

Green Synthesis and Characterization of TiO₂, ZnO and TiO₂/ZnO-Activated Maize Cob Composite Using *Tithonia Diversifolia* Leaf Extract as Capping Agent

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Abstract:

Green synthesis has emerged as an environmentally friendly and sustainable approach for producing metal oxide nanoparticles by eliminating the use of hazardous reducing agents and minimizing environmental pollution. This study focused on the green synthesis and characterization of titanium dioxide (TiO₂) and zinc oxide (ZnO) nanoparticles using *Tithonia diversifolia* leaf extract as a natural reducing, capping, and stabilizing agent, followed by the preparation and characterization of a TiO₂/ZnO-activated maize cob composite. The study aimed to develop an eco-friendly nanocomposite by integrating green nanotechnology with agricultural residue valorization. TiO₂ and ZnO nanoparticles were synthesized through a plant-mediated green synthesis route and subsequently immobilized onto chemically activated maize cob using the impregnation method to enhance structural stability and surface functionality. Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD) were employed to characterize the synthesized nanoparticles and the resulting composite. Visual observation confirmed successful nanoparticle formation through the appearance of white precipitates during synthesis. FTIR analysis identified characteristic O-H stretching bands at approximately 3750 cm⁻¹ for TiO₂ and 3800 cm⁻¹ for ZnO, together with C=O, C-O, Ti-O, and Zn-O functional groups, confirming phytochemical-mediated stabilization and successful nanoparticle incorporation into the activated maize cob matrix. XRD analysis confirmed the formation of highly crystalline phase-pure rutile TiO₂ and hexagonal wurtzite ZnO nanoparticles with average crystallite sizes of 1.89 nm and 15.33 nm, respectively. The TiO₂/ZnO-activated maize cob composite retained the characteristic crystalline phases of both metal oxides while exhibiting enhanced surface functionality and structural stability. The findings demonstrate that *Tithonia diversifolia* is an effective biological resource for the green synthesis of stable TiO₂ and ZnO nanoparticles and that activated maize cob provides an efficient, sustainable support matrix for developing environmentally friendly bio-nanocomposites.

Keywords: Green synthesis; *Tithonia diversifolia*; titanium dioxide; zinc oxide; maize cob; activated maize cob; nanocomposites.

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I. Introduction

Nanotechnology has become one of the fastest-growing fields in materials science because it enables the production of materials with unique physicochemical properties that differ significantly from their bulk counterparts. Among the various metal oxide nanomaterials, titanium dioxide (TiO₂) and zinc oxide (ZnO) nanoparticles have attracted considerable attention due to their high surface-area-to-volume ratio, excellent chemical stability, tunable surface chemistry, and remarkable optical and catalytic properties [1,2]. These characteristics make them suitable for numerous applications, including environmental remediation, catalysis, antimicrobial coatings, biomedical engineering, sensors, and energy conversion [3]. Their enhanced surface reactivity and abundance of active sites have stimulated extensive research into sustainable methods for synthesizing high-quality nanoparticles with controlled structural characteristics.

The performance of nanoparticles is largely influenced by their synthesis method, which determines particle size, crystallinity, morphology, surface functional groups, and degree of agglomeration [4]. Conventional synthesis techniques, including chemical precipitation, hydrothermal synthesis, solvothermal synthesis, microwave-assisted synthesis, and chemical reduction, have been widely employed for producing TiO₂ and ZnO nanoparticles [5]. Although these methods can produce nanoparticles with desirable properties, they often require toxic reducing agents, hazardous organic solvents, high energy input, and complex reaction conditions, resulting in environmentally harmful by-products and increased production costs [6]. These limitations have prompted the search for greener and more sustainable synthesis approaches that minimize environmental impacts while maintaining nanoparticle quality.

Green synthesis has emerged as an environmentally friendly alternative that aligns with the principles of green chemistry by replacing hazardous chemicals with naturally occurring biological materials [7]. Plant-mediated synthesis is particularly attractive because it is simple, economical, rapid, scalable, and environmentally benign. Unlike conventional chemical methods, green synthesis utilizes plant phytochemicals as natural reducing, capping, and stabilizing agents, thereby eliminating the need for toxic reagents and minimizing secondary pollution [8]. In addition to being safer, plant-mediated synthesis often produces nanoparticles with improved stability, controlled particle size, and reduced agglomeration because phytochemicals adsorb onto nanoparticle surfaces during formation.

