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Synthesis and Structural Characterization of Ethylene Glycol–Capped Cu-Doped TiO_2 Nanoparticles for Advanced Material Applications

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Abstract

Ethyleneglycol@Cu-doped TiO_2 nanoparticles were synthesized using the Sol–Gel method, which enables molecular-level mixing of precursors and provides in-situ control over nucleation and nanoparticle growth. Fourier Transform Infrared (FTIR) spectroscopy confirmed the presence of the capping agent on the nanoparticle surface. X-ray Diffraction (XRD) analysis revealed that the nanoparticles crystallized in the rutile phase, with a prominent diffraction peak observed at 27.5°. The average crystallite size, calculated using the Scherrer equation, was found to be 4.4 nm. Scanning Electron Microscopy (SEM) images showed predominantly spherical surface morphology. Elemental composition determined through Energy Dispersive X-ray (EDX) spectroscopy indicated atomic fractions of 76.22% oxygen, 20.95% titanium, and 0.92% copper, confirming successful Cu doping in TiO_2 lattice.

Keywords: Nanoparticles, Titanium dioxide, Doping, Capping, FTIR, XRD, SEM, EDX

Introduction

Titanium dioxide (TiO_2) has emerged as one of the most versatile metal oxide materials owing to its distinctive optical, electronic, and surface properties (Bensouici et al. 2017). Its chemical stability, non-toxicity, abundance, low cost, well-characterized optical and electronic properties, strong photocatalytic behaviour have enabled wide applications in sensing, environmental remediation, photovoltaics, and energy storage (Madadi et al. 2019). TiO_2 exists in two most studied polymorphs, anatase and rutile, exhibit band gaps near ~3.2 eV and ~3.0 eV, respectively, which enables strong UV activity but limits visible-light utilization; in addition, rapid recombination of photogenerated electron–hole pairs is a persistent bottleneck for many TiO_2 applications (Byrne et al. 2019) (Bhatia and Dhir 2016). However, the intrinsic wide band gap of TiO_2 limits its photo response primarily to the ultraviolet region, thereby reducing its efficiency under visible-light irradiation (Mathew et al. 2018). Efforts to overcome these intrinsic limitations have produced a large literature on defect engineering, sensitization, heterojunction formation, and purposeful doping (Chithira and Theresa

