Effect of Ni doping on the structural, optical and photocatalytic activity of MoS$_2$, prepared by Hydrothermal method

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Abstract

Transition metal dichalcogenides (TMDs) are promising materials for photocatalytic functions. In class of TMDs, MoS$_2$ is comprehensively explored as a co-catalyst due to the extraordinary activity for photocatalytic activity of organic dye degradation. But the catalytic activities of MoS$_2$ are generated through S ions on depiction edges. Also numerous of S ions existed on basal planes are catalytically inactive. The insertion of external metals in MoS$_2$ organism is extensive way for activation of basal planes surface to enhance concentration of catalytically active sites. For this purpose, nanoparticles of Nickel (Ni) doped MoS$_2$ are prepared by hydrothermal technique. Structural and morphological analysis are characterized by XRD and SEM, respectively. XRD results showed that Ni is completely doped into MoS$_2$. SEM showed that pure MoS$_2$ has sheet like structure and Ni doped MoS$_2$ has mix disc and flower like structure. Band gap energy was observed in declining range of 2.30–1.76 eV. The photocatalytic activity of pure MoS$_2$ and Ni doped MoS$_2$ were evaluated by degrading MB and RhB dyes under UV light irradiation. MB dye degradation of MB was 71% for pure MoS$_2$. For 1% to 5% Ni doping in MoS$_2$, MB dye degraded from 85% to 96%. It means that MB dye degradation of MB was enhanced continuously by increasing the concentration of Ni doping. RhB dye degradation of RhB was 62% for pure MoS$_2$. For 1% to 5% Ni doping in MoS$_2$, the RhB dye degraded from 77% to 91%.

1. Introduction

Renewable energy, photocatalysts and nanoscience have attracted the interests of many researchers from last couple of decades [1–5]. Various nanoscale semiconductors can be developed through different synthesis techniques. Organic or inorganic pollutant materials can be removed by the application of ZnO, Fe$_2$O$_3$, TiO$_2$, CdS and MoS$_2$ photocatalysis materials [6–11]. Currently, higher surface to volume ratio and quantum effects have boost the significance of nanomaterials in fields including pollution, smell prevention and renewable energies [12]. Compositional variations, structures, shapes and dimensions of semiconductors have defined such nanomaterials in technological applications [13]. Photo-oxidation and photo-reduction are numerous terms applied for oxidation and reduction executed in light. Band gap excitation appears by partitions of charges when light strikes in semiconductor materials. Oxidation and reductions on substrates can be initiated by the application of electrons and holes generated by light. Charge transformations amongst molecules at nanoscale, the materials have important part for photocatalysis development [14–16]. ZnO semiconductors have wide band gap, energetic under UV region of light and embrace just 5% of light [17]. Various researchers have reported the enrichment in photocatalytic performance through adjustment of hetrojunctions of ZnO with TiO$_2$, Ag$_2$O, Cu$_2$O etc semiconductors [18–21]. Recently for photocatalysis applications, transition metal
sulphides have become potential candidate and have successfully substituted the noble metals [22–24]. P-type semiconductor molybdenum disulphide (MoS2) having contracting hand gap (1.7 eV) have drawn the attention of researchers. MoS2 have powerful adsorption in visible range of solar spectra and is categorized as a stable layer metal dichalcogenides [25]. Feng-Jun Zhang et al have synthesized MoS2 photocatalyst having stronger photocatalytic hydrogen generation efficiency [26]. Youzi Zhang et al have prepared the MoS2 with 242% enhancement in production of H2 gas [27]. Said Ridene has investigated improving results by fabricating MoS2 and employing in laser emitting gadgets [28]. Adhimoorthy Saravanan et al have composed the MoS2 nanomaterials for future gas sensor and storage equipments [29]. Various metals can be doped into P-type semiconductor molybdenum disulphide (MoS2) to explore the potential for various applications. The insertion of external metals in MoS2 classification is an extensive way for activation of basal planes surface to enhance concentration of catalytically active sites. For this purpose, nanoparticles of Nickel (Ni) doped MoS2 are prepared by hydrothermal technique to expand photocatalytic activity of MoS2 nanomaterials to develop visible light state of solar spectra. In current research work MoS2 and Ni2+ doped MoS2 have been developed for photocatalytic applications. Ni was inserted with different ratios of 1–5%. Hydrothermal technique was employed to fabricate such nanomaterials. After synthesis XRD, SEM, UV and photocatalytic characterizations are examined of MoS2 and Ni (1–5%) doped MoS2.

