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Synthesis and Characterization of NiO/SnO_2 Nanocomposites Prepared via Sol-Gel Method for Enhanced Optical Properties and Ethanol Gas Sensing Applications

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Abstract

Nickel Oxide (NiO) and Tin Oxide (SnO_2) semiconductor nanomaterials have been a lot of attention recently due to their excellent optical, electrical and gas sensing characteristics. In this study, the NiO nanoparticles, SnO_2 nanoparticles and NiO/SnO_2 nanocomposites were successfully synthesized by a simple sol-gel process and then systematically characterized to investigate the structural, morphological, optical and gas sensing properties. The synthesized materials were characterized by X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), energy-dispersive X-ray spectroscopy (EDS) and UV-visible absorption spectroscopy. The XRD results were used for confirming the successful formation of the crystalline phases for individual oxides and nanocomposite structure. The average crystallites size of NiO, SnO_2 and NiO/SnO_2 nanocomposite was about 33.64nm, 13.80nm and 16.30nm respectively. The FE-SEM images showed nanoscale granular morphologies and higher grain aggregation in the nanocomposite sample. The existence of Ni, Sn and O was confirmed and their homogeneity was verified by EDS analysis.

Improved visible light absorption and red shift in absorption edge were observed with the incorporation of NiO into SnO_2, as indicated by optical characterization. The optical band gaps for NiO (3.7 eV) and SnO_2 (3.3 eV) were reduced to approximately 3.2–3.5 eV in NiO/SnO_2 nanocomposite, which results in better electronic interactions between both semiconductor phases. Moreover, as compared with the pristine NiO and SnO_2 gas sensors, the NiO/SnO_2 heterojunction sensor exhibited better gas sensing properties for ethanol gas sensing experiments. The nanocomposite displayed higher sensitivity, low operating temperature and excellent response and recovery properties with optimum sensing characteristics at around 225°C. This was believed to be due to the creation of p–n heterojunctions, an increase in oxygen adsorption sites, and charge transfer efficiency. The results obtained here show that NiO/SnO_2 nanomaterials prepared by sol-gel process have a potential as well as interesting application in advanced gas sensing and optoelectronic field.

Keywords: Nickel oxide; Tin oxide; NiO/SnO_2 nanocomposite; Sol-gel synthesis; UV-Vis spectroscopy; Optical band gap; Ethanol gas sensor; Semiconductor heterojunction

1. Introduction

The remarkable physicochemical properties of metal oxide semiconductors (MOSs) such as high thermal stability, excellent catalytic activity, large surface-to-volume ratio, tunable band structures and superior gas sensing capabilities, have made them one of the most important classes of functional nanomaterials [1]. The special properties make them highly suitable for applications in environmental monitoring, energy storage, photocatalysis, solar cells, biomedical devices, and optoelectronic devices [2].

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Tin oxide (SnO_2) has been widely studied as a semiconductor metal oxide among the various semiconductor metal oxides because of its good chemical stability, low electrical resistance, high optical transparency and high sensitivity to various reducing and oxidizing gases [3]. SnO_2 has a wide band gap of 3.5-3.8 eV (direct band gap) and good electron mobility, and is one of the most widely studied materials for gas sensing applications [4]. But, pristine SnO_2 has several drawbacks such as low selectivity, high operating temperature, slow response kinetics and also poor long-term stability during real operating conditions [5].

To achieve these limitations, many strategies have been suggested such as morphology engineering, noble metal decoration, defect engineering, and heterojunction construction by transition metal doping [6]. Semiconductor heterojunctions have been developed as one of the most promising techniques to improve the gas sensing performance due to the distinct effects of charge separation, oxygen adsorption and active surface sites [7].

The p-type semiconductor nickel oxide (NiO) has a wide band gap of 3.6-4.0 eV, excellent electrochemical stability, catalytic activity and charge transportability [8]. The electronic properties of NiO make it a material of particular interest for batteries, supercapacitors, electrochromic devices, photocatalysis and gas sensing systems [9]. The use of NiO and SnO_2 can generate p–n heterojunction structures, which plays significant role to improve the interfacial charge transfer processes and performance of the NiO–SnO_2 based sensors [10].

The fabrication of NiO/SnO_2 heterojunction nanocomposites has several benefits compared to the NiO and SnO_2 oxide systems. The first point is that interfacial Electric field created at the p-n junction facilitates efficient separation of electron and holes. Secondly, the higher amount of oxygen adsorption sites leads to more surface reactions with target gases. Thirdly, the synergistic interaction between both semiconductor phases increases the sensitivity and decreases the optimum operating temperature [11]. Thus, NiO/SnO_2 nanocomposites have gained significant interest as potential sensors for next-generation gas sensing applications.

A number of the synthesis routes are developed for the preparation of NiO/SnO_2 nanostructures: hydrothermal, co-precipitation, chemical vapor deposition, pulsed laser deposition, spin coating, solid state reactions and solvothermal methods [12]. However, the sol-gel process is still one of the most promising methods due to its simplicity, low cost, high compositional homogeneity, controllable particle size distribution and large scale production ability [13].

Precise control over the nucleation and growth of particles is possible with the sol-gel process, enabling researchers to control the structural and optical properties of semiconductor nanocomposites. In addition, this technique yields high purity materials and has very good reproducibility at relatively low processing temperatures [14]. Such advantages make sol-gel synthesis a suitable method for the synthesis of high performance gas sensing nanomaterials.

The optical properties of semiconductor nanomaterials are highly dependent on particle size, crystallinity and surface defects. The quantum confinement effects lead to variations in optical absorption and energy band gaps with changes in particle dimensions as a result of changes in the electronic band structure [15]. UV-visible spectroscopy is thus a crucial tool for characterization and analysis of optical transitions and understanding electronic interaction in semiconductor heterostructures.

Surface adsorption and desorption reactions are the key factor that dominate the gas sensing mechanism in Metal Oxide Semiconductors. Adsorbed oxygen molecules on the surface of the sensor capture free electrons to create oxygen species in the ionic state which in turn changes the electrical resistance [16]. These adsorbed oxygen species are reduced when they are exposed to reducing gases like ethanol, which frees up electrons in the semiconductor and thus lowers sensor resistance [17].

Although considerable progress has been made in the development of NiO/SnO_2 nanocomposites, the structures, optical band-gap modulations and gas sensing performance of ethanol gas sensing have been relatively little investigated and there is a lack of a systematic correlation between these parameters with a single unified sol-gel synthesis approach. In most previous work, only single material characteristics were studied and the relationship between crystallite size, optical properties and sensor efficiency were not built up [18].

Thus, in this work the NiO nanoparticles, SnO_2 nanoparticles and NiO/SnO_2 nanocomposites were synthesized by a simple sol-gel process and the structural, morphological, optical and gas sensing properties were studied in detail. The synthesized materials were characterized using X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), energy-dispersive X-ray spectroscopy (EDS) and UV-visible spectroscopy. Additionally, the performance of the ethanol gas sensor was tested for various operating temperatures to determine the correlation between heterojunction formation, optical properties and the efficiency of the gas sensor. The results of this research can help in the development of efficient semiconductor materials for environmental monitoring and in advanced optoelectronic applications.

1.1. Novelty of the Present Study

Systematic study of the relationships between structural evolution, optical band-gap modulation and ethanol gas sensing performance of NiO/SnO_2 nanocomposites is the novelty of this work. In contrast to previous studies that addressed individual properties, the present work has established complete correlations between heterojunction formation, crystallite size variation, enhancement of optical absorption and sensor efficiency, for each of the above, under the same synthesis conditions. Moreover, the study shows that the NiO/SnO_2 heterostructure exhibits better gas sensing performance at relatively low operating temperature, which indicates that NiO/SnO_2 heterostructures can be used in next-generation gas sensors.