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John 2020). To overcome these constraints, the modification of TiO_2 through metal doping and surface functionalization has become an important strategy for tuning its electronic structure, enhancing charge separation, and improving interfacial interactions (Ahadi et al. 2019). Its anatase and rutile forms are commonly used, but their wide band gaps and rapid electron–hole recombination limit visible-light performance (Alotaibi et al. 2020). To address these drawbacks, strategies such as dye sensitization, coupling with other semiconductors, introducing oxygen vacancies, and metal or non-metal doping have been explored (Safardoust-Hojaghan et al. 2020). Transition-metal doping is a widely explored route to extend light absorption and tailor charge dynamics (Ali et al. 2018). Among transition metals, copper (commonly present as Cu^{2+} and Cu^{+} under synthesis/operando conditions) is attractive because its multiple oxidation states and strong interaction with oxygen vacancies can introduce mid-gap states that absorb visible photons and participate in charge transfer processes (Ang et al. 2016). Experimental findings indicate that introducing small amounts of Cu leads to a red shift in the absorption band, enhances the generation of reactive oxygen species, and boosts photocatalytic efficiency under visible light (Ayim-Otu et al. 2020) (Reddy et al. 2019). However, the overall outcome is highly sensitive to factors such as dopant level, whether the Cu occupies substitutional, interstitial, or surface-cluster positions, and the existence of accompanying defects that may promote charge recombination (Alotaibi et al. 2020). Among various dopants, copper (Cu) has received considerable attention because of its ability to introduce intermediate energy levels within the TiO_2 band gap, promote visible-light absorption, and modulate redox activity (Bhatia and Dhir 2016) (Ayim-Otu et al. 2020). Experimental studies have shown that low to moderate Cu loadings can induce sub-bandgap absorption (visible activity), and can alter defect chemistry (oxygen vacancies, Ti^{3+} centres) . Cu-doped TiO_2 systems have shown promising performance in catalytic, antimicrobial, and optoelectronic applications due to their improved charge transfer pathways and altered structural characteristics (Mathew et al. 2018). The incorporation of Cu ions can also influence crystallinity, defect formation, and particle morphology, making structural analysis a crucial step toward understanding the material's functional behaviour (Abbas and Rasheed 2021). Surface capping agents further play a central role in prevention of nucleation, particle growth, dispersion, stabilizing nanoparticles, preventing agglomeration, and tailoring surface reactivity (Bensouici et al. 2017). Organic ligands such as amines, glycols, and thiols offer distinct coordination modes that significantly affect the nucleation, growth, and final architecture of metal-doped TiO_2 nanoparticles (Mai et al. 2020). Amine capping typically enhances dispersion and controls particle size through nitrogen coordination (Agasti and Kaushik 2014); glycols bind via hydroxyl groups and may act as mild reducing agents influence steric stabilization and modify surface polarity (Santos and Machado 2021); while thiol groups form strong metal–sulfur bonds that can alter surface chemistry and electronic interactions (Kumar et al. 2016). The use of capping ligands during or after synthesis exerts an outsized influence on nanoparticle nucleation/growth, colloidal stability, surface defect chemistry, and interfacial charge transfer (Gulati et al. 2018). Studies on the capping of nanoparticles mainly highlight how ligands coordinate with surface metal atoms to prevent additional particle growth, provide steric or electrostatic stability in colloidal systems, and adjust the surface electronic structure, thereby influencing adsorption and reactions at the interface (Mahvelati-Shamsabadi and Goharshadi 2017). The specific chemical groups present in a ligand define the geometric and electronic environment at the nanocrystal surface, which can modify dopant–ligand interactions as well as the behaviour of copper species anchored to the surface (Yang et al. 2021). Ethylene glycol are commonly used in solvothermal/sol–gel routes for TiO_2 as solvents, complexing agents, mild reducing agents

and structure-directing templates (Kumar et al. 2016). Glycols coordinate through hydroxyl groups to titanium precursors, slow hydrolysis/condensation rates, and can promote formation of small, uniform nanoparticles or sheet/mesoporous morphologies depending on protocol (Santos and Machado 2021). Their hydrogen-bonding networks and viscosity effects also control particle aggregation and porosity—attributes that frequently improve dispersion and accessible surface area for catalytic reactions (Santos and Machado 2021) (Bensouici et al. 2017). Achieving reproducible dopant incorporation and maintaining crystallinity are recurring synthetic challenges. However, excessive Cu or segregated CuO phases can act as recombination centres or change stability; thus, synthesis method and dopant distribution are critical. Methods such as precipitation, sol–gel, hydrothermal, solvothermal routes are commonly used because they allow molecular-level mixing of precursors and in-situ control over nucleation and growth (Athira et al. 2020). These methods have been shown to influence particle size, suppress aggregation, and sometimes partially reduce metal dopants under controlled conditions (Garzon-Roman et al. 2020). These synthetic variants play a major role in determining whether Cu remains atomically dispersed in the TiO_2 lattice or segregates to oxide phases or surface clusters (Gupta et al. 2020).

Most available studies emphasize photocatalytic performance rather than detailed structural relationships involving dopants, surface ligands, and crystal features. The present work addresses this gap by synthesizing copper-doped TiO_2 nanoparticles capped with Ethylene glycol ligands using a unified approach. By examining phase composition, crystallite size, dopant incorporation, functional groups, and surface interactions, the study clarifies how different capping environments shape the structural and functional properties of doped TiO_2. These insights support the targeted design of advanced TiO_2-based materials for photocatalysis, sensing, and related applications. The scope includes synthesizing doped TiO_2 through sol–gel or hydrothermal methods, applying functional capping agents, and thoroughly characterizing the materials.

