

RESEARCH PAPER

Structure and Optical properties of TiO₂@CuO Core-Shell Nanoparticles Thin Film Synthesised via Pulsed Laser Ablation

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ABSTRACT

The synthesis of core-shell nanomaterials has demonstrated substantial importance for both fundamental research and practical applications, encompassing cell imaging, surface enhanced Raman scattering, chemical sensing, light emission diode, catalysis, and solar cell. This study demonstrates the synthesis of TiO₂ as core and CuO as shell nanoparticles, they were synthesized using the laser ablation in two steps to control their size and the possibility of changing their characteristics, then deposited on glass substrate to get on TiO₂@CuO core-shell nanoparticles thin film. TiO₂@CuO core-shell nanoparticles thin film had an absorption spectrum at 372 nm and TiO₂ at 345 nm with band gap energy was estimated to be 3.3 eV for TiO₂ and 2.5 eV for TiO₂@CuO core-shell thin films. FESEM results confirm the particles had spherical shapes with average particle size of 15 nm for TiO₂ thin film and 25.5nm for TiO₂@CuO core-shell nanoparticles thin film. TEM image proves that coating the TiO₂ surface with CuO was successful. The zeta potential was used to confirm the stability of synthesized TiO₂ NPs and TiO₂@CuO core-shell nanoparticles that will be used in biomedical applications.

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INTRODUCTION

Titanium dioxide (TiO₂) and copper (II) oxide (CuO) are two extensively researched metal oxides. TiO₂ exhibits greater environmental stability and chemical durability than SiO₂, making it the most extensively researched semiconductor [1]. TiO₂ demonstrates significant oxidation capability upon the creation of electron-hole pairs, although its broad energy band gap (E_g = 3.2 eV for TiO₂ anatase and E_g = 3.0 eV for TiO₂ rutile) [2]. Consequently, TiO₂ can produce reactive species in the presence of hydrogen peroxide, facilitating its use for hydrogen generation. TiO₂ can be

employed in biomolecule detection either by using biomolecules as reagents or by quantifying a byproduct, such as hydrogen peroxide [3].

CuO is a monovalent copper oxide exhibiting electrical and in-situ catalytic capabilities, extensively utilized in catalysts, supercapacitors, biosensors, and other applications [4]. CuO is electroactive and enhances electron mobility, making it suited for diverse applications. CuO has a substantial energy band gap and can harness ultraviolet light. TiO₂-CuO photoanodes have been engineered for use as both anodes and cathodes in various applications [5].

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TiO₂@CuO core-shell architecture is suggested for multiple benefits. Initially, selective performance augmentation. The CuO coating on TiO₂ will facilitate accelerated electron transport, thereby enhancing TiO₂ performance. Secondly, stability: TiO₂-CuO has greater stability than other TiO₂ hybridizations under specific conditions, including acidic environments. Third, interfacial compatibility: CuO forms spontaneously on TiO₂ due to lattice matching [6,7]. TiO₂@CuO core-shell nanoparticles are applicable in various fields, including environmental, energy, and biological sectors. Core/shell particle architectures are advantageous for the development of sensor platforms [8].

Composite TiO₂@CuO nanoparticles can be synthesized using many techniques, including sol-gel, hydrothermal, chemical vapour deposition, supersonic spraying, laser ablation, and seed-mediated growth [8,9]. The duration, temperature, and pressure are controllable parameters of the processing conditions, and methodologies for scaling from small to large batch sizes have been established. The nanoparticles can be tailored for specific applications or functionalities due to the extensive flexibility each method offers for modification or regulation [10].

TiO₂@CuO core-shell nanoparticles, situated at the convergence of photocatalysis, environmental remediation, and energy storage, also exhibit potential for sensing and biological applications [8,11]. A TiO₂@CuO nanoparticle-chitosan

composite film biosensors facilitate optical quantum yield assessments and have been employed for the electrochemical quantification of glucose. Unmodified TiO₂@CuO nanoparticles can amplify optical and electrochemical signals from graphene oxide and exhibit biocompatibility with human breast cancer cell types. Diagnostic imaging applications utilize the nuclear medicine characteristics of TiO₂@CuO and Gd³⁺ co-deposited systems for concurrent tracking and delivery enhancement during transmembrane transport [12].

