

A Comparative Study of the Photocatalytic Properties of ZnO and ZnS Nanoparticles Combined with Carbon Quantum Dots

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Abstract ZnO/CQDs and ZnS/CQDs nanostructures were prepared by simply mixing ZnO and ZnS nanoparticles with carbon quantum dots (CQDs) to compare their photocatalytic performance in the degradation of methylene blue (MB). X-ray diffraction (XRD) analysis revealed that the presence of CQDs does not affect the crystalline structure of the ZnO and ZnS nanoparticles (NPs). Furthermore, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analysis revealed a porous structure resulting from the aggregation of NPs with average diameters of approximately 30 nm and 39 nm, respectively for the ZnO/CQDs and ZnS/CQDs nanocomposites. Furthermore, the study of reflectance in the visible spectrum of sunlight showed that the presence of CQDs reduces reflectance, with reflectance percentages of approximately 86% and 88%, respectively for ZnO/CQDs and ZnS/CQDs nanoparticles. Under optimal conditions between 11 a.m. and 3 p.m. with solar exposure, photocatalytic activity achieved 100% degradation of MB (10 mg/L) in aqueous solution in 80 minutes and 120 minutes, respectively for ZnO/CQDs and ZnS/CQDs. The best photocatalytic performance was achieved in the presence of ZnO/CQDs. Consequently, CQDs derived from charcoal can be used for enhancing the photocatalytic properties of semiconductors for the degradation of organic pollutants in aqueous media under irradiation.

Keywords: Nanoparticles (NPs), carbon quantum dots (CQDs), photocatalysts, methylene blue (MB), degradation

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1. Introduction

Water pollution caused by chemicals such as organic dyes in industrial settings has contributed to environmental pollution [1]. Indeed, many of these dyes are often discharged into water without pretreatment [2]. The presence of these dyes in water can significantly reduce light penetration and affect the photosynthesis process in aquatic environments, thereby posing a real threat to the aquatic ecosystem [1]. Furthermore, the presence of dyes in water also has toxic effects on human health due to their role in the development of certain diseases such as allergies, tumors, cancer, and heart disease [3].

Among these dyes, methylene blue (MB) is the most commonly used for dyeing cotton, wood, and silk, and has

a harmful effect on living organisms and water [4,5]. Furthermore, inhaling it can cause breathing difficulties, a burning sensation, nausea, vomiting, sweating, and high blood pressure [6,7]. Removing MB from industrial effluents is a major challenge for environmental protection. It is therefore urgent to develop reliable and environmentally friendly technologies for its removal before it is discharged into the environment. To achieve these objectives, several techniques such as chemical [8], physicochemical [9], and biological [10] methods are used to treat these dyes in aquatic environments. Among these methods, advanced oxidation processes (AOPs) specifically photocatalytic systems based on nanomaterials have been the subject of numerous scientific studies [8,9,10].

Among these nanomaterials, semiconductors such as zinc oxide (ZnO) and zinc sulfide (ZnS) have been used for the removal of organic pollutants [11]. They are well

known as promising photocatalysts for wastewater treatment due to their high capacity to completely oxidize organic pollutants at low cost, combined with their high chemical stability and low toxicity [12]. Nevertheless, due to their wide bandgap (3.37 eV [13] for ZnO and 3.72 eV and 3.77 eV [14,15] depending on the phase), ZnO and ZnS NPs are only sensitive to radiation with energy at least equivalent to ultraviolet (UV) radiation, which accounts for approximately 5% of the total solar spectrum [16], thereby limiting their applications in solar systems for the degradation of organic pollutants.

To address this challenge, several approaches such as metal and non metal doping [17] and the surface deposition of noble metals [18] have been tested. A new class of nanoscale carbon materials, carbon quantum dots (CQDs), with sizes smaller than 10 nm, is attracting significant interest. CQDs are highly soluble in water and can bind to other chemically reactive groups for surface passivation and functionalization [13,19]. In addition, they possess high conversion efficiency, high photon absorption efficiency, high biocompatibility, low toxicity, an excellent reservoir of free electrons, and a π -conjugated structure [16]. Studies on composite systems consisting of ZnO and ZnS NPs and carbon-based materials such as carbon nanotubes, fullerenes, graphene, and CQDs continue to be the subject of intensive research.

