Fabrication of NiO/SnO2 heterojunction based photocatalyst for efficient sunlight degradation of organic dyes

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ABSTRACT
A new composite oxide (NiO/SnO2) photocatalyst was synthesized by simple co-precipitation route. The structural elucidation of composite oxide photocatalyst was carried out via X-rays diffraction (XRD) technique. The morphology was determined by scanning electron microscopy (SEM). The Fourier transform infra-red (FTIR) and UV–visible spectroscopic techniques revealed the spectral and optical features. XRD analysis confirmed the formation of binary metal oxide nanocomposite comprising NiO in cubic and SnO2 in the tetragonal crystalline phase. The crystallite size for NiO/SnO2 calculated from XRD was <15 nm. The spectroscopic studies also supported the XRD data. The morphological study of NiO/SnO2 revealed the formation of relatively small rounded nanoparticles. The solar light driven photocatalytic degradation study showed about 54% degradation of methylene blue and 34% degradation of Congo red and the value of rate constant for NiO/SnO2 was found to be 0.0059 min⁻¹. The mechanism of degradation of dyes was also explored and discussed.

1. Introduction
The world is growing at a very faster pace. Different industries, research laboratories are revolutionizing the world in order to cope up with the demands of the ever-increasing population. These areas are adversely affecting our environment. These industries and research laboratories discharge their harmful effluents directly into freshwater canals, rivers, oceans, etc. without proper treatment which in return destroy our natural habitat as well as aquatic life. Different organic dyes, oxidizing agents, antibiotics, drugs are present in these effluents which is alarming. Thedyesinwaterareharmfultoplants/animalsandcausecarcinogenicity,teratogenicityandendocrinedisruptions in humans [1]. Quality of life and water is at serious threat if left untreated. A number of physical (screening, flotation, reverse osmosis, multimedia filtration, adsorption) [2–4] and chemical methods (neutralization, flocculants and coagulants, oxidation, ozonation, ion exchange) are being employed already [5,6]. However, these methods have their limitations such as expensiveness, uninterrupted power supply and high maintenance requirements. So, in this regard, environment-friendly green and light-driven photocatalysis has attracted much attention of researchers for the efficient removal of toxic effluents [7,8].

Semiconductors (transition metal oxides TMO’s) has the best potential for the degradation of toxic and virulent dyes as they are abundant in nature, easily available, have variable oxidation state, tunable bandgap, wide absorption range, stable and low cost [9,10]. Earlier, ZnO2, TiO2, PbO2, V2O5 were utilized for photocatalytic applications but soon they lost their significance because of short electron hole recombination time, wider bandgap, absorb UV radiation for the electrons to move from the valence band to conduction band [11–13]. Photocatalysis driven by visible light is the need of the hour. As the morphological study of nano particles reveals them to be extremely small in size and hence adhere to the surface of chemical pollutants eliminating them easily. Co3O4, Bi2O3, In2O3, SnO2 and NiO are considered more significant as sunlight contains ~43% visible light and ~4% UV light [14].

Among metal oxides, NiO is a p-type (hole-type) semiconductor with a broad energy bandgap that ranges between 3.6 and 4 eV [15], a well-known substance with distinguishing electrochemical and optical characteristics [16]. It is also a green nanomaterial with a fast-switching speed, excellent conductivity, well-defined uniform properties and steady Redox kinetics and an efficient charge-transport system [17]. SnO2 is n type semiconductor with a bandgap of 4.2 eV.
excellent electrical and chemical properties and cyclic stability [19].

Visible light-driven photocatalysts, NiO and SnO₂ are potent materials due to their non-toxic nature. SnO₂ is n-type and NiO p-type material widely used in lithium-ion batteries. SnO₂ has the high efficiency and potential to degrade the pollutants due to its strong oxidation power, but it has negative consequences on human health, such as chronic poisoning [20], toxicity of the lungs [21] and testicles [22] which has limited its use. When n-type SnO₂ is coupled with p-type NiO, a heterojunction is formed. Electrons move from SnO₂ to NiO which favours the separation of the electron with holes improving its photocatalytic activity.

Herein this report, we have synthesized two different metal oxides NiO/0.5 g SnO₂ and NiO/1 g SnO₂ nanocomposite via the coprecipitation route which is economically favourable, gives high yield and handling is easy. The main purpose of this study is to enhance the photocatalytic activity of metal oxides by finding the optimum concentration of SnO₂ and the findings have truly shown our achievement.

2. Experimental work

2.1. Materials and chemicals

Chemicals, solvents and reagents of high purity and analytical grade were procured from Sigma Aldrich and utilized as received, with no additional purifications: NiCl₂·6H₂O (99.9%), SnCl₂·5H₂O (98%) and NaOH (97%). All experiments were conducted using deionized water.