2. Experimentation

MoS2 solution was prepared by adding 0.72 g of MoO3 and 0.38 g of thiourea into 30 ml of de-ionized water. Similarly, 2 and 4 wt% nickel nitrate was added in this solution for the synthesis of 2% and 4% Ni doped MoS2. At room temperature all the solutions were stirred for 20 min for homogenious mixing. And then these three solutions were shifted into 50 ml Teflon stainless steel autoclaves at 200 °C for 9 h. The autoclaves were cooled down at room-temperature. These four samples were cleaned three times with de-ionized water and ethanol and then dried in vacuum at 80 °C for 5 h. Structural and surface morphology of these specimens were characterized by XRD and SEM, correspondingly. Photocatalytic activity (PCA) of Ni doped MoS2 was analyzed by degrading methylene blue (MB) and rhodamine blue dye (RhB). A 10 mg Ni doped MoS2 was mixed 30 mL of dye solution (1 × 10^{-5} mol l^{-1}). To make sure adsorption-desorption equilibrium stirred the mixture for the period of 1 h in absence of light. 500 W Hg lamp was utilized as a resource UV light. 100 mW cm^{-2} was adjusted irradiation intensity of UV light source and cutoff filter was employed to exclude the visible light. The suspension was irradiated to UV light for 240 min. After predetermined time period, 2 ml specimen was withdrawn. Then filtered and left over dye concentrations were anticipated at 665 nm and 554 nm (CE Cecil 7200, UK) for MB and RhB, respectively.

3. Results and discussion

3.1. Structural analysis

Figure 1 illustrates x-ray diffraction pattern for MoS2 and 2% Ni-MoS2 and 4% Ni-MoS2. XRD pattern shows lower angle reflections and distinguishing of fine order laminar structures. It can be revealed that Wurtzite—2 H hexagonal phases are formed and showing peaks at 29° and 44.1° with corresponding peaks (004) and (006) for MoS2, at 29.1° and 44.15° with corresponding peaks (004) and (006) for 2% Ni-MoS2 and 29.1°, 44.2° and 58.3° with (004), (006) and (110) corresponding peaks for 4% Ni-MoS2 [29]. It can be observed that more peaks with the increasing percentage of Ni as compared to the pure MoS2 are appeared. XRD patterns of all products are well matched with crystal arrangements of MoS2 JCPDS. Same kinds of MoS2 peaks were detected by Youzi Zhang et al [27]. Results obtained from XRD pattern are well agreed with the results obtained from FESEM analysis.

3.2. FESEM

SEM micro images of MoS2 are exposed in figure 2. Field Emission Scanning Electron Microscopy was exploited to establish morphological arrangements of nanoparticles, dispersion and distribution after different amendments. Layered structure of MoS2 is demonstrated in figure 2(A) from the cross-sectional view. From the FESEM image it can be observed that MoS2 nanocomposites are consisted of stacked thin nanosheets of nanometers thickness range. Hence gap spaces amongst MoS2 nanosheets are comparatively with lamella morphology. Figure 2(B) shows 2% Ni doped MoS2, Ni doped MoS2 shows 10 μm grains are formed having different shapes and sizes distributed on the surface of sample. The shapes of grains are spherical, small flower types and elliptical having different size. The sizes of grains are varying from 0.5 μm to 4 μm. In figure 2(B) some pores are existed on the left top corner of sample due to the less concentration of Ni contents. The grains are agglomerated to form bigger size particles at the lower surface which are also stacked on each other. Thus with
1% of Ni doped in MoS₂, Ni contents on the sample form different size of micro structures in the sample. Figure 2(C) shows 4% of Ni doped in MoS₂, it is evident that due to agglomeration big clusters are formed. These big size clusters are observed at the left bottom of the sample with the size of 13 μm. Figure also shows that the small grains are uniformly distributed having spherical shape and size with range from 0.2 μm to 0.4 μm. Some beads like structures observed at different position of samples. By increasing the concentration of Ni doping in MoS₂ the rate of agglomeration increases due to which the bigger size micro structures are observed. In 4% of Ni-doped MoS₂ the surface morphology of the sample shows that with increase in the concentration of Nickel small grains are started to agglomerates to form the bigger structures. Due to agglomeration the smallest structures form the bigger clusters due to which empty places are observed on the surface. Some nano-spheres are also observed at different positions of the sample.

