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CHARACTERIZATION OF Ni Doped CdZnS NANOPARTICLES AND THEIR USE IN METHYLENE BLUE DEGRADATION UNDER VISIBLE LIGHT IRRADIATION

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ABSTRACT

In this study, CdZnS nanoparticles (NPs) with different Zn concentrations were synthesized by chemical precipitation at room temperature. Zn concentration values used were determined as 1%, 2%, 5%, 10%. The photocatalytic properties of these synthesized NPs were investigated to determine the optimum Zn concentration in CdZnS NPs with different Zn concentrations. Thus, the optimum Zn concentration with the best photocatalytic activity was found to be 2% and this sample was named $Cd_{0.98}$ $Zn_{0.02}$ S.

In the second phase of our study, $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations were synthesized using the same method. The Ni concentration values used were determined as 1%, 2%, 5%, 10%. By examining the photocatalytic properties of these synthesized NPs, the optimum Ni concentration with the best photocatalytic activity was found to be 0.5% and this sample was named $Cd_{0.975}Zn_{0.02}$ $Ni_{0.975}$.

Finally, the structural, morphological and optical properties of $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}$ $Ni_{0.975}$ NPs were investigated, respectively, after the optimum Zn and Ni concentrations were determined.

KEYWORDS: Characterization, methylene blue dye, nanoparticles, photocatalytic activity, synthesis.

I. INTRODUCTION

The valence electrons in the transmission band become excited when a semiconductor material absorbs equal or greater light energy in its band gap. This technique, known as photocatalysis, has been used to solve many existing environmental and energy issues. [1-4]. A charge separation called electron-hole pairs can form free radicals in the system for redox of substrate. Free radicals, such as hydroxyl (OH), are highly effective oxidizers of organic materials and are used to degrade pollutants [5-6].

A catalyst for effective degradation of the dye must have a suitable band gap, flat band energy levels and good adsorption properties in the visible region [7]. Most group II–VI materials offer the potential to destroy organic contaminants [8]. Among them, cadmium sulfide (CdS) with a direct band gap of 2.4 eV and zinc sulfide (ZnS) with a with band gap of 3.7 eV are thought to be an effective catalyst, resulting from the production of fast electron-hole pairs and the highly negative redox potentials of excited electrons [9-10]. Cadmium Zinc Sulfide (CdZnS) semiconductors which are synthesized using appropriate Cd: Zn ratio, have fine and tunable absorption in the visible region of solar energy and excellent electrical conductibility, which have been widely used as capable wide band gap (2.4 -3.7 eV) materials for photocatalytic degradation [11-12].

The well-defined structure is needed to entirely obtain the photocatalysis properties of CdZnS. The structure and size are the most important factors which affect the photocatalytic activity of materials. The reactivity of the CdZnS nanoparticles (NPs) is generally improved in comparison with their bulk counterpart owing to size dependence of their redox potentials and high density of the active surface states related to large surface-to-volume ratio [13-14]. Furthermore, the size reduction in the semiconductor particle leads to decrease the recombination of electron-hole pairs [15-17]. Optical properties of catalyst can be tuned [16] and the intermediate bands [17] can be formed in band gap of catalyst in by doping. Furthermore, the doping can enhance the chemical stability by injecting the holes into the formed acceptor energy level and reduce their activity on photocorrosion [18]. Generally, transition metals such as Ni [19], Mn [20], Co [21], and Cu [22] have been used as dopants.

Methylene blue (MB) dye is widely used in textile, leather, paper, wood and plastic industries. In humans and animals, it is able to lead to permanent burning of eyes and eyes. In addition, nausea, vomiting, excessive sweating can cause rapid and difficult breathing and short-term mental confusion [23]. Given the possible harmful effects of MB dye, it is more vital to remove the dyes from the wastewater. Various physical, chemical and biological methods are used for wastewater treatment [24].

Pulse laser deposition/ablation [25-26], hydrothermal method [27], sol-gel processing [28] and chemical precipitation [29] techniques can be used as methods to successfully prepare semiconductor nanocrystals. Among these methods, the chemical precipitation method which is one of the most convenient techniques has been widely used by researchers because of its low cost, simplicity and ease.

In our present study, CdZnS NPs with different Zn concentrations were synthesized by chemical precipitation at room temperature. Zn concentration values used were determined as 1%, 2%, 5%, 10%. The photocatalytic properties of these synthesized NPs were investigated to determine the optimum Zn concentration in CdZnS NPs with different Zn concentrations. Thus, the optimum Zn concentration with the best photocatalytic activity was found to be 2% and this sample was named $Cd_{0.98}$ $Zn_{0.02}$ S.

In the second phase of our study, $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations were synthesized using the same method. The Ni concentration values used were determined as 1%, 2%, 5%, 10%. By examining the photocatalytic properties of these synthesized NPs, the optimum Ni concentration with the best photocatalytic activity was found to be 0.5% and this sample was named $Cd_{0.975}Zn_{0.02}$ $Ni_{0.975}$.

Finally, the structural, morphological and optical properties of $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}$ $Ni_{0.975}$ NPs were investigated, respectively, after the optimum Zn and Ni concentrations were determined.