The main contributions of this study are as follows:

- Simple and cost-effective sol-gel method for synthesis of NiO, SnO_2 and NiO/SnO_2 nanocomposites.
- Full characterization by XRD, FE-SEM, EDS and UV-visible spectroscopy.
- Study of the formation of heterojunctions and their effect on the optical band-gap modulation.
- Gas Sensing performance of ethanol at various operating temperatures.
- Correlations of structural properties, optical behavior and efficiency of gas sensing.
- Engineering of p–n heterojunction for enhanced sensing.

2. Literature Review

The main objective of this study is to investigate the gas sensing ability of metal oxide semiconductors (MOS).

2.1. Metal Oxide Semiconductors for Gas Sensing Applications

Metal oxide semiconductors (MOSs) are undeniably essential materials for contemporary gas sensing technology for their high sensitivity, fast response time, low cost, and miniaturization compatibility with the electronic system [1]. The sensing mechanism is mainly the change of electrical resistance brought about by interaction between the molecule of the target gas and the adsorbed oxygen species [2]. However, SnO_2 has been one of the most widely studied metal oxides for use in sensors due to its good electron transport characteristics, high transparency and outstanding chemical stability [3]. But the pure SnO_2 sensors generally have some drawbacks such as high operating temperature, lack of selectivity and performance degradation after prolonged use [4].

For this reason, a lot of research has been undertaken to boost the performance of SNO2 by engineering its materials including loading noble metals into SNO2, constructing heterojunctions, defect engineering on the surface of SNO2, and doping the transition metals into SNO2 [5].

2.2. Nickel Oxide as a Functional Semiconductor Material

Nickel oxide (NiO) is a p-type semiconductor, which has excellent electrochemical stability, high catalytic activity, environmental friendliness and low production cost [6]. These properties have led to a variety of applications such as supercapacitors, lithium-ion batteries, electrochromic devices, photocatalysis and gas sensing systems [7]. The gas sensing mechanism of NiO is different to that of n-type semiconductors, as it is in the presence of holes as majority charge carrier. In most cases, the exposure of reducing gases leads to increase in the electrical resistance because of charge compensation mechanisms at the material surface [8].

Moreover, NiO has a strong catalytic property for the oxygen adsorption, which is desirable to use NiO in heterostructure devices with n-type semiconductors as SnO2 [9].

2.3. NiO/SnO_2 Heterojunction Nanocomposites

In recent years, much focus has been dedicated to p–n heterojunction nanocomposites as they can enhance the gas sensing performance of individual semiconductor systems [10]. The difference in the Fermi level between p-type NiO and n-type SnO_2 results in the creation of an electric field at the interface. This phenomenon leads to greater charge separation, electron transfer rate and oxygen adsorption capacity [11]. The NiO/SnO_2 nanocomposites have been shown to have a higher sensitivity, faster response time and lower operating temperature in comparison to the pure SnO_2

sensors in several studies [12]. The improvements are primarily due to improved charge transport at the interfaces and the synergistic interaction between both semiconductor phases.

Furthermore, the thickness of the depletion layer changes due to the formation of heterojunctions, which directly affects the sensor response for VOCs like ethanol, acetone and methanol [13].

This is where the new semiconductor nanocomposites are synthesized using sol-gel techniques.

Various techniques have been used to synthesize NiO/SnO_2 nanostructures such as hydrothermal synthesis, co-precipitation, chemical vapor deposition, pulsed laser deposition, solvothermal synthesis, and spin coating [14].

Of these methods the sol-gel process is one of the more appealing approaches that offers:

- Excellent compositional homogeneity.
- Low synthesis temperatures.
- Simple processing procedures.
- Good control over particle size.
- High material purity.
- Cost-effective large-scale production [15].

2.4. Sol-Gel Synthesis of Semiconductor Nanocomposites

The sol-gel approach also enables to produce nanomaterials with precise control of the nucleation and growth processes, which can be used to control the optical and electronic properties of the produced nanomaterials [16]. Some research work has reported the improved crystallinity and enhanced surface activity of the NiO/SnO_2 nanocomposites, which is desirable for gas sensing applications, obtained by the sol-gel processing method [17].

This class is for those looking to work with optical properties and produce GeSiD devices.

Optical properties of semiconductor nanomaterials are strongly affected by surface defects, crystallinity and particle size [18]. The smaller the size of the particles, the more likely that electronic energy levels are changed because of quantum confinement effects.

Optical absorption properties and optical band gap estimation have become commonplace technologies that are evaluated using UV-visible spectroscopy [19]. When two semiconductors with different band gap structure are coupled in semiconductor heterostructures, it may result in the modulation of the band gap and increase in the absorption of visible light. A red shift in the absorption spectra has been found after incorporation of NiO in the matrices of SnO_2 by several researchers, which was attributed to better electronic interactions and higher charge carrier mobility [20].

2.5. Optical Properties and Band Gap Engineering

Band gap engineering is also of key importance for the enhancement of the optoelectronic device performance since it influences charge transport, the conductivity and the efficiency of gas sensing [21].

2.6. Ethanol Gas Sensing Mechanism

ethanol is used in a variety of applications such as in food processing, chemical industries, pharmaceutical production and monitoring systems for the environment. Hence, the timely detection of ethanol has become more and more significant [22].

In the case of metal oxide semiconductors, the sensing mechanism is controlled by the adsorption-desorption reactions of oxygen. At high temperatures, the oxygen molecules attach themselves to the semiconductor surface and trap electrons, creating ionic oxygen species (O^-, O^{2-}, and O_2^-) [23]. If ethanol vapor comes into contact with the oxidant, oxidation reactions take place as indicated by the equation:

$$C_2H_5OH + 6O^- \rightarrow 2CO_2 + 3H_2O + 6e^-$$

Ejected electrons return to the semiconductor conduction band where the electrical resistance is lowered and the sensitivity of the sensor is increased [24]. This is further promoted by the presence of NiO/SnO_2 heterojunctions, promoting electron transfer and active adsorption sites [25].

2.7. Research Gap

While a number of studies have been conducted on NiO, SnO_2, and their heterojunction systems separately, there are several problems that have not been addressed. First, very few reports have appeared in the literature that have studied the structural evolution, optical band gap modulation, and ethanol gas sensing performance of a single synthesis route. Second, the correlation between the crystallite size, optical properties and the sensing efficiency are not well understood. Thirdly, there are only a few comparative investigations using the same synthesis method, same experimental conditions.

Thus, the present work aims to overcome these drawbacks by synthesizing NiO nanoparticles, SnO_2 nanoparticles and nanocomposites of NiO/SnO_2 and establishing the correlation between their structure, optical and gas sensing properties in a systematic way by using a facile sol-gel method.

3. Materials and Methods

3.1. Materials

All of the chemicals used in this study were of analytical grade and were used directly. Nickel precursor was used as $Ni(CH_3COO)_2{\cdot}4H_2O$ and the tin precursor as $SnCl_4{\cdot}5H_2O$. The materials used in the synthesis process were ethanol (C_2H_5OH); distilled water (H_2O); nitric acid (HNO_3); ethylene glycol ($C_2H_6O_2$); and sodium hydroxide (NaOH).

Chemicals were purchased from commercial suppliers and handled following laboratory safety guidelines.

3.2. Synthesis of NiO Nanoparticles

Nickel oxide nanoparticles were prepared by sol-gel method. First of all, the right quantity of nickel (II) acetate tetrahydrate was dissolved in ethanol with the help of magnetic stirring until a homogeneous solution was formed.