All of these studies have demonstrated the sensitivity of ZnO/CQDs and ZnS/CQDs nanocomposites to solar radiation. In addition, several techniques using biomass [20] or citric acid via ammonium hydroxide (NH₄OH) at high temperatures (200 °C) in a hydrothermal method [21] as a carbon source to produce fluorescent CQDs have been evaluated.

This study builds on recent work on nanomaterials combined with carbon quantum dots for the degradation of organic pollutants [22,23], while proposing an innovative approach that compares the photocatalytic performance of ZnO/CQDs and ZnS/CQDs in the removal of organic pollutants from wastewater.

2. Materials and Methods

2.1. Reagents

The chemicals used in this study were of analytical grade and underwent no further purification (Table 1).

Table 1. Various chemicals used, their sources, and their purities

Chemicals	Sources	Purity (%)
KOH	Sigma Aldrich (St. Louis, MO, USA)	85
HCl	panreacquímica (Barcelona, Spain)	37
C ₃ H ₆ O	panreacquímica (Barcelona, Spain)	98
NH ₄ OH	Honeywell Fluka (Buchs, Switzerland)	28-30
Zn(NO ₃) ₂ ·6H ₂ O	Honeywell Fluka (Buchs, Switzerland)	98
Na ₂ S	Sigma Aldrich (St. Louis, MO, USA)	98

In addition, the deionized water used throughout this study was obtained using a water purification system from

Sichuan Zhuoyue Water Treatment Equipment CO., Ltd. (Chengdu, China). This system produces deionized water with a resistivity of 18.25 M Ω ·cm⁻¹. The CQDs were synthesized from commercially available charcoal (Abidjan, Ivory Coast).

2.2. CQD Synthesis Method

The CQDs were synthesized according to the procedure described in the literature [21], using the chemical fragmentation method at a relatively low temperature (50 °C). Specifically, 50 mL of ammonium hydroxide (NH₄OH) solution with a concentration of 2.5 mg/L was prepared from commercial NH₄OH by dilution in deionized water.

Next, 500 mg of powdered charcoal was added to this solution, which had been heated to 50 °C in a water bath under a fume hood, and stirred with a magnetic stirrer at 500 rpm for 24 hours. The resulting solution was cooled to room temperature and then centrifuged at 4,000 rpm for 5 min to finally obtain the CQDs.

2.3. Synthesis of ZnO/CQDs and ZnS/CQDs Nanoparticles

Nanocomposites consisting of CQDs and ZnO NPs (ZnO/CQDs) were synthesized according to the procedure described previously [24]. Briefly, 500 mg of ZnO NPs or ZnS NPs synthesized as described in the literature and subsequently modified [25,23,26] were dispersed in 250 mL of ethanol with stirring for 10 min. To this solution, 100 μ L of the prepared colloidal CQD solution was added. This mixture was stirred at 500 rpm for 30 min at room temperature. The resulting solution was collected by centrifugation, washed three times with ethanol, and dried at 100 °C for 3 h to ultimately obtain the ZnO/CQDs or ZnS/CQDs nanocomposites.

2.4. Sample Characterization

X-ray diffraction of the various samples was performed using a Siemens D5005 diffractometer (Siemens AG, Munich, Germany) equipped with a copper anode (λ K α 1 = 1.5406 Å). The incidence angle 2θ ranged from 5 to 90°, with a step size of 0.01. Absorbance and reflectance were measured using a Flame-S-XR1 UV-Visible spectrometer (Ocean Optics, Largo, USA). The fluorescence of the CQDs was studied using a Qiwei WFH-204B portable UV lamp (254/365 nm) (Hangzhou, China). The morphological characterization of ZnO NPs, ZnO/CQDs, and CQDs was performed by scanning electron microscopy (SEM) using a JSM-6010LA instrument (JEOL Ltd., Tokyo, Japan).

2.5. Photocatalytic Analyses of MB

The photocatalytic degradation test of MB (C₀ = 10 mg/L, at pH 11) in the presence of the various prepared photocatalysts was conducted between 11:00 a.m. and 3:00 p.m. at 28°C to maximize the benefits of solar irradiation. To this end, 150 mg of ZnO/CQDs or ZnS/CQDs was added to 100 mL of MB. The prepared solutions were stirred for 30 minutes in the dark and then

exposed to sunlight for intervals ranging from 20 to 120 minutes. The solutions are centrifuged at 4,000 rpm for 5 min. The recovered supernatant is measured using a UV-Vis spectrophotometer to determine the residual MB concentration at a wavelength of 664 nm.

