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Structural and Photocatalytic Analysis of Nanostructured CdO, ZnO and their Composite Useful to Remove Textile Dyes Waste from the Drainage System

Chitra Bhukkal[1,2] and Rachna Ahlawat[2]
FGM Government College, Adampur, Hisar[1]
Department of Physics, Chaudhary Devi Lal University, Sirsa[2]
chitrabhukal@gmail.com

Abstract: *In recent years, the fabrication of semiconductor nanostructures has increasingly been adopted as the stimulating mechanism in nanoscience and nanotechnology. The comprehensive investigation and environmental impact of CdO, ZnO, and their nanocomposite executed in this direction with the help of a profitable co-precipitate approach at room temperature. In the prepared sample, the diffractogram is having rock salt CdO and wurtzite ZnO crystalline phase while the composite sample has combinedpeaks of both.We have investigated the modified intensity, FWHM, crystalline size, and microstrain present in the synthesized samples. The texture of the surface is evaluated by SEM micrographs for prepared nanocrystallites. In the present study, Rhodamine B dye is being selected for decomposition and investigated the catalytic efficiency of prepared samples under visible light.*

Keywords: CdO-ZnO nanocomposite, Dye degradation, Environment impact, etc.

I. INTRODUCTION

The polycrystalline formation of II-IV semiconductors has primarily focused on current research areas due to applicability in the diverse field of modern photonics [1]. Not only photonics, but other branches of chemical and life sciences have also widely utilized the oxides of the II-IV series of elements, especially CdO and ZnO [2]. These compound semiconductors have n-type intrinsic behavior, high binding energy (above 50 MeV), large thermal and chemical durability, and can generate good quality native substrate. Also, CdO is another most important compound semiconductor with a rocksalt structure and possesses low electrical resistivity, large optical transmittance, and direct/indirect bandgap energy [3]. Polycrystalline ZnO has quite wide bandgap energy that is mainly good for modern short-wavelength devices [4]. Both CdO and ZnO have considerably shown high potentiality at nanoscale dimensions with exciting optical, electronic catalytic behavior [5-6]. Besides the single form of these oxidesthe binaries of CdO-ZnO are also explored.In material science, different structures of such nanomaterials have been investigated depending upon the requirements. In cleansing the air and waste management, II-VIsemiconductors are also acted as an atmospheric disinfectantdue to their significant applications as a water purifier.Large studies are available in the literature on semiconductor photocatalysts related to the degradation of organic dyes [7]. In this context, M_xO_y(Metal = Titanium, Cerium, Magnesium, etc), their binaries are investigated as the dynamiccatalyst that eliminatesharmful dyesfrom drainage water [8-9]. The mainproblemin single metal-oxidehas been noticed as the fast charge carrier's recombination rate that modified the photocatalytic efficiencyup to great extent. While their binary formations can affect the energy gap and move the excitonic band edge to a higher wavelength. Consequently, the separation in the e^--h^+ pair expandsunder an appropriate light source and as a result, enhances photocatalytic character [10]. Therefore, the preparation of CdO, ZnO, and CdO-ZnO nanocrystallites is systematically presented in this study. We have selected Rhodamine B dye to evaluate the photocatalytic character of prepared nanopowder and proposed that the nanocomposite sample is the best decomposer of such synthetic dyestuffs excreted by textile industries.

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II. EXPERIMENTAL

2.1 Methodology for Preparation

All chemical precursors used in this study are as follows:

- 0.6 mole fraction of $Cd\{(CH)_3COO\}_2.2H_2O$ (cadmium acetate dihydrate)
- 0.4 mole fraction of $C_4H_6O_4Zn.2H_2O$ (zinc acetate dihydrate)
- 0.5M sodium hydroxide (NaOH)

These chemicals with analytic grade standards are acquired through Sigma Aldrich Pvt Ltd which are refluxed in a conical flask according to their fixed molar ratio. The detail of the complete procedure has been published in our earlier publications [10-11].The synthesized samples are further characterized using experimental tools like XRD, SEM, UV-Vis, etc.

Tools for Characterizations

- For phase identification, an XPERT-PRO x-ray diffractometer was used and functioned at 45kV and 40 mA through Cu Kα monochromatic rays of 1.5406 Å wavelength in the step of two degrees.
- The surface texture has been evaluated by JEOL-JSM-6100 scanning electron microscope that was also linked with Energy Dispersive X-ray Spectrometer (EDX) used for compositional analysis of elementals.
- For recording absorption spectra, Lambda 750 spectrophotometer (Perkin Elmer)was employed in the UV-Visible-NIR range of wavelength. The device is also helpful in the investigation of dye degradation of Rhodamine dye under visible light illumination.