2 Methodology

2.1 Reagents:

Ethanol (absolute, >99.8%), tetrabutyl titanate ($Ti(OBu)_4$, 98%), hydrochloride acid (HCl, 37%), Cupric acetate (≥98%), Ethylene glycol also of analytical standard. All reagents were of analytical grade and used as received from Sigma-Aldrich. Triply distilled water was used for preparation of all the solutions.

2.2 Synthesis mechanism of Ethyleneglycol@Cu-doped TiO_2

Ethanol and Tetrabutyl titanate were added to the beaker in a volume ratio of 2:1. Polyethylene glycol, hydrochloric acid and distilled water in a volume ratio of 1:1:15 were added to a separatory funnel add appropriate amount of copper nitrate (4% of the total mole fraction of Titanium) solution into the separatory funnel. Then it is added to the above solution of Titanate dropwise, the mixture was heated at 200°C for 10min. During this mixing, hydrolysis occurred and condensation reaction between water loss and alcohol loss occurred to obtain the TiO_2 precursors. The obtained precursors gradually dissolved, nucleated, grew and crystallized to form TiO_2 products. Afterwards, the pure TiO_2 was obtained by washing the sample three times with distilled water and drying in an oven at 100 °C for 4 h as shown in Figure 1.

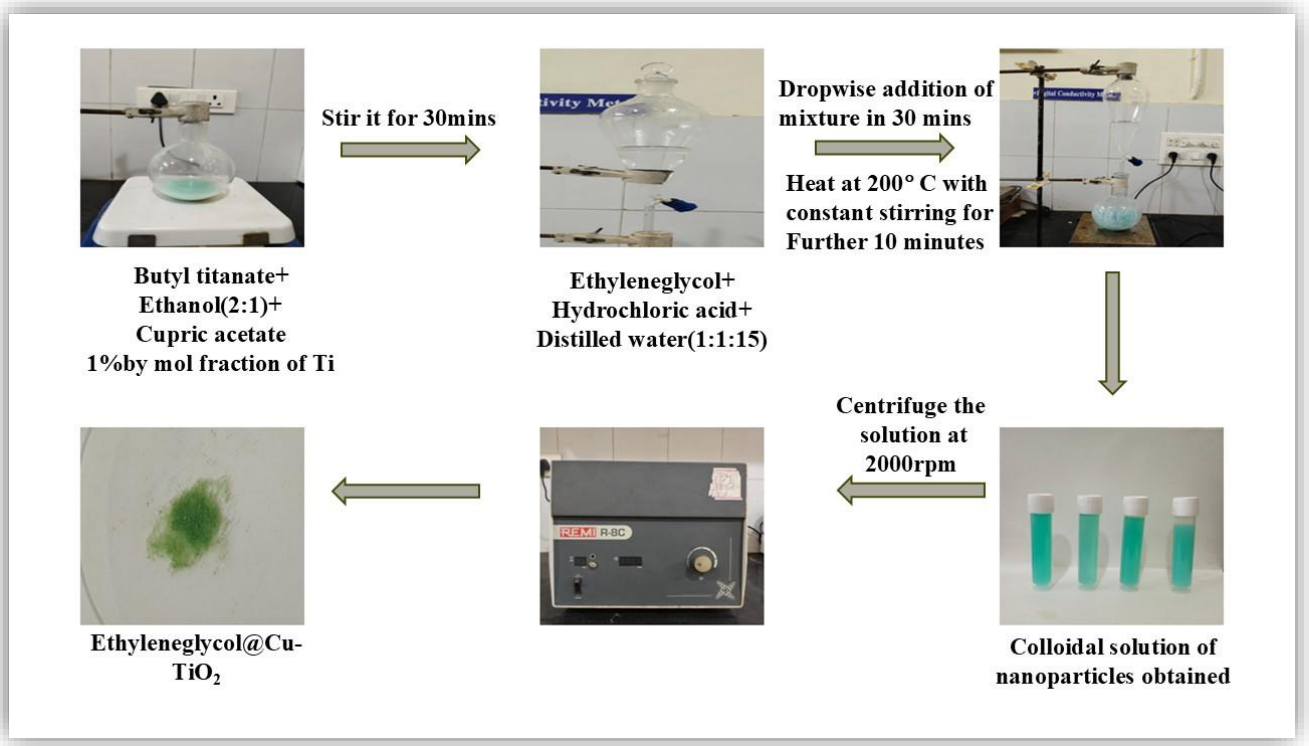


Figure 1: Schematic diagram showing the steps for the synthesis of Ethylene-glycol@Cu-TiO_2 nanoparticles.

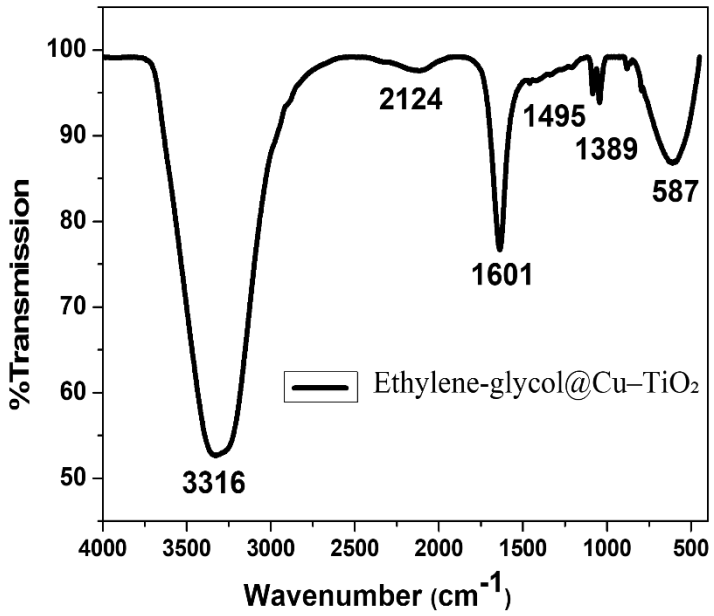
2.3 Characterization Techniques

