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Synthesis and Characterizations of CeO₂-TiO₂ Nanocomposites for Gas Sensing Applications

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ABSTRACT

TiO₂-CeO₂ nanocomposites were successfully synthesized using the sol-gel method, which is a flexible, cost-effective, and widely adopted chemical synthesis technique known for producing highly homogeneous and ultra-pure nanostructured materials. The sol-gel process allows precise control over composition, particle size, and microstructure, making it particularly suitable for preparing mixed metal oxide nanocomposites. In recent years, the TiO₂-CeO₂ system has attracted considerable research interest due to its synergistic physicochemical properties, which enhance its performance in applications such as photocatalysis, environmental remediation, gas sensing, and energy-related technologies. In the present study, titanium isopropoxide and cerium nitrate were used as precursor materials for titanium and cerium sources, respectively. These precursors were hydrolyzed under carefully controlled pH and temperature conditions to obtain TiO₂-CeO₂ nanocomposites with varying molar ratios of TiO₂ and CeO₂. Controlled hydrolysis and condensation reactions ensured uniform distribution of CeO₂ within the TiO₂ matrix. The resulting sols gradually transformed into gels, which were subsequently aged to improve network formation and structural stability. X-ray diffraction (XRD) analysis confirmed the successful formation of anatase TiO₂ and fluorite-structured CeO₂ phases. The absence of impurity peaks in the diffraction patterns indicated high phase purity and effective incorporation of CeO₂ into the TiO₂ lattice. The crystallite size of the nanocomposites was found to vary between 10 and 25 nm, depending on the CeO₂ content and the calcination temperature, suggesting that cerium doping influenced crystal growth and inhibited particle agglomeration. Fourier-transform infrared spectroscopy (FTIR) was employed to analyze the chemical bonding and functional groups present in the nanocomposites. The FTIR spectra exhibited characteristic metal-oxygen stretching vibrations corresponding to Ti-O-Ti, Ce-O, and Ti-O-Ce bonds. These features confirmed strong chemical interactions and successful coupling between TiO₂ and CeO₂ within the nanocomposite structure. Field emission scanning electron microscopy (FE-SEM) revealed nearly spherical nanoparticles with relatively uniform size distribution and reduced agglomeration. UV-visible diffuse reflectance spectroscopy (UV-Vis DRS) indicated enhanced optical absorption and band-gap modification due to CeO₂ incorporation.

1. Introduction

Titanium dioxide (TiO₂) is one of the most widely investigated photocatalysts for the degradation of organic pollutants due to its high photochemical stability, strong oxidizing capability, non-toxicity, low cost, and abundant availability [1]. TiO₂ exists mainly in three crystalline polymorphs: anatase, rutile, and brookite. Among these, anatase typically exhibits superior photocatalytic performance, attributed to its larger specific surface area and reduced rate of photoinduced charge carrier recombination [2,3]. However, the practical efficiency of pristine TiO₂ remains limited by its wide band gap (~3.2 eV), which restricts light absorption predominantly to the ultraviolet region of the solar spectrum, representing only a small fraction of incident solar energy [4,5]. Moreover, the rapid recombination of photogenerated electron-hole pairs significantly diminishes quantum efficiency [6]. To address these limitations, various modification strategies have been explored, including morphological control, surface sensitization, ion doping, and coupling with other semiconductors [7]. Among these approaches, forming heterostructures with cerium dioxide (CeO₂) has emerged as a promising route for enhancing photocatalytic activity [8]. CeO₂, a lanthanide oxide with reversible Ce³⁺/Ce⁴⁺ redox behaviour and high oxygen storage capacity, can facilitate charge separation and promote interfacial charge transfer when integrated with TiO₂ [9]. The creation of TiO₂-CeO₂ heterojunctions not only suppresses electron-hole recombination but also extends optical absorption toward the visible region [10,11]. Additionally, oxygen vacancies and defect sites introduced by CeO₂ contribute to

improved adsorption and surface reactivity [12]. In this context, the sol-gel method offers a versatile synthesis route, enabling precise control over composition, homogeneity, and particle size at relatively low temperatures [13]. The present study focuses on the sol-gel synthesis, characterization, and photocatalytic evaluation of CeO₂/TiO₂ nanocomposites to elucidate the influence of cerium incorporation on structural, optical, and photocatalytic properties [14]. It also has a high oxygen storage capacity. By creating heterojunctions at the TiO₂-CeO₂ interface, the addition of CeO₂ to TiO₂ not only increases light absorption into the visible spectrum but also efficiently reduces electron-hole pair recombination [15]. This cooperative relationship improves the composite material's photocatalytic effectiveness and helps keep it stable over several catalytic cycles [16]. Additionally, CeO₂ can introduce defect sites and oxygen vacancies that act as active foci for adsorption and interaction with pollutant molecules, increasing the total photocatalytic activity [17]. Due to its ability to precisely control composition, homogeneity, and particle size at relatively low temperatures, the sol-gel method of synthesizing TiO₂-CeO₂ nanocomposites has garnered increased attention in recent years [18,19].