III. RESULTS AND DISCUSSIONS

3.1 Phase Identification by (XRD)

To assess phase purity and crystal structure of prepared nanopowder crystallographic evaluation of synthesized CdO, ZnO, CdO-ZnO samples has been accomplished as displayed in Fig.1.

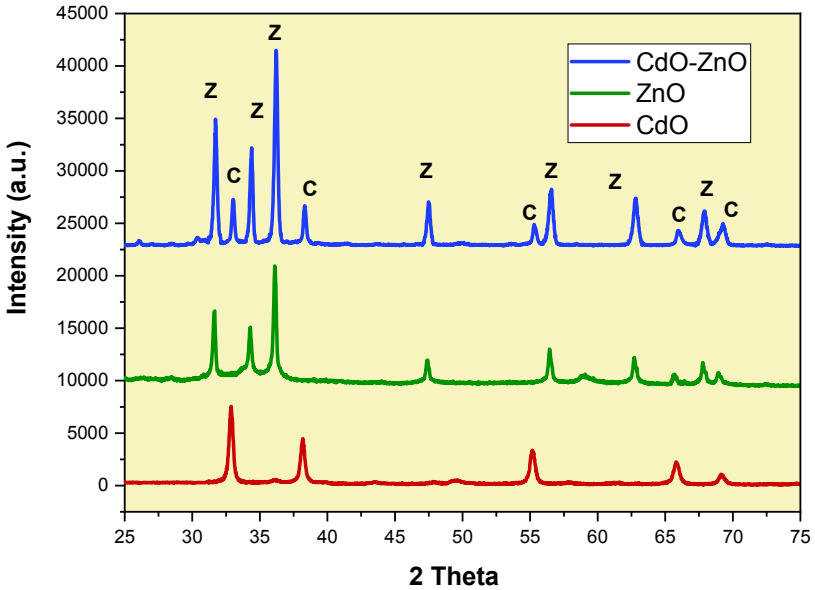

Figure 1: X-Ray Pattern of pristine CdO, ZnO, and theirnanocomposites.

The x-ray diffraction pattern for all prepared samples is exhibited in Fig. 1 in the 20-80°C range in two theta degrees. In pristine CdO sample, the characteristic peaks are evolved at different angles of 2θ given in table 1 which are attributed to [111], [200], [220], [311], and [222] reflection planes of pure cubic CdO. These observations are in an excellent match with standard JCPDS card no: 05-0640 [12]. The particular structure as an $Fm\bar{3}m\{225\}$ space group and lattice parameter 'a' = 4.695Å that ensures the rock salt crystalline structure of cubic CdO [13].For ZnO pristine sample, the detail of all diffraction peaks is given in table 1 with corresponding planes as [100], [002], [101], [102], [110], [103],

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[200], [112] and [201]. These peaks and planes emphasized the P63mc{186}space group- point group:6mm and acquires its lattice parameter as *'a'*and 'c' as 0.3249 nm and 0.5206 nm, respectively [14]. When binary nanocomposite is formed, its crystallographic pattern is having peaks of both CdO and ZnO phases as observed in Fig. 1. Here, one may notice symbol (**Z**) denoteswurtzite peaks of ZnO while (**C**) represents the standard peaks of rock salt CdO. Details of XRD peaks are summarized next in tabular form. In this study, one may notice that the establishment of the crystalline structure and analyzed results are similar to the previous literature [10-15]. Nanocrystalline size for the most prominent peak is evaluated using Debye-Scherrer's formula and found as 32, 35, and 38 nm, respectively for CdO, ZnO, and their nanocomposites.

Table 1: Details of XRD peaks of CdO, ZnO, and CdO-ZnO nanocomposites.

Sr. No.	CdO			ZnO			CdO-ZnO		
	2θ	I (%)	[hkl]	2θ	I (%)	[hkl]	2θ	I (%)	[hkl]
1	32.86	100	[111]-C	31.62	64	[100]-H	31.62	55	[100]-H
2	38.20	62	[200]-C	34.37	53	[002]-H	32.94	100	[111]-C
3	55.30	48	[220]-C	36.13	100	[101]-H	34.32	42	[101]-H
4	65.90	30	[311]-C	56.52	34	[110]-H	36.13	83	[101]-H
5	69.20	18	[222]-C	62.75	26	[103]-H	38.26	86	[200]-C
6				65.75	16	[200]-H	47.34	22	[102]-H
7				67.97	23	[112]-H	55.34	43	[220]-C
8				68.10	16	[201]-H	56.50	26	[110]-H
9							62.75	26	[103]-H
10							65.94	29	[311]-C
11							67.92	20	[112]-H
12							69.24	18	[222]-C

3.2 Scanning Electron Morphology

The surface morphology of CdO, ZnO, CdO-ZnO samples is displayed in Fig. 2 that is executed by scanning electron microscope.

