

Sustainable Photocatalytic Removal of Diclofenac from Aquatic Environments Using CdS Nanorods: Synthesis, Structural Characterization, and Performance Evaluation

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ABSTRACT

The widespread contamination of aquatic ecosystems through pharmaceutical pollutants, particularly diclofenac shows a critical challenge to conventional water treatment systems, which are often inadequate in addressing the persistent and harmful nature of these substances. This study evaluated the photocatalytic degradation of diclofenac using cadmium sulfide nanorods (CdS NRs) synthesized via a hydrothermal method at 363 K. The cost-effectiveness, availability, easy synthesis and exceptional redox activity of CdS NRs, demonstrated its promising potential to be used as photocatalysts for environmental cleanup. Several analytical tools were used to determine its surface morphology, structure, physicochemical and optical properties. FESEM analysis revealed an average particle size of 90.89 ± 0.01 nm along with the EDX spectra and elemental mapping. XRD analysis confirmed a face-centered cubic (FCC) fluorite-type structure with crystallite sizes of 6.838 ± 0.01 nm. UV-Vis spectroscopy showed two prominent absorbance peaks at 350 nm and 480 nm with a corresponding bandgap energy of 2.39 ± 0.01 eV. The presence of Cd-S bond and improved crystallinity as well as decreased surface adsorbates at increased synthesis temperatures were confirmed by FTIR spectroscopy. Photocatalytic degradation of CdS NRs was obtained to 85 percent in 210 minutes when xenon lamp ($\lambda > 300$ nm, 300 W) was used. The results proved a new and effective way of the photocatalytic removal of insistent pharmaceutical contaminants and offered a long-term opportunity to improve the quality of water and save the environment.

1. Introduction

The growing use of pharmaceuticals in the world today has resulted in a major problem of drug contaminants in water bodies [1]. A prominent example of such pollutants is Diclofenac a non-steroidal anti-inflammatory drug (NSAID) that is a widely used drug because of its persistence [2,3], high environmental stability, and low biodegradability [4]. Traditional wastewater treatment facilities have been ineffective to a great extent in getting rid of diclofenac, and this leads to a build-up of the same in water bodies [5]. This build up has some very dangerous effects to the aquatic life and the health of human beings, with the potential to bioaccumulate in aquatic life and interfere with the endocrine systems endangering not only the ecological balance, but also the health of the population [6].

This demonstrates that new, effective, and innovative methods of treatment using water are much needed. One of the prospective alternatives in breaking down the persistent organic pollutants and even the newly developed pharmaceutical, lies in the form of photocatalysis, which would take place under an onus of light energy to facilitate a chemical process [6,7]. Different studies have managed to establish the effectiveness of different compounds used as the photocatalysts, in the degradation of diclofenac and other medications [8,9,10]. As an illustration, titanium dioxide (TiO_2) nanoparticle photocatalysts have been widely discussed but can only work when in the UV light since they have wide bandgap [11,12]. Similarly, TiO_2 nanoparticles have also been shown to increase in their efficiency by getting silver-doped and have also raised concerns to do with their costs and their stability. Likewise, TiO_2 nanoparticles have demonstrated their increase in efficiency by being silver-doped and have raised issues to do with their costs and their stability [13]. Cadmium sulfide (CdS) nanorods are one of the many photocatalysts that have caused a lot of stir because of their narrow bandgap energy, incredible amounts of light they can absorb in the visible region, and great separation of the charges [14]. The new developments have demonstrated that the efficiency of CdS can be increased further by the combination with other substances such as titanium dioxide (TiO_2) or gold nanoparticles to slow down the electrons and holes recombination and maximize the separation between them over a longer period [15]. Despite such a prospect, hiccups still exist in the practical usefulness of CdS nanorods, not only does their enhanced catalytic action hinder the latter, but there are also environmental secondary issues as well [16]. When deciding how these limitations can

overcome, the recent studies have put in place some of the solutions which are surface modification, bio-functionalization, incorporation of co-catalysts which have been found to reduce photocorrosion and enhance the stability and reusability of CdS nanorods [17]. In addition, nanorods modelling and optical properties have also been enhanced by photocatalytic reaction and synthesis techniques [18,19] such as hydrothermal synthesis [20], pulsed laser ablation in liquid (PLAL) [21,22] and sol-gel [13,23]. The article presents a methodical discussion of the sustainable purification of aqueous environment against diclofenac by using CdS nanorods.