The characterization of the synthesized materials was carried out using different techniques including Fourier-Trans form Infrared (FT-IR) spectroscopy, powder X-ray diffraction (XRD) Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX). FT-IR spectra were recorded on a Bruker FT-IR spectrometer in the spectral range of 400-4000 cm^{-1}. The measurements were performed using KBr pellets. The XRD patterns of the powdered samples were recorded using XPERT PRO diffractometer with Cu-Kα radiation [(λ = 1.5406Å); 1,800 W (45 kV, 40 mA)]. The crystallite size was estimated by using the Scherrer equation. The data was collected in the 2θ range from 10°-80° with a step size of 0.05˚ and a counting time of 10s per step. For recording XRD, the samples were coated on glass substrates. The size of the synthesized material was determined by SEM using model JEOL JSM-IT210 For SEM analysis, the diluted nanoparticles were suspended in ethanol and introduced on a copper grid, and the analysis was carried out after drying them in air. EDX was used for determining the elemental compositional analysis using in column EDX detector. The samples were allowed to dry and then measured in reflection geometry.

3 Results and Discussions

3.1 FTIR spectra of Ethyleneglycol@Cu–TiO_2 nanoparticles

The FTIR spectrum of the Ethylene-glycol@Cu–TiO_2 nanoparticles is shown in Figure 2 and presents characteristic absorption bands confirming the successful capping of ethylene glycol molecules on surface of Cu–TiO_2 nanoparticles. The broad and intense band observed at 3316 cm^{-1} corresponds to the stretching vibration of hydroxyl (–OH) groups s of ethylene glycol coordinated with TiO_2 (Singh et al. 2020).