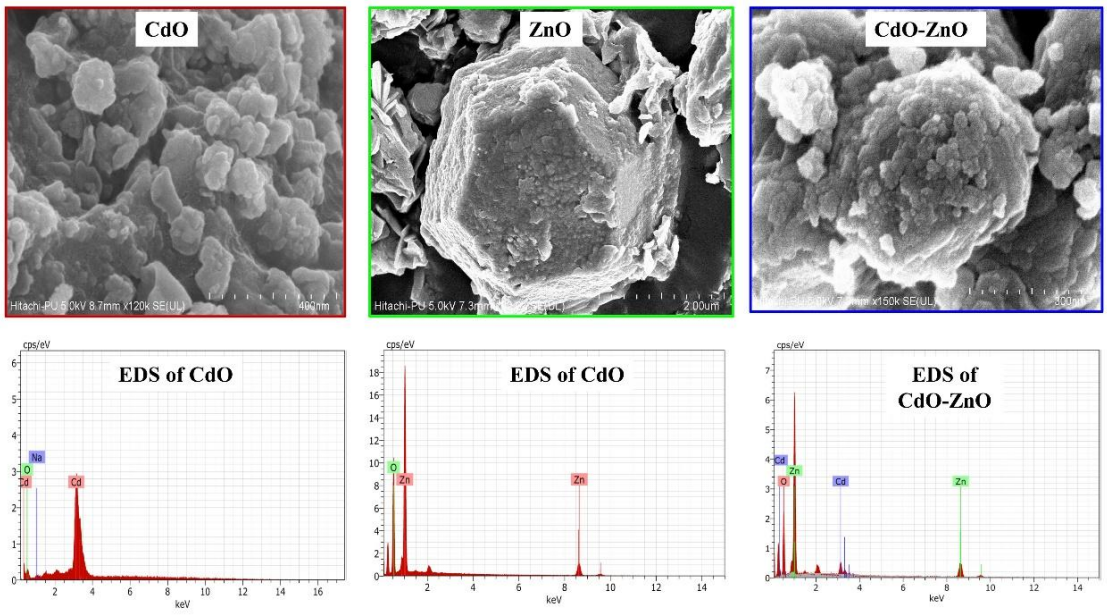

Figure 2: Scanning Electron Microscope of prepared samples of pristine CdO, ZnO, and CdO-ZnO nanocomposites.

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It is seen that the top surface of samples is covered with aggregated grains which generally formed due to their large surface energy. As pristine CdO has a compatible and well-organized lattice, therefore, in this nanopowder, mostly particles are evident on the surface with spherical morphology and consistently distributed. One may notice beautiful hexagonal-shaped ZnO nanocrystals that are clearly displayed in Fig. 2. The particular shape of these particles is potentially useful owing to the large surface area and great importance in numerous optoelectronic devices as well equally in particularly in dye degradations.

3.3 UV-Vis Spectroscopy

Fig. 3 displays the UV-Vis absorbance within the range of 200-800 nm for all prepared samples. UV-V is spectrum reported absorption band at 420 nm and its strong and sharp band edge absorbance appeared in the visible range forpristine CdO [2]. We have found an absorption band at 410 nm for pure ZnO sample for which band edge lies towards shorter wavelength side [5]. Moreover, in the composite sample, the absorption band exists at 380 nm while the band-edge is shifted more to the lower wavelength side. All these observed absorption bands assigned to excitonic transitions from lower energy band to higher energyband.

We have learned that a finite increment in absorbance has been generated via band-to-band transition. This absorbance spectrum is helpful to decide the bandgap(E_g) energy of the produced nano powder. To execute this, a graph is plotted that shows the variation of energy (hν) on the x-axis while on the y-axis $(\alpha h\nu)^2$ term is denoted and the linear portion of the graphensured the direct allowed electronic transition.To calculate the bandgap energy, we have extrapolated this straight portion on the x = 0 axes and frequently measured bandgap energy (E_g) of the pristine CdO which is = 2.55 eV.