2.2. Synthesis of NiO

Nickle chloride hexahydrate (NiCl₂·6H₂O) and sodium hydroxide (NaOH) were used as starting materials to synthesize NiO nanoparticles. At first, 4 g of NiCl₂·6H₂O was dissolved in 250 mL of deionized water as the solvent and the solution was magnetically agitated for 40 min at 323 K temperature. Following that 20 mL of 2 M NaOH solution was added dropwise until pH becomes 8. Thus, the obtained green gel was washed with deionized water and ethanol to remove by-products if any and dried at 333 K for 14 h and the dried samples were annealed at 773 K for 2 h to obtain NiO nanoparticles [23]

\[ \text{NiCl}_2 \cdot 6\text{H}_2\text{O} + \text{H}_2\text{O} + 2\text{NaOH} \cdot \text{O} + 8\text{H}_2\text{O} + 2\text{NaOH} \]

(1)

2.3. Synthesis of SnO₂

To achieve pure SnO₂ nanoparticles, 4 g of SnCl₂·5H₂O was dissolved in 200 mL of distilled water and the solution was stirred for 40 min by using a magnetic stirrer. Then 2 M NaOH solution was prepared and added dropwise until pH becomes 9. After 2 h the precipitates were washed with equimolar deionized water and ethanol. The obtained particles were oven-dried at 353 K and annealed at 773 K for 2 h in a muffle furnace before using for further analysis [24]

\[ \text{SnCl}_2 \cdot 5\text{H}_2\text{O} + 4\text{NaOH} \rightarrow \text{Sn(OH)}_2 \downarrow + 4\text{NaCl} \]  

(2)

\[ \text{Sn(OH)}_4 \rightarrow \text{SnO}_2 + 2\text{H}_2\text{O} \]  

(3)

2.4. Synthesis of nanohybrids

Metal oxide nanohybrids of varying concentrations were synthesized via the simple coprecipitation method. About 4 g of NiCl₂·6H₂O was dissolved in 250 mL of deionized water as a solvent and then 0.5 g SnO₂ was added to the same solution. Which is then magnetically agitated for 40 min at 323 K. After that, 20 mL of NaOH

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**Figure 1.** Schematic illustration of NiO, SnO₂ and nanocomposite oxide.
solution was added dropwise till a pH of 8. Thus, the resulting green gel was washed with deionized water and ethanol to eliminate any by-products and dried at 333 K for 14 h before being annealed at 773 K. The same NiO is also prepared in the presence of 1 g SnO2 and used further for photocatalytic activity (Figure 1).

2.5. Photocatalytic experiment

Photocatalytic degradation was carried out under sunlight. For experiment, 0.001 mg of the photocatalyst was added into 100 ml of aqueous solution of dye methylene blue (MB) having a concentration of 5 ppm. The solution was magnetically stirred for 120 min under dark to achieve adsorption–desorption equilibrium of methylene blue on the surface of the photocatalyst. Almost 3 ml of the aliquot part was taken from the reaction mixture after a time interval of 20 min and centrifugation was done to separate the catalyst. To observe the change in concentration of MB, the dye was analysed under natural sunlight.

3. Results and discussions

3.1. X-rays-diffraction analysis (XRD)

The X-ray diffraction (XRD) technique was employed to characterize the aforementioned nanoparticles. The as-synthesized NiO, SnO2, NiO/0.5 g SnO2 and NiO/1 g SnO2 nanocomposite were analysed via X-ray diffraction (6100) instrument from Schimadzu with Cu Kα radiation at 40 kV and 50 mA. XRD patterns were recorded in the range of 10–80° (2θ) at a scanning rate of 3° min⁻¹. The XRD library database was used to identify the structural composition and crystalline phases of samples. Figure 2 is in good accordance with the cubic phases of NiO standard peaks (JCPDS 01-073-1523). The strong peaks of NiO appear at 2θ, 37.1°, 43.1°, 62.6°, 75.1°, 79.3° corresponds to the diffraction planes at (111), (200), (220), (311) (222), respectively. While SnO2 shows characteristic peaks at 2θ = 26.1°, 33.4°, 37.1°, 51.3°, 64.6°, 70.7° at diffraction planes of (110), (101), (200), (211), (112), (202), (321), respectively, which confirms the tetragonal structure of SnO2 (JCPDS 0-001-0625) [25]. The broad and intense XRD pattern for nanocomposites show the characteristic peaks of both the components as shown in Figure 1. The crystallite size of the NiO is approximately 15 nm and SnO2 4.29 nm which is calculated by the famous Scherrer equation given as

\[ D = \frac{K\lambda}{\beta \cos \theta} \] (4)

where K is the Scherrer constant and its value is 0.9, β the full width half maxima (FWHM) of the peak in radians, D is crystallite size, λ is the wavelength (0.15406) of the X-ray source and θ denotes the peak position (Figure 2).

