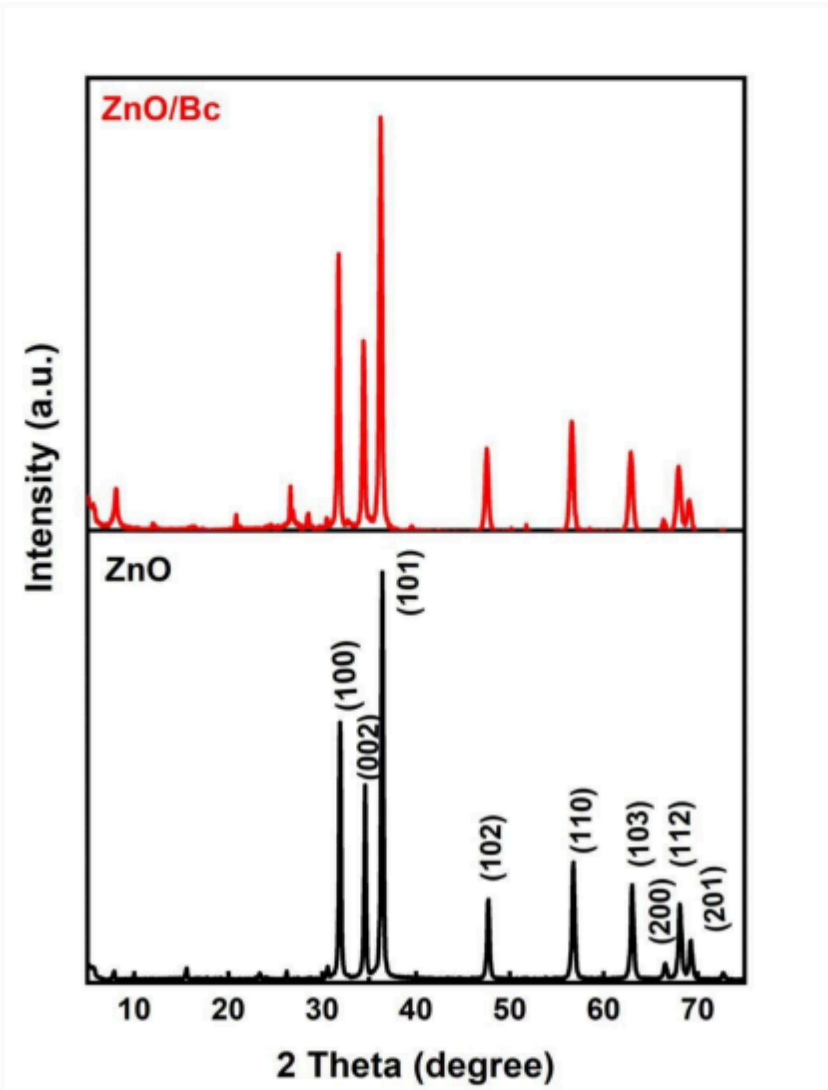

FIG 3.3 *X-Ray Diffraction graph of ZnO and ZnO / BC*

3.2 APPLICATION

3.2.1 ADSORPTION OF METHYLENE BLUE

Fig 3.4 gives the photograph of methylene blue and that of the mixture of Methylene blue and ZnO/BC composite for 1-2 hrs.

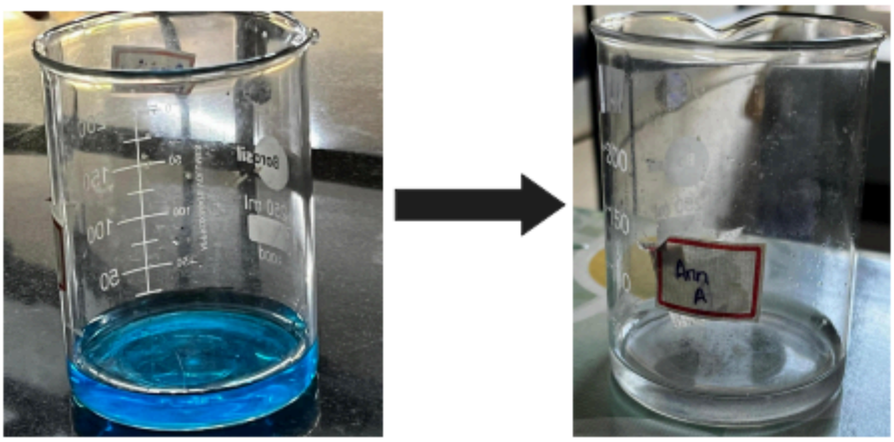

Fig 3.4 *Methylene blue solution before adsorption and after adsorption ZnO/BC composite*

