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SYNERGISTIC INTERACTION OF PANI AND TIO_2 NANOSTRUCTURES FOR HIGH-SENSITIVITY CO GAS SENSING APPLICATIONS

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Keywords

Polyaniline, Titanium dioxide , Nanocomposite , Sol–gel , Spin-coating , CO gas sensor.

Abstract

In this research, polyaniline–titanium dioxide (PANI/TiO_2) nanocomposite thin films were effectively synthesized and evaluated for their carbon monoxide (CO) gas sensing capabilities. Titanium isopropoxide was used as a precursor to prepare TiO_2 nanoparticles via the sol–gel method, while polyaniline (PANI) was synthesized through chemical oxidative polymerization. The PANI/TiO_2 nanocomposite was obtained by blending both components in m-cresol and spin-coating the resulting mixture onto glass substrates. The structural, optical, and thermal properties of the prepared materials were characterized using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), UV–Visible spectroscopy, and thermogravimetric analysis (TGA). XRD confirmed the formation of anatase-phase TiO_2 and revealed a reduced crystallite size in the composite (25 nm) compared to pure TiO_2 (30 nm), indicating polymer–oxide interaction and suppressed grain growth. FTIR and UV–Vis spectra confirmed strong interfacial bonding and charge transfer between PANI and TiO_2, enhancing visible-light absorption and stability. The PANI/TiO_2 sensor showed a rapid and reversible response to CO concentrations (0–1000 ppb), exhibiting high linearity (R^2 = 0.901) and a detection limit of 0.40 ppb. These findings suggest that the PANI/TiO_2 nanocomposite is a promising, cost-effective, and

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	highly sensitive material for CO detection in environmental and industrial applications.

1. INTRODUCTION

Hazardous gas sensors have attracted substantial interest in both industrial and research sectors because to the ubiquitous occurrence of dangerous gases such as CO_2, H_2S, NO_2, CO, and NH_3 in the environment. Continuous monitoring of these gases is vital to protect the safety of people, animals, and plants [1]. Among these gases, ammonia (NH_3) is one of the most extensively used industrial compounds, acting as a coolant and raw material in food processing, fertilizer, textile, paper, and chemical industries, as well as in environmental and automotive applications [2]. However, extended exposure to ammonia may create major health issues affecting the respiratory system, skin, and lungs. According to the National Institute for Occupational Safety and Health (NIOSH), the allowable exposure limit for ammonia is 25 ppm for an 8-hour period and 35 ppm for a 15-minute short-term exposure [3]. Hence, the creation of efficient and sensitive NH_3 gas sensors has become an essential topic of study.

In recent years, hybrid materials that combine organic polymers with inorganic nanomaterials have attracted considerable interest due to their synergistic properties, which integrate the flexibility and processability of polymers with the stability and functional characteristics of inorganic components [4–6]. Polyaniline (PANI), a well-known conducting polymer, has emerged as a potential material because of its environmental stability, simplicity of production, high electrical conductivity, and reversible redox activity [7–9]. It is generally a p-type semiconductor and has found uses in batteries, electromagnetic shielding, anticorrosion coatings, and different sensors [10]. The electrical and sensing properties of PANI may be modified by acid/base doping or dedoping and are very sensitive to environmental fluctuations, making it a viable material for gas sensing applications at room temperature [11,12].

On the other hand, titanium dioxide (TiO_2), an n-type semiconducting metal oxide, has been widely investigated for its unusual photocatalytic, electrical, and optical characteristics. TiO_2 occurs largely as anatase and rutile phases, with the anatase form having stronger photocatalytic activity. Its applications include photovoltaics, photocatalysis, photo-electrochromic devices, and gas sensors [13–15]. Due to its high surface-to-volume ratio and strong oxidative potential, TiO_2 can efficiently interact with gaseous molecules, making it a promising option for gas sensing applications [16].