The efficiency of green synthesis depends largely on the phytochemical composition of the selected plant extract. Bioactive compounds such as flavonoids, phenolic acids, tannins, alkaloids, terpenoids, proteins, and carbohydrates contain hydroxyl and carbonyl functional groups capable of reducing metal ions while simultaneously stabilizing the synthesized nanoparticles [9]. These biomolecules regulate nucleation and crystal growth, resulting in nanoparticles with enhanced crystallinity and surface functionality. Consequently, green synthesis has become an increasingly preferred approach for producing metal oxide nanoparticles intended for environmental and technological applications.

Tithonia diversifolia (Mexican sunflower) was selected in this study as the biological reducing and capping agent because it is abundant, inexpensive, rapidly renewable, and rich in phytochemicals [10]. The leaves contain flavonoids, tannins, phenolic compounds, sesquiterpene lactones, alkaloids, and saponins, all of which possess hydroxyl and carbonyl functional groups that effectively reduce titanium and zinc precursors while stabilizing the resulting nanoparticles [11]. These phytochemicals also prevent excessive particle agglomeration, producing well-dispersed nanoparticles with improved structural stability. Furthermore, the widespread availability of *T. diversifolia* in tropical regions, including Kenya, makes it a sustainable and locally accessible resource for green nanoparticle synthesis.

Although TiO₂ and ZnO nanoparticles possess excellent physicochemical properties individually, their practical application can be limited by nanoparticle agglomeration, reduced dispersion, and difficulties associated with handling free nanoparticles [12]. Incorporating nanoparticles into a solid support matrix is an effective strategy for overcoming these limitations while improving material stability and structural integrity. Agricultural biomass has emerged as an ideal support material because it is renewable, biodegradable, inexpensive, and rich in functional groups capable of interacting with metal oxide nanoparticles.

Maize cob is an abundant lignocellulosic agricultural residue composed mainly of cellulose, hemicellulose, and lignin, which provide numerous hydroxyl and carboxyl functional groups for nanoparticle attachment [13]. Chemical activation further enhances its porosity, surface area, and accessibility of active functional groups, making it an excellent support matrix for nanoparticle immobilization. The integration of TiO₂ and ZnO nanoparticles into activated maize cob produces a TiO₂/ZnO-activated maize cob composite with improved structural stability, enhanced nanoparticle dispersion, and increased surface functionality resulting from the synergistic interaction between the inorganic nanoparticles and the lignocellulosic biomass. In addition to improving material performance, the composite promotes agricultural waste valorization and supports sustainable resource utilization.

Comprehensive characterization is essential for confirming successful nanoparticle synthesis and composite formation. Fourier Transform Infrared Spectroscopy (FTIR) provides information on the functional groups responsible for nanoparticle stabilization and chemical interactions within the composite, while X-ray Diffraction (XRD) determines crystalline phases, crystallite size, and structural purity of the synthesized materials [14]. Together, these techniques provide comprehensive evidence of successful green synthesis, nanoparticle crystallinity, and effective incorporation of TiO₂ and ZnO nanoparticles into the activated maize cob matrix.

Therefore, this study focused on the green synthesis and characterization of TiO₂ and ZnO nanoparticles using *Tithonia diversifolia* leaf extract as a natural reducing and capping agent, followed by the preparation and characterization of a TiO₂/ZnO-activated maize cob composite. By integrating green synthesis with agricultural waste valorization, the study contributes to the development of sustainable bio-nanocomposite materials with enhanced structural properties while advancing environmentally friendly nanotechnology and circular bioeconomy principles.