Pulsed laser ablation in liquids (PLAL) is a physical method employed in this research. Laser ablation refers to the procedure of extracting a segment of material from the surface of a solid after laser irradiation [13]. This is crucial in the processing and organization of materials across several technological domains. Laser ablation of a solid target submerged in a liquid environment has emerged as a significant “top down” technique for the fabrication and production of metallic colloids, semiconductors, alloys, and oxide nanoparticles in deionized water and solvents [14,15]. The dimensions and morphology, along with nanoparticle concentration, can be regulated by varying the laser wavelength, energy, and pulse duration [14].

In this research, titanium powder and copper oxide powder were pressed into a pellet, then using laser ablation produce TiO₂@CuO core-shell nanoparticles. for testing, deposited solution on a

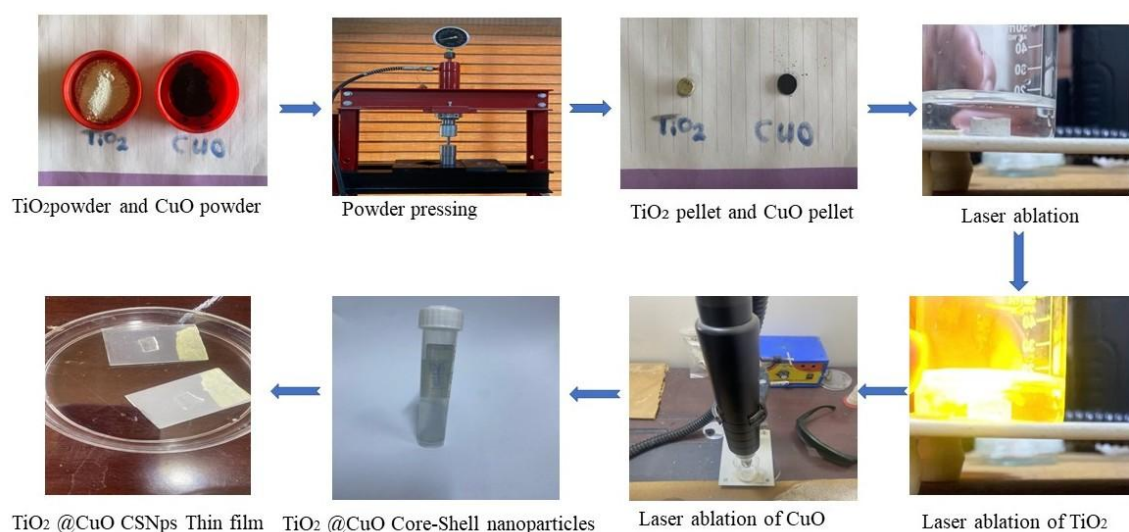


Fig. 1. Experimental work.

glass substrate using spin coating. The successful one-step preparation of TiO₂ nanoparticles via the fast, environmentally friendly platform of the freshly developed laser-ablation-in-liquid system, which translates a TiO₂ pellet (microparticle) to TiO₂ solution (nanoparticle) in 5 min. Laser ablation synthesis of titanium dioxide (TiO₂) nanoparticles in liquid represents a powerful “top-down” approach for controlling particle properties by systematically varying laser parameters.

MATERIALS AND METHODS

Preparation of TiO₂@CuO CSNPs Thin Film

Titanium dioxide-copper oxide (TiO₂@CuO) core-shell nanoparticles thin film were produced by a many-step process. First, each of titanium dioxide (99%) and copper (II) oxide powder (99.99%) was shaped into pellets that are 1 cm wide and 0.5 cm thick using a hydraulic piston press (SPECAC) with a force of 5 tons for 10 minutes, as shown in Fig. 1. Second, using an Nd:YAG laser (Huafei Tongda Technology-DIAMOND-288 pattern EPLS) to prepare TiO₂@CuO nanoparticles, providing pulses of 1064 nm wavelength with energy per pulse of 150 mJ, pulse width of 10 ns, repetition rate of 10 Hz, effective beam diameter of 5 mm, and number of pulses of

1000. The titanium dioxide pellets were first put in deionized water, The laser ablation process in deionized water was continuous for 3 minutes. During laser irradiation, become radiation readily visible, after that produce TiO₂ nanoparticle in deionize water. The second step, put copper (II) oxide pellet was immersed in a beaker containing a titanium nanoparticle solution. Then open the laser to ablate the particle of copper oxide. It was obtained TiO₂@CuO core-shell nanoparticles. TiO₂@CuO core-shell nanoparticles solution was deposited onto a glass substrate to obtain films in order to examine the physical properties of TiO₂@CuO core-shell nanoparticles thin film.

