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Synthesis and Characterization of Ni and Zn Doped MoS2 Nanoparticles by Hydrothermal Method for Photocatalyst Dye Degradation

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***Abstract*— In the field of environmental protection and energy generation, photocatalytic technology contributes wide range of applications such as the degradation of pollutants, water splitting, CO2 fixation/reduction, and organic synthesis. Here, a series of Zn and Ni-doped MoS2 nano-heterostructures were prepared using the hydrothermal method. The crystal structure and phase purity of the as-prepared Zn and Ni-doped MoS2 were confirmed by the powder X-ray diffraction method. The average crystallite size of all the samples was calculated using Scherrer's formula. Further, the surface morphology and elemental composition of stoichiometric compounds were analyzed using FE-SEM equipped with the EDX spectrum. The calculated bandgap of the pure and doped MoS2 varies from 3.5 eV, calculated from the Tauc plot using absorbance spectra of UV-visible data. Moreover, the photo-catalytic activity of Ni and Zn doped MoS2 was studied by the process of degrading MB dye under UV light irradiation. The roles of Zn and Ni doping on the photocatalytic activities of MoS2 hetero-structures were studied in detail and the experimental results will be presented.**

***Index Terms*— hydrothermal, MoS2,photocatalytic..**

I. INTRODUCTION

The entire biosphere including humans and living organisms is affected by polluted water that was ejected by industries and domestic such as toxic pollutants and colorants.

They are hard to degrade. It leads to environmental pollution due to industrial developments and a lack of awareness among people [1].

Worldwide 20% of water pollution is caused by textile dying processes according to the World Bank analysis [2]. This emergency needs to find a solution and development, it also must be eco-friendly, cost-effective, and renewable, such as Photodegradation which has grasped a large amount of attention in recent years due to its efficiency [3].Solution for the environmental problems and generation of renewable energies photocatalysts play a major role and also, and they are environment-friendly and cost-effective [4]. For these above applications, Semiconductor-based photocatalysts attract many researchers and industrialists [5].

The bandage positions, high surface-to-volume ratio, rapid charge separation rate, and high carrier mobility are important and needed characteristics for efficient photocatalytic properties [4]. To degrade the organic pollutants and dye from wastewater several semiconductor materials are used for degradation, they are TiO_2, SnO, ZnO, Nd_2O_5, ZnS, and MoS_2 they also have large bandgap so they undergo recombination of electron-hole pairs [5],[6]. Among those materials MoS_2 is widely considered by a researcher due to its unique physical, chemical, and optical properties, it has a broad range of applications like sensors, photocatalysis, dye-sensitized solar cells, batteries, and so on [5],[7]. Nanoscale MoS_2 nanoparticles attracted researchers because they are a good O_2-activation co-catalyst in forming superoxide radical anions (O_2) in photocatalytic reactions [5]. Physical properties like size and structure, along with synthesis methods, are important parameters in materials applications, elevating materials into suitable for various applications. The individual particles play a notable role in the material's properties. In the case of MoS_2 photocatalysts, the optical properties of a material, size, and shape are very important parameters [8].

MoS_2 is synthesized by using various techniques such as pulsed laser ablation, hydrothermal, spray pyrolysis, chemical vapor deposition (CVD), and sulfurization, these are slightly difficult and costly when compared with the hydrothermal method which is easy and cost-effective [9]. Methylene blue (MB) is a cationic dye,which was in 1878 invented by Caro, and has a C16H18N3SCl as its molecular formula and heterocyclic aromatic structure.

It is mainly used for dyeing industries, paper, cotton, and wood factories. Effects of MB in human beings are vomiting, irritation of the eyes, nausea, diarrhea, cyanosis, jaundice, quadriplegia, dyspnea, tachycardia, methemoglobinemia, and tissue necrosis [10]. Yunjin Yao et al. successfully developed and reported that Zn-MoS_2@PVDF membranes for the CrVI reduction. For heavy metal pollutants treatment, they give a promising design and fabrication of highly active catalytic membranes [11].