II. Materials And Methods

Materials

Fresh *Tithonia diversifolia* leaves were collected from Murang'a University of Technology, Murang'a County, Kenya and used as the green reducing, capping, and stabilizing agent during nanoparticle synthesis. Dry maize cobs were obtained from local farms within Murang'a County and served as the biomass support material for nanocomposite preparation. Titanium (IV) isopropoxide ($\geq 98\%$ purity) and zinc nitrate hexahydrate ($\geq 99\%$ purity), sourced from Lab Chemicals Limited, Nairobi, were used as precursor salts for TiO₂ and ZnO nanoparticle synthesis, respectively. Phosphoric acid (H₃PO₄, 5% v/v), obtained from Sigma-Aldrich distributors in Kenya,

was used for chemical activation of maize cobs, while absolute ethanol and deionized water were used during nanoparticle synthesis, composite fabrication, and washing procedures [15]. All reagents used in the study were of analytical grade and were used without further purification.

Preparation of *Tithonia diversifolia* Leaf Extract

200g of freshly collected *Tithonia diversifolia* leaves were thoroughly washed with tap water, rinsed with deionized water to remove dust particles, and left to air dry for 72 hours at room temperature. 10g of the dried leaves were grounded and heated at 60°C in 100 mL of deionized water for 30 minutes to extract bioactive compounds [16]. The mixture was allowed to cool to room temperature and subsequently filtered using Whatman No. 1 filter paper to remove solid residues. A brown plant extract was obtained and used immediately as a reducing and stabilizing agent in the green synthesis of nanoparticles.

Green Synthesis of TiO_2 and ZnO Nanoparticles

TiO_2 nanoparticles were synthesized using *Tithonia diversifolia* leaves extract. 5ml of titanium (IV) isopropoxide was mixed with 10ml absolute ethanol under stirring speed of 500rpm. Separately, 20 ml of leaf extract was mixed with 60ml of deionized water [16]. The later mixture was added dropwise to the titanium (IV) isopropoxide solution prepared initially under stirring speed of 500rpm at room temperature for 2 hours. The resulting mixture was aged for 24 hours at room temperature, washed with ethanol and deionized water, filtered, dried at 80°C for 8 hours, and finally calcined at 500°C for 4 hours to produce rutile-phase TiO_2 nanoparticles.

Likewise, *Tithonia diversifolia* leaf extract, as a green reducing and stabilizing agent, was used to synthesize ZnO nanoparticles. A 0.1M $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution was prepared separately by dissolving 2.97g of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in a 100ml beaker using deionized water and then transferred to a 100ml volumetric flask. The solution was made up to the mark with deionized water. The entire solution was subsequently transferred to a 250ml beaker, and 20 ml of leaf extract was introduced dropwise under constant stirring at 500rpm at 60°C for 2 hours, resulting in the formation of a white precipitate. The precipitate was filtered, washed with ethanol and deionized water, and dried at 80 °C for 12 hours. Calcination was then carried out at 450 °C for 3 hours to obtain crystalline ZnO nanoparticles [17].

Preparation of Activated Maize Cob Bio-Adsorbent

Maize cobs were thoroughly washed with tap water followed by deionized water to remove adhering soil particles and other contaminants. The cleaned cobs were chopped into smaller pieces and oven-dried at 60°C for 24 hours to eliminate moisture while preserving their lignocellulosic structure. The dried material was ground and sieved to obtain particles ranging from 0.5 to 2.0 mm. Chemical activation was performed by soaking 50 g of the sieved maize cob powder in 500 mL of 5% (v/v) phosphoric acid contained in a 1000 mL beaker. The suspension was stirred at 800 rpm for four hours and subsequently aged for twelve hours at room temperature to enhance pore development and introduce oxygen-containing functional groups onto the biomass surface [18]. The activated material was washed repeatedly with deionized water until neutral pH was achieved, filtered, oven-dried at 60°C for eight hours to constant weight, and stored in airtight containers until composite preparation.

Preparation of TiO_2/ZnO -Maize Cob Nanocomposites

Nanoparticle-maize cob composites were prepared using the impregnation method to ensure uniform distribution of nanoparticles on the activated biomass surface. Initially, 0.3 g of synthesized TiO_2 and ZnO nanoparticles were separately dispersed in 9 mL of absolute ethanol using ultrasonication for 30 minutes to break particle agglomerates and obtain stable nanoparticle suspensions. Separately, 0.6 g of activated maize cob was dispersed in 30 mL of absolute ethanol and stirred at 150 rpm for 20 minutes to achieve homogeneous suspension of the biomass particles. The nanoparticle suspensions were gradually introduced into the maize cob suspension under continuous stirring at 150 rpm for one hour at room temperature to facilitate uniform coating and strong interaction between nanoparticles and the lignocellulosic support [19]. The resulting nanocomposites were filtered and washed with deionized water to remove loosely attached nanoparticles before being oven-dried at 60°C for one hour. The dried composites were gently ground using a mortar and pestle to obtain uniform particle size and stored in airtight containers for characterization and adsorption experiments.