Experimental Section

To synthesize CdZnS NPs with different Zn concentrations in aqueous solution by chemical precipitation technique at room temperature, commercial $Cd(CH_3COO)_2.2H_2O$ as Cd source, Zn $(CH_3COO).2H_2O$ as Zn source, and Na_2S as S source, were of analytical grade and were used without further purification. In typical chemical precipitation method, to prepare $Cd_{0.98}Zn_{0.02}S$ three different clean beakers were used to prepare 1M aqueous solutions of $Cd(CH_3COO)_2.2H_2O$, 0.02M aqueous solutions of Zn $(CH_3COO).2H_2O$ and 1M aqueous solutions of Na_2S. Then, these three solutions cation and anion sources were transferred into fourth clean beaker, then stirred at certain time to get homogeneous mixture at room temperature. A filter paper was used to obtain the percipated sample from the solution. De-ionized water (DIW) and ethnaol were used to wash the resultant particles to get rid of unwanted compounds. The final solution was heated in oven for few hours at 80 ^{0}C. Then, a mortar was used to grind the obtained orange colour powders to get $Cd_{0.98}Zn_{0.02}S$ NCs. To synthesize CdZnS NPs with other Zn concentrations, 0.01M, 0.05M and 0.1M Zn $(CH_3COO).2H_2O$ are used, respectively.

For preparing $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations, the different concentrations of Ni $(NO_3)_2{\cdot}6H_2O$ were added into aqueous solutions of 1M Cd $(CH_3COO)_2.2H_2O$ and 0.02M Zn $(CH_3COO).2H_2O$, respectively.

Photocatalytic activities of CdZnS NPs with different Zn concentrations and $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations were tested by decolorization of MB dye in aqueous solution. The experiments were performed under visible light irradiation without any catalyst (blank), with catalyst in dark and in the presence of CdZnS NPs with different Zn concentrations and $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations. Reaction was set up by adding 30 mg of CdZnS NPs with different Zn concentrations and $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations into 50 mL of MB solution in the Pyrex glass beaker of 100 mL volume. To obtain adsorption/ desorption equilibrium before irradiating the light in the beaker, the suspension was magnetically stirred in dark for 30 minutes. Before CdZnS NPs with different Zn concentrations and $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations were exposed to UV light; oxygen was purged into the solution to keep the suspension of the reaction homogenous.

During the reaction, the solutions of CdZnS NPs with different Zn concentrations and $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations were kept at room temperature. 2 mL of the solution of CdZnS NPs with different Zn concentrations and $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations were drawn for 90 minutes for 15 minutes

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during the irradiation period after the light source was activated. The withdrawn suspension was centrifuged at 3000 rpm for 10 minutes to remove CdZnS NPs with different Zn concentrations and $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations. The wavelength at 664 nm was employed to measure the absorbance of the clear solution. The relation is given in Equation 1 was utilized to calculate percentage degradation of MB dye.

$$\% \text{ degradation of MB dye} = \frac{A_0 - A_t}{A_0} \quad (1)$$

A_0: Absorbance of dye at initial stage
A_t : Absorbance of dye at time t

The structural, morphological and optical properties of $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.975}$ NPs were studied by x-ray diffraction (XRD) on a Rigaku x-ray diffractometer with Cu K_α (λ= 154.059 pm) radiation, scanning electron microscope (SEM) (JEOL JSM 5800) and Perkin Elmer Lambda 2 spectrometer, respectively.

II. RESULTS AND DISCUSSIONS

Figure 1 indicates percentage (%) photocatalytic degradation of Methylene blue (MB) as a function of irradiation time (in min) using CdZnS NPs with different Zn concentrations with fixed MB concentration at pH 7.

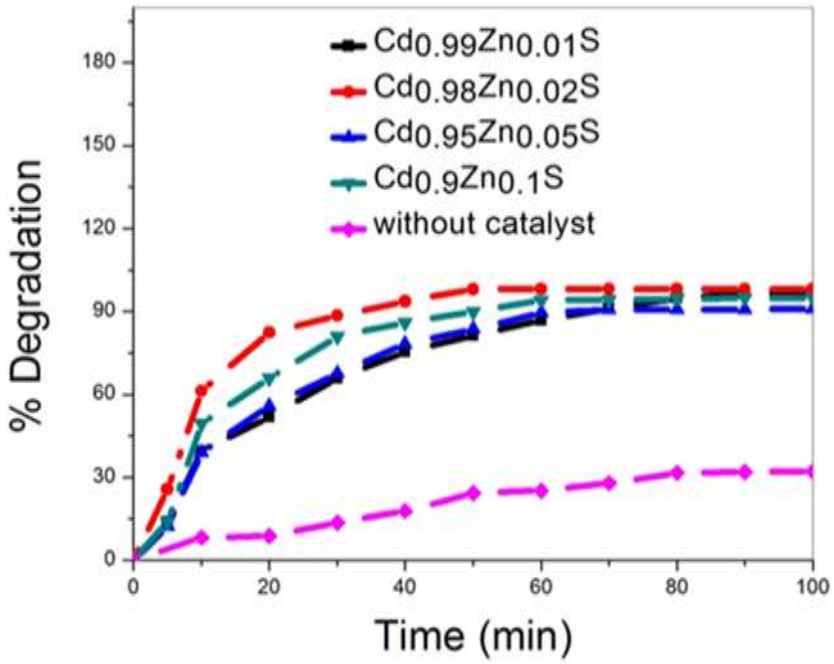

Figure 4 Plot of % photocatalytic degradation of Methylene blue (MB) as a function of irradiation time (in min) under visible light using CdZnS NPs with different Zn concentrations.