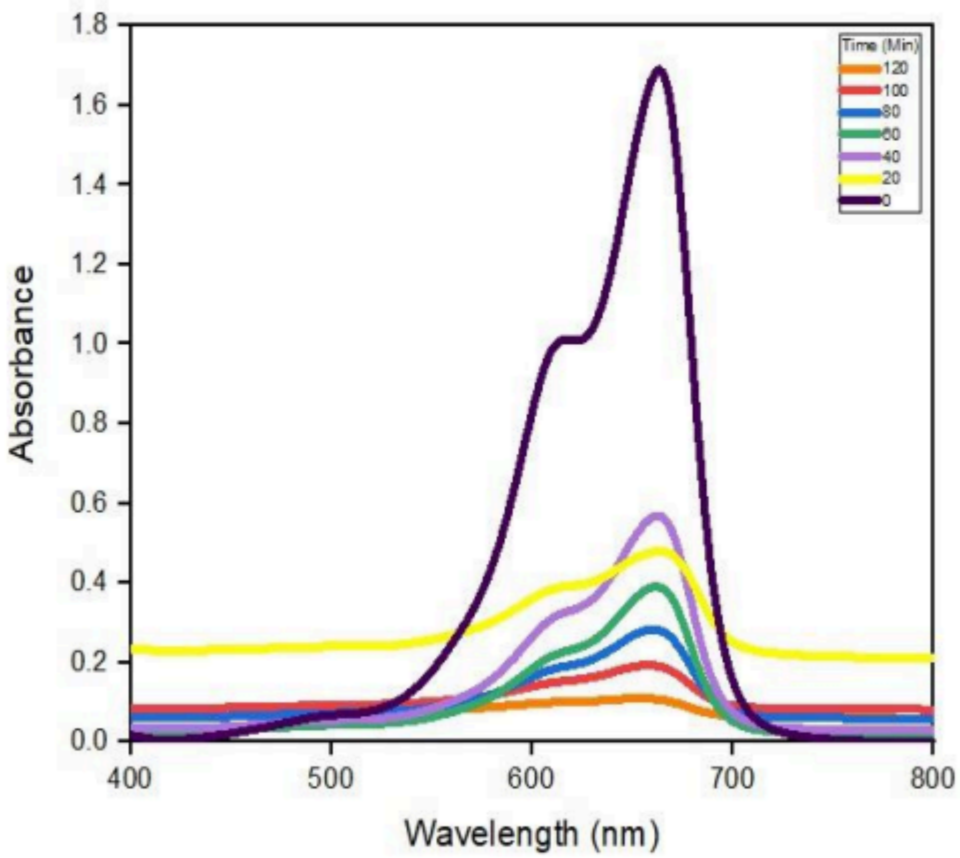

***Fig 3.5** UV-Visible spectrum of methylene blue*

- UV–Visible absorption spectrum shows the change in absorbance of the dye solution with irradiation time (0–120 min) in the presence of the ZnO/BC sample.
- A strong characteristic absorption band is observed in the visible region around ≈ 650–670 nm, which corresponds to the main chromophoric transition of the dye.

- At 0 min, the absorbance is maximum, indicating the highest dye concentration in the solution.

- As the reaction time increases from 20 to 120 minutes, the intensity of this peak gradually decreases without any noticeable shift in position.

- This shows a continuous reduction in dye concentration.

- The progressive decrease in absorbance confrms the effective photocatalytic degradation or removal of the dye by ZnO/BC over time.

- Meanwhile, the unchanged peak position suggests that the dye structure is being degraded instead of being transformed into another light-absorbing species.

- Observations and conclusions after photocatalytic degradation with ZnO/BC shows that the peaks found in Methylene blue dye absorption are clearly absent in that in the ZnO graph indicating that the ZnO/BC has adsorbed the Methylene blue dye.

Chapter 4

Conclusion

In this study, Zinc Oxide (ZnO) nanoparticles were successfully synthesized using a green synthesis method with *Aloe vera* extract, which acted as a natural reducing and stabilizing agent. The ZnO/BC composite was prepared via a sucrose-assisted pyrolysis method, enabling the direct conversion of ZnO nanoparticles into a ZnO/BC composite in a simple and eco-friendly manner. The synthesized ZnO/BC composite was characterized using UV-Visible spectroscopy, XRD, and FTIR. UV-Vis spectroscopy confirmed the formation of ZnO nanoparticles, while XRD analysis demonstrated the crystalline nature of ZnO and verified the successful formation of the ZnO/BC composite. FTIR analysis revealed the functional groups present, indicating an effective interaction between the ZnO and biochar. Finally, the photocatalytic performance of the ZnO/BC composite was evaluated through methylene blue dye degradation; the UV results confirm the strong photocatalytic activity of the synthesized ZnO/BC Composite.

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