Characterization of Synthesized Nanoparticles and Nanocomposites

The synthesized nanoparticles and prepared nanocomposites were characterized to confirm successful synthesis, evaluate structural properties, and determine surface chemistry relevant to adsorption applications.

Fourier Transform Infrared Spectroscopy (FTIR). Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify functional groups involved in nanoparticle formation, stabilization, and composite fabrication. Spectra were recorded over the range of 4000-400 cm^{-1} . Characteristic absorption bands

corresponding to hydroxyl (-OH), carbonyl (C=O), C-O, Ti-O, and Zn-O vibrations were used to confirm phytochemical-mediated nanoparticle stabilization and successful incorporation of TiO₂ and ZnO nanoparticles into the activated maize cob matrix. Changes in peak intensity and slight shifts in absorption bands were interpreted as evidence of chemical interactions between the nanoparticles and biomass support [14].

X-ray Diffraction (XRD). X-ray Diffraction (XRD) analysis was performed to determine crystal structure, phase composition, crystallinity, and structural stability of the synthesized nanoparticles and nanocomposites. Diffraction patterns were recorded over 2 θ scanning range and compared with Joint Committee on Powder Diffraction Standards (JCPDS) reference data. The Debye–Scherrer equation was used to estimate crystallite size from the most intense diffraction peaks. Preservation of characteristic rutile TiO₂ and hexagonal wurtzite ZnO diffraction peaks after immobilization confirmed successful nanocomposite formation without alteration of nanoparticle crystal structures [12].

III. Results And Discussion

Visual Observation of Nanoparticle Formation

The formation of TiO₂ and ZnO nanoparticles was initially confirmed through observable colour changes and precipitate formation during synthesis. Upon gradual addition of *Tithonia diversifolia* leaf extract into the metal precursor solutions, the reaction mixtures progressively became turbid, followed by the formation of white precipitates. This observation indicates successful reduction of titanium and zinc ions and the nucleation of metal oxide nanoparticles mediated by phytochemicals present in the leaf extract. Similar observations have been reported in plant-mediated nanoparticle synthesis, where naturally occurring flavonoids, tannins, alkaloids, and terpenoids function simultaneously as reducing, capping and stabilizing agents. The successful formation of nanoparticles without introducing synthetic reducing or stabilizing chemicals demonstrates the effectiveness of *T. diversifolia* leaves extract as a green synthesis medium [16]. Besides minimizing hazardous chemical usage, plant phytochemicals regulate nanoparticle growth by preventing excessive aggregation, thereby promoting the formation of smaller particles with improved surface properties. Such characteristics are particularly desirable for adsorption applications because smaller nanoparticles provide greater surface-area-to-volume ratios and a higher density of reactive adsorption sites.