As is seen, $Cd_{0.98}Zn_{0.02}S$ NPs have higher photocatalytic activity compared to other Zn concentrations. It is important to note that the $Cd_{0.98}Zn_{0.02}S$ NPs demonstrates enhanced activities than CdS and ZnS which are reported by Ahmed et al. [30] and Jabeen et al [31], respectively. This result suggests that the photocatalytic activity of CdS can be improved with presence of Zn.

The percentage (%) photocatalytic degradation of MB for Cd_1 $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations is demonstrated in Figure 2.

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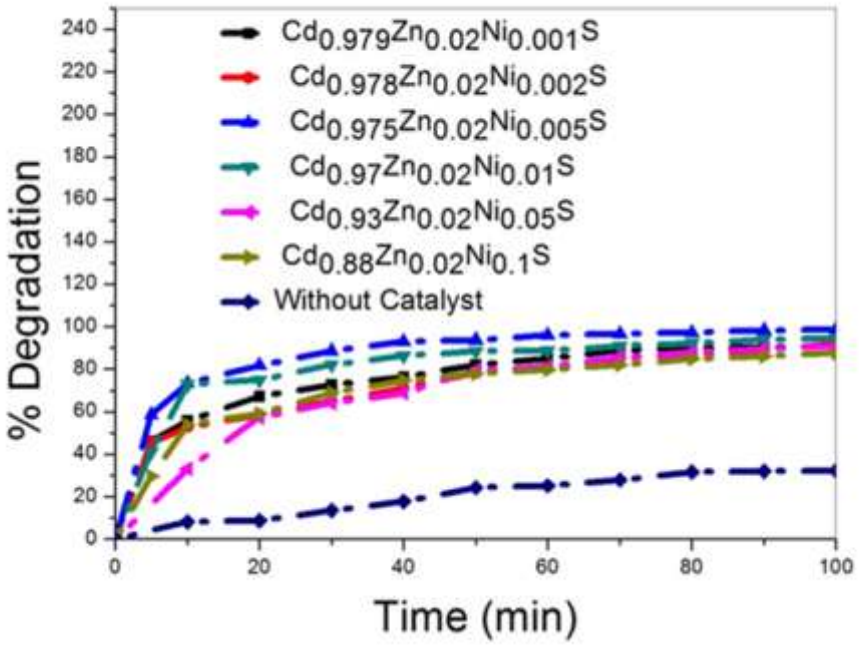


Figure 2 Plot of % photocatalytic degradation of Methylene blue (MB) as a function of irradiation time (in min) under visible light using $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations.

From Figure 2, it is important to note that a maximum degradation efficiency of MB dye is observed for $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NCs at initial pH of 7 after irradiation time of 100 min. The improvement in photocatalytic acticity is not proportional to the concentration of Ni doping. It is notewothy that $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs reveal more enhanced photocatalytic activity than that of $Cd_{0.98}Zn_{0.02}S$ NPs (see Figure 3).

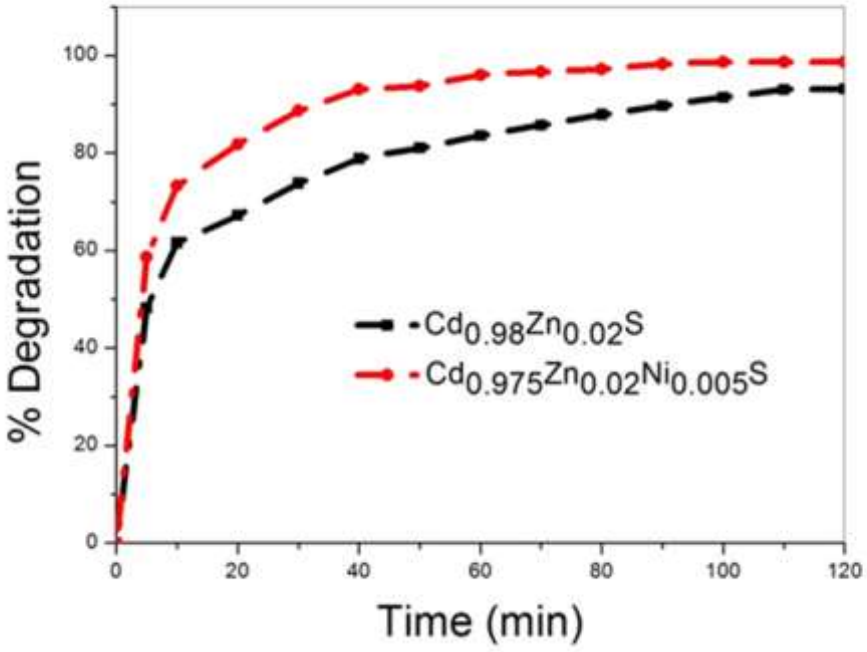


Figure 3 Comparison of % photocatalytic activity of $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs.