3.2. FTIR analysis

To observe the chemical groups present in the compound, the FTIR spectra were recorded. All FTIR measurements were carried out at room temperature. Regarding metal oxide, they showed lower absorption peaks below 1000 cm⁻¹. The spectral peaks at 424 and 408 cm⁻¹ correspond to the stretching vibrations of Ni–O [23] and IR absorption bands at 426 and 406 cm⁻¹ were assigned to the antisymmetric vibrations of O=Sn=O [27]. This FTIR study supported the XRD data and confirmed the existence of NiO and SnO2 in binary metal oxides nanocomposites as shown in Figure 3. All these measurements were recorded by an FTIR 6700 Nicolet™ Fourier transform infrared (FTIR) spectrometer.

3.3. SEM analysis

SEM was used to examine the morphology of NiO (a), SnO2 (b), NiO/0.5 g SnO2 (c), and NiO/1 g SnO2...
Figure 4. SEM images of NiO, SnO₂, NiO/0.5 g SnO₂, NiO/1 g SnO₂ in (a), (b), (c), (d), respectively. (d). Figure 4 shows the irregular shaped particles of NiO and SnO₂ and the size calculated is approximately 30 nm.

3.4. UV–visible analysis

The UV–visible spectra were recorded in the range of 200–800 nm to estimate the optical behaviour of binary nanocomposites. All recorded UV–visible graphs are recorded on AGILENT CARY 4000/5000/6000i OPTICS and shown in Figure 5. All these studies were carried out under ambient conditions. The solid particles were suspended in aqueous media by sonication for a few hours. After sonication, the settled particles were filtered off, the aqueous solution containing catalyst particles were subjected to UV–visible spectra. According to the literature NiO exhibits the absorption band at 344 nm [28] and SnO₂ shows an absorption band at 370 nm [29]. In the current study NiO showed the absorption spectra at 318 nm and SnO₂ showed absorption peak at 317 nm. The composite oxides such as NiO/0.5 g SnO₂ and NiO/1 g SnO₂ showed absorption peaks at 303 and 300 nm respectively.

![Figure 5. UV–visible absorption spectra of NiO, SnO₂, NiO/0.5 g SnO₂ and NiO/1 g SnO₂.](image)

3.5. Bandgap energy

It is essential to investigate the bandgap of photo catalytically active material in order to quantify its photocatalytic response. The bandgap energy of NiO/SnO₂ nanocomposites was calculated using the Tauc plot technique.

\[
\frac{1}{n} = \frac{A(h\nu - E_g)}{A(h\nu)}
\]

Figure 6 shows the indirect bandgap for NiO 3.29 eV, SnO₂ 3.39 eV, NiO/0.5 g SnO₂ 3.09 eV, NiO/1 g SnO₂ 3.0 eV. The bandgap we reported here is the lowest bandgap possible for NiO/SnO₂ as compared to their
Figure 6. (a) Bandgap energy of NiO (b) bandgap energy for SnO$_2$ (c) bandgap energy for NiO/0.5 g SnO$_2$ (d) bandgap energy for NiO/1 g SnO$_2$.

Figure 7. (a) Degradation profile of MB in the presence of NiO (b) SnO$_2$, (c) NiO/0.5 g SnO$_2$ and (d) NiO/1 g SnO$_2$. 
Figure 8. (a) $A/A_0$ plot for NiO, SnO$_2$ and NiO/SnO$_2$ composites (b) $-\ln(A/A_0)$ plot for NiO, SnO$_2$ and NiO/SnO$_2$ composites, (c) degradation (%) of NiO, SnO$_2$ and NiO/SnO$_2$ composites. (d) Rate constant for NiO, SnO$_2$ and NiO/SnO$_2$ composites.

Figure 9. (a) $A/A_0$ plot of MO, CR, RB, CV, (b) $-\ln(A/A_0)$ plot for MO, CR, RB, CV, (c) degradation plot for MO, CV, CR, RB and (d) rate constant for MO, CR, CV and RB.
oxides. This optical property makes our samples active in sunlight.

3.6. Photocatalysis

To study the photocatalytic degradation efficiency of the synthesized samples, the methylene blue (MB) solution was used. Figure 7(a–d) shows the visible spectra of MB in the presence of prepared catalysts (NiO, SnO₂, NiO/0.5 and NiO/1 g SnO₂). The spectra were recorded at a consecutive time interval of 20 min in the presence of sunlight. As the reaction proceeds, there is a constant decrease in the absorption spectra of methylene blue [30–32].