Haixia Qian et al. Zn–MoS_2–RGO ternary nanosheets were successfully prepared by a simple hydrothermal method. When compared with pure MoS_2 Zn–MoS_2–RGO exhibited enhanced catalytic HER performance. By adding Zn as a dopant, it does not disturb the structure and morphology of the MoS_2 instead it enhances the catalytic property and conductivity. This work, it confirmed that the Zn–MoS_2–RGO hybrid shows better performance up to 92% of Faraday efficiency for HER performance [12].M. I. Khan et al, synthesized and reported that the Ni-doped MoS2 and the doping resulting enhanced the photocatalytic property of a MoS2, and the doped material directly changed the morphology, the bandgap of the material also varied from 2.30 – 1.76 Ev. The MB dye and RhB dye percentage changes were also reported [13].

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KouroshRahimi et al, summarized that few-layer MoS_2 sheets were synthesized using the liquid exfoliation method and composited with ZnO nanorods. This work shows that photocatalytic activity of ZnO under sunlight enhanced after adding MoS2 by 74% (33%), they also treated organisms, and photocatalytic hydrogen evolution was also tested. From these results, ZnO/MoS_2 heterojunctions can be highly active photocatalysts [14]. Hongtao Lin et al, demonstrated that MoS2 nanorods fabricated using a very simple hydrothermal method with silicon tungstic acid as an additive. It shows the average diameters of MoS_2nanorods are 20–50 nm with lengths ranging from 400 to 500 nm. To prepare The present layered metal sulfide it can be extended [15].

In the present work we have chosen two different materials as dopants(Zn, Ni) to synthesis MoS_2 nanomaterials and characterize the structural and optical properties of prepared nanoparticles. It is important to achieve better results using these prepared nanoparticles for photocatalytic applications. Such as MB dye degradation and removal of organic pollutants from waste water. The sample was prepared by using Hydrothermal method, and they were characterized to identify whether they are suitable for further applications.

II. MATERIALS AND REAGENTS

All the chemical reagents were used as received without any further purification. Deionized water was used in all the experiments. Ammoniumheptamolybdate tetrahydrate (≥99%), Thiourea (99-101%), citric acid (99.7%), Zinc acetate dihydrate $(CH_2\ COO)_2$ Zn. $2H_2O$, Nickel (II) nitrate hexahydrate $(Ni(NO_2)_2.6H_2O)$, sulphuric acid (95-98%) and ethanol (99.9%) were purchased from the Merck.

A.Synthesis of zn doped mos$_2$(molybdenum disulphide)

To synthesize a Zinc (Zn) doped MoS_2 nanoparticles, a similar inexpensive hydrothermal technique is used. Chemical precursors$(NH_4)_6Mo_7O_{24}.4H_2O$, thiourea and $(CH_2\ COO)_2$ Zn. $2H_2O$ was dissolved in a 30 mL of distilled water and stirred for thirty minutes. The above solution was transferred into a 100 mL stainless steel autoclave and kept it at 100˚C for 20 h in an oven. The pressure inside the autoclave triggers the nucleation reaction. The sediments were collected and centrifuged with distilled water for three to four times and then followed by drying at 180˚C for 8h in oven.

B. Synthesis of Ni doped MoS$_2$(molybdenum disulphide)

To synthesize a Nickel (Ni) doped MoS_2 nanoparticles, a similar inexpensive hydrothermal technique is used. Chemical precursors$(NH_4)_6Mo_7O_{24}.4H_2O$, thiourea and Nickel (II) nitrate hexahydrate $(Ni(NO_2)_2.6H_2O)$ was dissolved in a 30 mL of distilled water and stirred for thirty minutes. The above solution was transferred into a 100 mL stainless steel autoclave and kept it at 100˚C for 20 h in an oven. The pressure inside the autoclave triggers the nucleation reaction. The sediments were collected and centrifuged with distilled water for three to four times and then followed by drying at 180˚C for 8h in oven.

C. Synthesis of Zn and Ni co-doped MoS$_2$

Synthesis of Zinc and Nickel co-doped MoS_2 nanoparticles was carried out through changing of many parameters to identify suitable condition by a hydrothermal method. Precursors$(NH_4)_6Mo_7O_{24}.4H_2O$, $(CH_2\ COO)_2$ Zn. $2H_2O$,$(Ni(NO_2)_2.6H_2O)$ and thiourea was dissolved in 30 mL of distilled water and stirred for 30 minutes. The suspension was transferred into a 100 mL stainless steel autoclave and kept it 100˚C for 20 h. The final black product was centrifuged with ethanol and then allowed it to dry for 8 h at 180˚C in an oven.