Characterization techniques

Including morphological, surface chemical, and optical analyses is essential to fully understand the as-synthesized TiO₂@CuO core shell thin film structures. Morphology and crystalline phase of TiO₂@CuO core shell nanoparticles thin film was characterized by X-ray diffraction (XRD), filed emission scanning electron microscopy (FESEM), and transmission electron microscopy (TEM). Optical properties are evaluated by UV-Vis spectroscopy. Also, the zeta potential was used to confirm the stability.

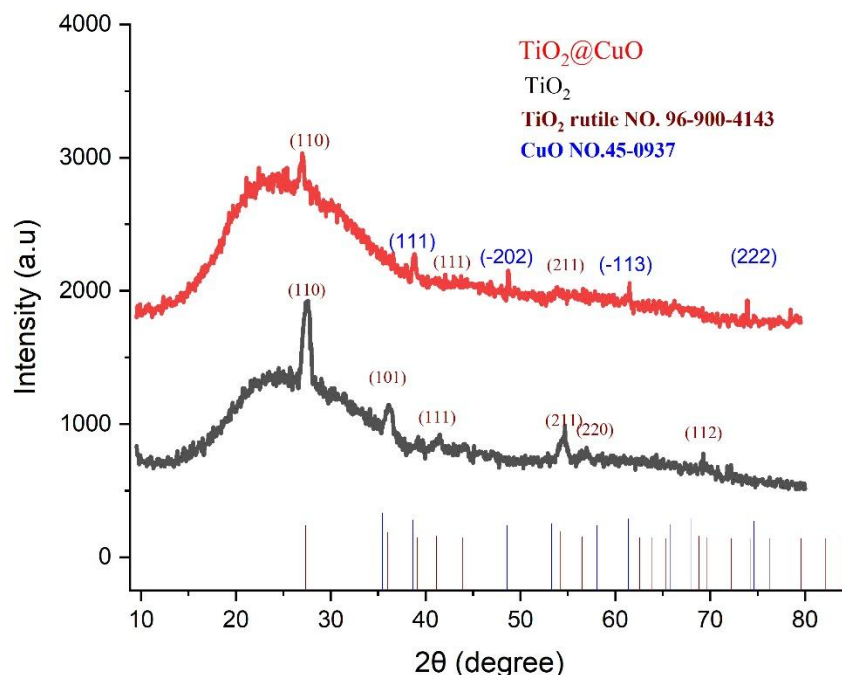


Fig. 2. XRD patterns of TiO₂@CuO core shell nanoparticles thin films.

RESULTS AND DISCUSSION

X-ray diffraction was completed to identify phase formation and crystallographic information of the samples. The X-ray diffraction (XRD) patterns of TiO₂@CuO core shell nanoparticles thin films are displayed in Fig. 2. All the peaks of XRD patterns were analyzed and indexed using the X'Pert

HighScore Plus program and compared with ICSD data standards. The pattern exhibits diffraction peaks which is due to the well crystallization. According to ICSD data, the peaks at $2\theta = 27.4306^\circ$, 35.959° , 41.25° , 44.07° , 54.44° , 61.997° , 69.6° which corresponds to the Bragg's reflection plane of (110), (101), (111), (210), (211), (220), (112).

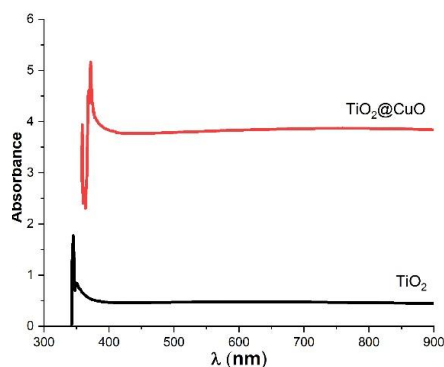
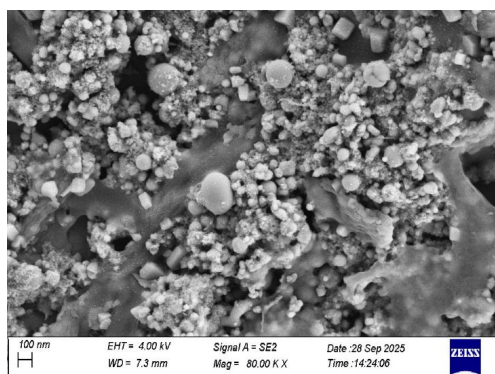
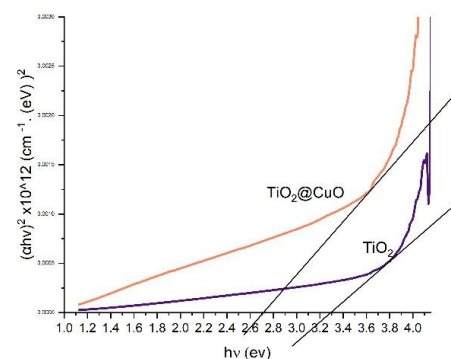
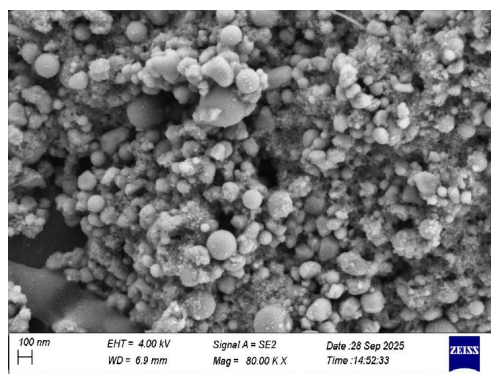