This result is higher than value obtained by Jin et al [32]. They reported that $Zn_{0.2}Cd_{0.8}S$ and $Zn_{0.1}Cd_{0.9}S$ hollow spheres show high photocatalytic activities and the photocatalytic conversion of Rhodamine B (RhB) with hollow $Zn_{0.2}Cd_{0.8}S$ spheres reaches as high as 96% afer 50 min of irradiation. This result suggests that the insertion of Ni^{2+} has enhanced the photocatalytic activity of CdZnS NPs considerably.

The Langmuir–Hinshelwood model is usually applied to investigate the sorption process, the pseudo-first-order and pseudo-second-order kinetic models were used to describe the kinetics of photocatalytic reactions in aqueous solutions for catalytic degradation of organic compounds through adsorption [33].

The pseudo-first-order model:

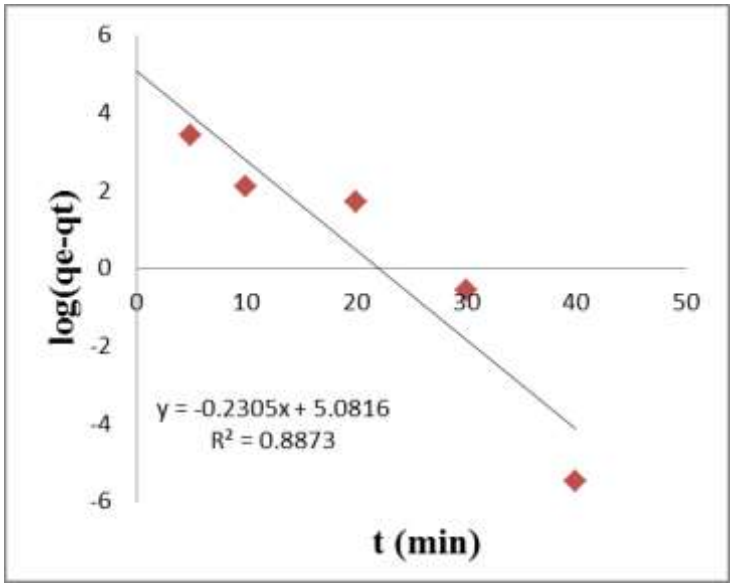
$$\log (q_e-q_t)=\log q_e- [k_1/2.303]t \quad (2)$$

The pseudo-second-order model:

$$(t/q_t)= 1/ (k_2q_e^2) + (1/q_e)t \quad (3)$$

Where q_t and q_e are the amount of MB adsorbed at certain time t and at equilibrium (mg g^{-1}); k_1 is the rate constant of the pseudo-first-order adsorption process (min^{-1}); k_2 is the pseudo-second-order rate constant (g mg^{-1} min^{-1}).

The plots of log (q_e-q_t) versus t and (t/q_t) versus t for $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs are indicated in Fig. 4a-d, respectively.



(a)

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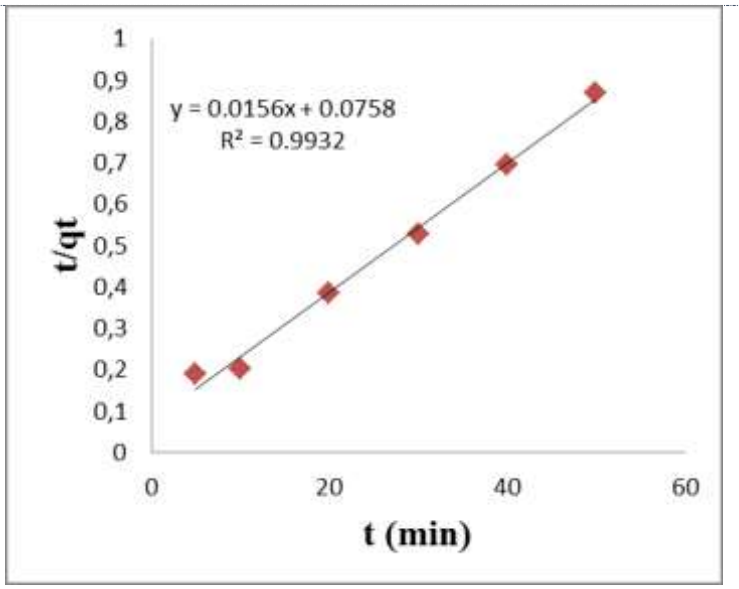
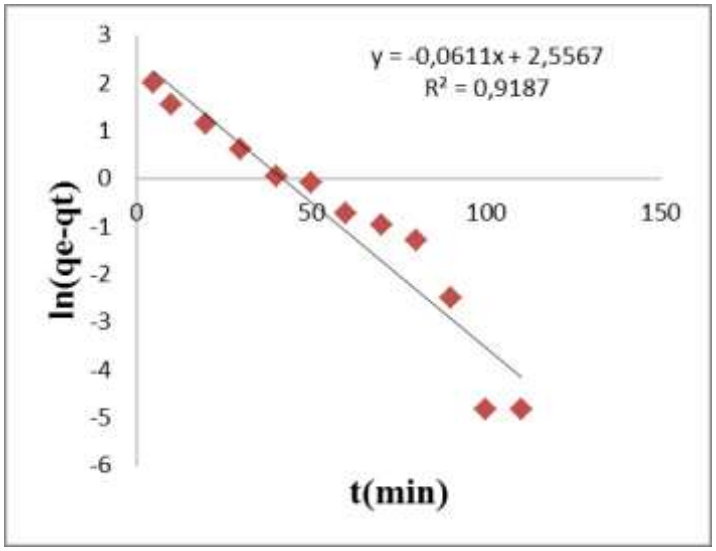