Furthermore, the MB removal rate in the presence of these photocatalysts was estimated using equation 1:

$$\%D = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

Where C_0 represents the initial dye concentration and C_t represents the dye concentration after solar irradiation for time t .

2.6. Kinetic Study

The kinetics of the photocatalytic reaction of MB as a function of its initial concentration were studied in a solution containing 150 mg of ZnO/CQDs or ZnS/CQDs nanoparticles. The kinetic order of the MB dye degradation was evaluated using Equations 2 and 3.

Pseudo-first-order kinetic equation:

$$\ln\left(\frac{C_0}{C_t}\right) = k_1 t \quad (2)$$

The pseudo-second-order equation can be expressed as shown in Equation (3):

$$\frac{1}{C_t} - \frac{1}{C_0} = k_2 t \quad (3)$$

where C_0 and C_t are the initial concentration and the concentration at various times t of MB, respectively. The constants k_1 and k_2 are the rate constants for the pseudo-first-order and pseudo-second-order models, respectively.

3. Results and Discussion

3.1. Characterization of CQDs, ZnO/CQDs, and ZnS/CQDs

To determine the crystalline structure of the ZnO/CQDs and ZnS/CQDs nanocomposites, X-ray diffraction (XRD) analysis was performed (Figure 1a). The resulting diffraction patterns were compared with the technical data sheet (JCPDS No. 36-1451, for ZnO/CQDs). The results show that the ZnO/CQDs NPs all exhibit distinct peaks at 2θ values of 31.77° , 34.42° , 36.25° , 47.54° , 56.60° , 62.86° and 67.96° , which can be attributed, respectively to the (100), (002), (101), (102), (110), (103) and (112) of the hexagonal wurtzite structure of ZnO with lattice parameters ($a = b = 3.2498 \text{ \AA}$ and $c = 5.2066 \text{ \AA}$). This result is in good agreement with the standard JCPDS data sheet and the literature [27]. Furthermore, the results clearly show that the prepared ZnO/CQDs nanocomposites have the same structure as the ZnO NPs alone. This suggests that the presence of CQDs does not affect the structure of the ZnO NPs, as previously noted by Liang et al. [27].

As for the ZnS/CQDs, XRD analysis reveals diffraction peaks that can be attributed to the (111), (220), and (311)

diffraction planes, observed at 2θ of 28.65° , 47.64° and 56.62° , respectively. The results show that the diffraction peaks obtained are associated with the cubic phase of the zinc blende structure, which is in good agreement with JCPDS data sheet No. 05-0566 for ZnS. Furthermore, for CQD-modified ZnS, no characteristic peaks of the CQDs were observed, which may be due to their relatively low content in ZnS. The results clearly show that the presence of CQDs does not affect the structure of the ZnS NPs [28]. However, the XRD analysis results for the ZnO/CQDs and ZnS/CQDs nanocomposites show much higher intensities of the diffraction peaks in ZnO/CQDs compared to those in ZnS/CQDs, suggesting higher crystallinity in ZnO/CQDs.

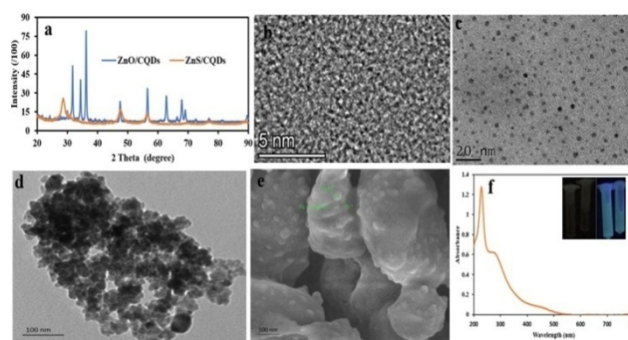


Figure 1. XRD spectra of ZnO/CQDs and ZnS/CQDs NPs (a). SEM images of CQDs (b). TEM image of CQDs (c) and ZnS/CQDs NPs (d). SEM image of ZnO/CQDs NPs (e) and UV-visible absorption spectrum of CQDs (f)

In addition, scanning electron microscopy (SEM) analysis of the CQDs and ZnO/CQD NPs, and transmission electron microscopy (TEM) analysis of the CQDs and the ZnS/CQD NPs were performed. The results show that the synthesized carbon quantum dots (CQDs) consist of nanoscale particles in a spherical shape with a uniform distribution and an average diameter of approximately 4 nm (Figures 1b and 1c). This clearly indicates that the carbon quantum dots were successfully synthesized using the described method.