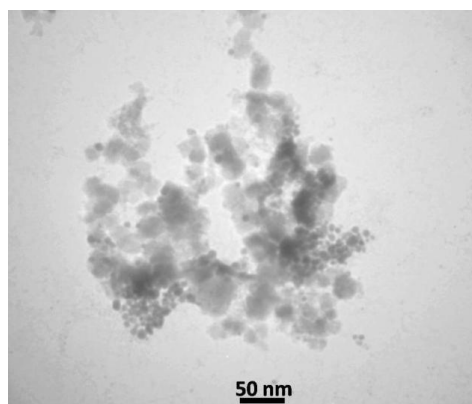
Fig. 3. UV-Vis spectroscopy of TiO₂@CuO core shell nanoparticles thin films.



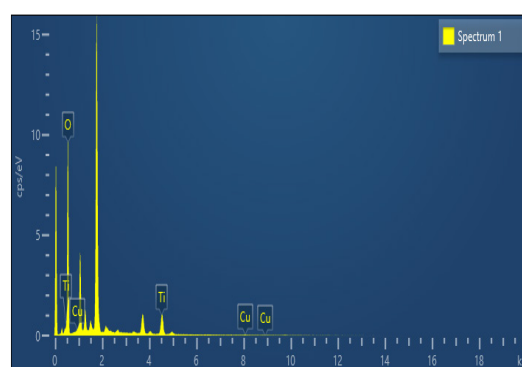
a. FESEM: TiO₂ NPs



b. FESEM: TiO₂@CuO core-shell nanoparticles thin films



c. TEM: TiO₂@CuO core-shell nanoparticles thin films



d. EDX: TiO₂@CuO core-shell nanoparticles thin films

Fig. 4. FESEM, EDX and TEM image of TiO₂@CuO core-shell nanoparticles thin films.

respectively are the characteristic reflections for the rutile phase TiO₂ Tetragonal crystal structure. However, The Presence of other peaks at $2\theta = 38.7^\circ$, 48.6° , 61.37° , 74.6° that matches the monoclinic phase of CuO (ICSD No 45-0937) which corresponds to the Bragg's reflection plane of (111), (-202), (-113), (222). The corresponding XRD patterns of TiO₂, and TiO₂@CuO are shown in Figure. All the peaks of TiO₂@CuO core shell nanoparticles thin films can be indexed to the rutile TiO₂ and the monoclinic CuO phases, except for the broad peak at $2\theta=15-35^\circ$, which due to the amorphous (glass) its substrate [16], indicating that the core-shell structure of TiO₂@CuO can be preserved during the CuO growth process.

The optical properties of TiO₂@CuO core shell nanoparticles thin films were examined by the uv-vis spectroscopy and the optical band gap can be determined by plotting $(\alpha h\nu)^2$ against $h\nu$, where α is the absorption coefficient were shown in Fig. 3A and Fig. 3B. From Fig. 3A, showing strong wavelength of the absorption band at 345 nm approximately for TiO₂ NPs (In the uv light absorption region), the peak shifted in red region (longer wavelength) due to CuO coats TiO₂ NPs approximately 372 nm. Because CuO has broader absorption band extends into visible region (700-800). The red shift confirms successful CuO coating. As seen in Fig. 3B, the band gap energy was estimated to be 3.3 eV for TiO₂ NPs and 2.5 eV for TiO₂@CuO core-shell nanoparticles thin films. The energy gap in the case of a core-shell usually manifests in two ways: either one energy gap appears, or two energy gaps appear. Fig. 3B confirms the core-shell has effective E_g about 2.5 eV. The reduced band gap indicates strong

interfacial interaction and charge transfer within the TiO₂@CuO heterostructure and also confirms the CuO shell that is coating the TiO₂ core: it is optically dominant.