(b)



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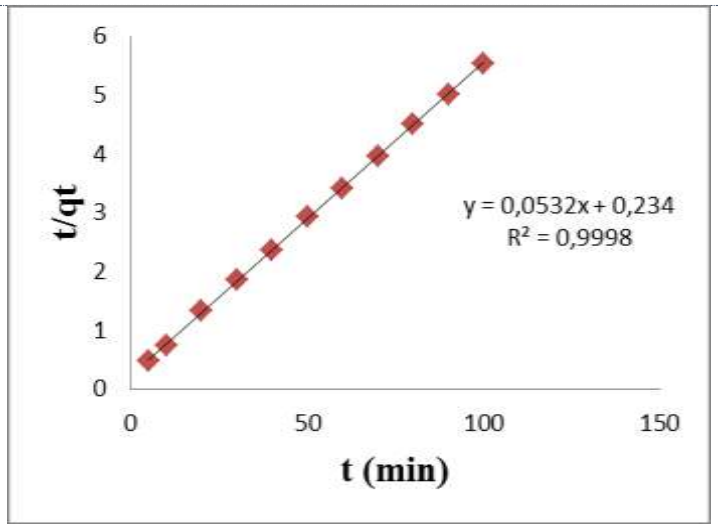

(d)

Figure 4 Adsorption kinetics of MB (a) pseudo-first-order model (b) pseudo-second-order model for $Cd_{0.98}Zn_{0.02}S$ NPs and (c) pseudo-first-order model (d) pseudo-second-order model for $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs.

Correlation coefficient (R^2) values obtained from the kinetic models for $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs are shown in Table 1.

Table 1 R^2 , k_1, and k_2 values for $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs.

Pseudo-first-order kinetic model		
Catalysts	k_1 (min^{-1})	R^2
$Cd_{0.98}Zn_{0.02}S$ NCs	0.1131	0.8824
$Cd_{0.975}Zn_{0.02}Ni_{0.005}$	0.0611	0.9187
Pseudo-second-order kinetic model		
Catalysts	k_2 (min^{-1})	R^2
$Cd_{0.98}Zn_{0.02}S$ NCs	0.0202	0.9913
$Cd_{0.975}Zn_{0.02}Ni_{0.005}$	0.0121	0.9998

As can be seen in Figure 4, the adsorption kinetics for both $Cd_{0.98}Zn_{0.02}S$ NPs and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs are in good agreement with psedu-second-order kinetic model. Furthermore, the correlation coefficient value of the pseudo-second-order kinetic model for two samples is higher than that of the pseudo-first-order kinetic model. It is important to note that the Ni doping results in the increase in the value of the correlation coefficient.

After $Cd_{0.98}Zn_{0.02}S$ NPs and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs indicates the maximum photocatalytic activity, were determined, respectively, the structural, morphological and optical properties of these samples were investigated.

The XRD patterns for the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs that were prepared at room temperature are shown in Figure 5.

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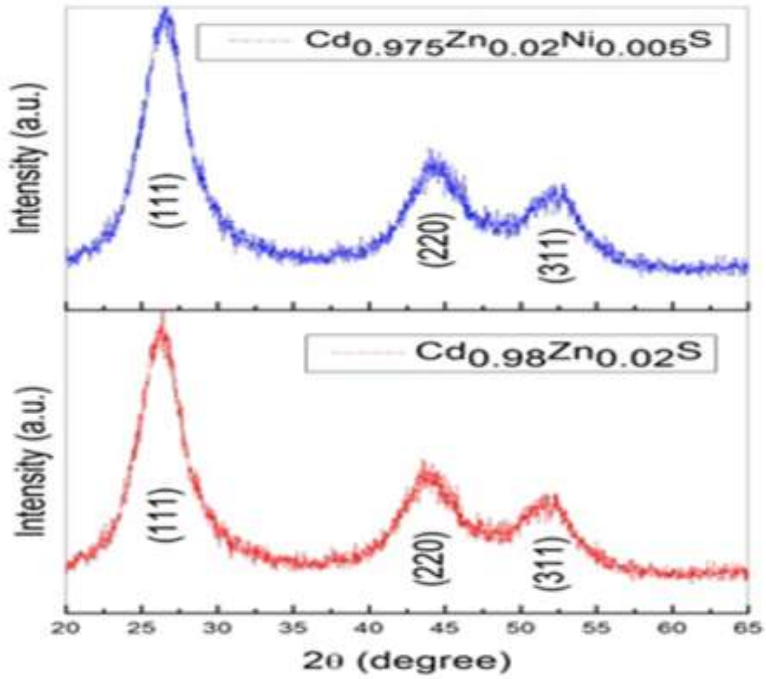

Figure 5 XRD patterns of the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs synthesized at room temperature.