FTIR Characterization of TiO₂, ZnO and Nanocomposites

FTIR was used to determine the functional groups of the nanoparticles involved in the synthesis process, and to verify the integration of TiO₂ and ZnO nanoparticles with activated maize cob. The spectra had characteristic absorption bands of hydroxyl (-OH), carbonyl (C=O), carbon-oxygen (C-O), and metal-oxygen (Ti-O and Zn-O) functional groups, indicating the successful green synthesis and the formation of composites. The broad absorption bands in the range 3200 to 3700 cm⁻¹ were attributed to the O-H stretching vibrations from hydroxyl groups of cellulose, hemicellulose, lignin, absorbed moisture and phytochemicals of *Tithonia diversifolia* as seen in Figure 1. Such hydroxyl groups contributed greatly to the reduction of metal ions and stabilization of the synthesized nanoparticles via hydrogen bonding. Peaks in the 1600 cm⁻¹ region were related to the C=O stretching vibrations of flavonoids and phenolic compounds, which suggests that these plant phytochemicals were still on the surface of the nanoparticles as a natural capping agent, thus preventing aggregation of the nanoparticles [14]. The increased exposure of the oxygen containing functional groups that lead to hydrogen bonding and electrostatic interactions, which lead to enhanced C-O stretching bands near 1100 cm⁻¹, was observed after chemical activation. The presence of Ti-O and Zn-O bonds was verified through the presence of distinct absorption bands around 400 cm⁻¹, which indicated successful synthesis of TiO₂ and ZnO nanoparticles and immobilization onto the activated maize cob matrix [10,14]. The strong chemical interactions between the nanoparticles and the lignocellulosic support were suggested by the minor shifts in hydroxyl and carbonyl absorption bands following the formation of the composite. Such interactions led to an increase in the surface functionality, which indicated the formation of chemically integrated TiO₂/ZnO- activated maize cob nanocomposites with high adsorption capacity for phenolic compound removal from wastewater.