Functional groups	Wavenumber (cm^{-1})
O-H Stretching	3316
C-H Stretching	2124
O-H bending	1601
C-O Stretching	1495 & 1389
Ti-O Stretching	587

Figure 2: FTIR spectrum of Ethyleneglycol@Cu–TiO_2 nanoparticles and wavenumbers of respective functional groups.

A distinct peak at 2124 cm^{-1} may be attributed to C–H stretching of Ethylene chain of the capping agent. The absorption at 1601 cm^{-1} corresponds to the O-H bending vibration. Peaks appearing at 1495 cm^{-1} and 1389 cm^{-1} indicate C–O stretching's. The strong metal–oxygen stretching band at 587 cm^{-1} is characteristic of Ti–O and Cu–O lattice vibrations. So, the FTIR results confirm the successful anchoring of ethylene glycol onto the surface of Cu–TiO_2 nanoparticles (Bhullar et al. 2015).

3.2 X-Ray Diffraction of Ethylene-glycol@Cu–TiO_2 nanoparticles

The crystalline structure of the Ethylene-glycol@Cu–TiO_2 nanoparticles was examined using X-ray diffraction (XRD, Figure 3). The diffraction pattern exhibits distinct and sharp peaks at 2θ values of approximately 27.5°, 36.4°, 41.5°, 54.4°, 63.1°, and 69.5°, which are indexed to the (110), (101), (111), (210), (220), and (310) crystal planes, respectively (Bhullar et al. 2013). These reflections are in good agreement with the standard JCPDS data for rutile TiO_2, confirming that the synthesized nanoparticles predominantly crystallize in the rutile phase (Wang et al. 2020). The presence of narrow and intense peaks indicates high crystallinity and well-developed long-range atomic ordering within the nanoparticles. The average crystallite size (D) of the TiO_2 phase was estimated using the Scherrer equation:

$$\boldsymbol{D = \frac{k\lambda}{\beta \cos\,\theta}} \qquad (\boldsymbol{i})$$

In equation (i) *k* is the shape factor (0.9), λ is the X-ray wavelength (1.5406 Å), β is the full-width at half-maximum (FWHM), and θ is the Bragg angle. The calculated crystallite size was found to be approximately 4.4 nm, indicating that the material consists of nanocrystalline domains. No additional peaks corresponding to copper or secondary impurity phases were clearly identified, which implies that copper ions are either present in very low concentration, highly dispersed on the TiO_2 surface, or embedded within the rutile lattice without forming a separate detectable crystalline phase (Singh et al. 2020). This observation is consistent with successful incorporation of Cu into the TiO_2 lattice

without significant structural distortion. Overall, the XRD results confirm the formation of highly crystalline rutile TiO_2 nanoparticles, with nanoscale crystallite size and good structural integrity after copper modification.

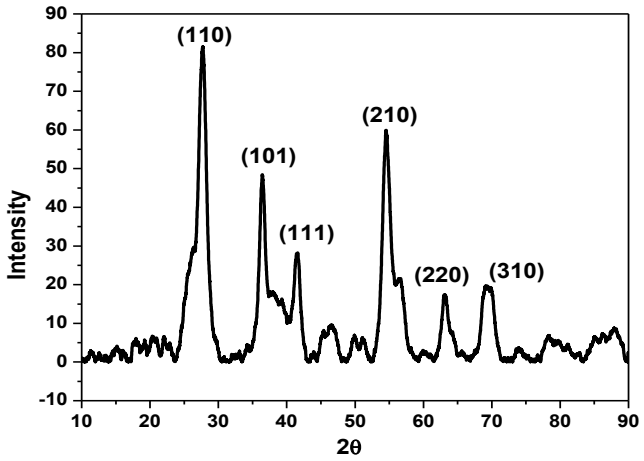


Figure 3: XRD spectra of Ethyleneglycol@Cu-TiO_2 nanoparticles

3.3 Morphology analysis of Ethyleneglycol@Cu-TiO_2 nanoparticles using Scanning electron microscopy

The surface morphology of the Ethyleneglycol@Cu–TiO_2 nanoparticles was examined using Scanning Electron Microscopy (SEM), as presented in Figure 4(a) and Figure 4(b). The micrographs show that the nanoparticles exhibit predominantly spherical to near-spherical morphology, which is typical of TiO_2 synthesized through sol–gel processing. The particles demonstrate dense packing and noticeable agglomeration due to the high surface energy and strong inter-particle forces generally associated with nanoscale metal oxides. Upon increasing the magnification (from micrograph **a** to **b**),