2. Experimental Methods

2.1 Synthesis of TiO₂-NPs

Nitric acid (HNO₃) and titanium tetra isopropoxide (TTIP, Sigma-Aldrich, 97%) were used as received without further purification. Ce(NO₃)₃·6H₂O and titanium butoxide [Ti(OBu)₄] served as cerium and titanium precursors, respectively. Initially, 10 mL of Ti(OBu)₄ was dissolved in 10 mL of anhydrous alcohol and ultrasonically dispersed. The

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Ti precursor solution was then added dropwise to the $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ solution at the desired Ce/Ti molar ratio (1/0.05/0.10). The mixture was stirred for 2 h at room temperature, and the pH was adjusted to 10 using ammonia solution, resulting in the formation of a yellow sol. The sol was aged for 24 h, followed by filtration, washing with deionized water and anhydrous alcohol, and drying at 100 °C for 12 h. The dried precursor was calcined at 600 °C for 4 h in air to obtain nanocrystalline $\text{CeO}_2/\text{TiO}_2$ composite powder.

2.2 Synthesis of CeO_2 NPS

Cerium oxide (CeO_2) nanoparticles were synthesized via the sol-gel technique using cerium nitrate hexahydrate, Cerium nitrate hexahydrate [$\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$], as the precursor. Initially, 4.34 g of $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (molar mass $\approx 434.22 \text{ gmol}^{-1}$) was accurately weighed and dissolved in 100 mL of deionized water under continuous magnetic stirring at room temperature to obtain a 0.1 M solution. A suitable chelating/stabilizing agent such as citric acid (molar ratio 1:1 with cerium nitrate; $\sim 1.92 \text{ g}$) or ethylene glycol (few mL, depending on desired viscosity) was added to promote homogeneous mixing and regulate hydrolysis. The solution was stirred until a clear and uniform sol was formed. The sol was then aged at room temperature for 12–24 h to allow gel formation via polycondensation reactions. The resulting gel was dried at 80–100 °C for 10–12 h to remove residual solvent and moisture. Finally, the dried gel was calcined at 600 °C for 3 h in air to obtain crystalline CeO_2 nanoparticles. The calcination step enabled decomposition of organic constituents and crystallization of CeO_2 . The synthesized nanoparticles typically exhibit high crystallinity, nanoscale particle size, and abundant oxygen vacancies, which are advantageous for catalytic and gas-sensing applications.

2.3 Synthesis of $\text{CeO}_2\text{-TiO}_2$ Nanocomposites

$\text{CeO}_2/\text{TiO}_2$ composite nanoparticles were synthesized via a controlled sol-gel method using titanium(IV) butoxide ($\text{Ti}(\text{O}i\text{Bu})_4$) and cerium nitrate hexahydrate ($\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$) as titanium and cerium precursors, respectively. Initially, $\text{Ti}(\text{O}i\text{Bu})_4$ was dissolved in anhydrous alcohol under continuous stirring and ultrasonication to ensure complete homogenization. Separately, an aqueous solution of $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ was prepared and added dropwise to the Ti precursor solution while maintaining the desired Ce/Ti molar ratio. The slow addition facilitated uniform mixing and controlled hydrolysis during sol formation. The pH of the reaction mixture was adjusted to ~ 10 using ammonia solution to promote gelation. The resulting gel was filtered and thoroughly washed with deionized water and anhydrous alcohol to remove residual impurities and unreacted species. Finally, the purified gel was dried at 100 °C for 12 h to obtain $\text{CeO}_2/\text{TiO}_2$ composite nanoparticles.