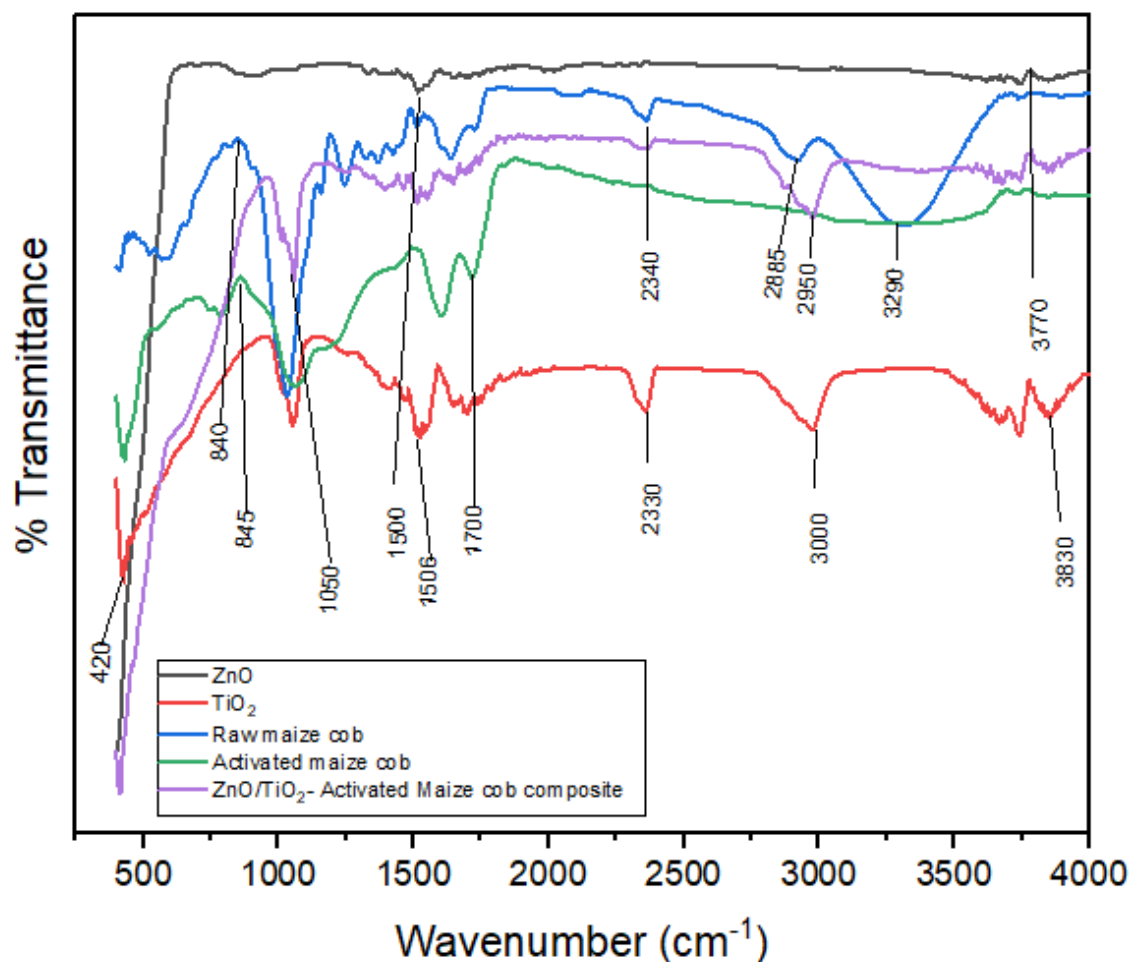


Figure 1: FTIR Spectra of TiO_2 , ZnO , and Maize Cob Nanocomposite

XRD Characterization of TiO_2 Nanoparticles

The XRD analysis was performed to determine the crystal structure, crystallinity, and phase purity of the synthesized TiO_2 nanoparticles. The diffraction pattern showed distinct sharp peaks corresponding to crystalline rutile TiO_2 , confirming successful formation of the desired metal oxide phase after calcination. The diffraction peaks observed corresponded to the crystallographic planes, as shown in Figure 2. These diffraction peaks align well with standard JCPDS reference data for rutile titanium dioxide [7,10,11].

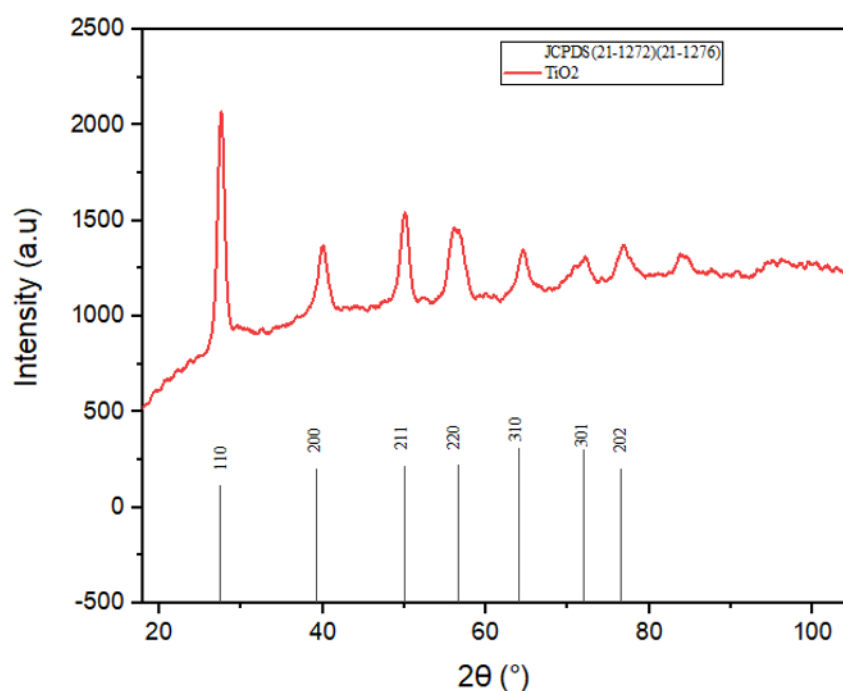


Figure 2: TiO₂ XRD Pattern

The Debye–Scherrer equation (Equation 1.1) was used to calculate crystallite size from the observed peaks.

$$D = \frac{K\lambda}{\beta \cos \theta} \dots\dots\dots \text{Equation 1.1}$$

Where D is crystallite size (nm), K is Scherrer constant (0.9), λ is X-ray wavelength for Cu K α radiation (0.15406 nm), β is full width at half maximum (FWHM) in radians, and θ = Bragg diffraction angle. The calculated values are presented in Table 1.

Table 1: Summary of Crystallite Sizes and FWHM of Synthesized TiO₂

2 THETA	PLANE	Estimated FWHM	Crystallite size (nm)
27.6	110	2.047	3.996
40.06	111	5.941	1.423
50.18	200	8.386	1.046
56.4	220	12.71	0.7092
63	204	6.297	1.480
77	301	3.779	2.686
			Average = 1.89

The results demonstrate that the synthesized TiO₂ nanoparticles possess crystallite sizes within the nanometre range, with average size being 1.89nm. High diffraction peak intensity indicates excellent crystallinity, while the small crystallite sizes suggest increased specific surface area. Such nanoscale dimensions enhance adsorption because they provide more active adsorption sites for contaminant interaction [6,13]. Preservation of the rutile crystal phase further indicates that calcination improved crystallinity without destroying nanoparticle integrity, thereby producing structurally stable nanoparticles suitable for composite fabrication.