D. Characterization techniques

The crystalline structure and phase purity of the samples were studied by X-ray diffraction (XRD), using Shimadzu XRD 6000 X-ray diffractometer Cu-Kα radiation k = 1.54 Å in the 2θ range of 5˚-80˚ at room temperature with a scanning rate of 0.06 deg s^{-1}. The Fourier transform infrared (FTIR) spectra were recorded using Brucker Tensor 27 spectrophotometer with resolution of 2cm^{-1}in the range of 4000-400 cm^{-1} at regular KBr phase. The JEOL JSM field emission scanning electron microscope (FESEM) equipped with energydispersive X-ray analysis (EDX) was used to find the surface morphology and elemental compositions presented in the prepared materials. The optical characterizations of the samples were performed using a UV-Visible spectroscopy (Perkin Elmer UV-Vis-NIR, spectrophotometry).

III. RESULT AND DISCUSSION

The X-ray diffraction (XRD) pattern of as-synthesized MoS_2 doped with Zn, Ni and Zn&Ni co-doped nanoparticles were shown in (figure. 1) . The XRD peaks were indexed at 2θ at 13^o, 25^o, 29^o and 33^o corresponds to (002), (201), (100), (004), (101) planes similar to the standard card (JCPDS cardNo.37-1492) which indicates that all the diffraction peaks can be readily confirmed as hexagonal(2H-MoS_2) crystal structure [space group $P6_3/MMC$ (194)] with lattice constants a= 3.16Å and c=12.20Å[16]. The strongest diffraction peak is indexed to the (002) plane of MoS_2, which means that a large number of grains are in the particular orientation, which is in good agreement with the previous report [17]. The peak 26^o was assigned to Zinc sulfide associated with MoS_2. The lattice parameters were calculated using the formula (eq. (1))

$$\frac{1}{d^2} = \frac{4}{3a^2}(h^2 + k^2 + l^2) + \frac{1}{c^2}l^2 \ldots . (1)$$

The calculated values of the lattice constant are in agreement with the MoS_2 lattice parameters [18]

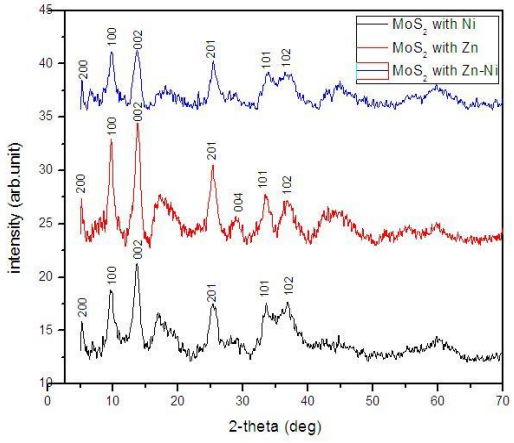

Fig. 1. XRD patterns of MoS_2 doped with Zn, Ni, and Zn&Ni co-doped nanoparticles.

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The morphological and elemental analyses of the prepared MoS_2 doped with Zn, Ni, and Zn&Ni co-doped nanoparticles are shown in Figure 2. From the FESEM analysis, it is seen that the grown particles are spherical in nature with high porosity. The porosity among the clustered nanoparticles helps to degrade organic dyes effectively [19]. The FESEM image of MoS_2 reveals that the MoS2microspheres were composed of Nanosheets with dimensions in the range of 300–400 nm assembled by densely packed petals and have a platelet-like shape [10], [20].

