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Original Research Article

Biosynthesis and Comprehensive Structural Characterization of TiO_2-Loaded Bentonite Nanocomposites using Orange Peel Extract

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ABSTRACT
Increasing contamination of water resources by industrial effluents has necessitated the development of low-cost, eco-friendly, and efficient materials for wastewater treatment. Among such materials, titanium dioxide (TiO_2) is recognized for its excellent photocatalytic activity, while bentonite clay provides high surface area and strong adsorption capacity. This study reports a green biosynthesis route for TiO_2-loaded bentonite nanocomposites (BTNCs) using orange peel extract as a natural reducing and stabilizing agent to integrate the photocatalytic and adsorptive properties of TiO_2 and bentonite respectively. Characterization of the synthesized nanocomposites was carried out using a suite of analytical techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), and Raman spectroscopy. Characterization via XRD, SEM, and Raman spectroscopy confirmed the successful incorporation of anatase TiO_2 nanoparticles (~17.5 nm) uniformly dispersed within the bentonite matrix, with evidence of Ti–O–Si bond formation indicating strong interaction between TiO_2 and bentonite. The eco-friendly synthesis method discourages the use of toxic reagents and offers multifunctional nanomaterials with promising applications in photocatalytic wastewater treatment.

INTRODUCTION

The advancement of nanotechnology has ushered in the development of multifunctional nanocomposites with enhanced physicochemical properties suitable for a wide range of environmental and industrial applications (Alshangiti *et al*., 2023). Among these materials, metal oxide-based nanocomposites, particularly those incorporating titanium dioxide (TiO_2), have attracted considerable attention due to their superior photocatalytic capability, antibacterial potentials, and adsorptive properties (Kumar *et al.,* 2023). However, conventional methods of synthesizing TiO_2 nanoparticles often involve toxic reagents and high-energy processes, which are not environmentally unfriendly (Patra and Baek, 2020).

To address these challenges, green synthesis approaches have emerged as sustainable alternatives, employing biological resources such as plant extracts, microorganisms, and natural polymers as reducing and capping agents (Ramesh *et al.,* 2023). Orange peel extract, rich in polyphenols, flavonoids, and citric acid, has demonstrated promising potential as a bio-reductant in the eco-friendly synthesis of metal oxide nanoparticles (Ali *et al.,* 2022). These phytochemicals facilitate the formation of stable nanoparticles under mild reaction conditions, thus reducing environmental impact while enhancing material functionality (Lawal *et al*., 2025).

Bentonite, naturally occurring aluminosilicate clay, is well-known for its high cation exchange capacity, layered structure, and large surface area(Maged,*et al*., 2020). These features make it an excellent support matrix for the dispersion of metal oxide nanoparticles, allowing synergistic interaction between components and preventing nanoparticles from agglomeration (Saeidi *et al.,*2021). The integration of biosynthesized TiO_2 with bentonite clay creates a hybrid nanocomposite—Bentonite-Based Titanium Oxide Nanocomposites (BTNCs)—that harnesses the photocatalytic activity of TiO_2 and the adsorptive capability of bentonite, making it highly effective for environmental applications, such as the removal of heavy metals and organic pollutants from wastewater (Ganguly *et al.,* 2023).

Furthermore, the structural and surface characteristics of BTNCs can be significantly enhanced through biosynthetic modification, which improves their performance in catalytic, antibacterial, and adsorption processes (Mustapha*et al*., 2020). The utilization of waste materials like orange peel provides a value-added route for agro-waste recycling where low-value agricultural residues are transformed into high-value products such as nanomaterials. Thus, through waste valorization, orange peels shift from being an environmental burden to becoming a sustainable resource, thereby aligning with circular economy goals of minimizing waste and maximizing resource efficiency (Bharath *et al.,* 2022).