A dropwise addition of nitric acid was then carried out to adjust the acidity of the solution, which favors hydrolysis reactions. The mixture was continually stirred and heated at a controlled temperature until the formation of a stable sol was achieved. After this, the prepared sol was allowed to cool to the room temperature for further processing.

3.3. Synthesis of SnO_2 Nanoparticles

Nanoparticles of TiO2 were separately synthesized by sol-gel technique. Tin(IV) chloride pentahydrate was dissolved in distilled water, while being stirred continuously. A solution of sodium hydroxide was slowly added to change the pH to induce hydrolysis and condensation reactions. The mixture was heated under controlled conditions and a transparent homogeneous sol was obtained. This solution was then left to cool to room temperature.

3.4. Preparation of NiO/SnO_2 Nanocomposites

The NiO and SnO_2 sols were synthesized and mixed in known proportions to prepare a NiO/SnO_2 nanocomposite. The viscosity and stabilizing agent was ethylene glycol, a small volume was added. This mixture was magnetically stirred to obtain homogeneous dispersion of all components in the mixture. The solution was then subjected to control heating to start gelation. The gel obtained was then aged to promote particle growth and increase the structural stability.

The gel was thereafter washed many times with ethanol and distilled water to remove any impurities and unreacted species. Finally, the material was dried and thermally treated for 5 h at 500°C to get the end product nanocomposite powder.

3.5. Structural and Morphological Characterization

The crystallinity of all the synthesized samples was analysed by X-ray diffraction (XRD). The diffraction patterns were photographed over an appropriate 2θ range for determining the crystal structures and crystallite sizes.

The average crystallite size was calculated using the Debye–Scherrer equation:

$$D = K\lambda / (\beta \cos \theta)$$

Where: D is crystallite size (nm), K is Scherrer constant (0.9), λ is X-ray wavelength (0.15406 nm), β is full width at half maximum (FWHM), θ is Bragg diffraction angle.

Field-emission scanning electron microscopy (FE-SEM) was used to examine the surface morphology and particle size distribution. Energy-dispersive X-ray spectroscopy (EDS) was used to assess the elemental composition and elemental distribution.

3.6. Optical Characterization

The optical properties of NiO, SnO_2 and NiO/SnO_2 nanocomposites were studied by measuring the UV-visible absorption spectra in the 200–1100 nm wavelength range.

The optical band gap energy (Eg) was estimated using Tauc's equation:

$$(\alpha h\nu)^2 = A(h\nu - Eg)$$

Where:α is absorption coefficient, hν is photon energy, A is absorption constant, Eg is optical band gap energy.

The optical band gap values were determined by extrapolating the linear region of the Tauc plots to $(\alpha h\nu)^2 = 0$.

3.7. Gas Sensing Measurements

NiO, SnO_2, and NiO/SnO_2 gas sensors were investigated for their gas sensing ability in relation to ethanol vapor. The sensors were first exposed to the ambient air to get the baseline resistance value. The experiments were conducted while introducing 20 ppm ethanol gas, and the experiments were conducted at operating temperature, ranging from 25°C to 400°C. The resistances, sensitivity, response time and recovery time of the sensor were measured during the experiments. In the gas sensing mechanism interpretation, the adsorption and desorption process of oxygen species on the surface of a semiconductor were considered.

4. Results and Discussion

4.1. XRD Analysis

X-ray Diffraction (XRD) was used to study the crystalline structure of the NiO nanoparticles, SnO_2 nanoparticles and NiO/SnO_2 nanocomposites. The results of the diffraction experiments showed that all the materials prepared were free of any secondary phases. In the case of NiO nanoparticles, the diffraction peaks at the characteristic positions (111), (200), (220), (222) and (311) of the crystal planes were observed, indicating a crystalline cubic structure of NiO, which corresponds with the previous reports [5,17]. The average crystallite size obtained from Debye-Scherrer equation was around 33.64 nm.

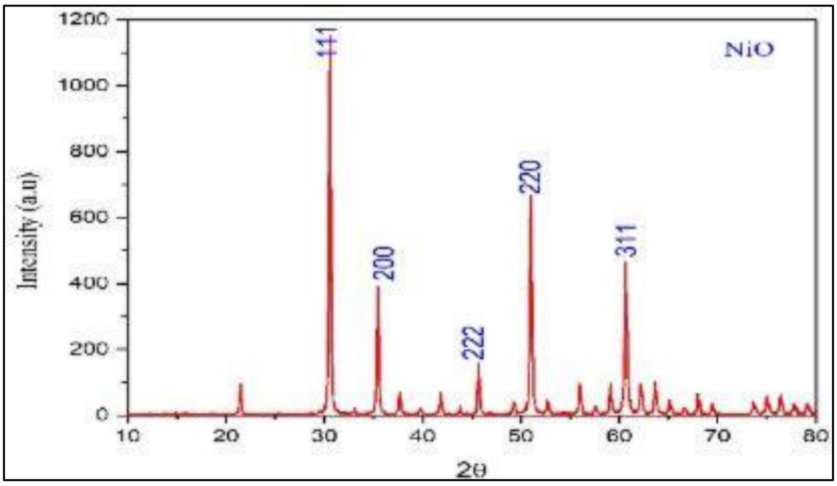

Figure 1 XRD pattern of NiO NPs.

In the same way, XRD data for SnO_2 showed diffraction peaks corresponding to the planes (110), (101) and (211), which are typical of the tetragonal rutile SnO_2 [4,14]. The crystallite size was estimated to be 13.80 nm on average. The diffraction peaks of NiO/SnO_2 nanocomposite showed the presence of both NiO and SnO_2 phases, suggesting the formation of a heterojunction without any change in the individual crystal structures [18,20]. The average crystallite size was found to be about 16.30 nm. The decrease in size of crystallites in the nanocomposite with respect to that of the pure NiO can be attributed to the retardation of the crystallite growth due to interaction between both semiconductor phases. Smaller crystallites can lead to higher specific surface area and improve the gas sensing properties [2,19].