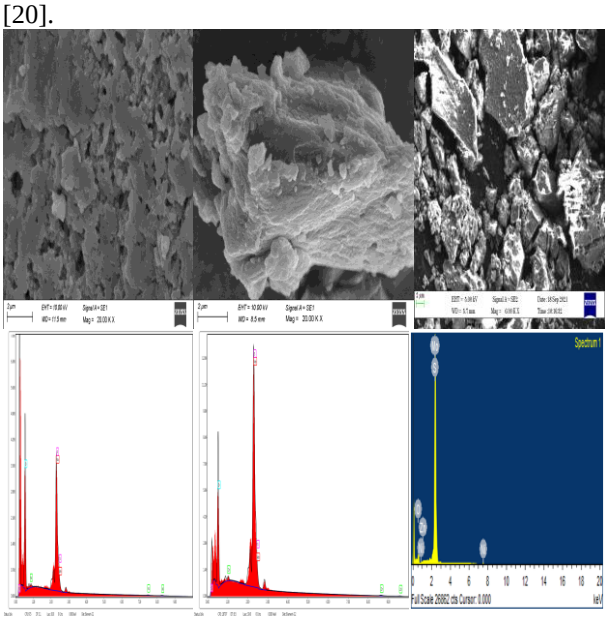

Fig. 2. FESEM and EDAXof MoS_2 doped with Zn, Ni, and Zn&Ni co-doped nanoparticles.

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[21]. The EDAX spectra of MoS_2 doped with Zn, Ni, and Zn & Ni co-doped nanoparticles are shown in Figure (2) it confirms the presence of the Mo, S, Zn, and Ni elements.

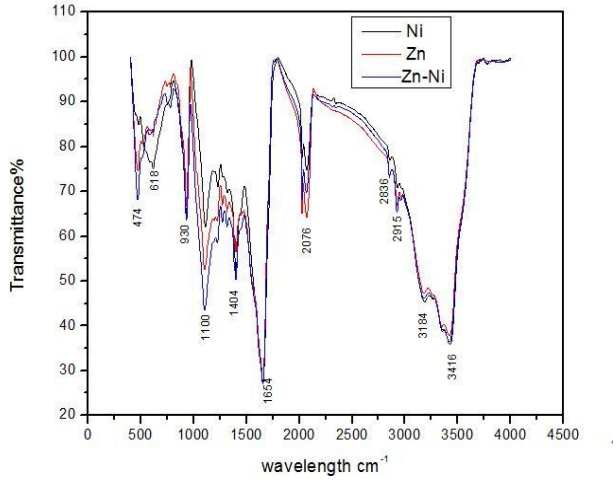

*Fig. 3.*FTIR spectraof MoS_2 doped with Zn, Ni, and Zn&Ni co-doped nanoparticles.

Figure 3, b shows the FT-IR spectra of MoS_2 doped with Zn, Ni, and Zn & Ni co-doped nanoparticles. There are broad absorption bands in the spectra at 474, 930, 1100, and 1654 cm^{-1} for both samples. The band at 480 cm^{-1} is due to the Mo–S bond. The band at 900 cm^{-1} can be attributed to the S–S bond. The absorption band between 1100 and 1650 cm^{-1} is due to the stretching vibrations of the hydroxyl group, the bands at around 1200 and 1650 cm^{-1} may be due to the Mo–O vibrations [19]. A peak at 1405 cm^{-1} might be accredited to the C=O bonding [21]. The peaks at 2848 cm^{-1} and 2917 cm^{-1} are assigned to the stretching vibrations of –CH2 groups [22]. The band at 3420 cm^{-1} is attributed to the OH vibration[23].

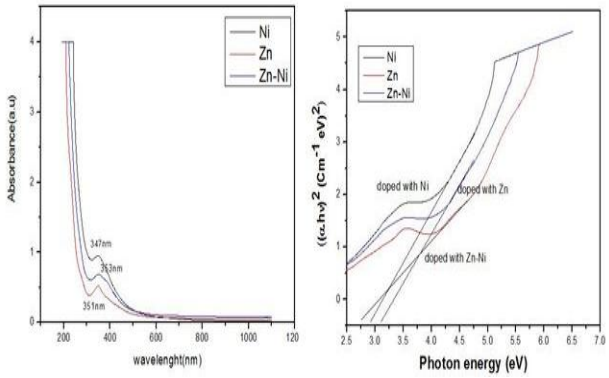

*Fig. 4.*UV-Vis spectraof MoS_2 doped with Zn, Ni and Zn&Ni co-doped nanoparticles.

