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Highly efficient fluorescent sensing of mercury contamination by Cu doped and capped TiO_2 nanoparticles

Seema Maheshwari[a], Gurpreet Kaur Bhullar[b], Shiwani Sharma[c], Kamaldeep Kaur[d], Bhupinder Kaur[e], Ramneek Kaur*[b]

[a] Department of Chemistry, Mata Gujri College, Fatehgarh Sahib-140406, Punjab, India
[b] Department of Physics, Mata Gujri College, Fatehgarh Sahib-140406, Punjab, India
[c] Department of Physics, Government College for Girls, Patiala-147001, Punjab, India
[d] Department of Physics, UIS Chandigarh University Gharuan,140413, Punjab, India
[e] Department of Physics, Akal Degree College Mastuana, Sangrur, 148001, Punjab, India
dhaliwalramneek@gmail.com

Abstract

Mercury poses a serious risk to living organisms after entering the food chain, it bioaccumulates and biomagnifies, leading to severe impacts at higher trophic levels. In aquatic and food matrices, mercury can also undergo chemical transformations that generate species with even greater toxicity, making its detection and remediation critically important. In this study, Cu-doped TiO_2 nanoparticles capped with ethylene glycol, ethylenediamine, and Triton X-100 were synthesized and comprehensively characterized to evaluate the effect of surface functionalization on their structural, optical, and sensing properties. FTIR spectra verified the effective binding of capping agents through characteristic hydroxyl, alkyl, amine, C–O/C–N, and metal–oxygen vibrational modes. UV–Visible analysis confirmed a clear red shift and reduced band gap, indicating enhanced visible-light activity due to copper doping and surface functionalization. SEM observations showed that different capping agents governed nanoparticle shape, yielding rod-like, spherical, and polyhedral structures for ethylene glycol, ethylenediamine, and Triton, respectively. Fluorescence results revealed strong emission linked to nanoscale size and surface defects, with Triton-capped Cu–TiO_2 showing the strongest fluorescence. In mercury sensing studies, ethylene glycol- and ethylenediamine-capped samples exhibited marked fluorescence quenching, whereas Triton-capped nanoparticles showed signal enhancement owing to defect passivation. Overall, the study demonstrates that surface capping critically controls optical behaviour and sensing efficiency, establishing Cu-doped, capped TiO_2 nanoparticles as promising materials for Hg^{2+} detection in water.

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

Introduction

Mercury (Hg) is an extremely poisonous heavy metal that threatens both human health and the environment (Devadiga et al. 2017). It enters ecosystems through natural events like volcanic eruptions as well as human-driven sources such as coal burning, gold extraction, and various industrial processes (Dold 2020). After being released, mercury undergoes several chemical changes and often ends up in aquatic systems, where microorganisms convert it into methylmercury (CH_3Hg^+)—its most hazardous form (Ma et al. 2019). Methylmercury is easily absorbed and tends to accumulate within living organisms, increasing in concentration as it moves up the food chain (Rand and Caito 2019). Even small amounts of exposure can damage the nervous, immune, Environmental Protection Agency (EPA) have set very strict limits for mercury in drinking water,