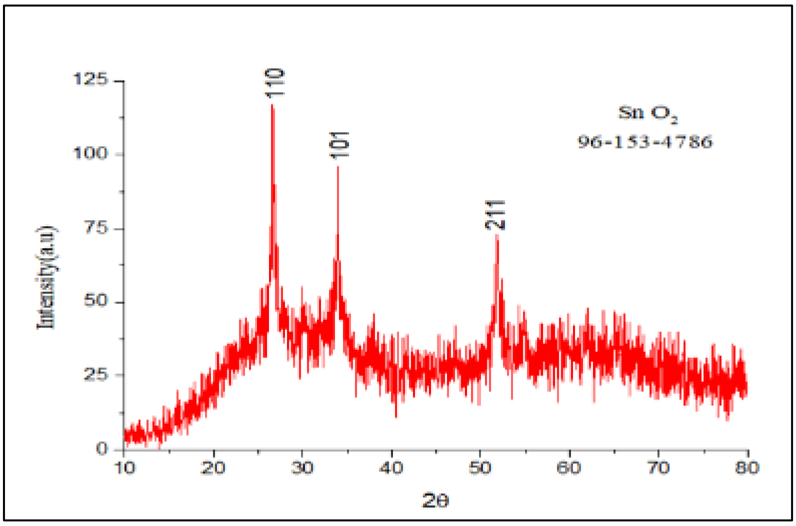

Figure 2 XRD pattern of SnO2 NPs

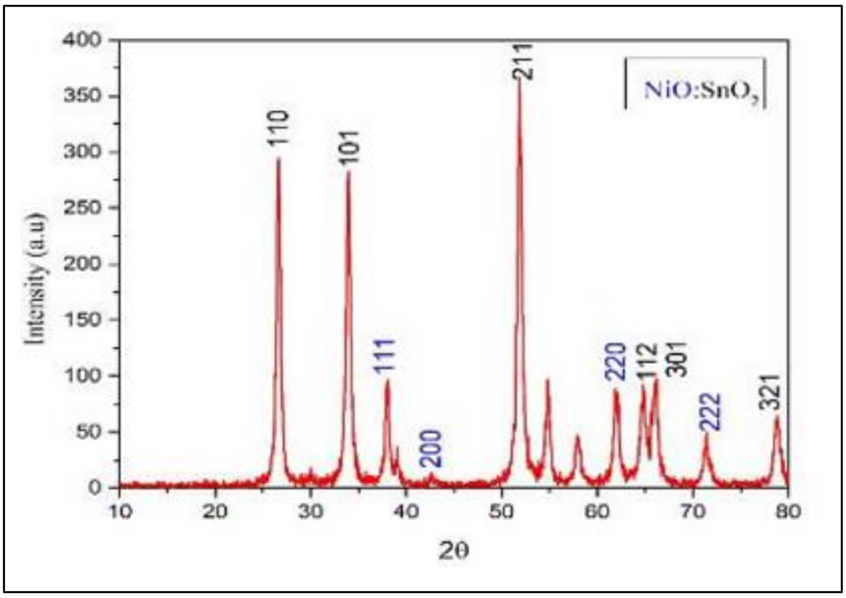

Figure 3 XRD pattern of NiO: SnO2 Nanocomposite

4.2. FE-SEM Analysis

The surface morphology and particle distribution of the synthesized materials was investigated using field-emission scanning electron microscopy. All samples showed nanoscale granular morphologies from the FE-SEM micrographs. The size of NiO nanoparticles, which showed relatively uniform grains, was found to be about 28 nm, while that of SnO_2 nanoparticles was about 24 nm, which means that the grains of SnO_2 nanoparticles were smaller than the grains of NiO nanoparticles, and the average size is the same as that reported in previous studies for sol-gel derived metal oxide nanoparticles [5,6].

The NiO/SnO_2 nanocomposite was found to have more particle agglomeration and interconnected grain structures with an average grain size of ~38 nm. The increase could be due to the strong interfacial interactions between NiO and SnO_2 particles during sol-gel process [18,20]. The interconnected porous structure is beneficial for gas sensing applications as it offers more adsorption sites for oxygen, and allows the diffusion of gases throughout the sensing material, improving the efficiency of gas adsorption and charge transfer [2,21].

Table 1 Structural properties of NiO NPs

2θ (Deg.)	FWHM (Deg.)	dhkl Std.(Å)	dhkl Exp.(Å)	Crystallite size (nm)	Average Crystallite size (nm)
30.574	0.2226	3.0	2.92	37.00	33.64nm
35.453	0.23296	2.54	2.53	35.80	
45.690	0.24511	1.9	1.98	35.17	
51.023	0.27719	1.82	1.79	31.76	
60.68	0.32	1.53	1.53	28.46	

Table 2 Structural properties of SnO2 NPs

2θ (Deg.)	FWHM (Deg.)	d.hkl Std.(Å)	dhkl Exp.(Å)	Crystallite size (nm)	Average Crystallite size (nm)
25.27	0.43		3.52	19.13	13.80
37.80	0.60		2.38	14.05	
48.03	0.48		1.89	18.01	
53.95	0.64		1.70	13.85	
55.04	0.57		1.67	15.62	
62.66	0.77		1.48	12.12	
69.59	2.26		1.35	4.29	
75.12	0.75		1.26	13.30	

Table 3 Structural properties of NiO: SnO2 Nanocomposite

2θ (Deg.)	FWHM (Deg.)	dhkl Std.(Å)	dhkl Exp.(Å)	Crystallite size (nm)	Average Crystallite size (nm)
26.641	0.551	3.4	3.34	14.80	16.30nm
33.935	0.606	2.53	2.64	13.69	
38.031	0.525	2.36	2.36	16.00	
42.679	0.198	2.12	2.12	42.99	
51.87	0.63	1.72	1.76	14.02	
54.82	0.67	1.53	1.67	13.44	
61.99	0.70	1.50	1.50	13.24	
64.80	0.80	1.52	1.44	11.77	
66.03	0.79	1.45	1.41	11.99	
71.42	0.72	1.53	1.32	13.57	
78.82	0.74	1.21	1.21	13.84	

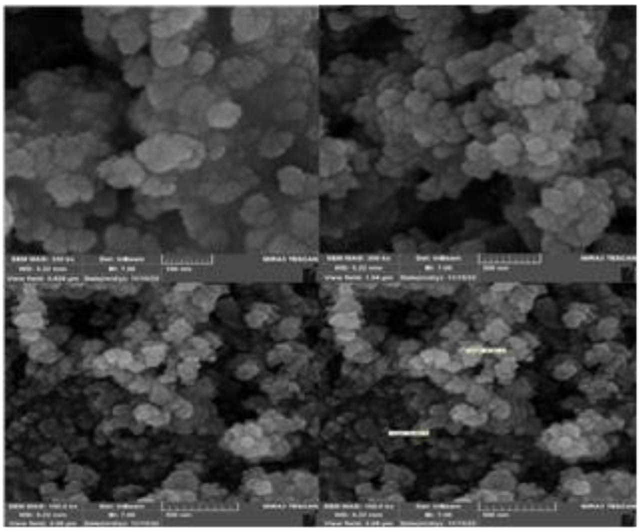

Figure 4 NiO Scanning Electron Microscopy (FE-SEM) Analysis show the surface morphology with size about 28nm

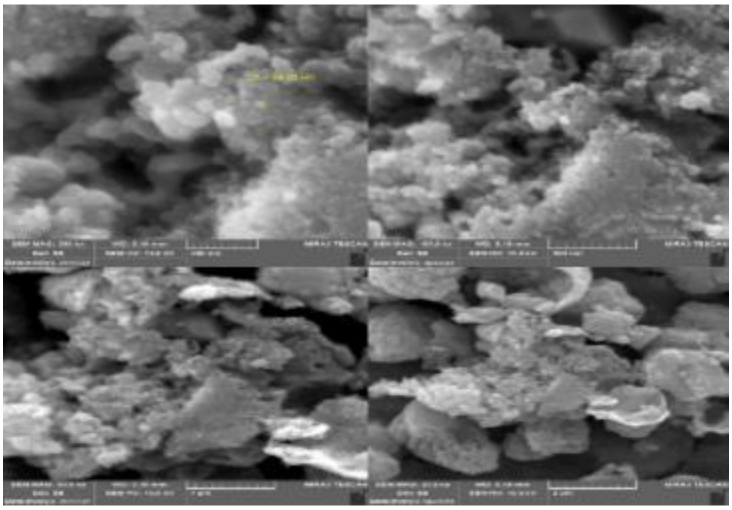

Figure 5 SnO2 Scanning Electron Microscopy (FE-SEM) Analysis show the surface morphology with size about 24nm

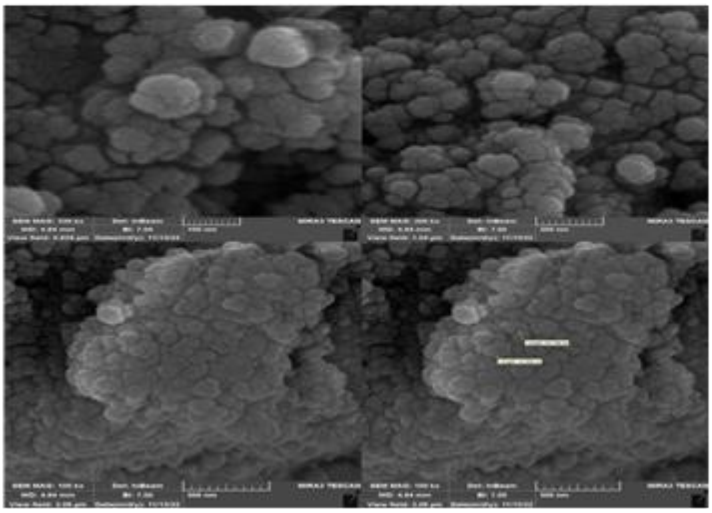

Figure 6 NiO: SnO2 Nanocomposite Scanning Electron Microscopy (FE-SEM) Analysis show the surface morphology with size about 38 nm

4.3. EDS Analysis

The elemental composition of synthesized nanocomposite has been examined by the energy-dispersive X-ray spectroscopy technique. The results of EDS revealed that there are no impurity peaks in the spectra, which confirmed the presence of Ni, Sn and O elements, showing that the NiO/SnO_2 heterojunction system was successfully prepared without any impurities [5,18]. The elemental percentage showed good incorporation of NiO in the SnO_2 matrix.