FESEM and TEM were used to analyse the surface, show particle images and determine particle sizes. Fig. 4a shows TiO₂ NPs thin films have many spherical particles and an average particle size of about 15 nm. The analysis confirms that the particle size increased to 25.5 nm when the coated by CuO shell, as illustrated in Fig. 4b. The TEM image proves that coating the TiO₂ surface with CuO was successful and had spherical shapes with an average particle size of 25 nm (Fig. 4c). The image shows dark and lighter regions. These regions are core-shell structures with a thin shell, darker particles indicate the core and lighter particles indicate the shell. EDX spectrum confirms the presence of Ti, Cu, and O elements that indicate the successful formation of TiO₂/CuO composite are shown in Fig.4d.

Zeta potential measurement provides details about particle stability, surface function, and how dissolved chemicals interact with the surface [17]. The zeta potential was utilized to analyze the stability of TiO₂NPs and TiO₂@CuO core-shell nanoparticles synthesized that will be used in biomedical applications. Fig. 5 shows the zeta potential of TiO₂ NPs and TiO₂@CuO core-shell nanoparticles are -40.07 mV and -55.6 mV in deionised water suspension. TiO₂ NPs has a low positive charge on its surface, indicating that it is less stable and has a propensity to agglomerate into clusters over time to create a core structure. After coating with a CuO shell, the stability and homogeneity of TiO₂ NPs become higher because

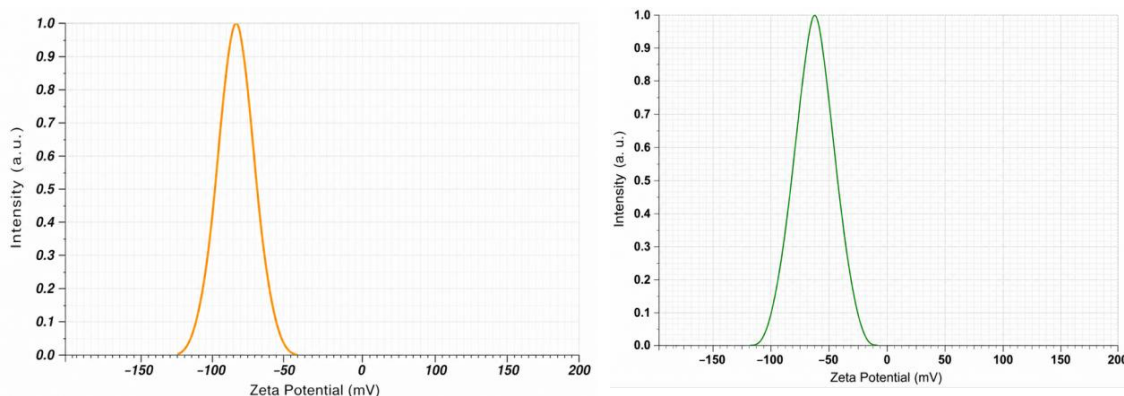


Fig. 5. Zeta potential measurement of A. TiO₂ NPs and B. TiO₂@CuO core-shell nanoparticles.

of CuO negative charge. Additionally, it causes a repulsive force among particles, which is indicated by the particle's huge negative surface charge, this proves that the CuO shell makes TiO₂ NPs stable and prevents them from agglomeration.

CONCLUSION

In this research, titanium powder and copper oxide powder were pressed into a pellet, then the fast, environmentally friendly method it used to produce TiO₂@CuO core-shell nanoparticles called laser-ablation-in-liquid, this method take just 5min to produce TiO₂@CuO core-shell nanoparticles. for testing, deposited solution on a glass substrate using spin coating. The UV-Vis, XRD, FESEM, and TEM analyses confirm that TiO₂ synthesized in nanoscales have particle size about 15 nm, also analyses confirm nanoparticles particle size increased with coated shells at 25.5 nm. the zeta potential of TiO₂ NPs and TiO₂@CuO core-shell nanoparticles are -40.07 mV and -55.6 mV in deionised water suspension, that confirm the stability of synthesized TiO₂NPs and TiO₂@CuO core-shell nanoparticles that will be used in biomedical applications.

CONFLICT OF INTEREST

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

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