The results obtained (Figure 1d) for the ZnO/CQDs NPs show that the sample consists of nanometer-sized particles in a compact, polydisperse form with a fairly diverse size distribution. A few agglomerates of varying sizes are observed, with an average diameter of approximately 30 nm. This finding was reported by Tabrizi [29]. In general, compact structures are formed through the aggregation of nanoparticles. This suggests that the presence of CQDs enhances the aggregation of ZnO NPs. It should be noted that these different forms of ZnO/CQDs are of great importance for their photocatalytic applications.

As for ZnS/CQDs, the TEM results (Figure 1e) show polydisperse particles with a compact hexagonal shape. The average particle diameter is approximately 39 nm. These different sizes could also play an important role in their photocatalytic application. It is important to note that the crystals of the synthesized ZnO/CQDs NPs are smaller in size (30 nm) compared to those of the ZnS/CQDs NPs. This difference could play a decisive role in catalytic applications, favoring the ZnO/CQDs NPs.

3.2. Measurement of the CQDs Absorption Spectrum

The absorption of the CQDs was measured using a UV-Vis spectrophotometer (Figure 1f). The results show two characteristic absorption peaks in the UV region: a strong peak at approximately 226 nm, which can be attributed to the $\pi-\pi^*$ transition of the C=C bonds, and a weaker peak at approximately 270 nm, which is attributed to the $n-\pi^*$ transition of the C-O or C-N bonds. These results suggest that the synthesized CQDs have end groups rich in carboxylic, carbonyl, amine, and alcohol functional groups [21]. Furthermore, when irradiated with a UV lamp at a wavelength of 365 nm, these particles emit green light, which may result from $n-\pi^*$ electronic transitions, thereby confirming the formation of the CQDs [30].

3.3. Reflectance Measurements of ZnO/CQDs and ZnS/CQDs

To evaluate the reflectance of ZnO/CQDs and ZnS/CQDs NPs, reflectance measurements were conducted in the UV-Vis range. The results obtained (Figure 2a) show that the reflectance of the two nanocomposites is similar, with reflectance values of 86% and 88%, respectively, for ZnO/CQDs and ZnS/CQDs NPs in the visible spectrum. The ZnO/CQDs exhibit slightly lower reflectance compared to the ZnS/CQDs in the visible spectrum. This lower reflectance of the ZnO/CQDs nanocomposites relative to that of the ZnS/CQDs could be attributed to the chemical bonds between the ZnO NPs and the CQDs [24]. Therefore, the photocatalytic activity of ZnO/CQDs could be better than that of ZnS/CQDs under visible light irradiation.

3.4. Photocatalytic Degradation of MB Using ZnO/CQDs and ZnS/CQDs

To determine the time required for the complete degradation of MB under optimal conditions ($C_0 = 10$ mg/L at pH 11 and $m = 150$ mg) using 150 mg of ZnO/CQDs or ZnS/CQDs NPs as photocatalysts, its degradation was studied in the presence of sunlight between 11:00 a.m. and 3:00 p.m at 28°C (Figure 2b). As presented in this figure, the MB absorption peak intensity in the presence of ZnO/CQDs NPs decreases more rapidly during the first 20 minutes of irradiation (Figures 2c). After 80 minutes of irradiation, the peak is no longer detectable, whereas in the presence of ZnO/CQDs. In contrast, the same peak decreases rapidly within 20 minutes and disappears completely within 120 minutes (Figure 2d). This disappearance of the peak would indicate the complete degradation of MB by ZnO/CQDs and ZnS/CQDs. This would suggest that ZnO/CQDs and ZnS/CQDs are excellent photocatalysts for the degradation of MB in an aqueous medium, as previously noted by Huiqin [27,28]. However, ZnO/CQDs are the best photocatalysts because their degradation time is shorter than that of ZnS/CQDs