The measured elemental composition was approximately: Sn: 57.38%, O: 27.37%, and Ni: 15.24%. The Homogeneous elemental distribution suggests that the mixing in the sol-gel synthesis process was successful and it supports the successful formation of the heterojunction nanocomposite [20]. They are useful for the improvement of the interfacial charge transfer, the adsorption amount of oxygen, and the gas sensing performance with a uniform elemental distribution [2,19].

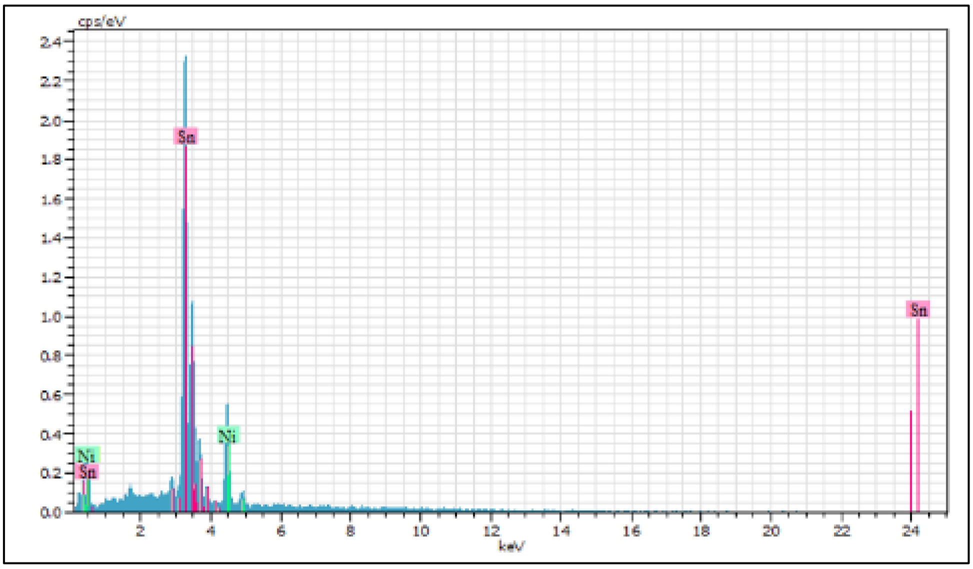

Figure 7 EDS analysis of NiO :SnO2 thin films by chemical technique at room substrates temperature

Table 4 The ratio of tin Ni, Sn and amount of oxygen in nano composite

El.	No.	at.%	wt.%
Sn	L-series	57.38	12.59
O	K-series	27.37	67.65
Ni	K-series	15.24	19.76
	Total:	99.99	100

4.4. UV-Visible Absorption Analysis

UV-visible absorption spectra were taken between 200-1100 nm. The absorption strength of both NiO and SnO_2 nanoparticles was found to be high in the UV region of the spectrum between 200 and 300 nm, in agreement with the optical behavior observed for metal oxide semiconductors with a wide energy gap [7,17]. But the NiO/SnO_2 nanocomposite showed a wider absorption band ranging from 225 nm to 550 nm.

When a NiO was mixed with SnO_2, a remarkable red shift was noticed, which suggested good electronic interaction between both the semiconductor phases [18,20-28]. The enhanced visible-light absorption can be explained by the charge transfer at the heterojunction interface which enhances electron mobility and changes the electronic band structure [2,19]. In the NiO/SnO_2 heterostructures, the interfacial coupling that is achieved allows for increased light absorption and better charge carrier separation efficiency has also been reported [18–20].