The mixing of TiO_2 with PANI may lead to the creation of an organic–inorganic hybrid composite that shows better gas sensing capabilities. In such composites, TiO_2 nanoparticles promote surface activity and electron transmission, while PANI offers a conductive network for charge transport. Moreover, the conduction band of TiO_2 aligns well with the lowest unoccupied molecular orbital (LUMO) of PANI, permitting efficient charge transfer and increasing electronic transport capabilities [17]. This synergistic interaction increases the sensor's sensitivity and selectivity toward target gases such as NH_3.

In this work, a PANI/TiO_2 (50%) nanocomposite was produced using in-situ chemical polymerization to examine its ammonia gas detecting capabilities. The produced materials—PANI, TiO_2, and the PANI/TiO_2 composite—were evaluated using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) to assess their structural, chemical, and morphological features. The gas sensing response of the composite was investigated for NH_3 at varied operating temperatures (20–70 °C), seeking to examine the impact of TiO_2 inclusion on the sensing features of PANI.

2. MATERIALS AND METHODS

2.1 Materials

Titanium isopropoxide (TTIP, 97%), methanol (99.8%), aniline (99.5%, AR grade), ammonium persulfate ($(NH_4)_2S_2O_8$, 98%, AR grade), hydrochloric acid (HCl, 35.4%, AR quality), and sulfuric acid (H_2SO_4, 96%, AR grade) were employed for the production of PANI, TiO_2, and PANI/TiO_2 hybrid materials. Deionized water was applied for hydrolysis throughout the synthesis procedure. All chemicals employed were of analytical reagent (AR) quality and were used without any additional purification.

2.2 Synthesis of Tio2

Titanium isopropoxide (TTIP) was exploited as a titanium precursor for the manufacture of TiO_2 nanoparticles using the sol–gel technique. In a typical process, 3.7 mL of TTIP was introduced dropwise to 50 mL of methanol under continuous stirring at 50 °C for 1 hour. The resultant white precipitate was collected and dried to create a fine powder. The dry powder was subsequently annealed in a tube furnace at 600 °C for 1 hour to obtain TiO_2 nanopowder [18].

2.3 Synthesis of polyaniline (PANI)

Polyaniline (PANI) was created utilising the chemical oxidative polymerisation process. Ammonium persulfate (APS) was used as an oxidising agent in this procedure, with hydrochloric acid (HCl) acting as a catalyst. Initially, 2 mL of aniline was added to 50 mL of 1 M HCl solution while continuously magnetically swirling. In a separate beaker, 4.9984 g of APS was dissolved in 50 mL of distilled water with 1 M HCl. The APS solution was then added dropwise to the aniline solution while constantly stirring. The reaction mixture eventually became green, suggesting the synthesis of polyaniline. To complete polymerisation, an ice bath was used to keep the reaction temperature between 0 and 8 °C for 5-6 hours. The precipitate was filtered and extensively washed with 1 M HCl, then distilled water, until it turned colourless. Finally, the product was vacuum-dried in an oven at 60°C for 12 hours, yielding pure PANI powder [18].

2.4 Synthesis of PANI/TiO_2 Nanocomposite Films

PANI/TiO_2 nanocomposite films were created by spin-coating method. To create the composite, the synthesised TiO_2 nanoparticles and PANI powder were thoroughly combined in an agate mortar and pestle for a homogenous mixture. The resultant mixture was disseminated in m-cresol

Bavane Ravindrakumar G (2025). *Synergistic Interaction of Pani and Tio₂ Nanostructures for High-Sensitivity Co Gas Sensing Applications. International Journal of Multidisciplinary Research & Reviews, 4(4), 128-138.*

and magnetically agitated for 12 hours to produce a uniform casting solution. Thin films were then deposited on clean glass substrates by spinning at 3000 rpm for 30 seconds and drying on a hot plate at 100 °C for 10 minutes. Silver paste electrodes, 1 mm wide and 1 cm apart, were placed to the film surfaces to make electrical connections. To compare, pure TiO_2 thin films were produced under same circumstances without PANI.