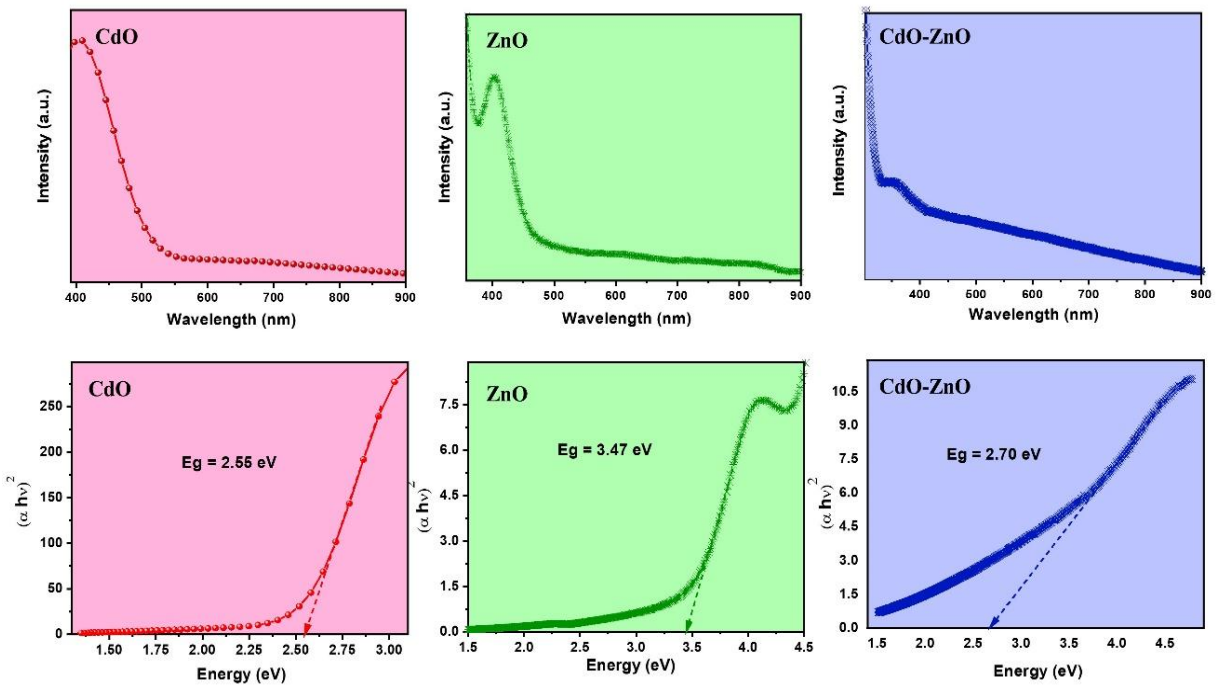

Figure 3: UV-Vis Spectra and corresponding bandgap energy for prepared nanocrystallites.

Using Tauc's plot, it is established that, the bandgap energy is 3.47 eV for ZnO pristine nanopowder. However, bandgap energy for nanocomposite is found at 2.70 eV that is a little more than the CdO and lower thannanocrystallites ZnO [14-15]. These results suggest that these semiconductors are verybeneficialin photocatalytic functions and capable to captivatethe particular wavelength due to nanocomposite formation of narrow and wideband materials.

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3.4 Dye Degradation of Rhodamine B

Decomposition of Rhodamine B(Rh B) dye is chosen for present investigation under white lightsinceit has an absorption in the solar range i.e. λ_{max} = 560 nm. The Rh B dye has been collected from Sigma-Aldrich Pvt. Ltd and formed achosen concentration (10 mg L^{-1}) with the help of deionized water as a solvent. 2.3 mg of Cdo, ZnO, and CdO-ZnO nanopowder was rigorouslymixed in 4 mL of Rhodamine Bsolution. To observe theresult, a white light source (150 W)was exposed to the sample holdercontaining Rhodamine B and photocatalyst powder. Then, an appropriate solution of degraded dye was takenevery15 minutes and examined its absorption spectra.