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and digestive systems (Zulaikhah et al. 2020). Both the World Health Organization (WHO) and the U.S. generally around 1–2 parts per billion (Ferreira da Silva and de Oliveira Lima 2020). Given these challenges, creating sensitive and selective systems for mercury detection is crucial. Fluorescence-based nanosensors have emerged as a promising solution because they enable fast analysis, are portable, and require minimal sample handling (Rasheed et al. 2018). Titanium dioxide (TiO_2) nanostructures—including nanoparticles, nanorods, nanotubes, and mesoporous films—have emerged as attractive platforms for chemical sensing due to their durability, biocompatibility, and surface functionalities that facilitate ligand attachment (Zhou et al. 2017). Their photocatalytic nature also enables self-cleaning and photoinduced signal enhancement (Rodríguez-González et al. 2020). However, early TiO_2-based Hg^{2+} sensors showed limited sensitivity and poor selectivity, largely because they relied on UV excitation and were susceptible to interference from natural water constituents (Macagnano et al. 2017). Subsequent advances involving noble metal modification and transition-metal doping improved optical activity and introduced defect-related fluorescence, yet challenges such as visible-light activation, matrix tolerance, and long-term stability persist (Zhang et al. 2019). These limitations have motivated the development of copper-doped and surface-capped TiO_2 nanomaterials. Copper doping has emerged as an effective strategy to enhance the electronic and optical performance of TiO_2 (Badawi and Althobaiti 2021). Introducing Cu^{2+} creates defect states and narrows the semiconductor band gap, allowing visible-light excitation and improving fluorescence efficiency that give rise to detectable fluorescence (Sittishoktram et al. 2020). Surface capping plays a crucial role in improving nanoparticle stability, dispersibility, and selectivity. Surface capping with hydroxy or amine group-based ligands enhances selectivity toward Hg^{2+} through strong metal–ligand interactions and improves colloidal stability in aqueous systems (Guan et al. 2020). Together, doping and capping offer complementary benefits, enabling highly sensitive, selective, and environmentally resilient detection suitable for field applications (Labeb et al. 2018). Titanium dioxide (TiO_2) nanoparticles are widely used in sensing applications due to their high surface area, chemical stability, and UV-responsive optical properties (Rashid and Tazri 2021). Their surfaces can be easily functionalized with selective ligands, enabling targeted detection of pollutant metal ions such as Hg^{2+} (Dai et al. 2021). Ethylenediamine binds to TiO_2 through its amine groups, controlling particle growth and aggregation while modifying surface charge (Fu et al. 2016). This results in smaller, more stable nanoparticles with altered optical properties and provides $–NH_2$ groups for further surface functionalization (Leong et al. 2021). Ethylene glycol binds to TiO_2 via hydroxyl groups, regulating particle formation and preventing aggregation (Jin et al. 2017). This leads to uniform, stable nanoparticles with tunable morphology, modified optical properties, and improved potential for subsequent surface functionalization (Fan et al. 2020). Triton binds to the TiO_2 surface as a non-ionic surfactant, limiting growth and aggregation of particles (Shondo et al. 2021). This produces uniformly dispersed, small nanoparticles with adjustable morphology, higher

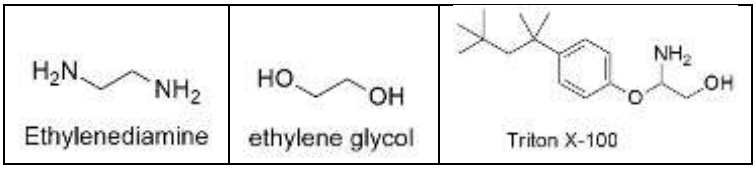

Figure1: Molecular Structure of capping agents ethylene glycol, ethylenediamine and Triton X-100

surface area, and improved stability and performance in surface-based applications (Gao et al. 2018). Prior studies have demonstrated that TiO_2 nanostructures functionalized with organic

dyes can achieve micromolar-level mercury detection (Parameswari et al. 2019). Several reports show that fluorescence quenching responses toward metal ions, attributed to charge-transfer interactions and trap-state modulation. Hydroxy capping agents, in particular, facilitate strong Hg–O interactions, producing highly selective turn-off fluorescence responses (Ding et al. 2015). Fluorescence-based detection with TiO_2 nanomaterials typically relies on mechanisms such as photoinduced electron transfer (PET), FRET, or inner filter effects, resulting in measurable quenching or enhancement (Muhr 2018).

The present research aims to exploit these synergistic effects to produce Cu-doped, ligand-capped TiO_2 nanoparticles capable of detecting mercury at ultra-trace levels, potentially down to the ppt range under simple blue LED excitation. The scope includes synthesizing doped TiO_2 through sol–gel, applying functional capping agents, and thoroughly characterizing the materials using optical, structural, and microscopic techniques. The sensor's fluorescence response will be evaluated across different environmental conditions and ion interferences. Enhanced selectivity over other metal ions has also been observed due to mercury's distinct redox interactions with Cu-modified surfaces. Key performance metrics—such as detection limit, linear range, selectivity, and response time—will be compared with conventional mercury detection technologies to assess advantages in sensitivity, portability, cost, and environmental compatibility.