This paper entails synthesis of CdS nanorods using hydrothermal method and its overall structural characterization using XRD analysis, FESEM, energy-dispersive X-ray spectroscopy (EDX), and transmission electron microscopy (TEM) along with efficiency of photocatalytic activity. The impact of other operational parameters on the photocatalytic degradation efficiency such as light intensity, pollutant concentration, and temperature is also discussed by the study. Additionally, the present work supplements earlier experiments in which the stability and reusability of the photocatalyst is critically evaluated and the mechanistic paths through which diclofenac is broken down explained. As an example, Ghamarpoor et al. [16] have shown the existence of intermediate compounds during photocatalytic degradation, which gives some information on reaction mechanisms, which are discussed in more detail herein.

This study is a general evaluation of the CdS nanorods as a potential, sustainable, and functional solution to the problem of pharmaceutical pollutants degrading in the water, which is the novelty of the paper. The findings of this paper is a contribution to the research of photocatalysis because it not only demonstrates the chance of applying CdS nanorods in environmental cleanup, but it also prepares the way towards the follow-ups of its material design and optimization. The results of the research have great data to inform the development of new generation technologies of wastewater treatment, which will minimize the negative impact of the environment caused by pharmaceutical contaminants.

2. Materials and Methods

2.1 Experimental Materials

Cadmium nitrate tetrahydrate ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$), ethylenediamine ($\text{NH}_2\text{CH}_2\text{CH}_2\text{NH}_2$), thiourea (NH_2CSNH_2) and deionized (DI) water were acquired

as chemical precursors of the same brand named Sigma-Aldrich at a purity of 99.9%. Synthesized products were exposed to a wide range of characterization techniques to investigate morphological, structural, and optical characteristics of the products. The crystal structure and phase composition of the nanocomposites were examined with the help of X-Ray Diffraction (XRD) and the surface morphology of nanostructures was examined in more detail with the assistance of FESEM. To confirm the elemental composition of the synthesized materials, Energy Dispersive X-ray Spectroscopy (EDX) was used, especially the detectability of cerium and cadmium, and Elemental Mapping was also made to investigate the spatial distribution of elements within samples. Further, to govern the functional groups and chemical bonding in the materials, FTIR spectroscopy was utilized. UV-visible spectroscopy (UV-Vis) has also been used, to observe the optical spectra of the made materials and to establish its band gap. All the characterization experiments done were reliable and accurate at the University-Industry research Laboratory Services, UTM.

2.2 Synthesis of CdS NRs

CdS nanorods were prepared in a hydrothermal, solvothermal way. In this procedure, 0.398 g of thiourea and 0.537 g of cadmium nitrate tetrahydrate were homogeneous in 40 mL of ethylenediamine solution with an aggressive stirring procedure of 1 hour. The resulting solution was added to 50 mL Teflon-lined stainless-steel autoclave and then treated solvothermally at 364 K for 24 hours. After this reaction, the precipitates, consisting of CdS nanorods, were collected using centrifugation, and extensively cleaned with ethanol, and deionized water for several times, to eliminate the remaining impurities and made to dry overnight in an oven at 335 K.

2.3 Photocatalytic Activity

The same methodology as in our previous investigations [24] was followed, altered slightly to test the photocatalytic activity. A model pollutant solution was made up using 35 mg of raw diclofenac dissolved in 1 liter of deionized (DI) water, to make a final concentration of 35 mg/L. A one-hour stirring was performed at 400 rpm to make the solution fully dissolved. Under degradation tests, the solution obtained was mixed with 20 mg of CdS nanorods (NRs). This was then sonicated in dark to allow system to achieve adsorption-desorption equilibrium for 30-

minute. To perform the photocatalytic reaction, a 100 mL quartz reactor was used, and this quartz reactor was treated with the Xenon lamp (300 W) at approximately 10 cm above the liquid. To monitor the degradation process, aliquots (2 mL) were collected at a given time interval and then the analysis of the UV-visible spectroscopy carried out.