The UV–Vis absorption spectra of prepared of MoS_2 doped with Zn, Ni and Zn&Ni co-doped nanoparticles at normal temperature in the 200-1200 nm wavelength range are shown in the figure 4. The absorbance spectrum is also used to calculate the band gap energy using the Tauc plot through the following equation [2]:

$(\alpha\hbar\nu)½ = A(\hbar\nu - Eg)$…… (2)

Where α is the absorption coefficient, h is the Planck constant, v is the light frequency, A is the proportional constant, and Eg is the band gap energy. In addition, the broad band absorption centered at 300nm (4.13 eV)–450 nm (2.76 eV) for Ni doped MoS_2, 310 nm (4.00eV)–420 nm (2.95 eV) for Zn doped MoS_2 and 320 nm (3.80 eV)–410nm (3.02 eV) for Zn & Ni co-doped MoS_2 nanoparticles. From theplot the calculated indirect bandgap energies of the respected samples are 3.5 eV for MoS_2 doped with Ni, 3.5eV for MoS_2 doped with Zn and 3.57eV for MoS_2 doped with Zn&Ni sample.

The photocatalytic reaction is performed under the light radiation with an energy bigger thanor equal to the band gap energy (Eg), wherethere will be a movement of electrons from valence band to the conduction band leaving behind holes in the valence band. The electrons in the conduction band combine with O_2 to form superoxide radicals ($O^{\bullet}$-). Subsequently the holes in the valence band oxidize with water to form hydroxyl radicals ($OH^{\bullet}$-). It leads to two possibilities: a) due to the absence of suitable electron scavenging there will be a recombination of electron-hole pair producing thermal energy b) redox reaction takes place by avoiding the recombination. Although the lifetime of an (e^- - h^+) pair is few nanoseconds, it is still long enough for promoting/initiating redox reactions. Thus, the series of chain oxidative reductive reactions that occurs at the photon

activated surface leads to degradation of organic molecules present in the pollutant [24].

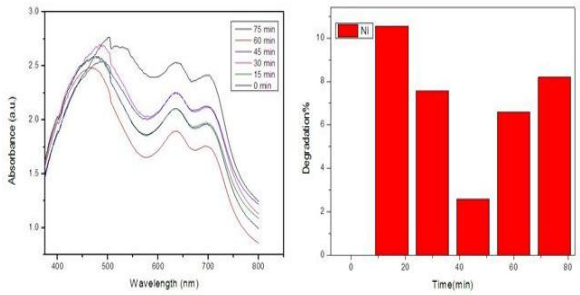

Fig .5. a) Photocatalytic activity of samples MoS_2 doped with Ni in MB dye degradation b) initial methylene blue (MB)concentration towards the degradation of MB under visible light using MoS_2 doped with Ni.

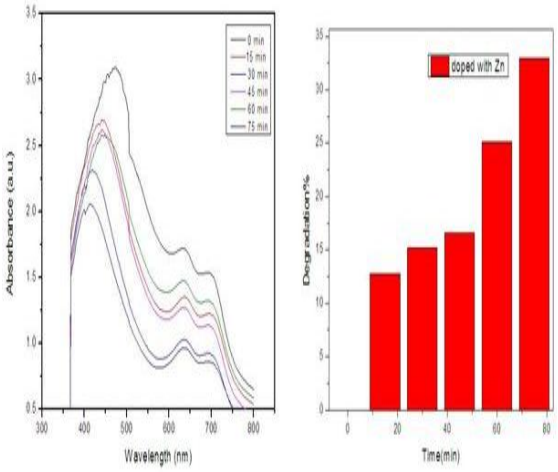

Fig.6. a) Photocatalytic activity of samples MoS_2doped with Zn in MB dye degradation b) initial methylene blue (MB)concentration towardsthe degradation of MB under visible light using MoS_2doped with Zn.

The corresponding photocatalytic activity of synthesized MoS_2 doped with Zn, Ni and Zn&Ni co-doped nanoparticles prepared by hydrothermal method investigated via the photo degradation of chosen organic pollutant as methylene blue dye under the illumination of visible light. The prepared samples of around 50mg as catalyst material were dispersed in the ratio of 2:1 with methylene blue dye in aqueous solution and the respective suspension was stirred for 60 min in order to achieve adsorption equilibrium under dark. Further, the obtained suspension was irradiated continuously under the visible light.