3.1 Reagents and Solutions

All chemicals employed in this study were of analytical grade and were used without any further purification, as supplied by Sigma-Aldrich. Triply distilled water was utilized for preparing all solutions. Ethanol (absolute, >99.8%), tetrabutyl titanate ($Ti(OBu)_4$, 98%), hydrochloric acid (HCl, 37%), cupric acetate (≥98%), ethylene glycol (≥97%), ethylenediamine (≥98%), and Triton X-100 were also procured in analytical grade and used as received.

3.2 Apparatus

The characterization of the synthesized materials was carried out using different techniques including UV-visible spectroscopy, fluorescence spectroscopy, Fourier-Trans form Infrared (FT-IR) spectroscopy, Scanning Electron Microscopy (SEM). 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 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. The optical absorption spectra of all the samples in the triply distilled water were recorded using Shimadzu-1700 UV-Vis spectrophotometer. All fluorescence measurements were carried out using a Shimadzu RF-5301PC spectrophotofluorometer (Shimadzu, Japan) equipped with a 150 W Xenon Arc Lamp. The slit widths for measurements were adjusted to 5 nm for excitation and emission monochromators. Ultra sonication was performed on ultrasonic bath (Sarthak Scientific Services, India). All fluorescence spectra were recorded using Shimadzu RF-5301 PC spectrophotometer with a 150 Xenon short Arc Lamp as excitation source and using 1 cm path length quartz cells. The slit width of the excitation and emission

monochromators were both set at 10 nm. The excitation and emission widths for measurements were adjusted to 10 nm. All optical measurements were carried out under ambient temperature.

3.3 Methods for synthesis of Cu-doped TiO_2 nanomaterials:

Synthesis of Cu doped Ethyleneglycol/Ethylenediamine/Triton capped TiO_2 (Ethyleneglycol@Cu-TiO_2, Ethylenediamine@Cu-TiO_2, Triton@Cu-TiO_2)

Ethanol and titanium isopropoxide were first mixed in a 2:1 volume ratio. Separately, ethylene glycol/ethylenediamine/Triton along with hydrochloric acid and distilled water were taken in a 1:1:15 volume ratio and placed in a separatory funnel. An appropriate amount of copper nitrate solution (1M), corresponding to 4 mol % with respect to titanium, was added to this mixture. The resulting solution was then introduced dropwise into the titanate solution. The reaction mixture was heated at 200 °C for 10 min, during which hydrolysis and subsequent condensation reactions involving the elimination of water and alcohol took place, leading to the formation of TiO_2 precursors. These precursors underwent dissolution, nucleation, growth, and crystallization, yielding TiO_2 products. Finally, the synthesized TiO_2 was washed three times with distilled water and dried in an oven at 100 °C for 4 h.

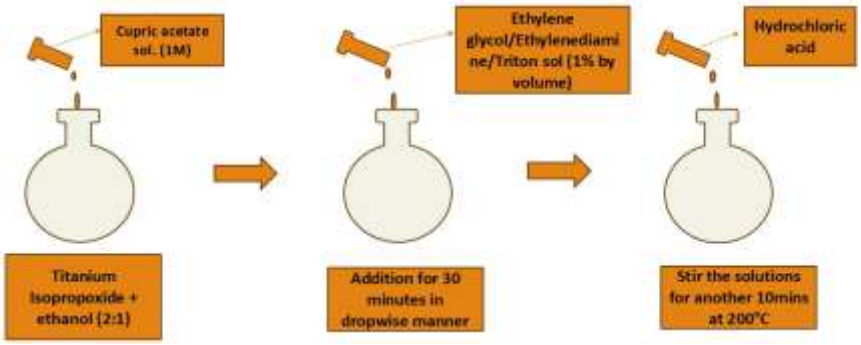

Figure 2: Schematic diagram showing method of Synthesis of Ethyleneglycol@Cu-TiO_2/ Ethylenediamine@Cu-TiO_2, Triton@Cu-TiO_2.

