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Structural, Morphological and Gas Sensing Characteristics of Chemically Synthesized Ni-Pd Nanocomposite

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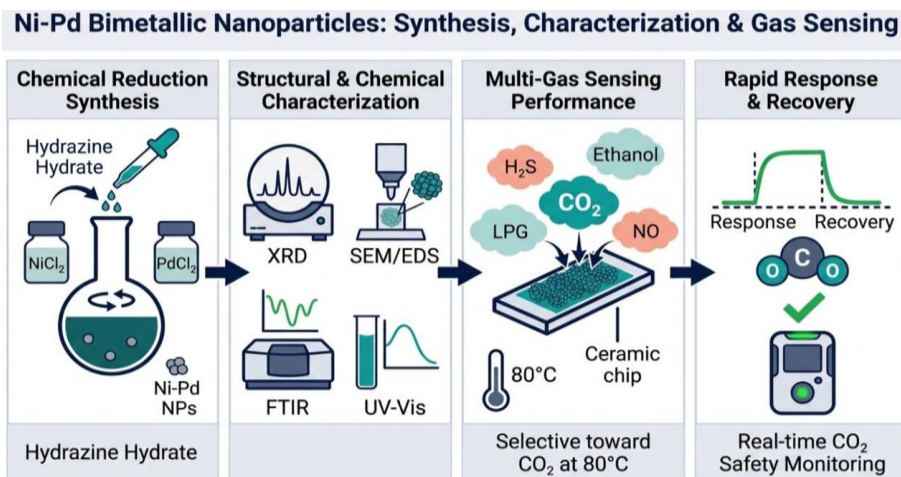
Chemical Synthesis

Hydrazine Hydrate

ABSTRACT

Nickel-palladium (Ni-Pd) bimetallic nanoparticles were fabricated using hydrazine hydrate by chemical reduction. The synthesized nanoparticles were characterized by employing various spectroscopic techniques to determine the structural, morphological, elemental, and optical properties. The crystalline nature, purity of phase, and crystallite size of the synthesized Ni-Pd nanoparticles were confirmed by XRD. The average crystallite size was found to be 5.45 nm. SEM revealed highly agglomerated, spherical, cluster-like nanoparticles with an average particle size of 50.277 ± 19.727 nm; EDS analysis showed the composition of Ni (64.32 wt.%), Pd (14.58 wt.%), and other detected elements; the oxygen signal was attributed to surface oxidation. Fourier transform infrared spectroscopy (FTIR) revealed metal-oxygen bonds and the presence of surface functional groups, and UV-Visible spectroscopy showed a characteristic absorption peak at 291 nm, confirming nanoparticle formation. The gas sensing performance of the synthesized Ni-Pd nanoparticles was examined towards the H_2S , ethanol, LPG, CO_2 , and NO_2 gases under optimized operating conditions. Ni-Pd nanoparticles offer high gas sensing performance with rapid and reversible response-recovery characteristics, strong selectivity toward CO_2 , and optimal operation at 80 °C with 93.56% response and 72 s recovery time, making them promising candidates for reliable, real-time gas detection and safety monitoring applications.

GRAPHICAL ABSTRACT



1. Introduction

The synthetic control of nanosized materials has gained more attention in various research fields, including catalysis [1], electronics, sensing [2], and energy fields, because of their non-toxicity, larger surface area, controlled particle size, and good stability. To increase the activity of nanoscale materials, precise synthesis methods must be used to control the surface structure of nanomaterials throughout their creation. Since the active sites of Ni and Pd are comparable, Ni is more frequently employed in the production of Pd-based bimetallic nanoparticles. This successfully lowers the cost of producing Pd-based bimetallic nanoparticles and improves their stability in an alkaline medium. The differential potentials of both Ni and Pd are different from each other ($Ni/Ni^{2+} = -0.257$ V, Pd/Pd^{2+}

$= +0.915$ V [at 298K, 1 M, and 1 atm]). Therefore, Pd-based nanoparticles formed as aggregated [3]; otherwise, small-sized bimetallic nanoparticles would be obtained in the synthesis.