Efforts to develop greener TiO_2–bentonite composites have gained increasing attention, as researchers aim to combine the photocatalytic potential of TiO_2 with the adsorption capacity of bentonite while minimizing environmental impacts. For instance, Cao *et al*. (2020) reported the green synthesis of a reusable multifunctional γ-Fe_2O_3/bentonite composite modified by doped TiO_2 hollow spheres for the removal of bisphenol A. Their study demonstrated that TiO_2–bentonite architectures can be fabricated under environmentally benign conditions, with the additional advantage of magnetic separability for easy recovery and reuse. Similarly, Krishnan *et al*. (2017) synthesized an Ag/TiO_2/bentonite nanocomposite via a green route and evaluated its antibacterial and cytotoxic properties. This work served as one of the early proofs of concept for multi-component TiO_2 systems supported on bentonite using green methods.

Bio-functionalization has also been explored in TiO_2–bentonite systems. Khansili *et al*. (2022) developed a curcumin-functionalized TiO_2-modified bentonite composite for aflatoxin B1 determination, highlighting the potential of natural biomolecules to enhance composite performance. Ulhaq *et al*. (2021) investigated TiO_2 supported on CTAB-modified bentonite for simultaneous photo-oxidation and adsorption of hydrocarbons in refinery wastewater. Although not entirely biogenic, the mild process conditions adopted in their study indicate compatibility with biosynthesized TiO_2, thus aligning with green chemistry principles.

Conventional synthetic routes remain a reference point for green adaptations. Mustapha *et al*. (2020) further reinforced this direction in their review of TiO_2/ZnO nanoparticles immobilized on clays for water treatment, noting that clays serve as excellent supports by preventing nanoparticles agglomeration and exposing additional active sites—factors that are critical when integrating green-synthesized TiO_2. Despite the increasing studies in green nanotechnology, there is still limited research on the biosynthesis of BTNCs using fruit-based extracts, especially with comprehensive physicochemical characterization and environmental relevance (Antunes Filho *et al*., 2023).

This study aims to biosynthesize and characterize BTNCs using orange peel extract as a green reductant and stabilizer. The synthesized nanocomposites are systematically characterized using XRD, SEM, and Raman spectroscopy to understand their structural and morphological properties. The outcome is expected to contribute to the development of sustainable nanomaterials with promising applications in wastewater remediation and other environmental fields.

MATERIALS AND METHODS

Materials

Natural bentonite clay was sourced from a local mining site in Lokoja, Nigeria. Fresh orange peels were sourced locally from the sellers in Lokoja Central Market, while titanium (iv) isopropoxide (TTIP) of 97% purity, purchased from Sigma-Aldrich, served as the titanium source for TiO_2 nanoparticles synthesis due to its high reactivity (Verma *et al.,* 2022).

Methods

Samples Pre-treatment

Before use, the bentonite clay was purified thoroughly with water to remove impurities such as organic matter and quartz. The raw bentonite clay was sun-dried, ground using a mortar and pestle, and sieved to obtain uniform particle sizes. The sieved bentonite clay was washed repeatedly with distilled water to remove soluble salts and dried at 105 °C for 6 hours. It was then calcined in a muffle furnace at 500 °C for 3 hours to increase surface activity and remove organic impurities (Sarkar *et al.,* 2022).

Also, the fresh orange peels were thoroughly washed, dried in the shade, and ground into powder. Subsequently, the orange peel extraction was carried out using heat-assisted extraction method. 10 g of the powdered peels was heated at 70°C with gentle stirring in 100 cm^3 of distilled water for 45 minutes. The mixture was filtered and the extract stored at 4 °C for future use.

Biosynthesis of BTNCs

During the biosynthesis, 10 g of bentonite clay was dispersed in 100 cm^3 of distilled water, stirred and treated with 1 M NaOH for 2 hours at room temperature to enhance surface activation and remove impurities. The mixture was filtered, washed, and dried at 80 °C.

Subsequently, 10 g of the activated bentonite clay was added to the TTIP precursor in 4:1. The mixture was stirred constantly at 450 rpm in an ice bath (0–5 °C), followed by drop wise addition of 20 cm^3 orange peel extract. The pH of the current mixture was controlled at pH using 0.5M NaOH. Thereafter, the mixture was stirred at 450 rpm for 2 hours until a whitish deposit was formed which indicates TiO_2 formation on bentonite surface. The formed nanocomposite was centrifuged, washed with distilled water and ethanol, dried at 80 °C, and then calcined in a static air atmosphere between 120-450°C (2°C min^{-1}) for 2 hours to obtained crystalline nanocomposites. After calcination, the sample was allowed to cool naturally in the furnace and subsequently stored in a desiccator under airtight amber containers to prevent rehydration prior to characterization.