According to the literature, individual oxide photocatalytic degradation is poor due to high optical bandgap energy and electron hole–pair recombination; nevertheless, studies have revealed that binary composites have outstanding degradation efficiency. NiO/0.5 g SnO₂ which has 54% efficiency, this might be attributed due to the formation of heterojunction between NiO and SnO₂ which lessens the electron–hole pair recombination. Figure 6 shows 0.8% degradation of methylene blue under dark and 2.2% degradation under sunlight without catalyst. The photocatalytic process was further investigated by studying kinetics, in order to study the kinetics, pseudo first-order rate equation was considered [33,34]

\[
\ln \frac{A_0}{A_t} = K_t t
\]

The results obtained using this equation are illustrated in Figure 8 and the rate constant for NiO/0.5 g SnO₂ is 0.00598 min⁻¹ and NiO/1 g SnO₂ is 0.00611.

A comparative study with Congo red, crystal violet, Rhodamine B, methyl orange was also established to evaluate the photocatalytic degradation of NiO/0.5 g SnO₂. The results showed the maximum degradation of Congo Red which is 34% and it is degraded to the

Table 1. Kinetic parameters and percentage degradation of synthesized samples.

| Photocatalyst   | Degradation time (min) | Catalyst amount (mg) | %Degradation (%) | Rate constant (min⁻¹) | Adj.R²   |
|-----------------|------------------------|----------------------|------------------|------------------------|---------|
| NiO             | 120                    | 1                    | 34%              | 0.0035                 | 0.9456  |
| 0.5 g NiO/SnO₂  | –                      | –                    | 54%              | 0.0059                 | 0.9014  |
| 1 g NiO/SnO₂    | –                      | –                    | 52%              | 0.006                  | 0.9888  |

Figure 10. A/A₀ plot of EDTA, AgNO₃, 2-propanol, (b) -lnA/A₀ plot of EDTA, AgNO₃, 2-propanol, (c) degradation % of EDTA, AgNO₃, 2-propanol and (d) rate constant for EDTA, AgNO₃, 2-propanol.
Table 2. Comparative performance of 0.5g NiO/SnO₂ with previously reported analogous photocatalysts.

| Photocatalysts    | Degradation efficiency (%) | Reference |
|-------------------|----------------------------|-----------|
| EDA–SnO₂          | 96.3                      | [36]      |
| CdO–NiO           | 78                        | [37]      |
| NiO–CdO–ZnO       | 98                        | [38]      |
| CTAB–SnO₂         | 95                        | [36]      |
| 0.5gNiO/SnO₂      | 54                        | Present work |

maximum among others. Kinetics of NiO/0.5g SnO₂ is also shown in Figure 9 which showed a rate constant of 0.0030 min⁻¹ with Congo red.

### 3.7. Photocatalytic mechanism

To examine the primary active species involved in the degradation reaction accelerated by solar radiations, a quenching experiment was carried out. Silver nitrate (AgNO₃) was used to quench the electrons for this purpose. Ethylenediaminetetraacetic acid (EDTA) was employed as a hole quencher, 2-propanol was also utilized to quench superoxide anion radicals (·O₂⁻) and hydroxyl radicals (·OH). Figure 10 shows the degradation mechanism of methylene blue with NiO/0.5 g SnO₂. Conduction band (E₉) and valence band (Eᵥ) edge potentials of NiO and SnO₂ are proposed for understanding the mechanism of the photocatalysed process [35]. The various kinetic parameters and comparison of reported present catalyst with previously reported catalysts are given in Tables 1–2. In Table 3, the comparison of different organic dyes us given. The schematic mechanism of photocatalysis is given in Figure 11.

$$E₉ = X − Eₑ − 1/2E₉$$  
$$E₉ = Eᵥ−E₉$$

The photocatalytic reaction mechanism can be described as

$$\text{NiO/SnO₂} + \text{hv} \rightarrow \text{NiO(h}^+\text{)} + \text{SnO₂(e}^−\text{)}$$  
$$\text{SnO₂(e}^−\text{)} + \text{O₂} \rightarrow \text{SnO₂ + O}^2−$$  
$$\text{O}^2− + \text{H₂O} \rightarrow \text{HO}^− + \text{OH}^−$$  
$$\text{HO}^− + \text{H₂O} \rightarrow \text{HO}^− + \text{H₂O₂}$$  
$$\text{H₂O₂} \rightarrow 2\text{OH}^−$$  
$$\text{MB + OH}^− \rightarrow \text{Degradation of methylene blue}$$

### 4. Conclusion

The study was aimed to compare the photocatalytic degradation of methylene blue, Congo red, crystal...
violet, methyl orange and rhodamine B by as-prepared composite oxide catalyst. To estimate the main active species involved in the degradation of the photocatalyst, the quenching experiment was also designed. The composites oxide NiO/SnO2 have been synthesized by the facile coprecipitation route. The XRD broad and intense dual peaks of the composite has shown their formation. The SEM morphology showed irregular morphology. The NiO/SnO2 showed 54% degradation of MB and 34% of Congo red. The quenching experiments have shown that transfer of electrons is the major active species involved in the degradation mechanism.

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