Over past ten years, several techniques for creating bimetallic nanoparticles have been devised, its properties were investigated. Such nanoparticles are frequently employed in photochemical, electrochemical [4-6], and catalyst-mediated [7-9] processes. The nanoparticles impact on the electro-optical properties of other substances, which enables their use in photoelectrochemical cells, is related to improving the properties of photochemical processes. Certain electrochemical capabilities and energy characteristics of nanoparticles are the basis for improving sensor device operational features [10,11]. Because of a synergistic impact, the employment of bimetallic nanoparticles boosts their operational stability and decreases the consumption of costly metal in their varied activities.

The growing need for quick, accurate, and sensitive detection of hazardous gases across a variety of industries has made nanomaterial-based gas sensors increasingly significant. Ni-Pd nanoparticles were superior in terms of electrical conductivity and potent catalysts, making

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them attractive as gas sensors [12]. Ni-Pd nanoparticles are useful materials for gas sensing activities because Ni contributes to improved charge transfer and Pd improves gas adsorption and dissociation, improving sensor sensitivity, selectivity, fast response, and recovery properties [13,14].

Because of their fascinating synergistic qualities and numerous uses in nanoscience and nanotechnology, bimetallic nanoparticles have drawn much focus in the study [15]. This paper describes bimetallic Ni-Pd nanoparticles that could be employed as efficient CO₂ sensors. These nanoparticles were created using a quick and simple chemical reduction method and characterised using structural and morphological techniques.

2. Experimental Methods

2.1 Materials

All the solvents and chemicals were A.R. grade and were used as received. Hydrazine hydrate served as a reductant, and sodium hydroxide was used to maintain an alkaline medium.

2.2 Preparation of Precursor Solutions

A 0.1 M nickel chloride solution was obtained by dissolving the required amount of nickel chloride in distilled water and stirring. Similarly, a 0.1 M palladium chloride (aq.) was prepared separately in distilled water. Since palladium chloride is sparingly soluble in water, a few drops of hydrochloric acid were added to facilitate dissolution.

The dissolution process may be represented as: $\text{PdCl}_2 + 2 \text{Cl}^- \rightarrow [\text{PdCl}_4]^{2-}$

2.3 Synthesis of Nickel–Palladium Bimetallic Nanoparticles

Equal volumes of nickel chloride and palladium chloride precursor solutions in a 1:1 molar ratio was added under continuous stirring. Then, 7.0 mL of trisodium citrate was added dropwise under continuous stirring. 2.5 mL hydrazine hydrate was added. The pH of the reaction mixture was adjusted to basic conditions (pH 8–9) using sodium hydroxide solution. The stirring of the reaction mixture was carried out for 1.5 h at room temperature. The gradual colour change from a greenish-yellow solution to a dark brown-black colour was an indication of the formation of nickel-palladium bimetallic nanoparticles [16].

2.4 Collection and Purification

The obtained black precipitate was separated by filtration using Whatman paper and repeatedly washed with distilled water, then ethanol to eliminate impurities and other residual chemicals. The purified nanoparticles were dried in an oven at 60–80 °C and stored to perform characterization and applications. The hydrazine hydrate reduction method is a simple and efficient synthesis for the well-dispersed Ni-Pd bimetallic nanoparticles [17].

3. Results and Discussion

3.1 Characterization

3.1.1 UV-Visible Spectroscopy Study

The UV–visible absorption spectra of the chemically produced Ni-Pd nanoparticles, shown in Fig. 1, was obtained by choosing a wavelength range of 200–800 nm. At about 291 nm, a wide absorption band revealed the production of Ni-Pd nanoparticles. Because of their strong inter-band electronic transitions and large damping effect, Ni and Pd nanoparticles do not exhibit a noticeable localized surface plasmon resonance (LSPR) band in the visible range, as Au and Ag nanoparticles do.