All XRD diffraction peaks, correspond to (111), (220) and (311) planes, can be defined as those of zinc blende crystal structure of ZnS ((JCPD No: 65-0309) and CdS (JCPD No: 65-2887). No other peak reated to impurities was observed in the diffraction patterns. It is approved that Ni has been succcesfully doped in the lattice site rather than interstitial ones. Lattice constants of the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$NCs were calculated from the position of the peak (111) using the formula as given in Equation 4.

$$a = \frac{\lambda}{2\sin\theta}\sqrt{h^2 + k^2 + l^2} \qquad (4)$$

a : Lattice is constant,
θ : The Bragg's diffraction angle,
λ : The wavelength of x-ray,
(hkl): Miller indices.

The lattice constant for the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs, was found as 5. 86 Å and 5.84 Å, respectively which is smaller than lattice constant of CdS NPs obtained by Rodriguez et al [34]. This reason can be explained by Vegard's law. It means that the diffraction peaks shift towards to higher Bragg's diffraction angle because Ni^{2+} has a smaller ionic radius (0.62 Å) than Cd^{2+} (0.97 Å). Thus, the Ni dopant cannot create individuals peak by side of host peak.

Three broad diffraction peaks of the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs represent that the size of the NPs become smaller. The average particle sizes of the NPs were estimated from the peak widths of the relatively strong (111), (220) and (311) diffraction peaks using Debye- Scherrer's formula as given in Equation 5:

$$t = \frac{0.9\lambda}{\beta\cos\theta} \qquad (5)$$

t: The mean size of the NPs,
θ : The Bragg's diffraction angle,
λ : The wavelength of x-ray,
β : The broadening measured as the full width at half maximum (FWHM) in radians

The sizes of the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs were found as 2.3 and 2.1 nm, respectively. This result proves that the doping affect the size of NPs, results in the increase in the β of the XRD peaks of the NPs.

The shape and morphology properties of the $Cd_{0.08}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs were investigated by the SEM analysis. Figure 6a and 6b demonstrates the typical SEM image of the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs, respectively.

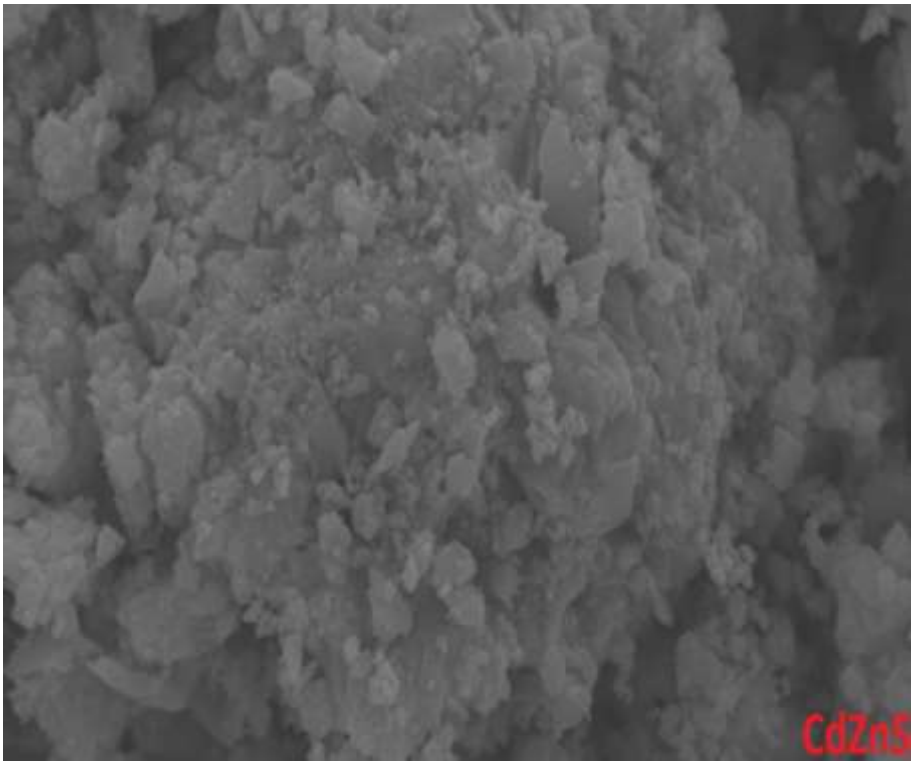

Figure 6a Typical SEM photograph of the $Cd_{0.98}Zn_{0.02}S$ NPs synthesized at room temperature.