3. Results and Discussion

3.1 UV-Visible Analysis

The UV-Visible absorption behaviour of $\text{CeO}_2\text{-TiO}_2$ nanoparticles is presented in Fig. 1. The UV-Vis absorption spectrum of the $\text{CeO}_2\text{-TiO}_2$ nanocomposite exhibits a strong and broad absorption band in the deep-UV region, beginning around 200 nm with an absorbance of approximately 1.45–1.50, which reflects the intrinsic electronic transitions of both TiO_2 and CeO_2 . The high absorption intensity arises mainly from $\text{O}^{2-} \rightarrow \text{Ti}^{4+}$ and $\text{O}^{2-} \rightarrow \text{Ce}^{4+}$ charge-transfer transitions, and it signifies the formation of nanoscale semiconductor domains with a wide band gap. As the wavelength increases, a gradual decrease in absorbance occurs, followed by a prominent drop near 350–380 nm, corresponding to the fundamental absorption edge of the composite. This region represents the combined band-gap behavior of TiO_2 ($\approx 3.2 \text{ eV}$) and CeO_2 ($\approx 3.1\text{--}3.2 \text{ eV}$).

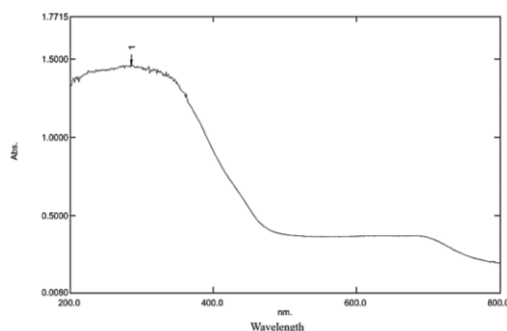


Fig. 1 UV-visible absorption spectrum of synthesized $\text{CeO}_2\text{-TiO}_2$ nanoparticles

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The slight tailing of the absorption edge into the visible region indicates the presence of Ce-induced defect states, oxygen vacancies, and electronic coupling at the $\text{CeO}_2\text{-TiO}_2$ interface. Such effects often result in marginal band-gap narrowing, improved light harvesting, and enhanced charge separation. Beyond 450 nm, the spectrum flattens at a lower absorbance level, with a weak hump observed near 700–750 nm, which can be attributed to defect-related transitions or localized Ce^{3+} states. Overall, the UV-Vis profile confirms successful formation of the $\text{CeO}_2\text{-TiO}_2$ nanocomposite, improved interaction between the two oxide phases, and the presence of structural defects that can significantly enhance photocatalytic activity, UV-driven processes, and gas-sensing performance due to better electron-hole separation and increased surface reactivity.

3.2 FE-SEM Analysis

The surface morphology and particle size distribution of the synthesized $\text{CeO}_2\text{-TiO}_2$ nanoparticles are shown in Fig. 2. The FE-SEM micrographs of the $\text{CeO}_2\text{-TiO}_2$ nanocomposite exhibit a highly agglomerated surface morphology composed of densely packed nanosized particles. At lower magnification, the sample shows irregular clusters and grain-like structures, indicating strong inter-particle interaction driven by high surface energy and the inherent tendency of CeO_2 and TiO_2 nanoparticles to agglomerate. The TiO_2 component appears as flower-like or plate-like crystalline aggregates in some regions, while CeO_2 nanoparticles form smaller granular structures surrounding and interconnecting these clusters. This mixed morphology confirms successful composite formation, where TiO_2 crystallites act as a structural backbone while CeO_2 nanoparticles deposit on or between TiO_2 surfaces, increase surf roughness and active sites.

Higher-magnification images further reveal that the nanocomposite consists of uniformly distributed spherical to near-spherical nanoparticles with an average particle size in the range of $\sim 20\text{--}33 \text{ nm}$, as indicated by the measurements shown in the images (values such as 21.6, 22.9, 24.3, 25.6, 27.0, 28.4, 33.0 nm). The particle size variation suggests the coexistence of both CeO_2 and TiO_2 nanoscale domains, where TiO_2 typically forms slightly larger crystalline grains and CeO_2 contributes smaller particles. The rough and porous texture observed throughout the microstructure indicates high surface area, which is beneficial for catalytic and gas-sensing applications, because it allows easier gas diffusion and more active adsorption sites.

Overall, the FE-SEM analysis confirms the successful synthesis of the $\text{CeO}_2\text{-TiO}_2$ nanocomposite with nanoscale particle distribution, heterogeneous morphology, and strong agglomeration. The combination of granular CeO_2 nanoparticles with plate- or flower-like TiO_2 structures significantly enhances the surface complexity, which is expected to improve gas sensitivity and catalytic reactivity due to better charge transfer across $\text{CeO}_2\text{-TiO}_2$ heterojunctions and increased surface-active regions.