XRD Characterization of ZnO Nanoparticles

The XRD pattern of ZnO nanoparticles confirmed successful synthesis of crystalline zinc oxide with a characteristic hexagonal wurtzite crystal structure. The diffraction peaks observed correspond to the crystallographic planes as shown in Figure 3. The absence of secondary phases or impurity peaks demonstrates that green synthesis produced highly pure ZnO nanoparticles [10, 13].

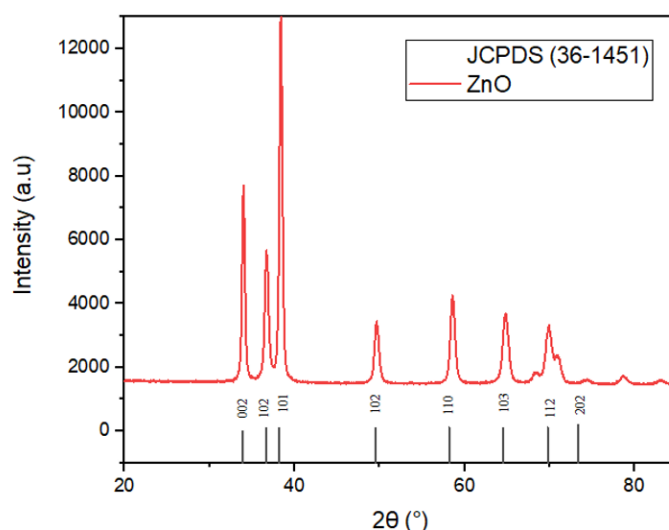


Figure 1: ZnO XRD Pattern

Crystallite sizes calculated using the Equation 1.1 are presented in Table 2.

Table 2: Summary of Crystallite Sizes and FWHM of Synthesized ZnO

2 THETA	PLANE	Estimated FWHM	Crystallite size (nm)
34	002	0.4252	19.54
36	101	0.5928	14.09
38	102	0.4515	18.61
49	102	0.6210	14.06
58	110	0.6528	13.91
64	103	0.7954	11.78
70	112	1.6451	5.895
			Average = 15.33nm

The ZnO nanoparticles similarly exhibited excellent crystallinity, as evidenced by sharp diffraction peaks characteristic of the wurtzite structure. The calculated average crystallite size was 15.33nm confirming successful nanoscale synthesis. Nanoparticles within this size range are advantageous for environmental remediation because their large surface area facilitates adsorption and photocatalytic degradation of organic contaminants [12].

XRD Characterization of TiO_2/ZnO -Maize Cob NanoComposite

XRD analysis of the TiO_2/ZnO -maize cob nanocomposite was performed to evaluate its structural properties and confirm the presence of nanoparticles within the biomass matrix. The diffraction pattern of the composite exhibited a combination of sharp and broad peaks, reflecting both crystalline and amorphous components, as shown in Figure 4. The characteristic diffraction peaks of TiO_2 (rutile phase) and ZnO (wurtzite structure) were clearly retained in the composite, indicating that the nanoparticles maintained their crystalline structure after incorporation into the maize cob matrix [20]. The presence of these peaks confirms that the synthesis and loading processes did not alter the fundamental crystal phases of the nanoparticles.