4. Characterization techniques for capped Cu- doped TiO_2

The characterization of nanostructures is essential for understanding their optical and morphological properties and assessing their suitability for specific applications. A comprehensive evaluation of nanoparticles requires the use of multiple analytical and spectroscopic techniques. In the present study, the characterization of the synthesized nanoparticles was carried out using the following methods:

4.1 Fourier Transform Infrared (FTIR) spectroscopy

The FTIR spectra of the ethyleneglycol@Cu–TiO_2, ethylenediamine@Cu-TiO_2, Triton@Cu-TiO_2 nanoparticles are shown in Figure 2 and presents characteristic absorption bands confirming the successful capping of ethyleneglycol, ethylenediamine, Triton molecules on surface of Cu–TiO_2 nanoparticles. The broad and intense band observed at 3299 cm^{-1}, 3292 cm^{-1}, 3262 cm^{-1} in the spectra of Triton@Cu-TiO_2, ethyleneglycol@Cu–TiO_2, ethylenediamine@Cu-TiO_2, corresponds to the stretching vibration of hydroxyl (–OH) groups of ethylene glycol coordinated with TiO_2. A distinct peak at 2112 cm^{-1}, 2124 cm^{-1}, 2130 cm^{-1} may be attributed to C–H stretching of Ethylene chain of the triton, ethyleneglycol and

ethylenediamine capping agent. The absorption at 1638 cm^{-1}, 1628 cm^{-1}, 1627 cm^{-1} corresponds to the O-H bending vibration. Peaks appearing at 1045 cm^{-1}, 1084 cm^{-1} and 1099 cm^{-1} indicate C–O/C-N stretchings (Urmi et al. 2020). The strong metal–oxygen stretching band at 688 cm^{-1}, 698 cm^{-1}, 699 cm^{-1} is characteristic of Ti–O and Cu–O lattice vibrations. So, the FTIR results confirm the successful anchoring of Triton /ethylene glycol/ Ethylenediamine onto the surface of Cu–TiO_2 nanoparticles.

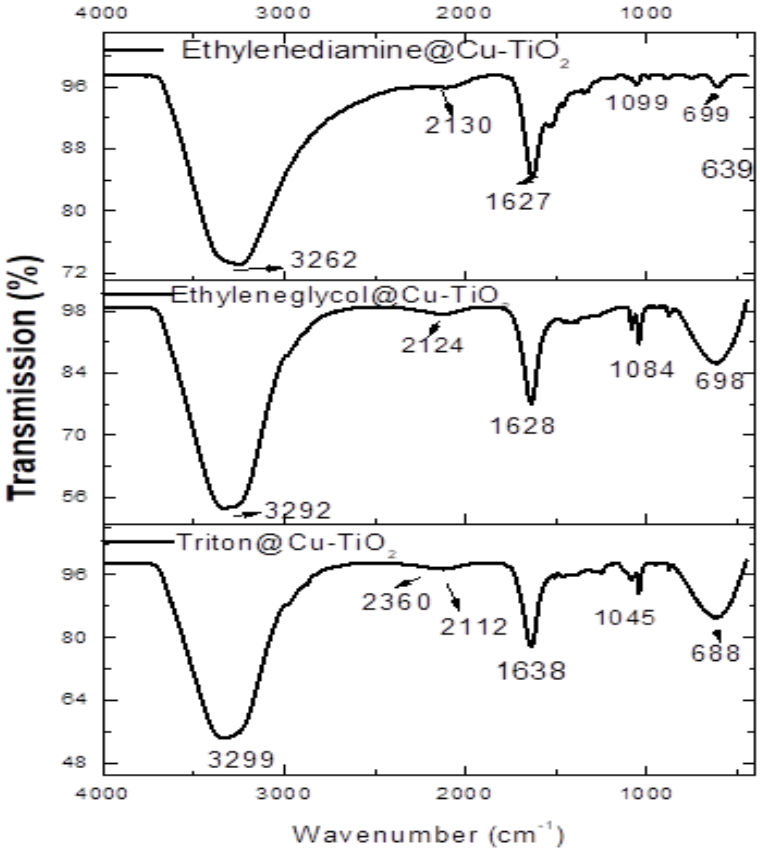

Figure 3: FTIR spectra of Ethyleneglycol@Cu-TiO_2 nanoparticles, Triton@Cu-TiO_2, and Ethylenediamine@Cu-TiO_2