3. CHARACTERIZATION TECHNIQUES

3.1 X-ray diffraction analysis

Figureure 1 displays the X-ray diffraction patterns of the synthesized materials, used to analyze their structural characteristics. The diffraction peaks of pure TiO_2 match well with the standard anatase phase (JCPDS No. 78-1285). In contrast, the TiO_2/PANI nanocomposite exhibits broader and less intense peaks. This broadening and decrease in intensity indicate the presence of the poorly crystalline PANI phase and a reduced volume fraction of TiO_2 within the composite. The interaction between TiO_2 nanoparticles and PANI chains appears to restrict grain growth, as scattering from the polymer chains hinders TiO_2 crystallization. Furthermore, when PANI molecules adsorb onto the TiO_2 surface, the confinement effects imposed by the inorganic phase cause slight disruption to the structural order of PANI. The inhibition of grain growth in the hybrid material was further confirmed by the average crystallite size, calculated using Scherrer's formula [19], which was found to be approximately 30 nm for pure TiO_2 and 25 nm for the TiO_2/PANI nanocomposite films.

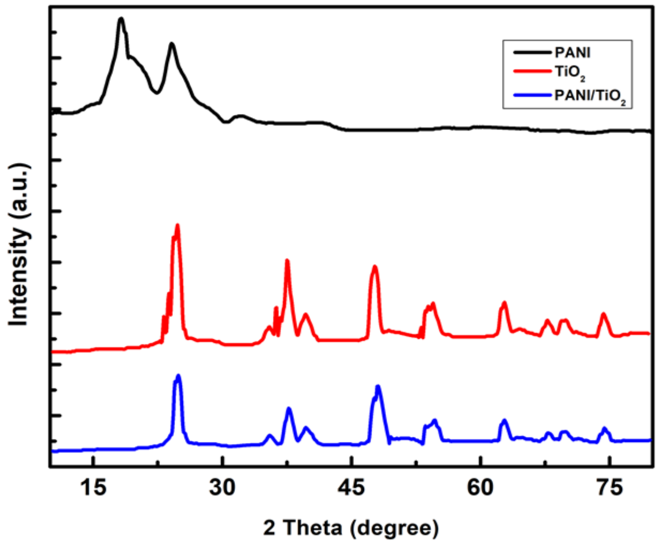

Figure. 1 X-Ray Diffraction (XRD) pattern of, TiO2, PANI and TiO2 /PANI composite films

Bavane Ravindrakumar G (2025). *Synergistic Interaction of Pani and Tio₂ Nanostructures for High-Sensitivity Co Gas Sensing Applications. International Journal of Multidisciplinary Research & Reviews, 4(4), 128-138.*

3.2 Fourier transform infrared (FTIR) analysis

The FTIR spectra of TiO_2, PANI, and the TiO_2/PANI composite are given in Figure. 2. Pure TiO_2 has a significant absorption band below 700 cm^{-1}, corresponding to Ti–O–Ti stretching vibrations, which indicates the production of TiO_2 nanoparticles [20]. The spectra of PANI exhibits distinctive peaks at 1560 and 1480 cm^{-1}, allocated to the quinoid and benzenoid C=C stretching vibrations, respectively, coupled with a band at 1290 cm^{-1} attributable to C–N stretching of aromatic amines. A characteristic band about 1140 cm^{-1} corresponds to the protonation state of PANI, whereas the peak around 800 cm^{-1} derives from C–H out-of-plane bending, both of which are consistent with past research [21, 22]. The composite spectrum clearly contains the properties of both PANI and TiO_2, with notable shifts and changes in band intensities. These spectrum alterations support the effective integration of TiO_2 into the PANI matrix and show significant interfacial interactions between the polymer chains and TiO_2 nanoparticles, as earlier observed in comparable composite systems [23, 24]. Such bonding improves electron transport, making the composite particularly promising for sensing applications.