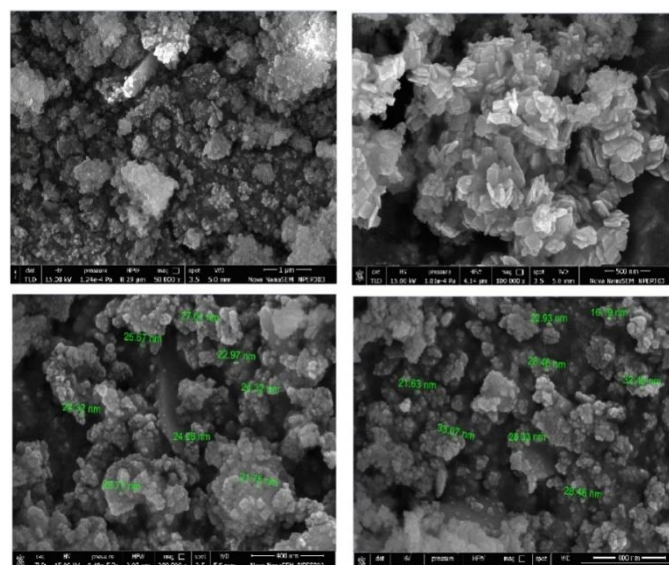


Fig. 2 SEM micrographs of synthesized $\text{CeO}_2\text{-TiO}_2$ nanoparticles

3.3 EDS Analysis

The EDS spectrum clearly displays characteristic peaks corresponding to cerium (Ce), titanium (Ti), and oxygen (O), confirming the successful formation of the $\text{CeO}_2\text{-TiO}_2$ nanocomposite without any notable impurity elements. A strong Ti peak appears around 4.5 keV, along with a secondary

Ti signal near 3.2 keV, both corresponding to Ti K-series emissions typical of TiO₂. Cerium shows multiple well-defined peaks in the 0.9–1.2 keV and 4.8–5.5 keV regions, indicating the presence of Ce L α and Ce L β transitions, which are expected for CeO₂. Oxygen contributes a prominent low-energy peak near 0.5 keV, confirming oxide formation and matching the expected stoichiometry for mixed metal oxides.

The proportional intensities of Ce, Ti, and O peaks suggest that both metal oxides are well-distributed within the sampled region, supporting the FE-SEM observations that showed an integrated and uniform composite structure. The absence of unwanted elements such as carbon, sodium, chlorine, or transition-metal contaminants indicates that the synthesis process was clean and free from residual precursors. The elemental composition was confirmed by EDX analysis (Fig. 3).

Overall, the EDS analysis provides strong evidence for the successful incorporation and coexistence of CeO₂ and TiO₂ within the nanocomposite, reinforcing its suitability for applications like gas sensing, catalysis, and UV-driven processes where purity and elemental homogeneity significantly influence performance.

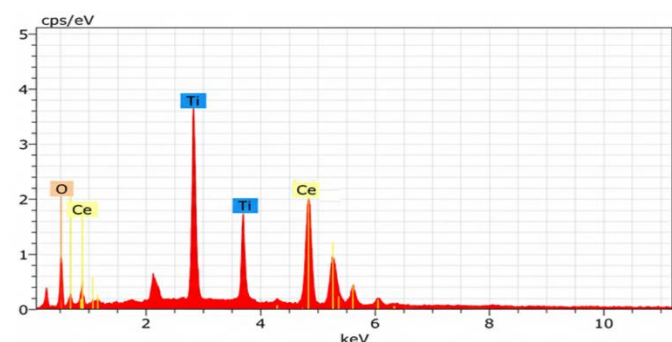


Fig. 3 Energy dispersive X-ray (EDX) spectrum of synthesized TiO₂-CeO₂ nanocomposite showing characteristic peaks of Ti, Ce, and O elements, confirming the elemental composition of the prepared material

3.4 FT-IR Analysis

The FT-IR spectrum of the synthesized CeO₂-TiO₂ nanocomposite displays characteristic vibrational bands that confirm the formation of the mixed-oxide structure and the presence of metal-oxygen linkages. The formation of metal-oxygen bonds was confirmed by FTIR analysis (Fig. 4).

A broad absorption band around 3430–3690 cm⁻¹ corresponds to O-H stretching vibrations, which originates from physically adsorbed water molecules and surface hydroxyl functional groups. These hydroxyls are commonly present on the surface of metal oxides and play an important role in enhancing gas-sensing and photocatalytic responses by providing active adsorption sites. A weak band near 3100–3000 cm⁻¹ may be associated with C-H stretching or residual organic traces from precursors or solvents used during synthesis.