4.2 UV- Visible spectroscopy

The UV–Visible spectra of ethyleneglycol@Cu–TiO_2, ethylenediamine@Cu–TiO_2, and Triton@Cu–TiO_2 nanoparticles were recorded, and the corresponding absorption maxima (λ_{max}) were observed at 276 nm, 279 nm, and 237 nm, respectively (Figure 3). All the capped Cu–TiO_2 nanoparticles exhibited a noticeable red shift in comparison with bulk TiO_2 (387 nm), indicating enhanced light-harvesting capability due to surface modification and Cu incorporation (Singh and Mehata 2019). The optical band gap (Eg) of the synthesized nanoparticles was evaluated from the UV–Visible absorption data using the Tauc relation, which is expressed as:

$$(\alpha h\nu)^{1/n} = A\,(Eg - h\nu) \qquad \ldots\ldots (i)$$

where *A* is a constant related to the transition probability, α is the absorption coefficient, *hv* is the photon energy, and *n* characterizes the nature of the electronic transition. For the capped TiO_2 nanoparticles, n = 2, corresponding to an allowed direct transition. From the Tauc plots (Figure 4a, 4b, 4c), the estimated optical band gap values for ethylene glycol@Cu–TiO_2, ethylenediamine@Cu–TiO_2, and Triton@Cu–TiO_2 are 3.0 eV, 3.0 eV, and 2.81 eV, respectively. The reduction in band gap, particularly for Triton-capped Cu–TiO_2, suggests improved visible-light response resulting from surface capping and Cu doping.

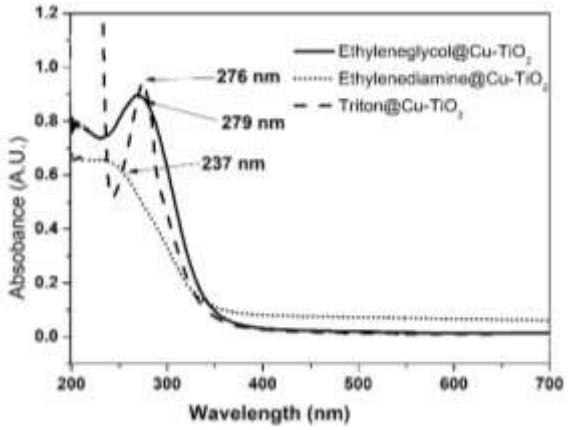

Figure 4: UV-Visible spectra of Cu- TiO_2 nanoparticles with capping agents.