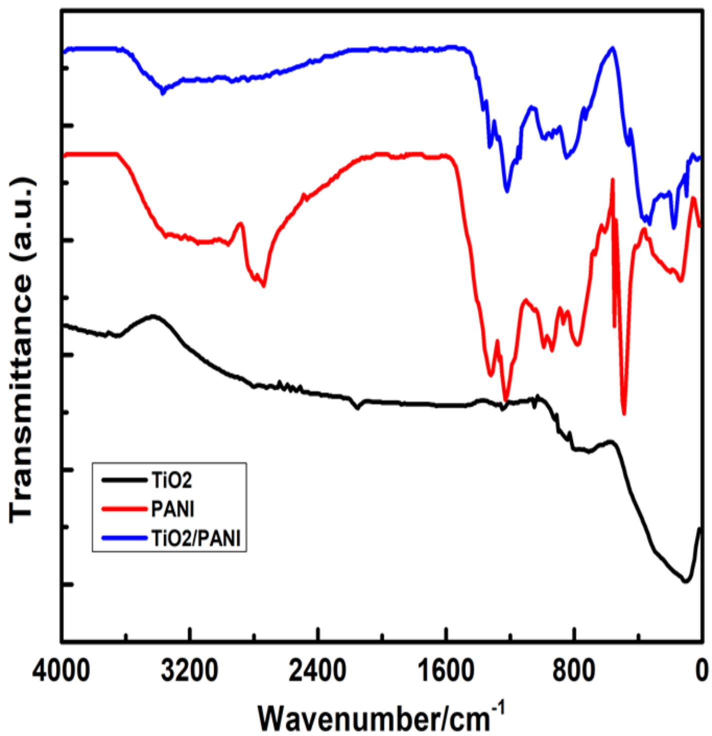

Figure. 2 FTIR spectra of TiO2, PANI and TiO2/PANI composite films respectively

Bavane Ravindrakumar G (2025). *Synergistic Interaction of Pani and Tio₂ Nanostructures for High-Sensitivity Co Gas Sensing Applications. International Journal of Multidisciplinary Research & Reviews, 4(4), 128-138.*

3.3 UV–Vis spectroscopy

The UV–Vis spectra clearly reveal the distinctive absorption properties of TiO_2, PANI, and their composite in Figure. 3. Pure TiO_2 (black curve) has a significant absorption edge in the UV area below 400 nm, which is related to its broad band gap transition (~3.2 eV). Pure PANI (red curve) displays extensive absorption bands spanning from the UV to the visible area, owing to π–π* transitions of the benzenoid rings (~250–300 nm) and polaron–π* transitions in the longer wavelength range, confirming its conductive emeraldine salt form. Interestingly, the PANI/TiO_2 composite (blue curve) exhibits mixed properties of both TiO_2 and PANI, with increased and expanded absorption in the visible area. This red-shift in absorption shows substantial contact and charge transfer between PANI and TiO_2 nanoparticles, enhancing visible light consumption. Such behavior shows that the PANI/TiO_2 composite has improved optical characteristics compared to the individual components, making it more appropriate for sensing and photocatalytic applications.

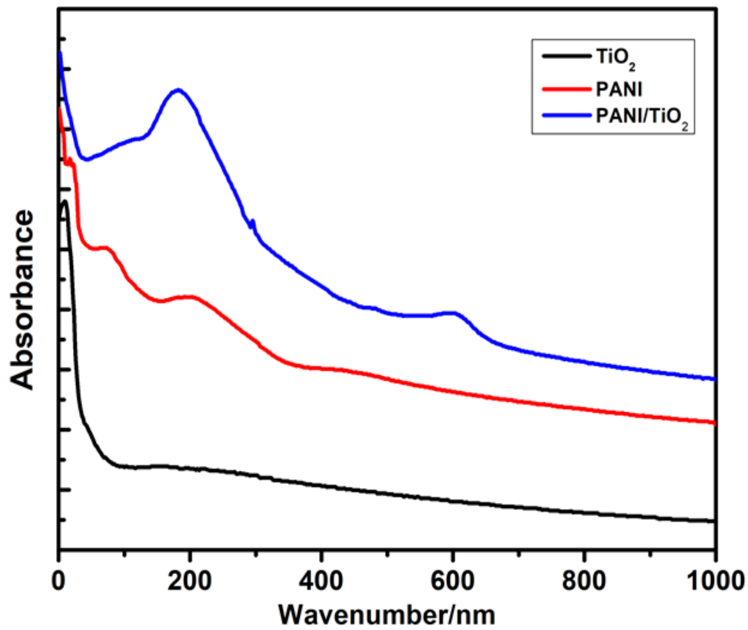

Figure. 3 UV-Vis Spectra of PANI, TiO_2 and PANI/TiO2.