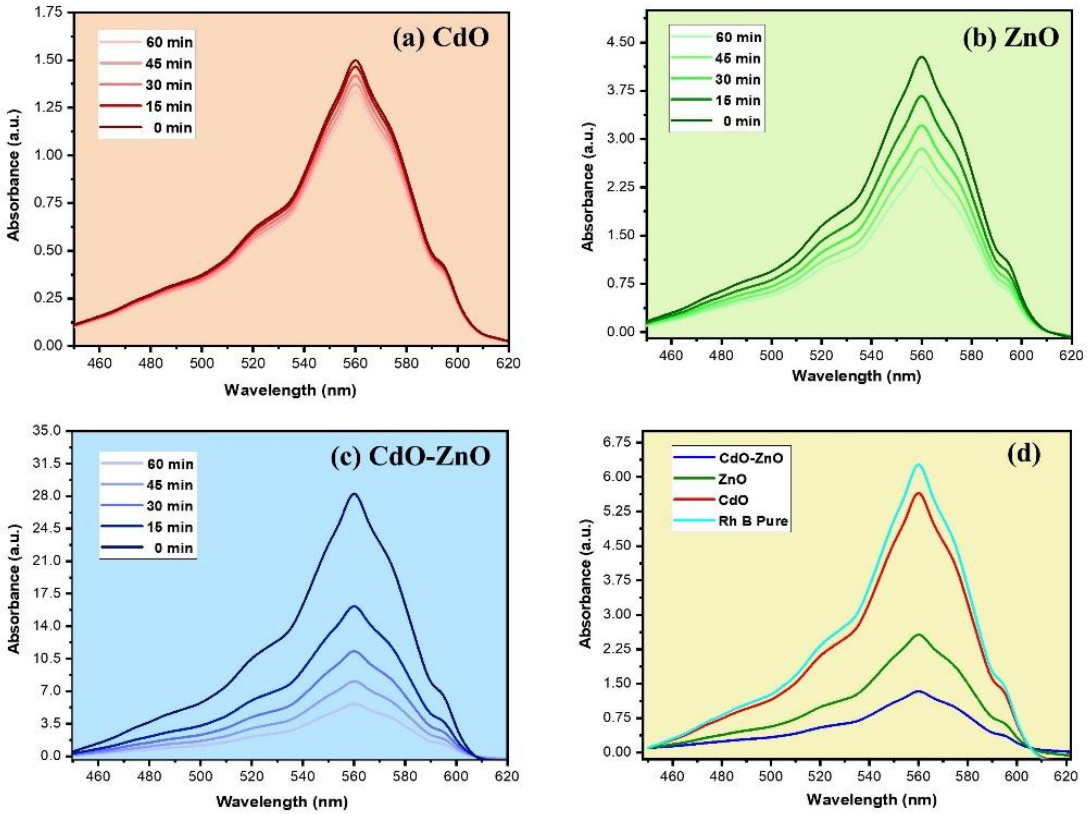

Figure 4: Degradation of Rhodamine B dye within subsequent time intervalsusing (a) CdO, (b) ZnO, (c) CdO-ZnO nanocomposite, and (d) comparative analysis of prepared samples.

Fig. 4s hows the depletion in the absorption of Rh B dye in the irradiation time 0 to 60 min under visible lightusing all prepared samples. When pristine CdO is being used as a photocatalyst, the maximum (λ_{max} = 560 nm) absorbance peak of Rh B is only slightly modified with time exposure up to one hour i.e. 60 minutes). When ZnO pristine sample is used for degradation, the Rh B dye is significantly decomposed under visible light exposure within different time slots. One may notice in Fig. 4 (c) that the nanocomposite of CdO-ZnO has the highest efficiency to degrade the Rh B dye under the same conditions. The decomposition is nearly zero without any photocatalyst and consequently, white light did not affect the dye. The degradation efficiency is estimated with the help of the following formula.

$$\text{Degradation Ratio } (\%\ \eta) = \left(1 - \frac{A_f}{A_i}\right) \times 100$$

Here absorbance before irradiation of white light is taken as $A_i$and after a fixed irradiation time it is $A_f$as shown in the above formula.

IV. Conclusion

The presented sample are successfully fabricated using chemical route and characterized by XRD, SEM and UV-Vis spectroscopy. Pure cubic structure is identified for pristine CdO and wurtzite phase is obtained for ZnO while nanocomposite has mixed peaks of both phases. SEM analysis revealed the spherical shape for CdO particles,

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hexagonal shape for ZnO and mixed shape for their nanocomposite with comparative large surface area. UV-Visible results analyzed that all nanopowders are translucent in the whole solar portion. The degradation of Rhodamine B istested successfully to explore the photocatalytic nature of the synthesized nanopowder. It is evaluated that the nanocomposite sample has the maximum efficiency owing to its huge surface/volume ratio, suitable bandgap, and most ordered crystalline structure.

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