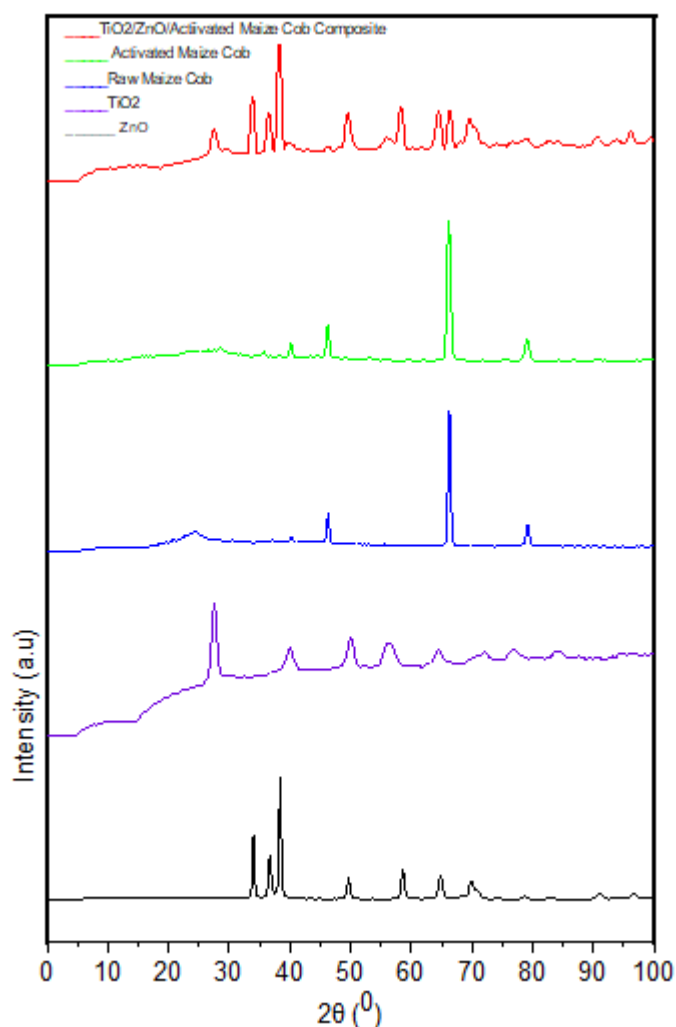


Figure 4: TiO_2/ZnO -Activated Maize Cob Composite XRD Pattern

A successful formation of a hybrid material is proven by the coexistence of crystalline nanoparticle peaks and amorphous biomass signals. Importantly, there were no significant peak shifts or disappearance of characteristic peaks, indicating that structural integrity of nanoparticles was maintained as seen in Figure 4. This mixture of crystallized & amorphous phases is beneficial for adsorption. The active adsorption sites are provided by the crystalline nanoparticles and amorphous maize cob provides a very high surface area and porosity [4]. The XRD results, in view of this, therefore confirm that the composite structure is stable and suitable for the adsorption applications [21].

IV. Conclusion

The present study successfully demonstrated the feasibility of using *Tithonia diversifolia* leaf extract as a natural reducing, capping, and stabilizing agent for the green synthesis of TiO_2 and ZnO nanoparticles. The use of plant-derived phytochemicals eliminated the need for hazardous synthetic chemicals, providing an environmentally friendly and sustainable approach to nanoparticle production. Visual observations confirmed successful nanoparticle formation through the development of characteristic white precipitates during synthesis, while FTIR analysis verified the presence of hydroxyl, carbonyl, and metal-oxygen functional groups responsible for nanoparticle stabilization and surface functionality. XRD characterization confirmed the successful formation of highly crystalline rutile TiO_2 and hexagonal wurtzite ZnO nanoparticles with nanoscale crystallite sizes, demonstrating that the green synthesis approach produced structurally stable materials suitable for environmental applications. The study further achieved the successful preparation of TiO_2/ZnO -maize cob nanocomposites by immobilizing the synthesized nanoparticles onto chemically activated maize cob using the impregnation method. Chemical activation significantly improved the porosity and surface functionality of the maize cob, while composite characterization confirmed effective nanoparticle incorporation without altering the crystalline structures of TiO_2 and ZnO . The coexistence of crystalline nanoparticles and the porous lignocellulosic support produced a stable hybrid material with enhanced surface properties that are favourable for adsorption. Overall,

the study demonstrates that integrating green nanotechnology with agricultural waste valorization provides a sustainable strategy for developing low-cost adsorbents. The synthesized TiO₂/ZnO-maize cob nanocomposites possess physicochemical characteristics as a promising alternative to conventional adsorbents. The findings contribute to the advancement of environmentally sustainable nanomaterials and support future application of green-synthesized nanocomposites.

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Conflict of Interest Statement

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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