Characterization of the BTNCs

In this study, Raman spectroscopy, X-ray diffraction (XRD), and Scanning Electron Microscopy (SEM)/Energy Dispersive X-ray Spectroscopy (EDS) were used to characterize the biosynthesized BTNCs sample. For the phase structures of the sample by XRD, 0.5 g of each powdered sample was homogenised through a 10 µm mesh sieve, placed on a sample holder, and scanned with filtered Cu-Kα radiation over a 2θ range of 5°–80° using a Shimadzu XRD-6000. For the SEM/EDS, the sample was gold-coated to prevent charging particle morphology and its micrograph was obtained at 10 KX magnification using a ZEISS Crossbeam 550 while EDS analysis complemented SEM confirmed the elemental composition of the sample surface. Meanwhile, Raman spectroscopy was subsequently performed using a ProRaman-L-785-B1S system, where the sample was placed on a slide positioned 7 mm from the lens, and scanned to obtain the characteristic vibrational peaks.

RESULTS AND DISCUSSION

Raman Spectroscopy

The Raman spectrum of the biosynthesized BTNCs was recorded in the range of 0 – 3000 cm^{-1}. The spectrum revealed distinct vibrational bands corresponding to both bentonite (montmorillonite) and the anatase phase of TiO_2. The prominent observed peaks are shown in Figure 1.

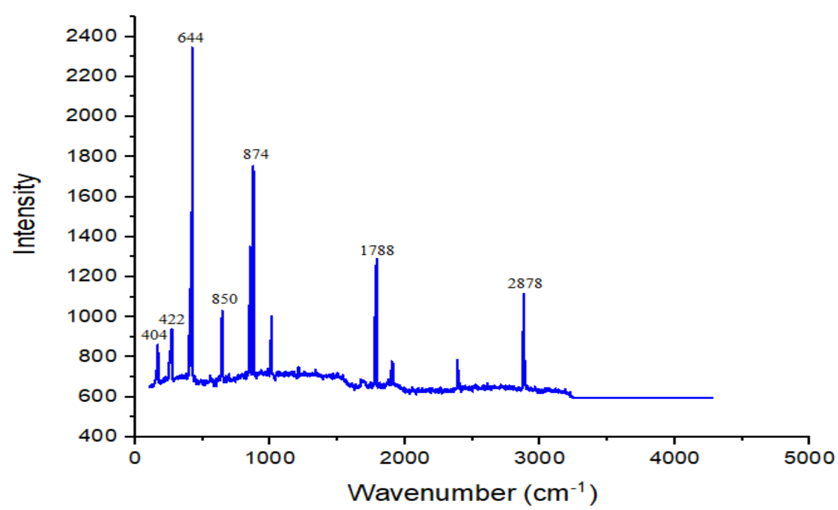

Figure 1: Raman Spectrum of the biosynthesized BTNCs

The most intense peak at 644 cm^{-1} corresponds to the Eg symemetric stretching of anatase TiO_2 also known as fingerprint of anatase which signifies highly crystalline anatase phase (Luma, 2025).

The peaks clearly observed at 404 $cm^{-1}$and 422 cm^{-1}confirm anatase TiO_2 at slight shift of Eg or B1g modes caused by particle size reduction or stress in the lattice and Ti–O–Si bonding resulting from strong interaction between TiO_2 and bentonite respectively (Taudul, Tielens, and Calatayud, 2023).

Also, the peaks observed at 850 cm^{-1} and 874 cm^{-1} are attributed to the Si–O–Si bending vibrations and Al–O–Si linkages of the bentonite (montmorillonite) structure. These characteristics confirm the existence of the bentonite clay matrix, and the difference in their intensity depends on the amount and extent of interaction of TiO_2 (Kgabi and Ambushe, 2023).