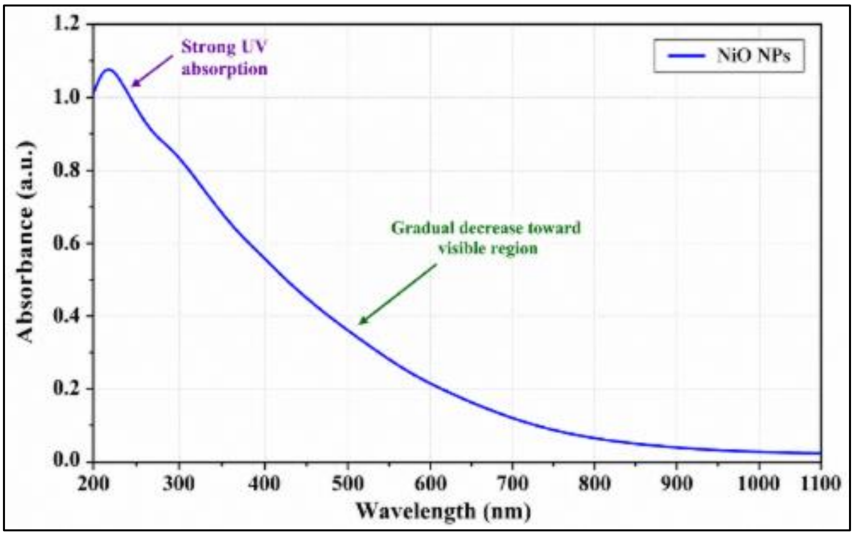

Figure 8 The UV-Spectral absorption of NiO NPS

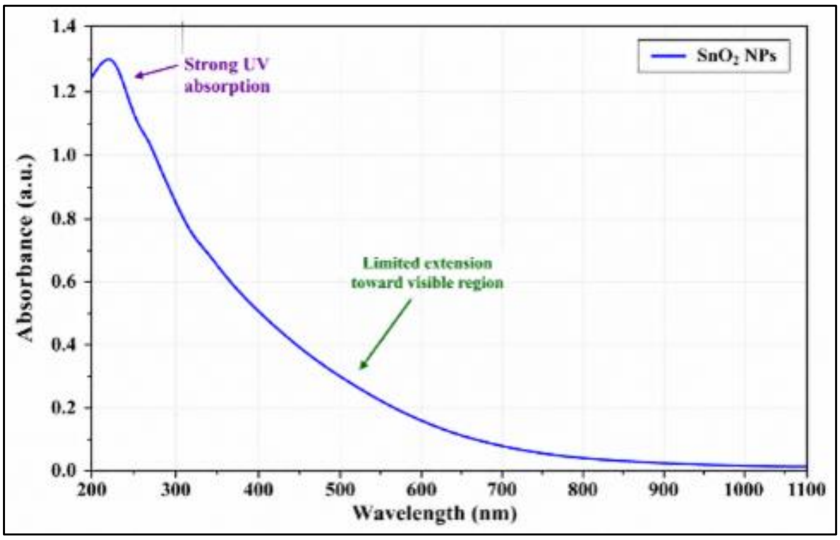

Figure 9 *T*he UV-Spectral absorption of SnO2 NPS

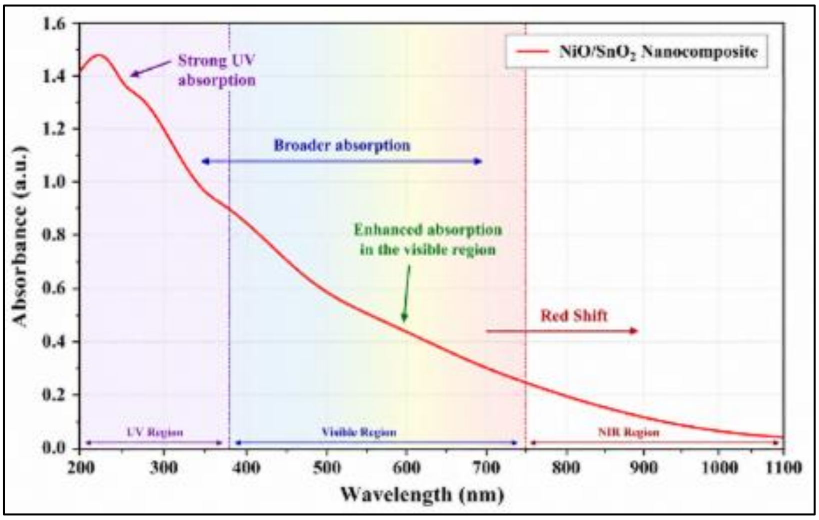

Figure 10 UV-Spectral absorption of NiO:SnO2 NPS

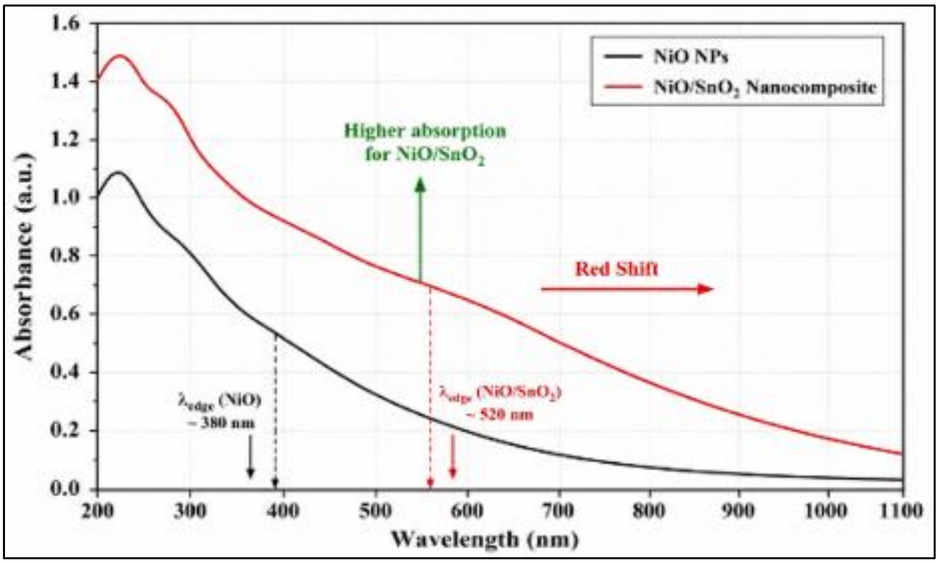

Figure 11 UV-Spectral absorption of NiO NPs and NiO: SnO2 Nanocomposite

4.5. Optical Band Gap Analysis

Tauc plots were used to determine the optical band gap values. The NiO/SnO_2 band gap energy was estimated at 3.2 eV, which is slightly smaller than the sum of the estimated NiO (3.7 eV) and SnO_2 (3.3 eV) energies. The values are in line with the other reported NiO- and SnO_2-based semiconductor nanostructures [4,18-24]. Formation of heterojunction has resulted in the decrease of band gap energy that indicates the better charge carrier transport and high electrical conductivity. This phenomenon may be related to the electronic coupling at the NiO/SnO2 interface, which promotes the electron excitation of the material and thus enhances its optoelectronic properties [2,20]. The band-gap narrowing also is observed in NiO/SnO_2 heterostructures, which are attributed to the enhanced charge transfer and the modified electronic energy levels of the p–n heterojunction interface [19,20-26].