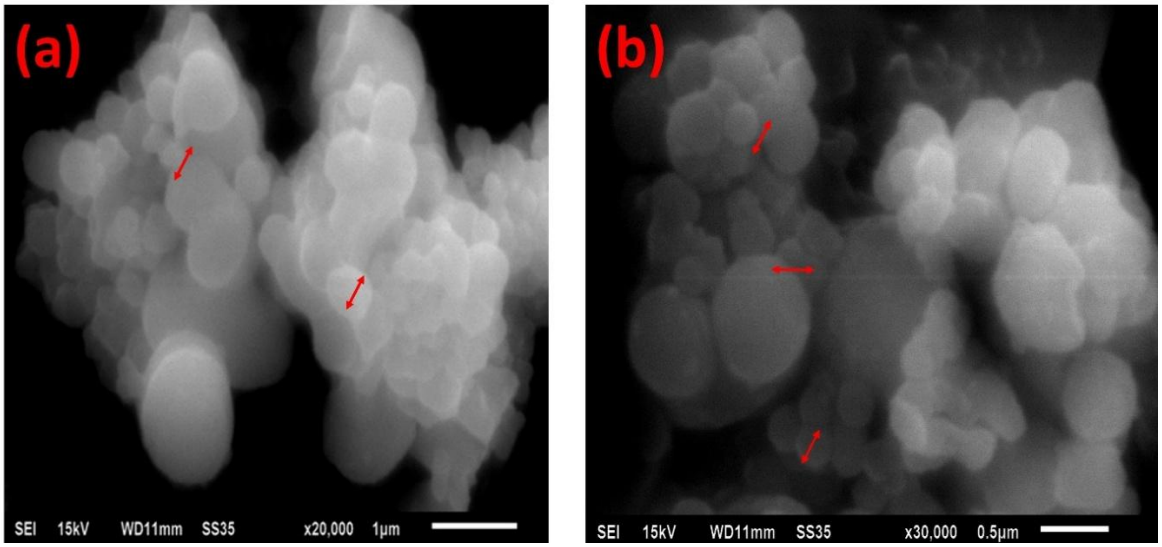


Figure 4: SEM micrographs of Ethyleneglycol@Cu–TiO_2 nanoparticles showing surface morphology at (a) 20,000× and (b) 30,000× magnification.

individual nanoparticles become more distinguishable, confirming their nanocrystalline nature with an overall average particle size in the range of 15-35 nm, depending on the degree of clustering at different locations in the sample. The smooth surface features indicate uniform nucleation and growth,

suggesting that for device applications. The difference between crystallite size (XRD nearly 4.4 nm) and particle size (SEM) suggests that each particle observed in SEM is composed of multiple smaller crystallites aggregated together a common phenomenon in sol–gel derived metal oxide nanoparticles due to high surface energy and inter-particle attractive forces (El-Desoky et al. 2018).

3.4 Elemental compositional analysis of Ethyleneglycol@Cu-TiO_2 nanoparticles using Energy Dispersive X-Ray (EDX) Spectrum

The EDX spectrum (Figure 5) obtained for the Ethylene-glycol@Cu–TiO_2 nanoparticles confirms the elemental composition observed in the SEM micrographs. The spectrum is dominated by strong peaks of titanium and oxygen, verifying TiO_2 as the primary phase, supported by the oxide framework evident in the SEM images. Quantitative EDX analysis shows that the nanoparticles contain 76.22% oxygen, 20.95% titanium, and 0.92% copper (atomic fraction), (also depicted from XRD analysis) indicating that approximately 4% of Ti (IV) sites are substituted by Cu (II) ions in the lattice. The relatively lower intensity of the Cu peaks is consistent with its moderate doping concentration and uniform incorporation into the TiO_2 matrix. The detection of carbon originates from the ethylene glycol capping layer, which enhances nanoparticle stability and minimizes aggregation. Minor traces of elements such as iron are attributed to precursor impurities, instrument background, or sample mounting materials and do not significantly alter the chemical composition (Aziz et al. 2020).