Furthermore, the peak at 1788 cm^{-1} may be attributed to organic carbonyl (C=O) stretching in flavonoids or phenolics as possible phytochemicals responsible for the biosynthesis while the peak at 2878 cm^{-1} is a familiar region for C–H stretching of $-CH_3$ and $-CH_2-$ groups confirming the presence of hydrocarbon moieties of the phytochemicals (Hayat *et al.*, 2020).

The presence of both bentonite and TiO_2 peaks in the nanocomposite spectrum suggests a physical interaction or weak chemical bonding via hydrogen bonding or van der Waals forces rather than complete structural alteration.

X-ray Diffraction (XRD) Analysis of BTNCs

The crystalline structure and phase composition of the biosynthesized BTNCs were analyzed using X-ray diffraction (XRD). The XRD pattern of the BTNCs is shown in Figure 2, and the key peaks showing the presence of bentonite in the diffractogram were observed at 2θ = 19.6°, 27.3°, 34.6°, 35.20° indexed as (110), (021), (130), and(131) which correspond to the characteristic indication of silicate layer structure, higher-order montmorillonite and overlap of the clay with feldspar while the absence peak at 2θ between 6 and 7.2 signifies the lack of swelling layer structure of the bentonite clay(Maged et al., 2020).

The absence of a strong and sharp diffraction peak at 2θ ≈ 25.3° indexed as (101) plane of anatase TiO_2 indicates formation of mixed-phase TiO2 nanoparticles in the biosynthesized nanocomposite. Though peaks at 2θ ≈ 39.4°, 47.5°, 62.9, 69.3°, 70.2°, and 73.0° attributed to (004), (200), (204), (116), (220), and (215)planes, suggest the presence ofa significant anatase phase as compared with JCPDS No. 21-1276. Also, the peaks at 2θ ≈ 54.5° and 56.6° indexed as (211) and (220) signify the existence of rutile phase TiO_2 nanoparticles while a peak at 2θ ≈ 48.5° indexed as (103) indicates the presence of brookite phase TiO_2 nanoparticles.

The formation of mixed-phase TiO_2 nanoparticles, as shown from the diffractogram, may be due to synergistic charge separation effects (Liu *et al.,* 2021).

Furthermore, the relatively low-intensity diffraction peaks at 2θ = 19.6°, 27.3°, 34.6°, 35.20° ,which correspond to montmorillonite as the main component of bentonite clay not only confirm the presence of the clay matrix in the nanocomposite but also suggest partial intercalation of TiO_2 nanoparticles into the bentonite layers.

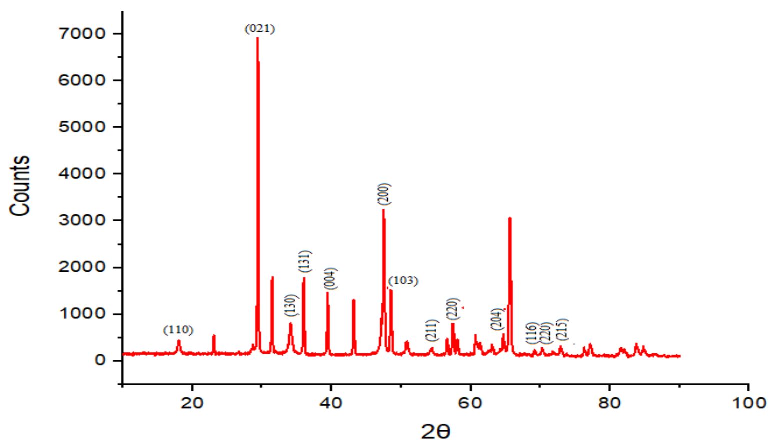

Figure 2: XRD pattern of the biosynthesized BTNCs

The average crystallite size of the TiO_2 nanoparticles in the composite was estimated using the Debye–Scherrer equation:

$$D = \frac{K\lambda}{\beta\cos\theta}$$

Where: D is the crystallite size, K is the shape factor (typically 0.9), λ is the X-ray wavelength (0.154 nm for Cu Kα), β is the full width at half maximum (FWHM) in radians, and θ is the Bragg diffraction angle.