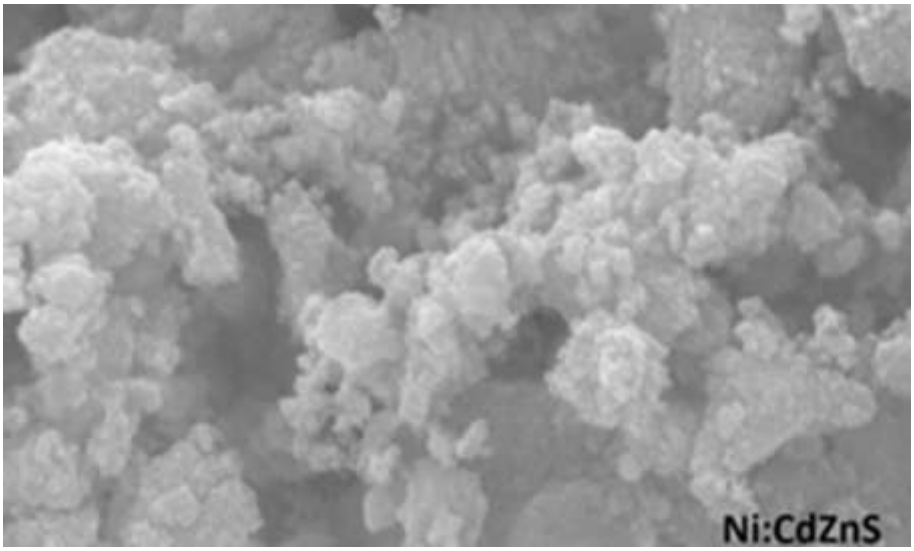

Figure 6b Typical SEM photograph of the $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs synthesized at room temperature.

It was observed that the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs poss tightly packed surface morphology which is consistent with the investigations of Theerthagiri et al [35]. In addition, Theerthagiri et al [35] reported

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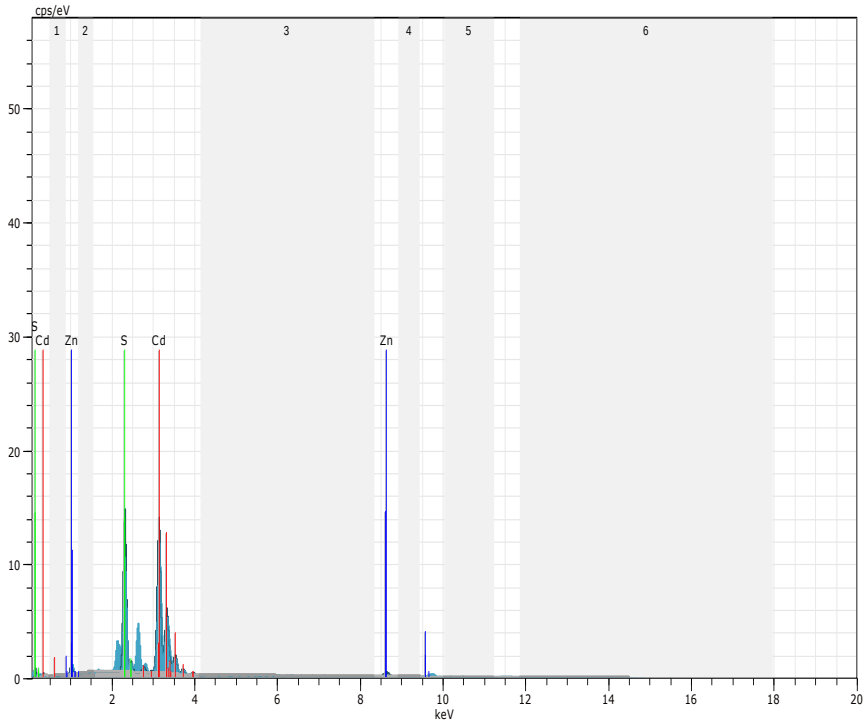
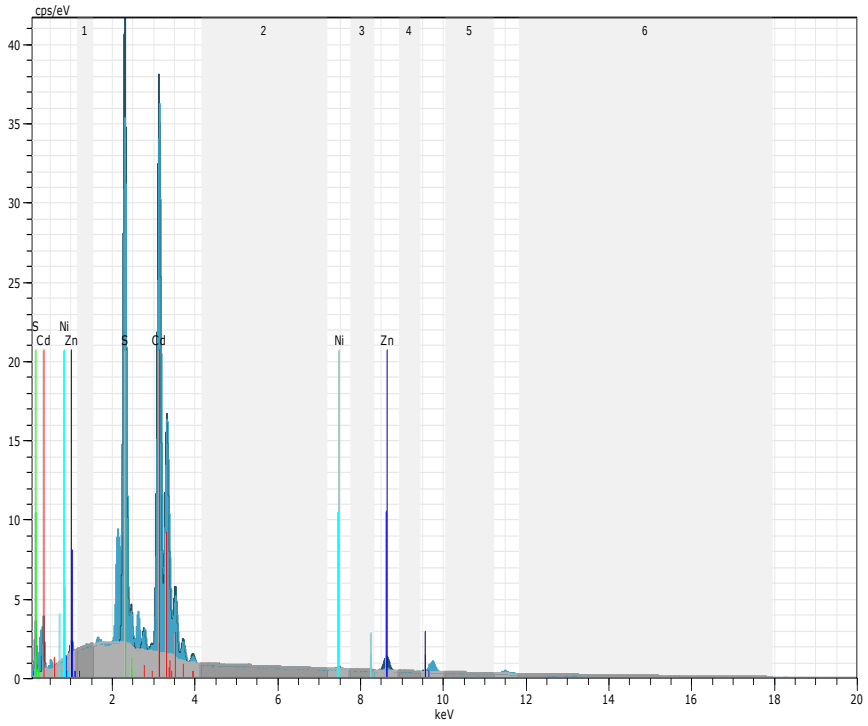
that the tightly packed surface morphology of the composite materials is beneficial for the efficient charge carrier separation and useful for efficient photocatalytic activity.