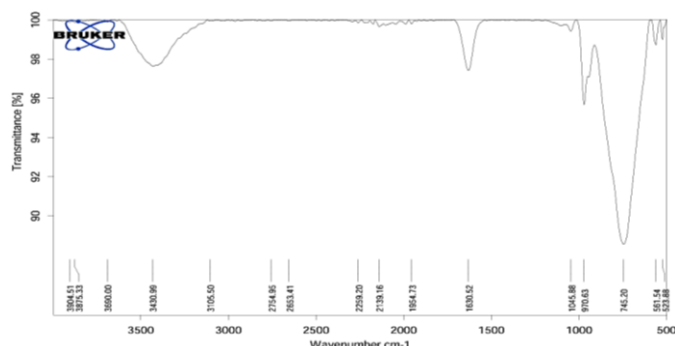


Fig. 4 FTIR spectrum of synthesized TiO₂-CeO₂ nanocomposite recorded in the range 4000–500 cm⁻¹ showing characteristic metal-oxygen vibrational bands

The spectrum shows a minor but noticeable feature around 1630 cm⁻¹, which corresponds to H-O-H bending vibrations of molecular water bound to the nanoparticle surface. The small peaks at 2139 cm⁻¹ and 1954 cm⁻¹ may be attributed to atmospheric CO₂ absorption or weak overtones from surface species, commonly detected in metal-oxide FTIR spectra. No strong organic functional groups (such as C=O or C-N) are observed, indicating successful removal of organic residues during calcination.

The fingerprint region from ~400–800 cm⁻¹ shows strong and broad transmittance dips, which are the most significant features of the spectrum. Peaks around 745 cm⁻¹, 970–1045 cm⁻¹, and the intense band at ~520–560 cm⁻¹ correspond to combined Ti-O, Ti-O-Ti, and Ce-O stretching modes. The broad metal-oxygen absorption in this region is

typical for nanosized CeO₂ and TiO₂, and the overlapping nature of these bands indicates the formation of an integrated CeO₂-TiO₂ nanocomposite. The slight shift in Ce-O and Ti-O bands relative to pure oxides suggests lattice distortion and strong interfacial interaction between Ce⁴⁺ and Ti⁴⁺ ionic sites, confirming composite formation at the nanoscale.

Overall, the FT-IR spectrum confirms the successful synthesis of the CeO₂-TiO₂ nanocomposite. The presence of strong metal-oxygen bands, surface hydroxyl groups, and the absence of undesired organic impurities affirm the purity of the sample and validate the structural integration of the two metal oxides. These structural features enhance surface reactivity, making the material suitable for applications in photocatalysis and gas sensing due to improved adsorption and charge-transfer capabilities.

3.5 XRD Analysis

The XRD pattern of the CeO₂-TiO₂ nanocomposite displays a prominent and sharp diffraction peak centered near 2 θ \approx 38 $^\circ$, indicating the presence of a highly crystalline phase. The additional smaller peaks between 30 $^\circ$ –35 $^\circ$, 55 $^\circ$ –60 $^\circ$, and 65 $^\circ$ –70 $^\circ$ correspond to mixed phases of CeO₂ and TiO₂, suggesting successful incorporation of both metal oxides. The intense peak around 38 $^\circ$ may correspond to the (111) plane of CeO₂ or the (004)/(112) reflections of TiO₂ depending on the exact structural ratio, indicating the dominance of one crystalline phase.

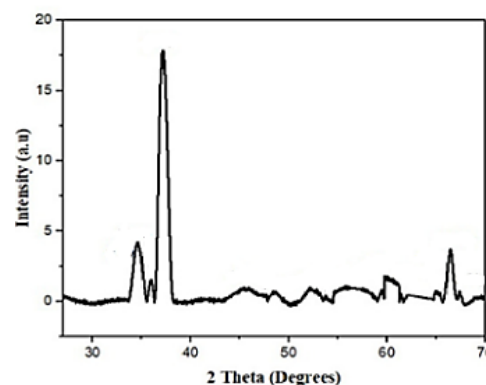


Fig. 5 X-ray diffraction (XRD) pattern of synthesized TiO₂-CeO₂ nanocomposite

The slight broadening of some peaks suggests nanocrystalline behaviour with small particle size, consistent with FE-SEM observations showing particle sizes around 20–33 nm. The crystalline structure was confirmed by XRD analysis (Fig. 5) The absence of impurity-related peaks confirms the high purity of the synthesized composite. The structural features, combined with peak broadening, imply lattice strain and strong CeO₂-TiO₂ interfacial interaction, which can significantly enhance photocatalytic and gas-sensing activity by facilitating charge separation and increasing defect density.