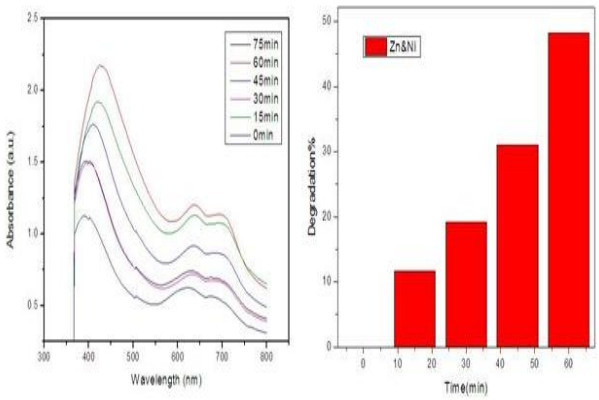

Fig.7. a) Photocatalytic activity of samples MoS_2 doped with Zn &Ni in MB dye degradation b) initial methylene blue (MB)concentration towards the degradation of MB under visible light using MoS_2 doped with Zn &Ni.

The process was continued for every 15mins time interval and the changes in the concentration of methylene blue in the suspension were monitored using UV-Vis (Ocean Optics, USA) spectrophotometer. The maximum absorption of the chosen dye (Methylene blue) was found to be 665 nm. The linear degradation nature of the proposed samples shows that the dye is degrading under visible infrared region (665 nm).

To calculate the absorption of Methylene blue (MB) dye detection of its removal efficiency of MoS_2 and doped mixtures were used at different time intervals. From the initial values its percent derivatives were calculated to determine the removal of pollutants using MoS_2 and doped mixtures. The reactions were performed at 15 mins interval of time under the visible light, and the absorption peak of MB dye appears at 490nm for MoS_2 doped with Ni, 470nm for MoS_2 doped with Zn and 430nm for MoS_2 doped with Zn & Ni. At the time of the reaction theabsorbance does not shows any difference but the dye concentration is proportional to its absorption.

Degradation efficiency was determined by employing the equation as,

$$\%\ degradation = \left(\frac{C_0 - C}{C_0}\right) \times 100 \ldots\ldots\ldots (3)$$

where C_0 is the MB concentration at the initial state and C is the MB concentration after illuminating under visible light at a specified time [21]. Using MB dye as the organic pollutant the photocatalytic materials prove its efficiency through cationic dyewhich has an organic compound of clear absorbance properties in the visible light region it helps remove the organic pollutants from the water resources thus making it one of the most efficient methods [23]. Using MoS_2 doped with Ni, MoS_2 doped with Zn, and MoS_2 doped with Zn&Ni as a photocatalyst material the MB dye wassuccessfully removed around 30%shown in the Figure.5-Figure 7. In 75 minutes, the degradation was increased up to 48% by adding the suitable dopant material to the source material. Using as a dopantmaterial the removal was enhanced from 34% shown in Figure 6. The total percentage of methylene blue dye removals was calculated for MoS_2 doped with Ni (30%), MoS_2 doped with Zn (34%), and MoS_2 doped with Zn&Ni (48%). According to the plotted diagrams, it is observed that the amount of photocatalytic degradation for MoS_2 doped with Zn&Ni nanomaterials has increased by 48% in comparison with MoS_2 doped with Ni and MoS_2 doped with Zn (34%). The results of methylene

blue dye degradation under visible light irradiation over the prepared samples are demonstrated in Figure. From the figure, it can be found that the MoS_2 doped with Zn&Ni (48%) nanomaterials show a higher photocatalytic activity.