3.4 Thermogravimetric Analysis (TGA)

The Figure.4 TGA graph demonstrates that pure TiO_2 is thermally stable with nearly little weight loss up to 1000 °C, but pure PANI experiences considerable multi-step deterioration, losing most of its weight by 600 °C and leaving just approximately 10% residual. In contrast, the PANI/TiO_2 composite showed superior thermal stability compared to PANI, with slower weight loss and

Bavane Ravindrakumar G (2025). *Synergistic Interaction of Pani and Tio₂ Nanostructures for High-Sensitivity Co Gas Sensing Applications. International Journal of Multidisciplinary Research & Reviews, 4(4), 128-138.*

greater residue (~30%) at 1000 °C. This implies that the inclusion of TiO_2 increases the heat stability of PANI by preventing polymer chain breakdown and providing structural support.

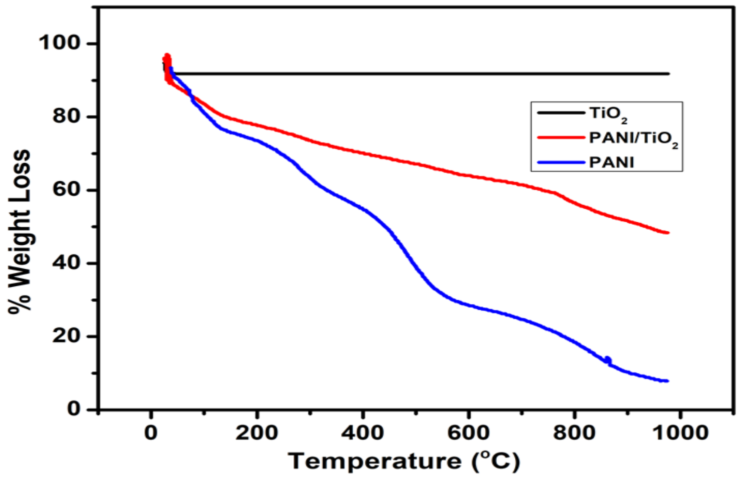

Figure. 4. TGA (Thermogravimetric Analysis) graph comparing PANI, TiO_2 and PANI/TiO_2 composite

4. CARBON MONOXIDE (CO) SENSING BY PANI/ TIO2 ELECTRODE

The Figure. 5 illustrates the dynamic sensing response of the CO sensor. In the dynamic response, the sensor displays stepwise rises and declines in output when subjected to varied CO concentrations (1000, 700, 400, 100, 80, 40, and 0 ppb). The distinct and steady plateaus at each concentration demonstrate outstanding repeatability, rapid response, and recovery behavior of the sensor.

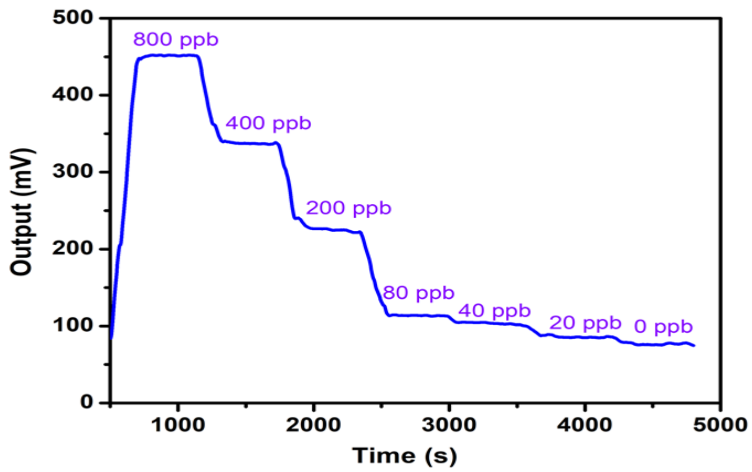

Figure. 5 Dynamic Sensing Response of Carbon Monoxide (CO).