3.6 Gas Sensing Results

The CeO₂-TiO₂ nanocomposites exhibit excellent gas-sensing performance toward multiple gases, showing strong temperature-dependent sensitivity behavior. As the operating temperature increases from 40 $^\circ$ C to 200 $^\circ$ C, the overall sensitivity improves significantly. Among all tested gases, NO₂ shows the highest response, reaching more than 70% sensitivity at 200 $^\circ$ C, indicating strong oxidizing-reducing interactions between NO₂ molecules and the CeO₂-TiO₂ surface. H₂S also shows a notable response that gradually increases with temperature, achieving around 40% sensitivity at 200 $^\circ$ C. The temperature-dependent sensitivity is shown in Fig. 6a.

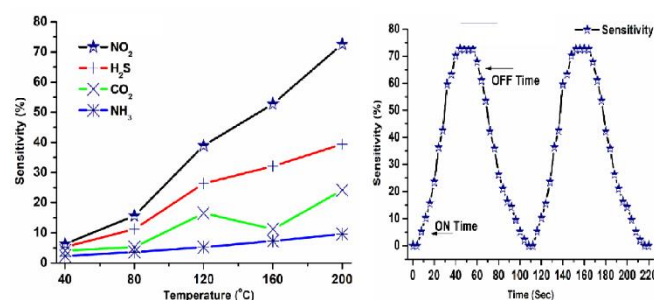


Fig. 6 a) Variation of gas sensitivity (%) of TiO₂-CeO₂ nanocomposite sensor toward NO₂, H₂S, CO₂, and NH₃ gases as a function of operating temperature (40–200 $^\circ$ C); and b) Dynamic response-recovery characteristics of the TiO₂-CeO₂ gas sensor showing sensitivity variation with time and indicating ON and OFF response cycles

CO₂ and NH₃ display moderate responses, with CO₂ showing a maximum near 120 °C followed by slight decline, while NH₃ shows a steadily increasing but comparatively lower sensitivity. This selective enhancement shows that the nanocomposite surface chemistry favours strong adsorption and reaction with NO₂ and H₂S gases.

Dynamic response curves further confirm the fast response and recovery behaviour of the CeO₂-TiO₂ sensor. During the ON cycle, sensitivity rapidly rises to its peak (~70%), demonstrating quick adsorption kinetics. In the OFF cycle, the signal smoothly returns to baseline, indicating efficient desorption and excellent reversibility. The dynamic sensing behaviour is illustrated in Fig. 6b. Repeated ON/OFF cycles maintain nearly identical peak heights, confirming high stability, reproducibility, and reliable long-term sensing performance. Overall, the CeO₂-TiO₂ nanocomposites show high sensitivity, fast response-recovery times, selective detection behaviour, and good cycling stability, making them promising candidates for practical gas-sensing applications, especially for NO₂ and H₂S detection.

4. Conclusion

Crystalline CeO₂/TiO₂ nanocomposites were synthesized via the sol-gel method and systematically characterized. XRD analysis confirmed well-defined crystalline phases with average crystallite sizes of 14–17 nm. At Ce/Ti ratios below 8/100, no distinct CeO₂ peaks were observed, indicating high cerium dispersion within the TiO₂ lattice, whereas higher Ce content (8/100–50/100) enhanced CeO₂ peak intensity and reduced anatase TiO₂ peak intensity. Lattice parameter variations confirmed structural modification due to Ce incorporation. XPS revealed Ti⁴⁺ as the dominant titanium species and mixed Ce³⁺/Ce⁴⁺ states, promoting oxygen vacancies and improved charge transfer. UV-Vis spectra showed strong absorption at 350–380 nm, attributed to wide band-gap characteristics and defect states. FE-SEM analysis demonstrated porous nanosized grains (20–33 nm), while EDS and FT-IR confirmed chemical purity and successful composite formation. The CeO₂/TiO₂ nanocomposites exhibited enhanced photocatalytic performance compared to pure TiO₂, particularly during the initial reaction stage, demonstrating their potential for photocatalytic and gas-sensing applications.

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