Based on the anatase peaks (004), (200), (204), (116), (220), and (215) at 2θ ≈ 39.4°, 47.5°, 62.9, 69.3°, 70.2°, and 73.0° the calculated average crystallite size was found to be approximately 19.4 nm, indicating nanocrystalline nature. This is relatively in good agreement with previous reports on green-synthesized TiO_2 nanoparticles (Rahman *et al.,* 2023) but higher compared to 14.91 nm, 12.01 nm, and 9.15 nm crystallite sizes obtained by Saheed*et al* (2023) for the biosynthesized TiO_2 at pH 8, 10, and 12, respectively. The nanocrystalline nature of TiO_2 with a crystallite size of ~19.4 nm suggests promising surface properties for environmental remediation applications such as heavy metal removal and photocatalysis.

Meanwhile, the use of orange peel extract as a green reducing and stabilizing agent introduced foreign peaks as unindexed peaks, indicating the formation of unwanted secondary phases during the biosynthesis process.

Furthermore, narrow peaks observed in the XRD pattern of BTNCs suggest reduced crystallinity due to the interaction of TiO_2 with the bentonite matrix and the organic components from the plant extract. Such narrowing is useful for adsorption and photocatalytic applications as it often correlates with higher surface area and more active sites (Chakraborty *et al.,* 2022). The XRD analysis confirms the successful formation of a bentonite–TiO_2 nanocomposite with predominantly anatase phase and minor rutile TiO_2. The presence of bentonite clay was evident from the presence of higher-order montmorillonite.

Scanning Electron Microscopy (SEM)/Energy Dispersive X-ray Spectroscopy (EDS)

SEM micrograph (Figure 3) of the BTNCs surface revealed an irregular, porous, and layered morphology typical of bentonite embedded with uniformly dispersed spherical TiO_2 nanoparticles. This is similar the findings reported by Krishnan and Mahalingam (2017) for Ag/TiO2/bentonite nanocomposite

The surface of BTNCs appeared rough, with visible grain boundaries and textural changes induced by TiO_2 deposition. The TiO_2 nanoparticles filled the pores and adhered to the clay layers, enhancing surface heterogeneity and thereby potentially increasing the number of active adsorption sites (Zhou *et al.,* 2023). The presence of Ti, as shown in micrograph suggests successful biosynthesis (Singh *et al*., 2022).

The EDS spectrum (Figure 4) verified the elemental composition of the BTNCs, showing intense peaks of Si, Al, and O corresponding to the bentonite structure, along with clear signals for Ti arising from the deposited TiO_2 nanoparticles. In addition, weaker peaks of Fe, K, Ni, and Ta were detected, which are naturally occurring constituents of bentonite clay (Cuevas *et al*., 2022). These findings are consistent with the EDS profiles previously reported by Aljeboree and Alkaim (2024).