3. Results and discussion

3.1 Structural Analysis by XRD

The XRD patterns were meticulously analyzed to determine the crystal-like phases, and the composition of cadmium sulfide (CdS). The characteristic diffraction peaks for CdS were observed at 24.9° (100), 26.6° (002), 28.4° (101), 36.7° (102), 43.7° (110), 47.8° (103), 51.9° (112), and 66.8° (203), which align closely with the standard JCPDS data (No. 41-1049) [16,17]. To help make a better comparison, the standard superposition on diffraction (SSD) pattern of CdS was overlapped in Figure 1. Such comparative study made it possible to offer a thorough analysis of the synthesized CdS, which is phase pure and contains a crystallographic structure. Also, the lattice parameters of the CdS synthesized nanorods were not dissimilar to the hexagonal wurtzite as the a-parameter was measured out to be 4.09 Angstrom and the c-parameter 6.68 Angstrom in the crystallographic plane

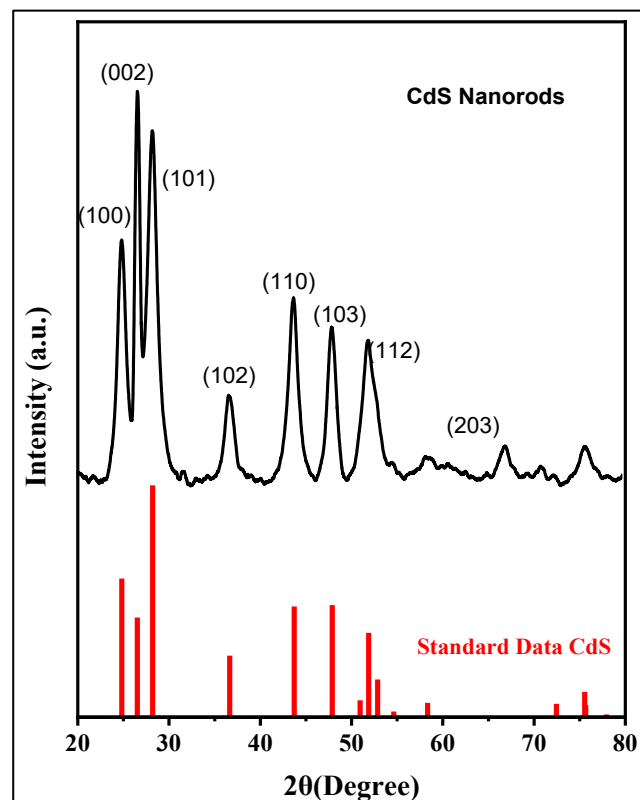


Fig. 1. XRD patterns of as-synthesized CdS NRs, the standard line patterns of CdS are given at the bottom

of (002), especially. These results assure, the structural stability, crystallinity and the proper orientation of the developed CdS nanorods, which make them usable as photocatalysts.

3.2 Surface Morphology and Elemental Analysis

To explore the surface morphology of the synthesized nanostructures, FESEM was utilized. The Figure 2(a) shows the morphology of the synthesized CdS NRs and Figure 2 (a1) shows their respective size distribution histogram. The synthesis of nanorods is proven to be successful, as the analysis shows that their average diameter is around 90 nm. There are also the elemental mapping images, which were achieved by EDX, and which give an idea of the composition and spatial distribution of the elements in CdS nanorods (Figures 2(b) and 2(b1)). The EDX data also supports the XRD data when it comes to phase purity, since the synthesized sample can be classified as highly pure. This high oxygen concentration in the EDX spectra can probably be explained by the presence of surface oxygen on the nanorods. The even distribution of both cadmium (Cd) and sulfur (S) exhibited through the elemental mapping, as well as the EDX spectra, shows the successful and uniform synthesis of CdS nanorods.

3.3 Optical Properties by UV-Vis DRS

To evaluate the optical properties of CdS NRs, related to light absorption, UV-vis DRS was employed, as depicted in Figure 3. The maximum absorption predominantly occurs in the UV-Vis region, peaking at approximately 342 nm and 456 nm, which matched the literature quite well [18–20]. Furthermore, the absorption range extends into the visible region at around 537 nm, surpassing the UV spectrum, as shown in Figure 3(a). This extended absorption range highlights the potential of CdS NRs for enhanced photocatalytic activity, attributed to the successful synthesis of the composite material and its nanoscale dimensions. The bandgap of the material was determined using the Tauc relation [21]. Figure 3(b) presents the corresponding Tauc plot of $(\alpha h\nu)^{1/n}$ Vs $h\nu$, CdS is a direct band gap semiconductor, and therefore, in the Tauc plot analysis, the exponent $n = 0.5$ was used, corresponding to a direct allowed electronic transition. The calculated bandgap of the synthesized CdS NRs was found to be 2.39 eV. This narrow bandgap is advantageous for extending the

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absorption spectrum, thereby boosting the activity of photocatalysis and efficiency in the photodegradation of pollutants.