Elemental analysis of $_{.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs was performed by making the EDX measurements indicated in Figures 6c and 7d. Both Zn and Ni have been successfully synthesized at room temperature by the chemical precipitation method, which is clearly shown in the figures.



Şekil 6c EDX spectrum of $Cd_{0.98}Zn_{0.02}S$ NPs.



Şekil 6d EDX spectrum of $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs.

To be carried out optical absorption measurement, the certain amount of the $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs were dispersed in ethanol. Figure 7 exhibit the optical absorption spectra for $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ Nps in ethanol solution.

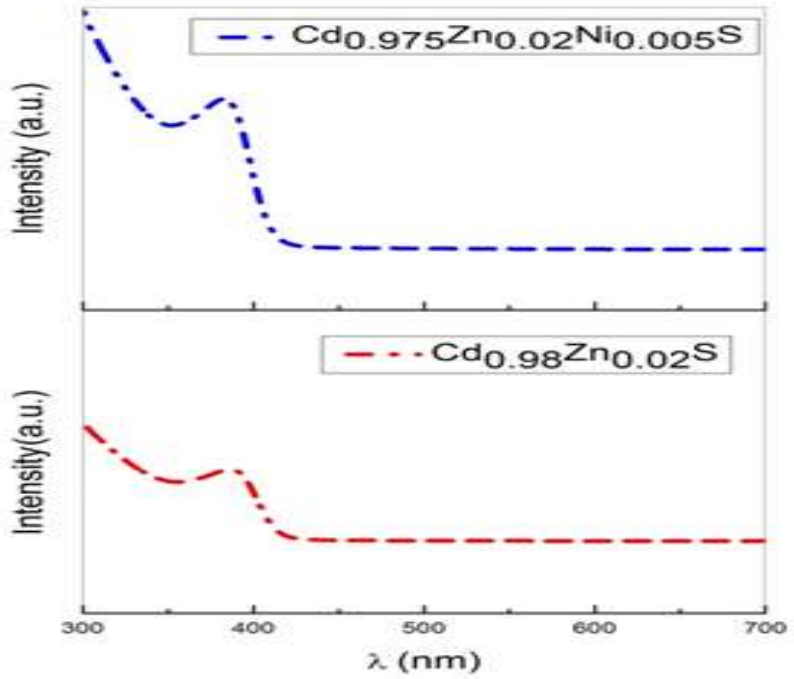


Figure 7 The UV-Vis absorption spectra of $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NCs were dispersed in ethanol.

The absorption peaks of $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs were observed at 387 nm and 384.12 nm, respectively. It was observed that the peak of $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs is blue shifted compare to the UV of spectrum of $Cd_{0.98}Zn_{0.02}S$. It was found that the band gap of $Cd_{0.98}Zn_{0.02}S$ NPs (E_g=3.13 eV) shows an increase when they are doped with Ni content (E_g=3.2 eV). The obtained results demonstrate that the doping plays a very significant role in the band gap assignment. Using E_g values obtained from Figure 7, The calculated particle size for $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}Ni_{0.005}S$ NPs using the equation given in Reference [36] was found as 2 and 1.95 nm, respectively.. This result is in good agreement with result obtained from the XRD measurements.

III. CONCLUSİONS

In our present study, CdZnS NPs with different Zn concentrations were synthesized by chemical precipitation at room temperature. Zn concentration values used were determined as 1%, 2%, 5%, 10%. The photocatalytic properties of these synthesized NPs were investigated to determine the optimum Zn concentration in CdZnS NPs with different Zn concentrations. Thus, the optimum Zn concentration with the best photocatalytic activity was found to be 2% and this sample was named $Cd_{0.98}$ $Zn_{0.02}$ S.

In the second phase of our study, $Cd_{0.98}Zn_{0.02}S$ NPs with different Ni concentrations were synthesized using the same method. The Ni concentration values used were determined as 1%, 2%, 5%, 10%. By examining the photocatalytic properties of these synthesized NPs, the optimum Ni concentration with the best photocatalytic activity was found to be 0.5% and this sample was named $Cd_{0.975}Zn_{0.02}$ $Ni_{0.975}$.

Finally, the structural, morphological and optical properties of $Cd_{0.98}Zn_{0.02}S$ and $Cd_{0.975}Zn_{0.02}$ $Ni_{0.975}$ NPs were studied.

IV. ACKNOWLEDGEMENT

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