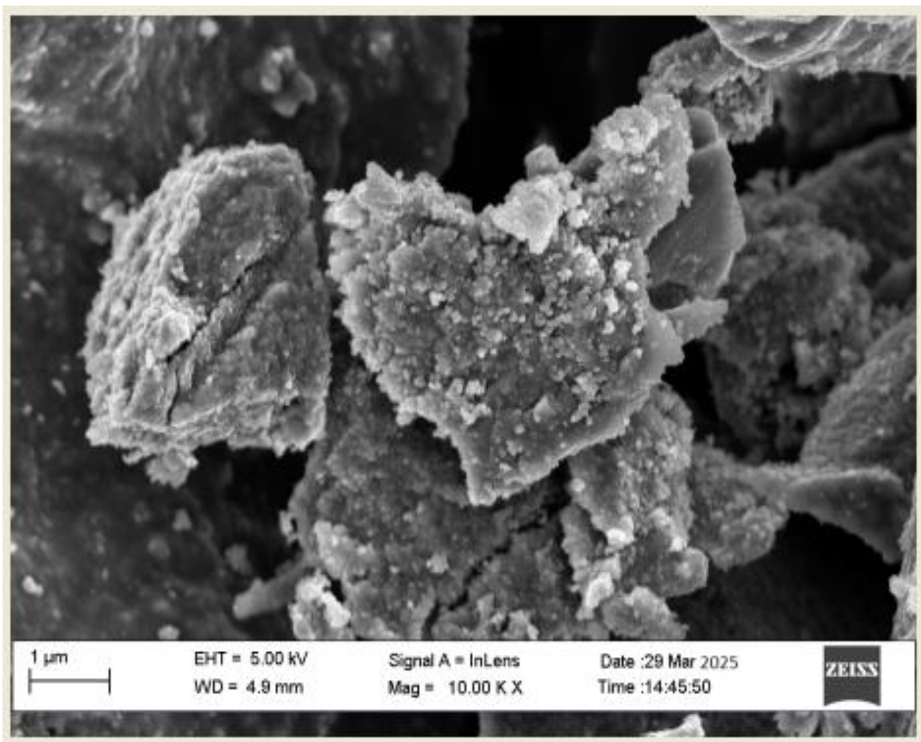

Figure 3: SEM micrograph of the biosynthesized BTNCs

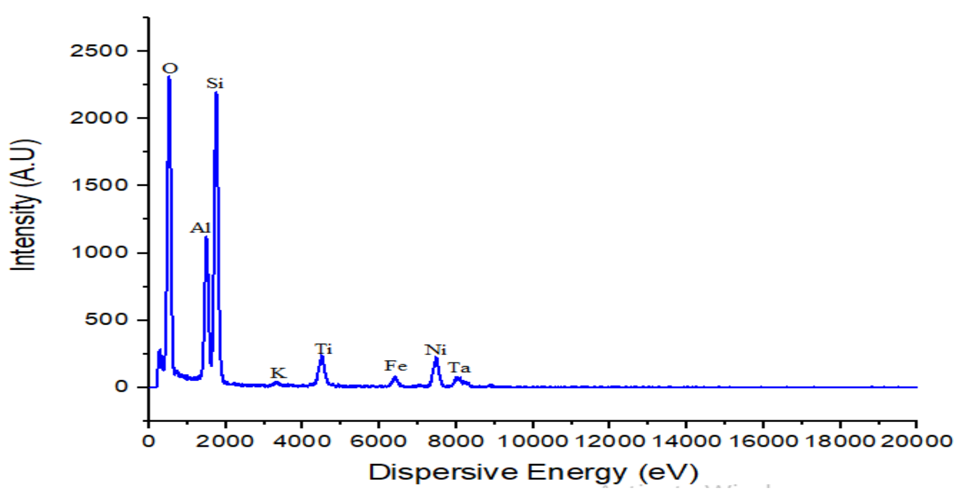

Figure 4: EDX spectrum of the biosynthesized BTNCs

CONCLUSION

This study successfully achieved the green synthesis of bentonite-based titanium dioxide nanocomposites (BTNCs) using orange peel extract, as confirmed by Raman spectroscopy, which revealed distinct vibrational bands corresponding to both bentonite (montmorillonite) and the anatase phase of TiO_2. Structural and morphological analyses further confirmed the incorporation of anatase-phase TiO_2 into the bentonite matrix, with uniformly dispersed titanium nanoparticles exhibiting low agglomeration and strong Ti–O–Si interactions between TiO_2 and bentonite. These characteristics highlight BTNCs as promising multifunctional materials for photocatalytic degradation of dyes and the adsorption of heavy metals, owing to the synergistic integration of TiO_2's photocatalytic properties with bentonite's adsorption capacity. Compared to conventional materials obtained through traditional synthesis, the eco-friendly BTNCs present a sustainable alternative for wastewater treatment and related applications. This work advances the development of cost-effective and environmentally sustainable nanomaterials for both environmental and industrial use, while also identifying the need for further research to test the composites in real wastewater systems, assess their reusability, and explore additional functionalities such as antimicrobial activity and detailed photocatalytic degradation behaviour.

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