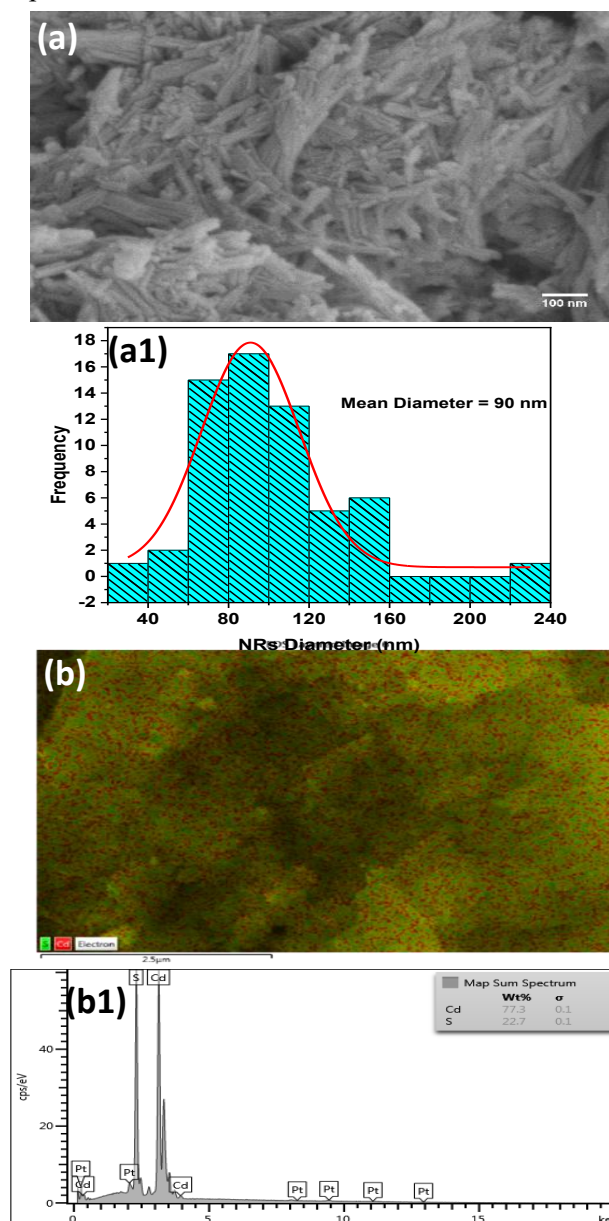


Fig. 2. (a) FESEM Micrograph **(a1)** the Histogram **(b)** Elemental Mapping Image **(b1)** EDX Analysis Image of as-synthesized CdS NRs

3.4 Functional Group and Bond Analysis by FTIR

Fourier Transform Infrared Spectroscopy (FTIR) was conducted to investigate the functional groups and bonding characteristics of the synthesized CdS nanorods. The spectrum, as shown in Figure 4, displays several characteristic peaks that provide insights into the material's structural and surface properties. The observed peaks at 1319 cm^{-1} , 986 cm^{-1} , and 807 cm^{-1} are attributed to vibrational modes associated with sulfur or cadmium in CdS, confirming the presence of Cd–S bonds that are essential to the crystalline structure of cadmium sulfide [22]. These peaks align well with previously reported CdS vibrational signatures, substantiating the successful synthesis of CdS nanostructures. In the region between

2650 cm^{-1} and 2750 cm^{-1} , peaks were detected nearly match to O–H stretching vibrations, or these peaks are more likely be associated with aldehyde or C–H stretching vibrations, which could originate from residual organic species or surface modifications introduced during the synthesis process. Additionally, the spectral region spanning 1684–1447 cm^{-1} exhibits peaks consistent with C=O and C–H stretching vibrations [23].