3.3. UV-visible spectroscopy
UV–vis or ultra violet visible spectroscopy is engaged to investigate optical possession of MoS₂ and Ni doped MoS₂ nanomaterials. UV–vis spectroscopy outcomes for MoS₂ and Ni doped MoS₂ (2% and 4%) to investigate band gap energy (E_{g}) of nanoparticles are demonstrated in figure 3. From acquired values of UV–vis
spectroscopy absorption coefficient ($\alpha$) can be computed. Following relation can be employed to inspect absorption coefficient as,

$$\alpha = 2.303 \frac{\log(A)}{t}$$  \hspace{1cm} (1)

where, ‘A’ is absorbance and ‘t’ is thickness of nanomaterials. To compute optical bandgap energy ($E_g$) Tauc’s relation can be used as [30],

$$E_g = \frac{hv}{\lambda}$$  \hspace{1cm} (2)

$$(\alpha hv) \frac{1}{\tau} = B(hv - E_g)$$  \hspace{1cm} (3)

where B is constant, h is Planck constant, $\nu$ is frequency and m is also constant depending on valance and conduction band’s transition. The transition value (n) can be established in form of (1/2, 3/2, 2, 3) consigned to direct transition, forbidden direct transition, indirect transition and forbidden indirect transition respectively. From figure 3 it can be observed that band gap energy $E_g$ for MoS$_2$ is 2.30 eV, for 2% Ni-MoS$_2$ is 1.76 eV and for 4% Ni-MoS$_2$ is 1.90 eV. The trends exhibited by nanoparticles are lying in visible region of spectrum. Same kinds of outcomes were determined by Sadegh Azizi Darsara et al for MoS$_2$/CdS composites [31].

3.4. Photo catalytic activity

The UV–Vis absorbance spectra of the pure MoS$_2$ and Ni doped MoS$_2$ are shown in figures 4 and 5. The photocatalytic activity (PCA) of Ni doped MoS$_2$ was estimated by degrading methylene blue (MB) and rhodamine blue dye (RhB). A 10 mg Ni doped MoS$_2$ was mixed 30 mL of dye solution ($1 \times 10^{-5}$ mol $l^{-1}$). To make sure adsorption–desorption equilibrium the combination was stirred for the period of 1 h in absence of light. 500 W Hg lamp was utilized as a resource UV light. 100 mW cm$^{-2}$ was adjusted irradiation intensity of UV light source and cutoff filter was employed to exclude the visible light. The suspension was irradiated to UV light for 240 min. After predetermined time period, 2 ml specimen was withdrawn. Then filtered and left over dye concentrations were anticipated at 665 nm and 554 nm (CE Cecil 7200, UK) for MB and RhB, respectively. Percentage dye degradation was determined by employing the equation (4) as,

$$\% \text{ dye Degradation} = \left( A_0 - \frac{A_t}{A_0} \right) \times 100$$  \hspace{1cm} (4)

where, $A_0$ is the absorbance at time $t = 0$ (min), $A_t$ is absorbance after time $t$ (min.) of treatment and $A_0$ and $A_t$ are recorded at $\lambda_{max}$ of dye. According to Fei Ye et al (2017) that pure MoS$_2$ shows a weak visible absorbance in visible ranges and doped MoS$_2$ exhibits an intense visible absorbance between visible ranges [32]. This is possible because Ni doping increased the numbers of un-coordinated atoms at the edge sites of MoS$_2$ to increase the number of electron transfer across MoS$_2$ inter-faces that’s why doping increased the photocatalytic activity of MoS$_2$ [33]. Pure MoS$_2$ and Ni doped MoS$_2$ PCA were approximated by degrading MB and RhB dyes under UV light irradiation and these results are discussed one by one in figures 4 and 5. The MB dye degradation of MB was 71% for pure MoS$_2$. For 1% Ni doped MoS$_2$ MB dye degradation of MB was noted 85%. For 2% Ni doped MoS$_2$
MB dye degradation of MB was 89%. For 3% Ni doped MoS2 MB dye degradation of MB was 92%. For 4% Ni doped MoS2 MB dye degradation of MB was 94% and 5% Ni doped MoS2 MB dye degradation of MB was 96%. It means that MB dye degradation of MB was enhanced continuously by increasing the concentration of Ni doping for 1%–5%. All behaviors are shown in figure 4.