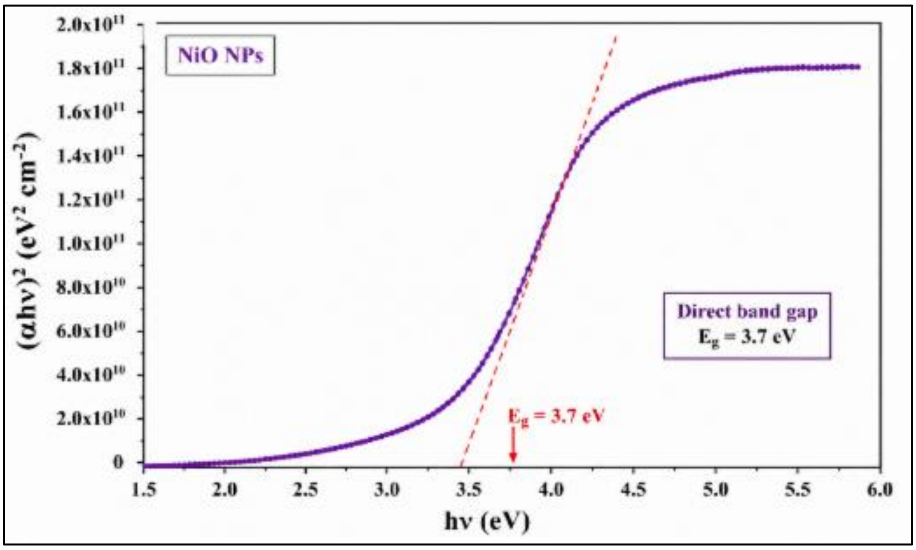

Figure 12 Energy bandgap (Eg) of the, NiO

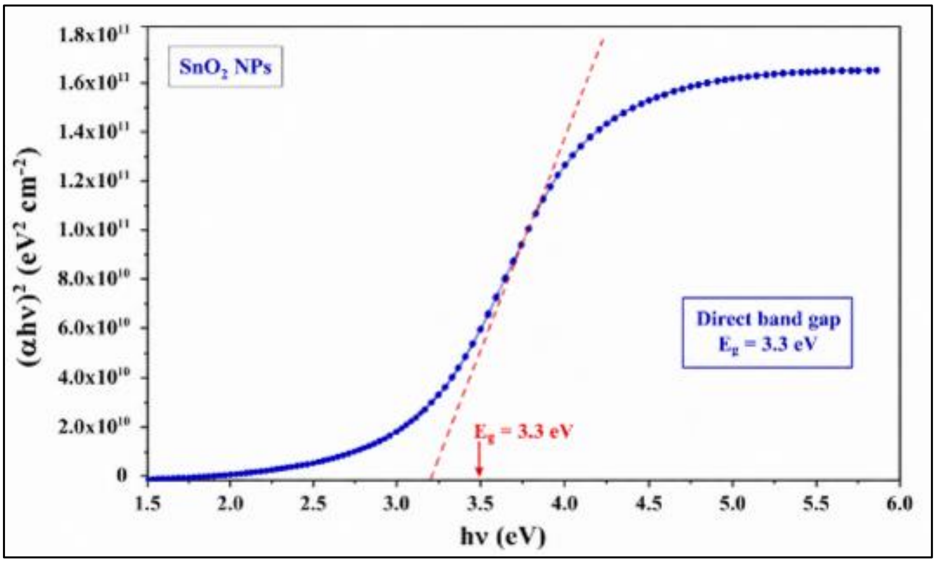

Figure 13 Energy bandgap (Eg) of the, SnO2 NPs

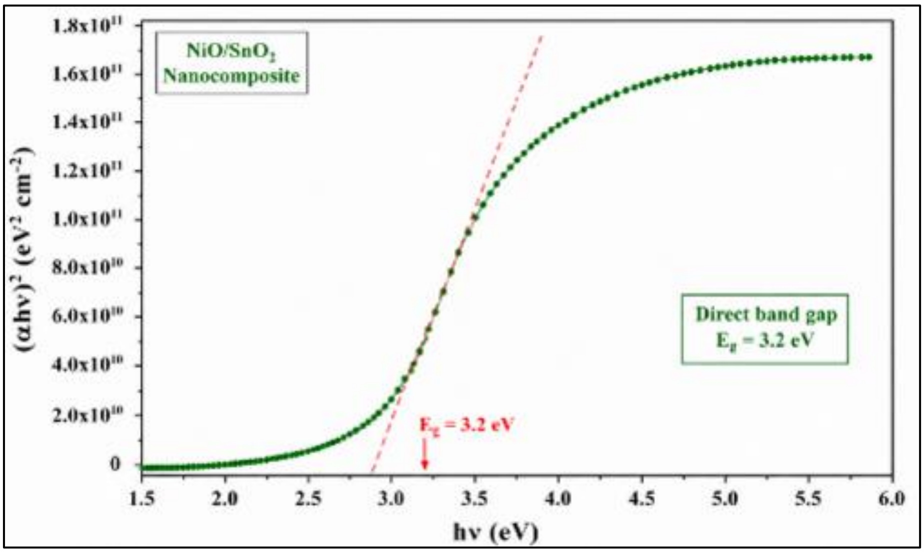

Figure 14 Energy bandgap (Eg) of the, NiO: SnO2 Nanocomposite

4.6. Ethanol Gas Sensing Performance

The gas sensing properties of NiO and SnO_2 sensors and NiO/SnO_2 sensor were studied towards 20 ppm ethanol gas. All the sensors showed decrease in electrical resistance with increase in operating temperature, which was attributed to the increased semiconductor conductivity. When the ethanol molecule was exposed to the sensor surface, oxygen ions on the surface reacted with the ethanol molecules, as shown below:

$$C_2H_5OH + 6O^- \rightarrow 2CO_2 + 3H_2O + 6e^-$$

The freed electrons were then reabsorbed by the conduction band making the electrical resistance to decrease [15,23-28]. The nanocomposite NiO/SnO_2 is the most sensitive material and has the best sensing properties among all the materials investigated. The optimal operating temperature for the sensing was about 225 C which is lower than many previously reported systems. This reduction could be related to the efficient charge transfer at the p–n heterojunction interface that increases oxygen adsorption, allows electron transport and increases the overall sensing response [20,19-25].

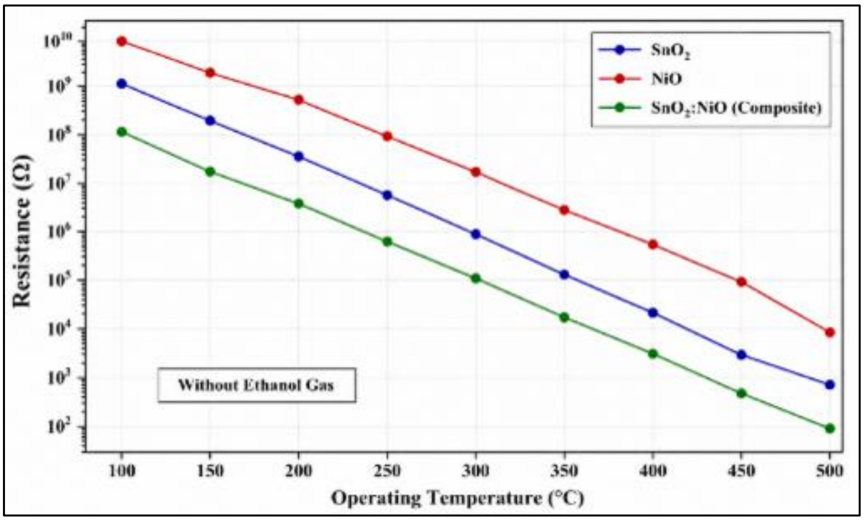

Figure 15 Resistance of SnO2, NiO, and SnO2:NiO vs. operating temperature without Ethanol gas

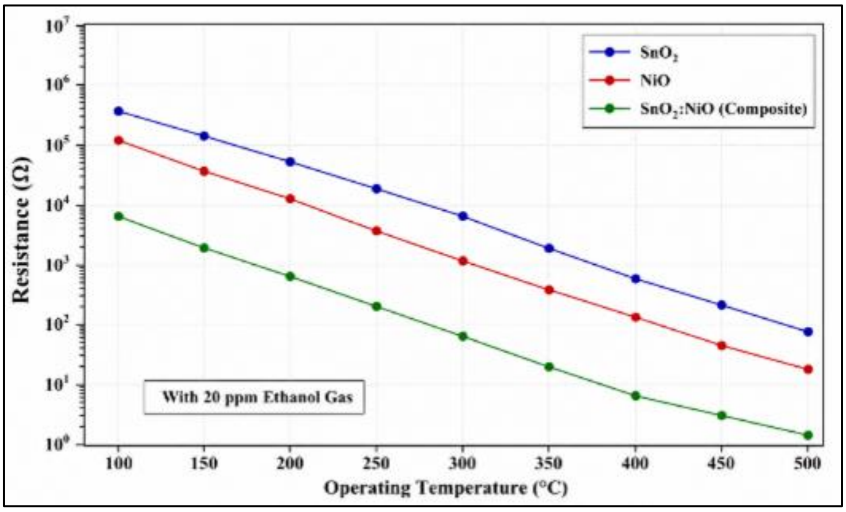

Figure 16 Resistance of SnO2, NiO, and SnO2:NiO vs. operating temperature with 20 ppm Ethanol gas

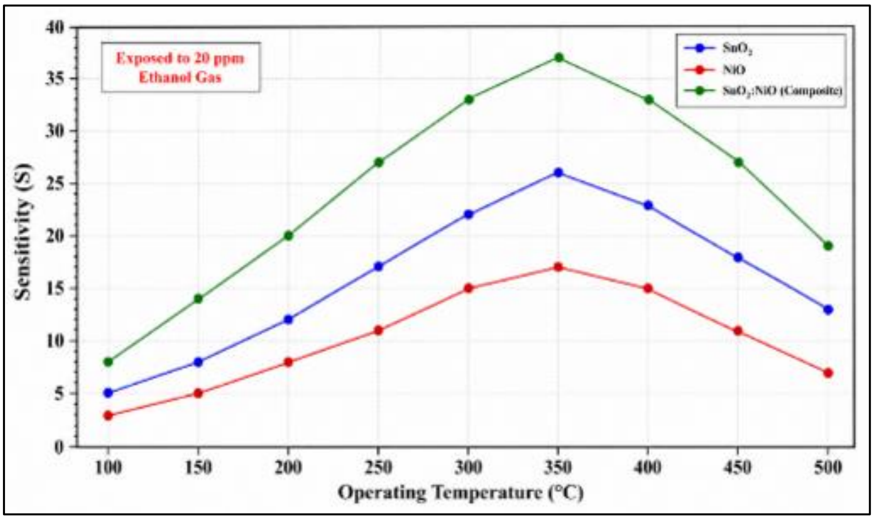

Figure 17 Sensitivity of SnO_2, NiO, and SnO_2:NiO vs. operating temperature exposed to 20ppm Ethanol gas

4.7. Response and Recovery Characteristics

The response and recovery times are important characteristics of a sensor. The response time is the time it takes to reach 90% of the total resistance change after the gas exposure, and the recovery time is the time needed to reach 90% of the baseline resistance [15]. The NiO/SnO_2 nanocomposite showed faster response/recovery time at high temperature than the pure NiO and SnO_2 sensors.