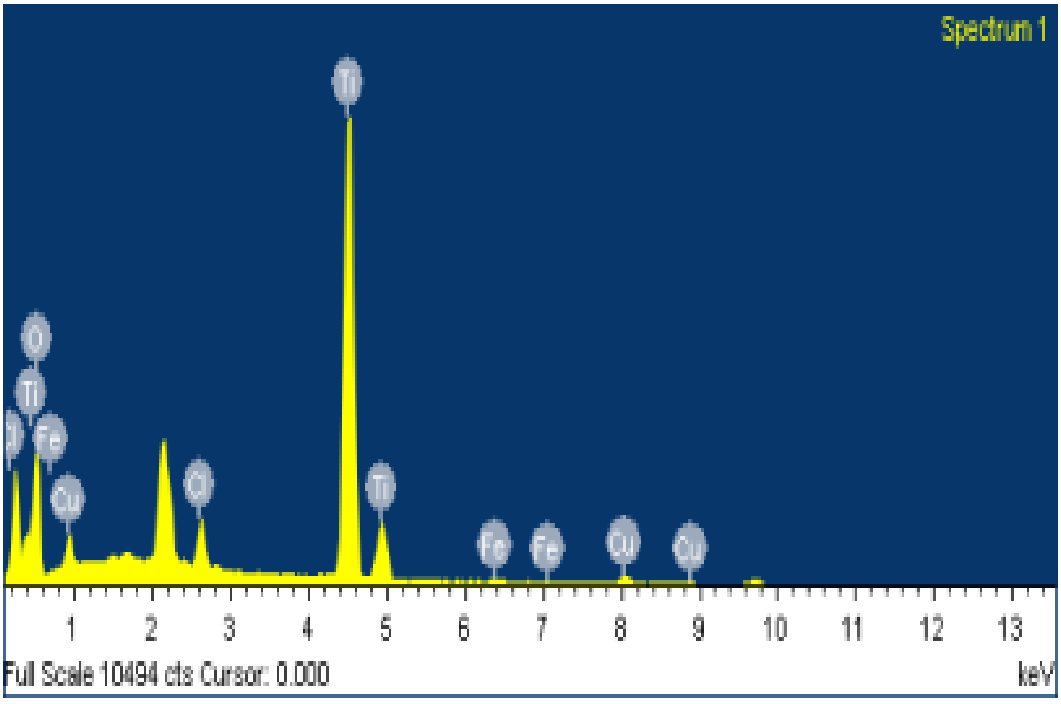


Figure 5: EDX spectra of Ethyleneglycol@Cu-TiO_2 nanoparticles

Overall, the combined SEM–EDX analysis confirms that the synthesized nanoparticles consist of crystalline TiO_2 successfully doped with Cu (II) ions and coated with ethylene glycol, demonstrating the effectiveness of the adopted synthesis approach.

4. Conclusion

The study successfully demonstrates the synthesis of Ethylene glycol@Cu-doped TiO_2 nanoparticles using the Sol–Gel method, enabling homogenous capping on surface of nanoaprticles. The presence

of the capping agent was verified through FTIR analysis, while XRD confirmed crystalline nature. SEM results revealed predominantly spherical morphology, and EDX analysis verified the elemental composition and effective incorporation of copper into the TiO_2 lattice. These findings collectively indicate that the suitability of adopted synthesis route for producing well-defined Cu-doped TiO_2 nanoparticles with controlled structural and morphological characteristics for various device applications.

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