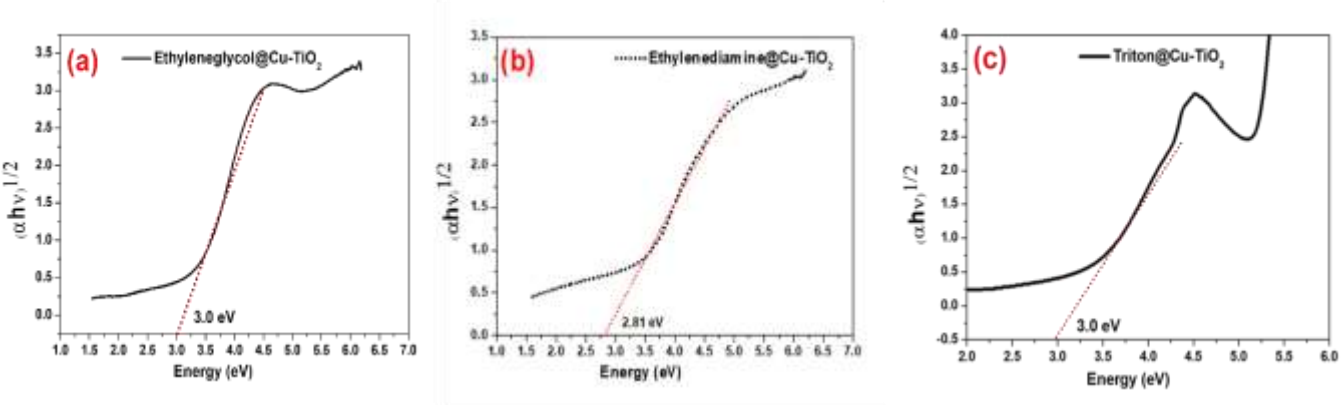

Figure 5: UV bandgap analysis of Ethyleneglycol@Cu-TiO_2 nanoparticles (a), ethylenediamine@Cu-TiO_2 (b), and Triton@Cu-TiO_2 (c) by using Tauc plot.

4.3 Scanning electron microscopy (SEM)

The surface morphology of the synthesized Cu-TiO_2 nanomaterials was examined using SEM. Distinct morphological variations were observed depending on the surfactant/capping agent employed during synthesis. The sample prepared using ethylene glycol (Figure 5a) exhibits predominantly elongated, rod-like, and plate-shaped particles, indicating anisotropic crystal growth. Ethylene glycol acts as a mild reducing and capping agent, slowing ion diffusion and enabling controlled nucleation. As a result, the particles show moderate agglomeration but retain well-defined anisotropic features. In contrast, the ethylenediamine-assisted sample (Figure 5b) displays dense agglomerates composed of fine, spherical nanoparticles. Ethylenediamine, a strong bidentate chelating ligand, coordinates efficiently with metal ions,

facilitating rapid nucleation and the formation of numerous ultrafine crystallites (Tvspv et al. 2020). This high nucleation rate suppresses directional growth and leads to the formation of compact, cluster-like aggregates with a rough surface texture (Singh and Dutta 2018). The sample synthesized in the presence of Triton X-100 (Figure 5c) reveals irregular polyhedral and block-like nanostructures, with a comparatively lower degree of agglomeration than the ethylenediamine sample. Triton X-100, a nonionic surfactant, forms micellar structures that provide steric hindrance and moderate control over nucleation and particle growth.

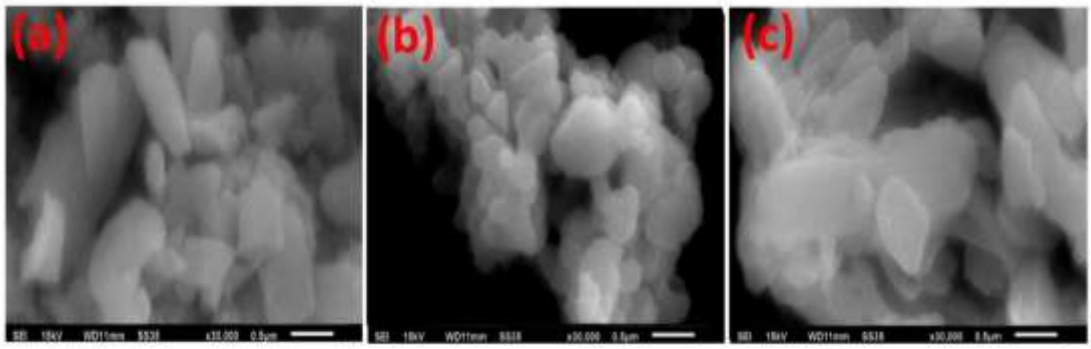

Figure 6: Figure: FSEM images showing morphology of ethyleneglycol@Cu–TiO_2 (a), ethylenediamine@Cu–TiO_2(b), and Triton@Cu–TiO_2(c) nanoparticles.

The approximated sizes were 18 nm, 24 nm and 35 nm for ethyleneglycol@Cu–TiO_2**,** ethylenediamine@Cu–TiO_2**,** and Triton@Cu–TiO_2 nanoparticles respectively. Overall, the SEM analysis confirms that the choice of surfactant significantly influences the nucleation kinetics, growth directionality, and aggregation behavior of Cu–TiO_2 nanoparticles.