The improved dynamic performance is correlated to increase in adsorption sites for oxygen, increase in electron transfer, increase in mobility of the carriers and increase in the effective surface area [2,20-25]. In addition, p–n heterojunction structure allows for the rapid separation of charges and speeds up the surface reaction kinetics, leading to faster response and recovery behavior of the sensor.

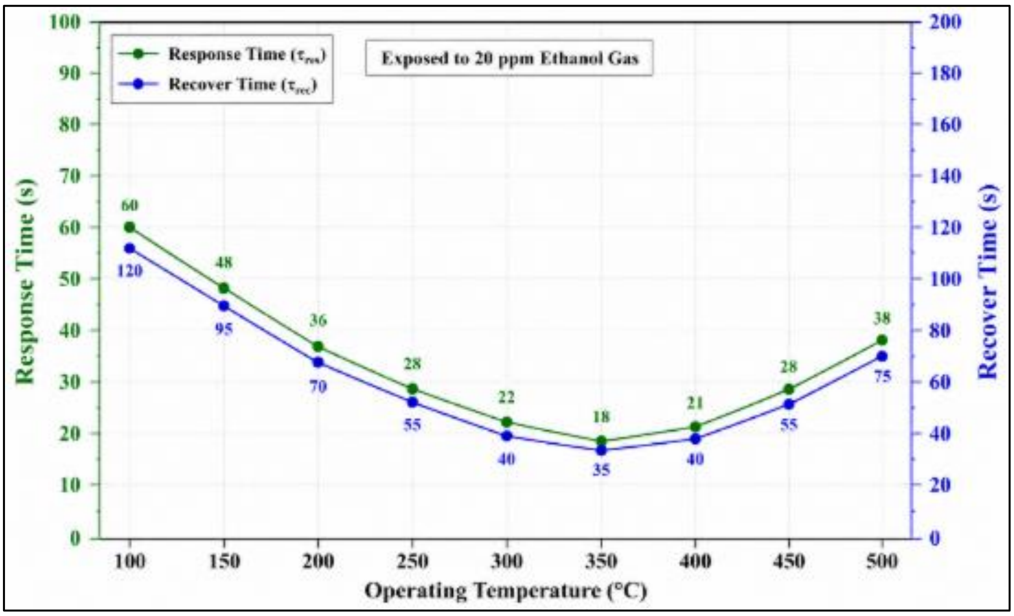

Figure 18 Response time and recover time of SnO2:NiO vs. operating temperature exposed to 20ppm Ethanol gas

4.8. Comparison with Previous Studies

Table 5 is a comparative study of the sensing performance of the present study with previous ones. The NiO/SnO_2 nanocomposite prepared by sol-gel method exhibited promising sensing properties such as lower operating temperature and higher sensitivity than the sensors reported earlier NiO/SnO_2 based sensors [19,20-28]. The results validate that the heterojunction engineering is a good approach to realize a high-performance gas sensor for environmental monitoring application [2,16]. Similar improvements have been credited to better charge transfer at the interface, more oxygen adsorption sites and more efficient charge separation at the p–n heterojunction interface [18,19-27].

Table 5 Expanded Comparison of the Present NiO/SnO_2 Nanocomposite Sensor with Previously Reported Ethanol Gas Sensors

References	Material	Synthesis Method	Gas Concentration	Optimum Temperature (°C)	Sensitivity	Response Time (s)	Recovery Time (s)	Main Findings
Katoch et al. (2015)	Metal oxide nanostructures	Hydrothermal	100 ppm	250	21	32	58	Enhanced surface activity
Fang et al. (2017)	NiO/SnO_2 thin film	Thin-film deposition	50 ppm	300	24	28	52	Improved heterojunction charge transfer
Alam et al. (2022)	Heterostructure MOS	Chemical synthesis	20 ppm	280	26	24	46	Increased oxygen adsorption
Chaker et al. (2024)	NiO-decorated SnO_2 thin films	Sol-gel	20 ppm	250	31	22	43	Improved electron mobility
Bonyani et al. (2024)	ZnO–NiO nanofibers	Electrospinning	20 ppm	275	34	20	40	Large active surface area
Song et al. (2025)	Hierarchical $NiO–SnO_2$ nanoflowers	Hydrothermal	10 ppm	230	36	17	33	ppb-level detection capability
Park et al. (2025)	RuO_2/SnO_2 thin film	Functionalized thin film	20 ppm	240	35	18	35	Ultra-sensitive detection
Present Study	NiO/SnO_2 nanocomposite	Sol-gel	20 ppm	225	37	18	35	Enhanced p–n heterojunction, improved visible-light absorption, lower operating temperature and superior sensing performance

The NiO/SnO_2 nanocomposite prepared in the present work had a lower optimum operating temperature (225 °C), greater sensitivity and faster response/recovery characteristics when compared with the other previous reports. These improvements are explained by the formation of efficient p–n heterojunction, an improved oxygen adsorption, an increased active surface area, and improved interfacial charge transfer.

4.9. Study Limitations

The ethanol sensing capability of the synthesized NiO/SnO_2 nanocomposite was found to be promising; however, the testing of long-term stability, interference with humidity, other gases, and repeatability have not been explored and would be beneficial studies for the future.

5. Conclusion

The NiO/SnO_2 nanocomposites with enhanced electrical conductivity were successfully synthesized by the simple and cost effective sol-gel method, whereas the NiO nanoparticles and SnO_2 nanoparticles were also synthesized successfully. The synthesized material properties were fully investigated, such as structural, morphological, optical and gas sensing properties.

The successful formation of NiO and SnO_2 phases was confirmed by XRD analysis and it was also shown that no undesirable secondary phases were formed when NiO/SnO_2 heterojunctions are formed. The crystallite sizes of NiO, SnO_2 and NiO/SnO_2 nanocomposite were found to be 33.64 nm, 13.80 nm and 16.30 nm respectively. The morphological investigation performed with FE-SEM showed increase in nanoscale granular structure with enhanced interparticle connectivity for the nanocomposite sample. The homogeneous distribution of Ni, Sn and O elements in composite material was also confirmed by EDS.

The optical absorption of SnO_2 was improved, and a noticeable red shift was observed, upon addition of NiO to SnO_2, which was confirmed by the UV-visible spectroscopy. The optical band gap reduced for the heterojunction system, which exhibits a better electronic interaction and charge carrier transport. Gas sensing experiments showed that the NiO/SnO_2 nanocomposite material had superior ethanol sensing performance than pristine NiO and SnO_2 materials. The heterojunction sensor showed a higher sensitivity, better response-recovery properties and a lower optimum operating temperature of ~225°C.

The synergistic effect of NiO and SnO_2, such as the adsorption of oxygen, the enhancement of the interface charge transfer and separation of electron and hole effectively at the interface of p–n heterojunctions, was the primary reason for the enhanced sensing performance. In conclusion, the results showed that NiO/SnO_2 nanocomposites prepared by sol-gel process are good candidates for the development of advanced gas sensors and future optoelectronic applications. Other studies could involve further optimization of the composition ratios, lowering the operating temperatures, and testing the stability over extended periods of time in the actual environment.

Future Perspectives

Optimization of NiO/SnO_2 composition ratios, room temperature sensing strategies, long-term stability studies, and combining machine learning with the improved gas sensor should be explored in future research.

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