Similar trends were observed for RhB dye as in figure 5; The RhB dye degradation of RhB was noticed 62% for pure MoS2. For 1% Ni doped MoS2 RhB dye degradation of RhB was 77%. For 2% Ni doped MoS2, RhB dye degradation of RhB was 81%. For 3% Ni doped MoS2 RhB dye degradation of RhB was 85%. For 4% Ni doped MoS2 RhB dye degradation of RhB was 89%. For 5% Ni doped MoS2 RhB dye degradation of RhB was 91%. It means that RhB dye degradation of RhB enhanced continuously by increasing the concentration of Ni doping. The increasing of MB and RhB dyes degradation by increasing the concentration of doping indicates that the photocatalytic activity of MoS2 increased. Doping is used to increase the S active sides of MoS2 by controlled morphology of MoS2. By increasing the active sides the photocatalytic activity of MoS2 increased [34].

The maximum degradation of RhB dye for 5% Ni doped MoS2 was observed 91% and maximum degradation of MB dye for 5% Ni doped MoS2 was observed 96%. It means that degradation of RhB was slightly lower than the MB dye. Using stronger visible light PCA results indicate that photocatalytic properties of doped MoS2 are larger than pure MoS2, means significantly enhanced electron–hole pair’s separation and increased the photocatalytic properties of MoS2 because doping increases the active sites of MoS2 [35].

Table 1 shows the summary of MD dye (%) and RhB dye (%) for pure MoS2 and Ni-doped MoS2 (1%, 2%, 3%, 4% and 5%). It can be examined that increasing trends are shown by all nano-composites. Figure 6 also demonstrates the behaviors shown by all compositions for MB dye (%) and RhB dye (%). The photodegradation presentation of MoS2 can be improved by the increasing doping of Ni at appropriate treatment time. Such occurrences can be revealed by the generation of Ni nanoparticles in MoS2 structure. The enhancements in

![Figure 4](image1.png)

**Figure 4.** The Photocatalytic spectra of pure and Ni-doped MoS2 (1%, 2%, 3%, 4% and 5%) for MB dye.

![Figure 5](image2.png)

**Figure 5.** Plot of Photocatalytic spectra of pure and Ni-doped MoS2 (1%, 2%, 3%, 4% and 5%) for RhB dye.
photodegradation for both MB and RhB dyes are illustrated in figure 6. Also band gap is decreasing with the increase of Ni contents. It means distance amongst valance band and conduction band is decreasing. Such phenomena are leading the excited electrons to move from valance to conduction band in limited period of time. Hence with the increment of Ni contents the photodegradation for both MB and RhB dyes are enriching [36, 37].

Figure 7 shows plot of $C/C_0$ versus irradiation time ($C$ and $C_0$ are the residual and initial concentration dyes) for MB and RhB dyes. Under light irradiation, the photocatalytic activity was enhanced and it was observed that the degradation rate was fast for first 45 min, which slowed down with time. Mg doped MoS$_2$ furnished the MB and RhB dyes degradation of 91% and 84% for 240 min UV irradiation

### Table 1. Results of MB dye (%) and RhB dye (%) for pure MoS$_2$ and Ni-doped MoS$_2$ (1%, 2%, 3%, 4% and 5%).

| Compositions   | MB dye (%) | RhB dye (%) |
|----------------|------------|-------------|
| MoS$_2$        | 71         | 62          |
| Ni-MoS$_2$ (1%)| 85         | 77          |
| Ni-MoS$_2$ (2%)| 89         | 81          |
| Ni-MoS$_2$ (3%)| 92         | 85          |
| Ni-MoS$_2$ (4%)| 94         | 89          |
| Ni-MoS$_2$ (5%)| 96         | 91          |

Figure 6. Plot of MB dye (%) and RhB dye (%) for pure MoS$_2$ and Ni-doped MoS$_2$ (1%, 2%, 3%, 4% and 5%) against compositions (%).

4. **Conclusions**

Transition metal dichalcogenides (TMDs) are potential materials for photocatalytic applications. MoS$_2$ and Ni doped MoS$_2$ have been successfully prepared by hydrothermal technique. XRD analysis confirmed the formation of Ni doped MoS$_2$ nanoparticles. The doping of Ni has effected on the morphology of nanoparticles, according to FESEM. Band gap energy was observed in variable trends with the range of 2.30–1.76 eV with the addition percentage of Ni contents. The improvement in photocatalytic activity of MoS$_2$ has been observed when Ni was doped in it. MB dye and RhB dye percentages demonstrated increasing trends with the addition of nickel contents.

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Figure 7. Kinetic plot of dye degradation (MB and RhB).

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