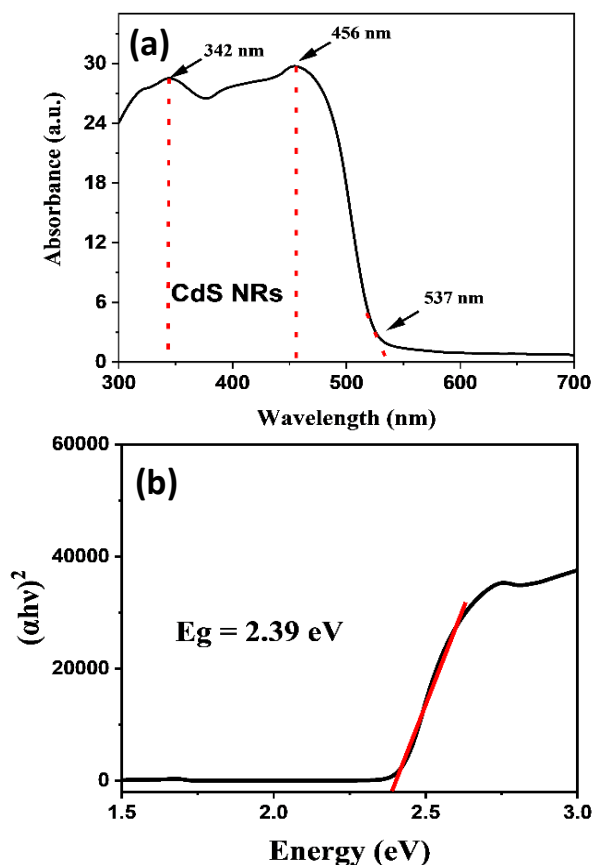


Fig. 3. (a) Diffusive reflectance spectra (DRS) of CdS NRs along with (b) the respective bandgap

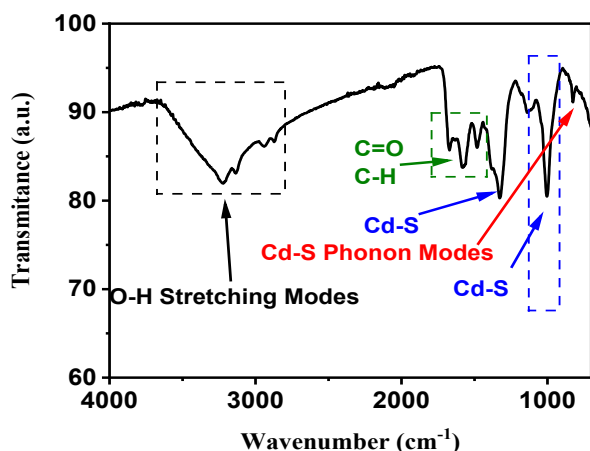


Fig. 4. FTIR Spectra of as prepared CdS nanorods

These are likely due to adsorbed organic molecules or surface-bound carbonyl groups that may have originated from the decomposition of precursors or incomplete removal of organic remainders during the process of synthesis. Such surface modifications or

adsorbed organic species can affect the photocatalytic properties of the material by altering the surface chemistry and electron transfer dynamics. Hence, the FTIR analysis validates the successful formation of CdS and provides valuable insights into the surface and functional group characteristics of the synthesized nanorods. These findings also demonstrate how surface functionalization may be essential for improving photocatalytic activity.

3.5 Photocatalytic Degradation Performance

The proposed photocatalytic mechanism for diclofenac degradation over CdS nanorods is illustrated in Figure 5. When exposed to light irradiation, CdS nanorods absorb photons and generate electron-hole pairs. Electrons in the conduction band reduce oxygen to superoxide radicals

($\cdot\text{O}_2^-$), while holes in the valence band oxidize water molecules to hydroxyl radicals ($\cdot\text{OH}$). These reactive species play a vital role in the stepwise breakdown of diclofenac molecules into less harmful intermediates and eventually mineralized byproducts [33]. The synergistic action of both radicals ensures efficient degradation and highlights the photocatalytic potential of CdS nanorods for pharmaceutical wastewater treatment.

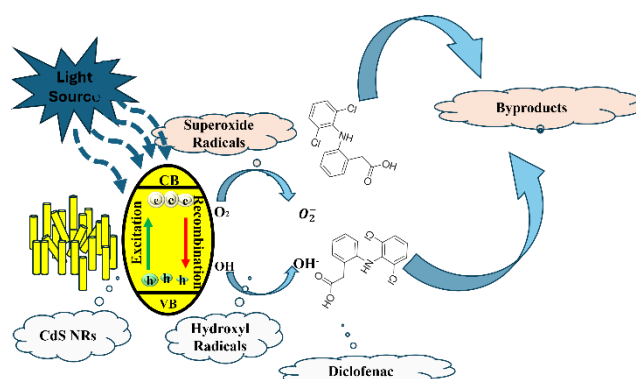


Fig. 5. Schematic illustration of the proposed photocatalytic degradation mechanism of diclofenac using CdS nanorods under UV irradiation.