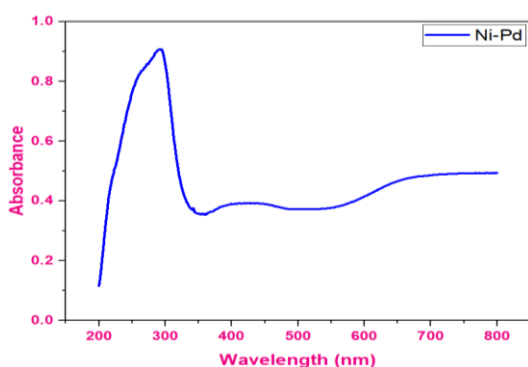


Fig. 1 UV-visible spectrum of Ni-Pd bimetallic nanoparticles
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Therefore, one characteristic of Ni-Pd bimetallic nanoparticles is their extensive absorption in the UV range. The lack of further absorption peaks in the visible area indicates that the nanoparticles are evenly distributed and somewhat aggregated.

3.1.2 FTIR Analysis

To identify the functional groups linked to the creation and stabilization of nanoparticles, the FTIR spectrum of Ni-Pd nanoparticles, as displayed in Fig. 2, was recorded in the region of 4000–400 cm⁻¹. The presence of surface functional groups and metal–oxygen interactions were confirmed by the FTIR spectrum of chemically produced Ni-Pd nanoparticles, which showed multiple distinctive absorption bands. The production of nickel and palladium-containing nanoparticles with a thin surface oxide layer generated upon exposure to air is confirmed by the distinctive bands at 626.75, 526.47, 493.69, and 456.08 cm⁻¹, which can be linked to Ni-O and Pd-O stretching vibrations. The effective synthesis of the bimetallic nanoparticle system is confirmed by the presence of the distinctive metal-oxygen bands.

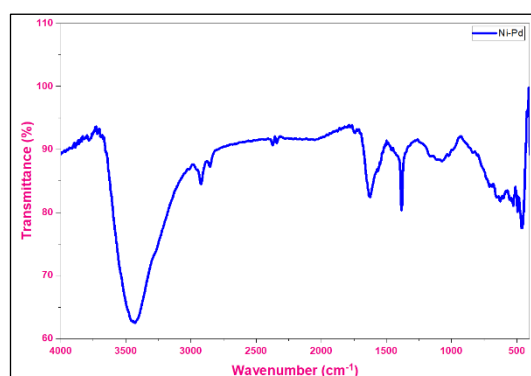


Fig. 1 FTIR spectrum of Ni-Pd bimetallic nanoparticles

3.1.3 SEM-EDS Analysis

The surface morphology of the synthesized Ni-Pd bimetallic nanoparticles was determined by using scanning electron microscopy (SEM), as shown in Fig. 3a–f. The SEM micrographs revealed that the synthesized nanoparticles are densely packed and form aggregates with a spherical-like irregular morphology. The particles appeared to be highly agglomerated, forming spherical, cluster-like structures distributed throughout the surface of the sample. This type of agglomeration is generally observed in chemical reduction or the coprecipitation method of synthesis. A rough and porous surface texture could provide a large surface area, which is suitable for nanoparticle applications such as catalytic activity and gas sensing performance. The morphology of nanosized particles that are obtained by the hydrazine reduction process suggests that rapid formation occurred, which results in the subsequent aggregation to give a cluster-like nanostructure. The successful nanostructured material was confirmed by the fact that there are no well-defined bulk crystalline structures or large micron-sized particles.