4.4 Fluorescence spectra

The bulk TiO_2 are not fluorescent but due to its nano range or size and by doping it with copper it becomes fluorescent. The fluorescence spectra of Cu doped TiO_2 nanoparticles were recorded at an excitation wavelength 320 nm. The λ_{max} for Ethyleneglycol@Cu-TiO_2, Triton@Cu-TiO_2, Ethylenediamine@Cu-TiO_2 were obtained at 344.4nm, 350.2nm, and 342.2nm respectively.

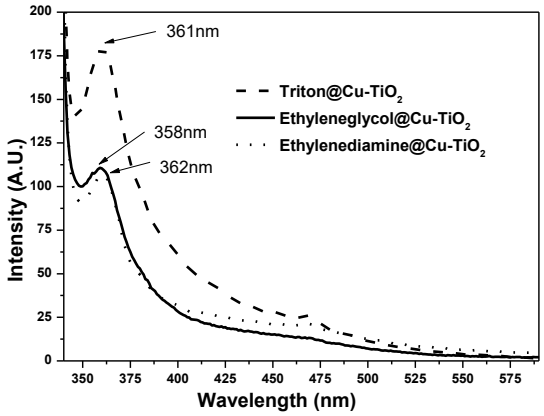

Figure 7: Fluorescence spectra of Cu- TiO_2 nanoparticles with ethyleneglycol, ethylenediamine and Triton as capping agents.

The fluorescence shown by all capped samples is attributed to the synthesis in nano range and surface defects on the nanoparticles (Mustapha et al. 2021). This enhancement in fluorescence could be due to better charge carrier separation as dopant copper acts as electron sink, thus reducing recombination rate of electron and hole. The highest intensity is observed in case of Triton@Cu-TiO_2, which depicts strong emission of light due to efficient absorption and radiative relaxation of excited molecules. Also, it indicates that decreased particle size, well crystallized nanoparticles and optimum dopant concentration.

4.5 Application of Capped Cu-TiO_2 nanoparticles

4.5.1 Application of Hg sensing with Ethyleneglycol@Cu-TiO_2

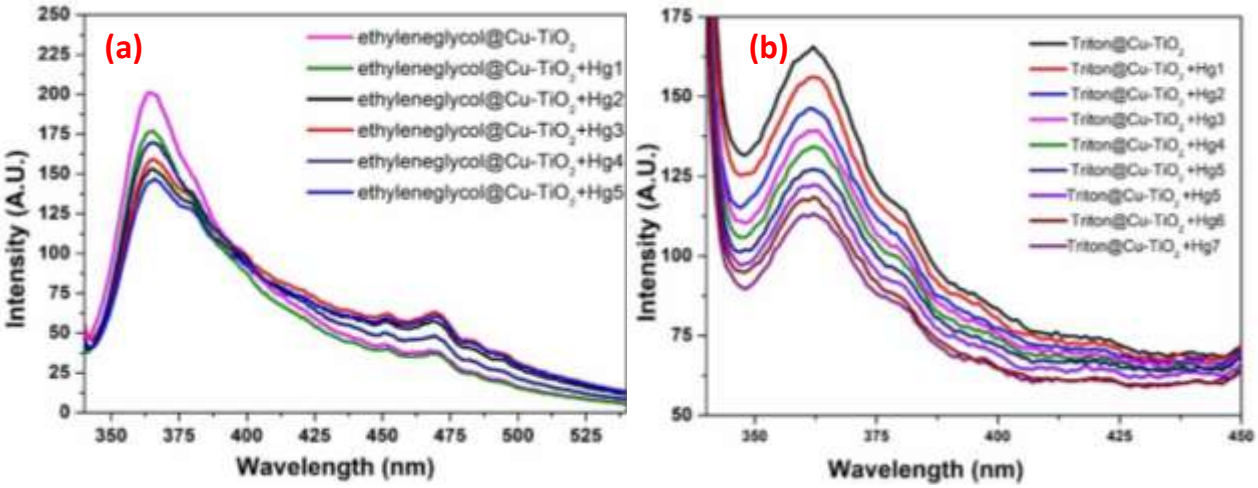

Figure 8: Fluorescence Quenching of signal by addition of Hg(II) ions in the sample of ethyleneglycol@Cu-TiO_2 and ethylenediamine@Cu-TiO_2

Ethylene glycol contains hydroxyl (-OH) groups, which can bind Hg (II) ions via coordination. Upon binding, Hg (II) acts as an electron acceptor, facilitating charge transfer from TiO_2 to Hg (II) (Al Shehab and Patra 2021) . This process reduces the radiative recombination of excitons, leading to fluorescence quenching. The quenching was observed up to the concentration of 7.8µM concentration. Further no more decrease in concentration was observed.