The photocatalytic diclofenac degradation was monitored under UV lamp irradiation for a total duration of 210 minutes, with the degradation efficiency measured at 30-minute intervals. The degradation percentages observed were 20%, 34%, 45%, 56%, 64%, 72%, and 85%, respectively as shown in Figure 6 (a), indicating a steady increase in the removal of diclofenac with prolonged exposure to UV light. The kinetics of the degradation were observed using the pseudo-first-order kinetic model, a widely applied in photocatalytic studies, findings are shown in Figure 6 (b). The degradation rate constant (k) was calculated to be 0.0081 min^{-1} , demonstrating a significant degradation rate of diclofenac under the given experimental conditions. Additionally, the

coefficient of determination (R^2), also known as the goodness of fit, was found to be 0.96157, signifying a strong correlation between the experimental data and the pseudo-first-order kinetic model. These results highlight the efficacy of CdS nanorods as photocatalysts for diclofenac degradation under UV irradiation. The steady increase in degradation efficiency over time and the high R^2 value confirm the reliability of the kinetic model and the potential of the photocatalyst in wastewater treatment practical applications.

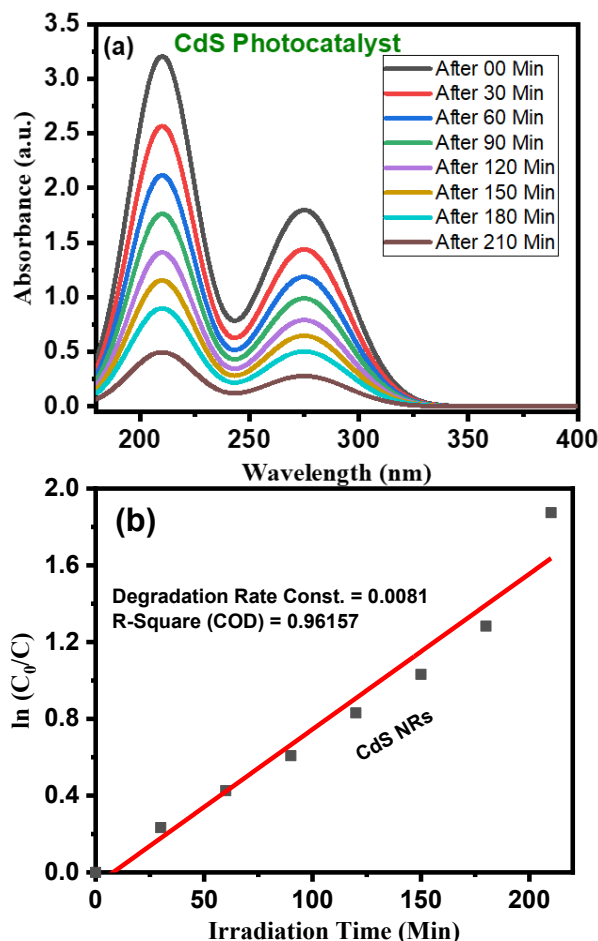


Fig. 6. (a) UV Spectra to observe the Degradation of diclofenac under UV Lamp in the presence of CdS NRs **(b)** Reaction kinetics to find the degradation rate constant

4. Conclusions

This study emphasizes the efficient preparation and characterization of cadmium sulfide nanorods (CdS NRs) and their usefulness in the photocatalytic degradation of diclofenac, a recalcitrant pharmaceutical pollutant. The FESEM and XRD, respectively, revealed high quality CdS nanorods of an average NR size of 90.89 ± 0.01 nm and crystallite size of 6.838 ± 0.01 nm, respectively. The substance had a hexagonal wurtzite structure and a bandgap energy of 2.39 ± 0.1 eV which was considered to be effective in absorbing light at the visible wavelength as confirmed by UV-Vis DRS analysis. The FTIR spectroscopy confirmed the existence of Cd-S bonds and improved

crystallinity, which showed that pure CdS nanorods are formed with minimum surface impurities. A photocatalytic reaction under a xenon lamp ($\lambda > 300$ nm, 300 W) confirmed a constant degradation of diclofenac, reaching 85% efficiency in the removal process in 210 minutes. Kinetics of the degradation followed a pseudo-first-order reaction that had a rate constant (k) of 0.0081 min^{-1} and an R^2 of 0.96157 which showed the accuracy of the model. The findings highlight the promise of CdS NRs as an economical, efficient, and environmentally friendly photocatalyst in the purification of pharmaceutical impurities in the water systems. Therefore, the study is a unique and viable answer to the problem of pharmaceutical pollution which will open the path to the creation of sophisticated water treatment systems. The capacity of CdS NRs to scale and their functionality in a variety of environmental conditions can be investigated in future research to make them even more applicable in practical settings.

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