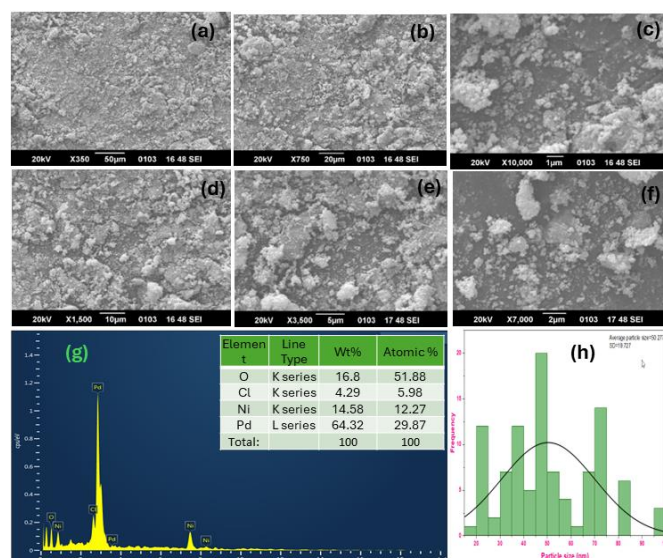


Fig. 3 (a–f) SEM images at different resolutions, (g) EDS spectrum and (h) SEM images obtained from Ni-Pd particle size distribution

The energy-dispersive X-ray spectroscopy (EDS) (Fig. 3g) was used to investigate the composition of nanoparticles. The EDS spectra give information about nickel (Ni) and palladium (Pd) as the major metallic constituents of the synthesized nanomaterial. The EDS spectrum displayed Pd and Ni contents of 64.32 wt.% and 14.58 wt.%, respectively, which confirms successful incorporation of both metals within the bimetallic system.

The higher palladium content indicates that the synthesized nanomaterial is Pd-rich, possibly due to differing reduction kinetics and deposition behaviour of the metal precursors during synthesis when the hydrothermal method or strong reagents are used.

Ni undergoes oxidation very easily as compared to Pd during the synthesis, which leads to lower Ni content in synthesized bimetallic nanoparticles [3]. In the hydrothermal method or in the presence of strong reagents, Ni-Pd nanoparticles undergo a complex process which accomplished the higher Ni content in the synthesized bimetallic nanoparticles. The oxygen peak in the EDS spectrum can be attributed to the surface oxidation of Ni nanoparticles or to adsorbed oxygen-containing species formed during sample handling and exposure to the ambient atmosphere.

The EDS spectrum further revealed that the oxygen (16.80 wt.%) signal detected in the sample may be due to partial surface oxidation of nickel nanoparticles upon air explosion. The oxygen signal may be due to the formation of a thin oxide layer at the time of synthesis and drying of nanoparticles. The minor residual signal of chloride indicated that a trace number of precursor-derived species remained adsorbed on the nanoparticle surface despite the washing procedure.

Overall, the SEM-EDS analysis displayed the successful synthesis of Ni-Pd bimetallic nanoparticles that are predominantly distributed within the nanoscale range, with an average size of 50.277 ± 19.727 nm (Fig. 3h).

3.1.4 XRD Analysis

The XRD pattern of the synthesized Ni-Pd bimetallic nanoparticles is displayed in Fig. 4. The crystalline nature of the produced Ni-Pd nanoparticles was confirmed by the peaks at $2\theta = 40.7^\circ$, 46.6° and 68.29° . The diffraction peak at 40.7° was uniquely assigned to the (111) plane, 46.6° (200), and 68.29° (200) plane of nickel-palladium according to JCPDS No. 46-1043 for Pd and JCPDS No. 04-0850 for Ni. The EDS spectrum and the diffraction pattern together indicated the coexistence of Ni and Pd, confirming the successful synthesis of crystalline Ni-Pd bimetallic nanoparticles. The crystallite size was estimated using the Debye-Scherrer equation. The range of calculated crystallite size is 4.83 nm to 6.04 nm, which indicates the crystalline nature of the synthesized nanomaterial.