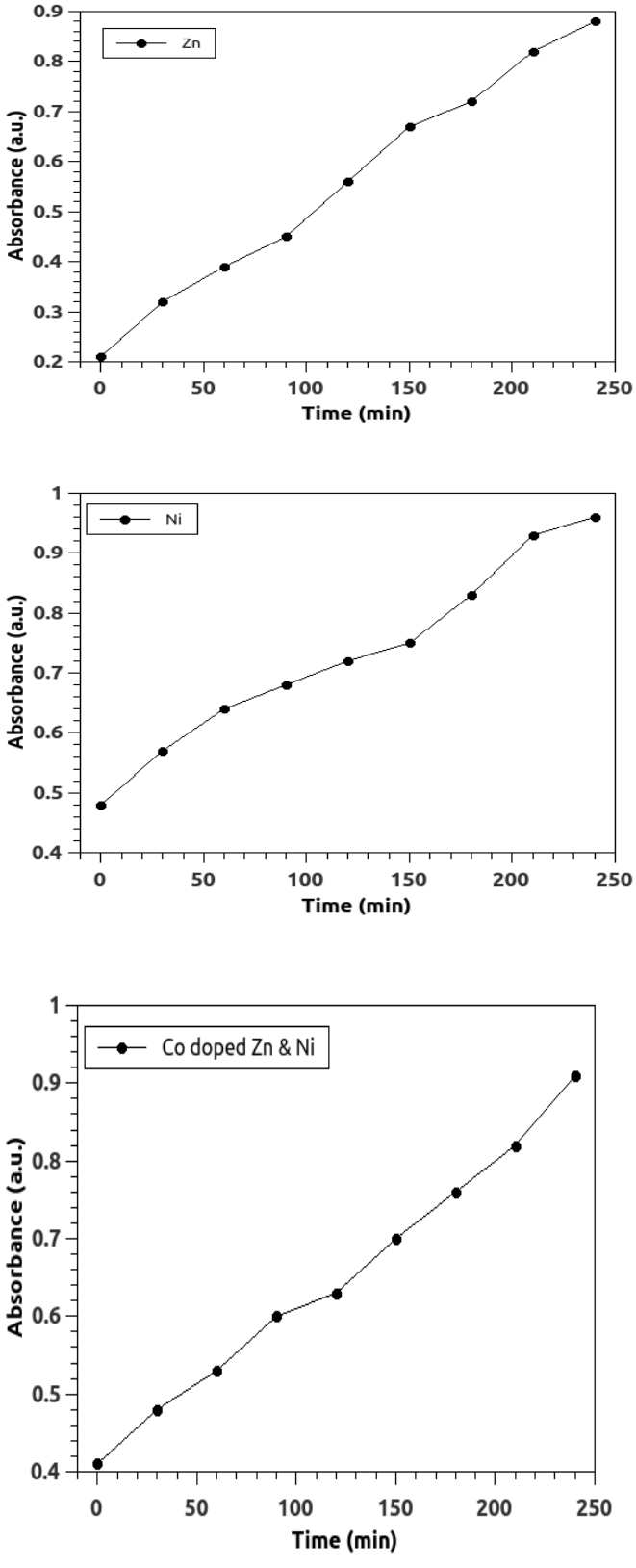

Fig. 8. Degradation activity of Organic pollutants MoS_2 doped with Zn, Ni, and Zn&Ni co-doped nanoparticles.

During the past decade, the technology of semiconductor nanocatalysts as a photocatalyst has endured an unpredictable growth for sustainable environmental remediation using both natural and artificial UV–-vis sources. Therefore, it is noteworthy to prepare visible light-active photocatalysts by tuning the operating conditions, so that the industrial implementation of photocatalytic degradation of organic pollutants can be attained over the conventional treatment techniques [20]. The total percentage of wastewater organic pollutants dye removals was calculated for MoS_2 doped with Ni (30%), MoS_2 doped with Zn (34%), and MoS_2 doped with Zn&Ni (48%). According to the plotted diagrams, it is observed that the amount ofphotocatalytic degradation for MoS_2 doped with Zn&Ni nanomaterials has increased by 48% in comparison with MoS_2 doped with Ni and MoS_2 doped with Zn (34%). It can be found that the MoS_2 doped with Zn&Ni (48%) nanomaterials show a higher photocatalytic activity same as the Methylene blue dye degradation.

IV. CONCLUSION

The current study demonstrates the facile synthesis of MoS_2 doped with Zn, Ni, and Zn & Ni co-doped nanoparticles using eco-friendly Hydrothermal methods, and XRD and FESEM analysis confirmed that the synthesized samples havea hexagonal crystal structure. The structural and optical analysis confirmed the formation of effective MoS_2 doped with Zn & Ni (48%) nanomaterials showing a higher photocatalytic activity under visible light. Using developing applicable nanotechnology, as a result,using the Hydrothermalmethod synthesized MoS_2 doped with Zn, Ni, and Zn&Ni co-doped nanoparticles have potential applications in the removal of organic pollutants from wastewater.

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