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The Figure. 6 illustrates the calibration curve of CO gas detecting using TiO_2/PANI, where the sensor output (mV) is plotted against CO concentration (ppb). The response of the sensor rises with the growth in concentration, demonstrating a nearly linear behavior at higher concentrations, while small deviations are observed at lower concentrations. The regression line fitted to the experimental data produces the equation y=72.048+0.522x with an adjusted R2=0.901, which demonstrates a reasonable degree of linearity. The slope of the line, 0.522 mV/ppb, reflects the sensitivity of the sensor, whereas the detection limit derived from the calibration is roughly 0.40 ppb. This implies that the TiO_2/PANI sensor can detect CO even at extremely low levels, making it appropriate for sensitive applications, while the linearity is significantly reduced compared to optimum calibration behavior.

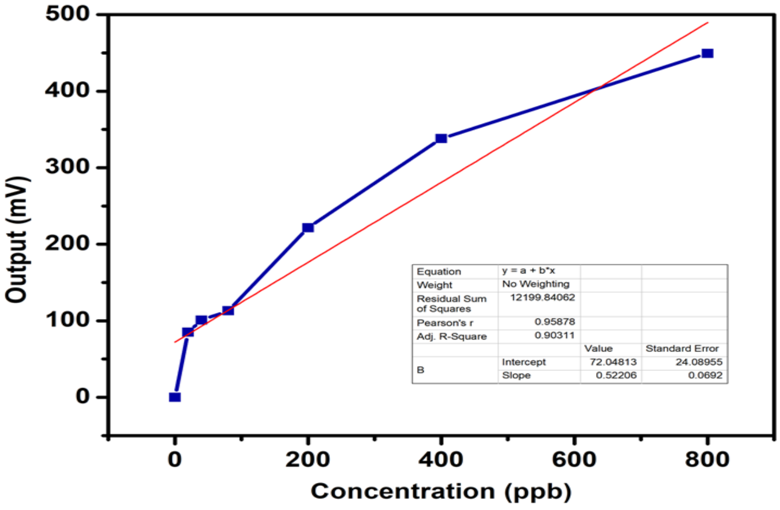

Figure. 6 Calibration curve Sensing Response of Carbon Monoxide (CO).

5. CONCLUSION

The effective fabrication and characterisation of PANI/TiO_2 nanocomposite films have revealed the synergistic benefits of mixing an organic conducting polymer with an inorganic metal oxide. XRD, FTIR, and UV–Vis investigations indicated significant interfacial contacts between PANI chains and TiO_2 nanoparticles, which resulted to decreased crystallite size, increased charge transfer, and enhanced visible-light absorption. Thermal study demonstrated that TiO_2 inclusion greatly increased the thermal stability of PANI. The PANI/TiO_2 sensor displayed outstanding CO detecting properties with high sensitivity (0.522 mV/ppb), strong linearity (R^2 = 0.901), and a low detection limit (0.40 ppb). The rapid reaction and recovery characteristics further indicate the applicability of this composite for real-time gas monitoring. Overall, the PANI/TiO_2 nanocomposite provides a promising, cost-effective, and stable material for future gas sensor

Bavane Ravindrakumar G (2025). *Synergistic Interaction of Pani and Tio₂ Nanostructures for High-Sensitivity Co Gas Sensing Applications. International Journal of Multidisciplinary Research & Reviews, 4(4), 128-138.*

development, especially for low-concentration CO detection in industrial and environmental applications.

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The writers affirm that they have no connections to, or engagement with, any group or body that provides financial or non-financial assistance for the topics or resources covered in this manuscript.

8. CONFLICTS OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

9. PLAGIARISM POLICY

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