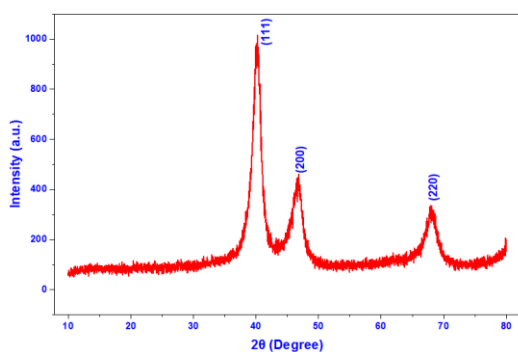


Fig. 4 XRD pattern of Ni-Pd bimetallic nanoparticles

3.2 Gas Sensing Performance

The gas-sensing characteristics of Ni-Pd nanoparticles reveal a strong temperature-dependent response toward H_2S , ethanol, LPG, CO_2 , and NO_2 , with sensitivity increasing from 40°C up to an optimum of around 160°C , after which it decreases due to reduced surface oxygen adsorption at higher temperatures (Fig. 5a).

Fig. 5c represents the sensor readings for the above gases, which are shown in the form of bar graphs. The temperature-dependent gas sensing response of Ni-Pd nanoparticles is illustrated. CO_2 exhibits the highest sensitivity (reaching $\sim 93\%$) because the $\text{Ni}^{2+}/\text{Pd}^{2+}$ redox mechanism and abundant oxygen vacancies promote effective chemisorption and rapid electron exchange between CO_2 molecules and the Ni-Pd surface (Fig. 5c).

The response increased from 5.16% at 40°C to the greatest value of 93.56% at 80°C and decreased dramatically at 200°C . It indicates that the optimum operating temperature for CO_2 detection is 80°C .

The selectivity study additionally demonstrated that the Ni-Pd sensor has a preference for CO_2 gas. The relative selectivity values for CO_2 , NO_2 , H_2S , ethanol, and LPG were 100%, 15.90%, 11.82%, 8.83%, and 3.78%, respectively, and this revealed that the sensor had much higher sensitivity to CO_2 than to other interfering gases. The enhanced selectivity could be explained by the significant interaction of CO_2 molecules with the active surface sites of Ni-Pd nanoparticles.

The effect of gas concentration was studied in the range 100–500 ppm (Fig. 5d). The response increased gradually from 32.58 % at 100 ppm to 93.56 % at 300 ppm, indicating the increase in the number of adsorbed gas molecules and active surface reactions. The response slightly decreased at 400 ppm (88.42%), which might be attributed to the saturation of available adsorption sites on the sensor surface.

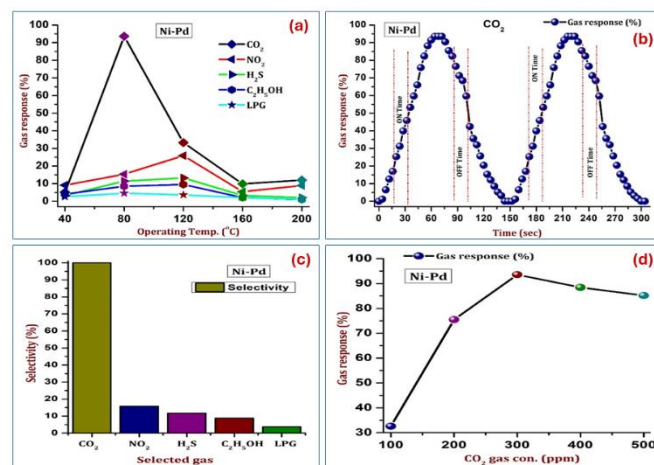
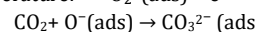
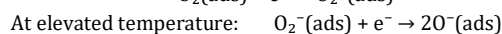
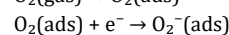
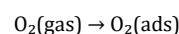