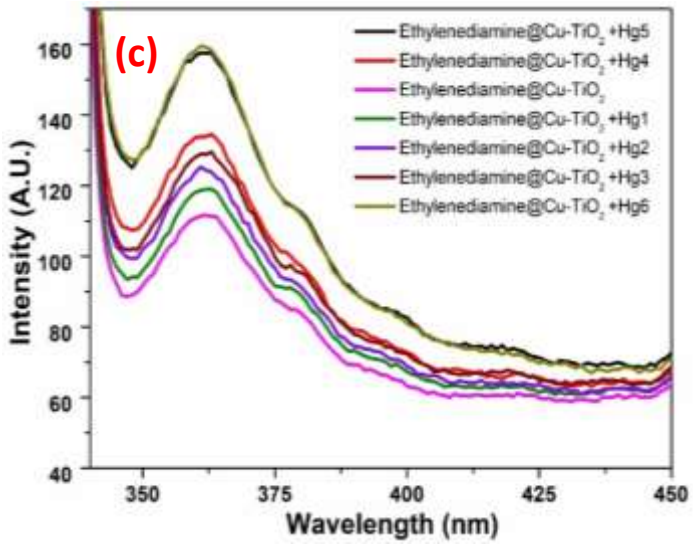

Figure 9: Enhancement in the signal by addition of Hg(II) ions in the sample of Triton@Cu-TiO_2

In case of Ethylenediamine@Cu-TiO_2, Ethylenediamine possesses two amino groups bearing lone-pair electrons, which enable it to function as an effective chelating ligand. Hg (II), being a soft Lewis acid, shows a strong tendency to bind with nitrogen-containing donors (Wang et al. 2020). Upon introduction of Hg(II), it preferentially complexes with the ethylenediamine molecules anchored on the nanoparticle surface (Wang et al. 2019). This interaction alters the surface passivation and generates additional defect or trap sites, which favour non-radiative charge recombination pathways, resulting in a pronounced quenching of fluorescence (Dey 2020). The quenching was observed up to the concentration of 12.5μM concentration. Further no more decrease in fluorescence intensity was observed.

For Triton-capped Cu-doped TiO_2 nanoparticles, the presence of Hg(II) results in an increase in fluorescence intensity by passivating surface defects and minimizing non-radiative recombination processes (Rajabathar et al. 2021). At the same time, the lack of strong Hg(II)–ligand coordination and inefficient photoinduced electron transfer pathways prevent emission quenching (Ahmed et al. 2019). The enhancement in fluorescence signal is up to the limit of 9.4μM.

5. CONCLUSION

This work demonstrates that surface capping plays a decisive role in regulating the physicochemical characteristics and Hg^{2+} sensing performance of Cu-doped TiO_2 nanoparticles. Combined FTIR, UV–Visible, SEM, and fluorescence investigations confirm that ethylene glycol, ethylenediamine, and Triton X-100 effectively modulate surface functionalities, particle morphology, size distribution, and defect states in Cu–TiO_2 nanostructures. These capping-induced variations are directly reflected in their optical behavior and interaction with mercury ions. Nanoparticles capped with ethylene glycol and ethylenediamine showed marked fluorescence quenching in the presence of Hg^{2+}, attributed to coordination-assisted charge transfer and enhanced non-radiative recombination, whereas Triton-capped samples exhibited fluorescence enhancement resulting from efficient passivation of surface defects.

The sensing performance clearly shows that surface-modified TiO_2 nanoparticles therefore represent a stable, low-cost, and eco-friendly sensing platform with advantages over conventional materials. Further studies should focus on dopant optimization, recyclability, and testing in real wastewater systems.

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