Fig. 5 a) Variation of gas sensitivity (%) of Ni-Pd nanoparticles toward gases as a function of operating temperature (40–200 °C) b) Dynamic response recovery characteristics of the Ni-Pd gas sensor showing sensitivity variation with time and indicating ON and OFF response cycles c) Sensor response vs different test gases d) Sensor response vs different gas concentrations

The dynamic response-recovery curve shows fast and reversible sensing behaviour. The response increased to the maximum value of 93.56% after about 64–68 s upon exposure to CO_2 gas and recovered to near its original baseline in about 72 s after elimination of the target gas. The response-recovery behaviour and repeatability studies demonstrated stable sensing performance. H_2S and ethanol showed moderate sensitivities because their molecular structures enable interaction with surface oxygen species, yet their adsorption energies and reaction kinetics are lower compared to CO_2 . LPG shows the weakest response due to its lower affinity toward Ni-Pd and faster desorption rate at elevated temperatures. The dynamic response-recovery study further demonstrates that Ni-Pd possesses excellent switching behaviour, with a sharp rise in sensitivity immediately after gas exposure (ON time) and a quick return to baseline once the gas is removed (OFF time), confirming efficient oxygen re-adsorption and surface regeneration. The repeated cycling pattern indicates highly stable operation, good reproducibility, and minimal degradation over time (Fig. 5b). Overall, the results underline that Ni-Pd nanoparticles offer high gas sensing performance with rapid and reversible response-recovery characteristics, strong selectivity toward CO_2 , and optimal operation at 80°C , making them promising candidates for reliable, on-site gas detection and safety screening implementation.

3.2.1 Gas Sensing Mechanism

The adsorption-desorption mechanism of oxygen species on the nanoparticles' surface and their reaction with CO_2 molecules provide the basis of the gas sensing mechanism of Ni-Pd bimetallic nanoparticles. Depending on the operating temperature, oxygen molecules are adsorbed on the surface of Ni-Pd nanoparticles upon exposure to air by taking electrons from the conduction band and creating chemisorbed oxygen species such as O_2^- , O^- , and O^{2-} .



The adsorbed oxygen species create an electron depletion region at the sensing surface. This enhances the sensor resistance at high operating temperatures. When exposed to CO_2 gas, the gas molecules react with the adsorbed oxygen species and release the electrons back to the conduction band, causing a notable change in electrical resistance that produces the sensing response.)

The complementary effect across the Ni-Pd nanoparticles strengthens the surface reactivity, electron transport, and adsorption capability towards CO₂ molecules, resulting in the optimum sensing performance. Furthermore, combining the nanoscale particle size and the agglomerated porous morphology offers a highly active surface area and the presence of abundant adsorption sites for gas interaction. The heterojunction interaction between Ni and Pd species and the presence of oxygen vacancies further promote effective electron transport and enhance the sensor's response-recovery properties. The maximum sensing response of 80 °C indicates optimum adsorption-desorption equilibrium along with efficient surface reaction kinetics of CO₂ molecules alongside chemisorbed oxygen species. The decrease in sensing response at higher temperatures may be due to fast desorption of gas molecules and a lowering in adsorbed oxygen concentration on the sensor layer.

4. Conclusion

The successfully synthesized Ni-Pd bimetallic nanoparticles by a simple hydrazine hydrate-assisted method were characterized to confirm their crystalline nature, morphology, elemental composition, and optical properties. The characterization techniques XRD, SEM, EDS, FTIR, and UV-Visible spectroscopy confirmed their crystalline nature, morphology, elemental composition, and optical properties. The gas sensing performance showed excellent selectivity towards CO₂ gas. The improved activity can be attributed to the combined effect of nickel and palladium, which enhance surface reactivity and charge transfer. Overall, chemically synthesized Ni-Pd bimetallic nanoparticles show promising potential for environmental remediation.

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