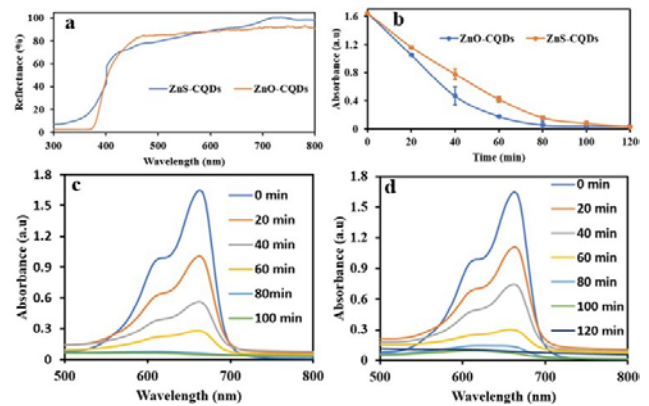


Figure 2. Reflectance spectrum of ZnO/CQDs and ZnS/CQDs NPs (a). Degradation of MB under solar irradiation using ZnO/CQDs and ZnS/CQDs (b). UV-vis spectra at different times of MB using ZnO/CQDs (c) and ZnS/CQDs (d)

3.5. Kinetic Study of MB Degradation

A study of the MB degradation rate in the presence of ZnO/CQDs and ZnS/CQDs nanostructures was conducted to evaluate the degradation kinetics of MB and compare their performance. The values of the kinetic constants for the photodegradation of the MB dye were determined using Equations 2 and 3 for pseudo-first-order (Figure 3a) and pseudo-second-order (Figure 3b) reactions, for ZnO/CQDs and ZnS/CQDs NPs, respectively.

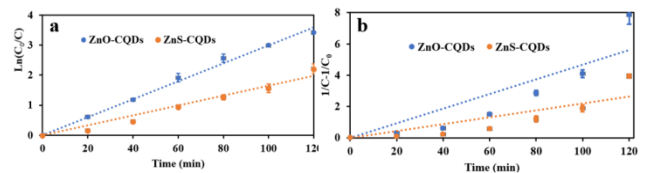


Figure 3. First-order (a) and second-order (b) kinetic models for the degradation of MB at a concentration of 10 mg/L using ZnO/CQDs and ZnS/CQDs

The results for the rate constants k_1 and k_2 and the various coefficients of determination R^2 are presented in Table 2.

Table 2. Kinetic parameters of MB degradation

Initial Concentration of MB (mg/L)	Pseudo-first order				Pseudo-second order			
	$k_1(\text{min}^{-1})$		R^2		$K_2(\text{min}^{-1})$		R^2	
	a	b	a	b	a	b	a	b
10	0,0299	0,0165	0,9977	0,9873	0,0468	0,0221	0,8895	0,8458

a: ZnO/CQDs; b: ZnS/CQDs

The R^2 values are clearly closer to 1 for the pseudo-first-order model than for the pseudo-second-order model, where R^2 is relatively far from 1 in the presence of both photocatalysts (ZnO/CQDs and ZnS/CQDs). Consequently, the photocatalytic degradation of MB would follow apparent first-order kinetics. The k_1 values for ZnO/CQDs are twice as high as those obtained with ZnS/CQDs NPs, confirming the excellent photocatalytic

properties of ZnO/CQDs compared to ZnS/CQDs NPs. Furthermore, the k_2 values for ZnO/CQDs are also twice as high as those obtained with ZnO NPs, which also reflects the high performance of the photocatalytic properties of ZnO/CQDs compared to ZnS/CQDs.

3.6. Study on the Reuse of Synthesized Photocatalysts

Catalyst regeneration is an important economic factor in wastewater treatment. Indeed, after obtaining the best degradation results, recycling tests were conducted using the catalysts with the highest performance to verify the stability of the photocatalysts. To this end, repeatability experiments were conducted over four continuous cycles. After each test, the catalysts (ZnO/CQDs and ZnS/CQDs NPs) were recovered and rinsed several times with distilled water and ethanol, then dried at 100 °C for 24 h before the start of the next cycle, while maintaining the same conditions for the concentration and initial volume of the waste water. The results are shown in figure 4.

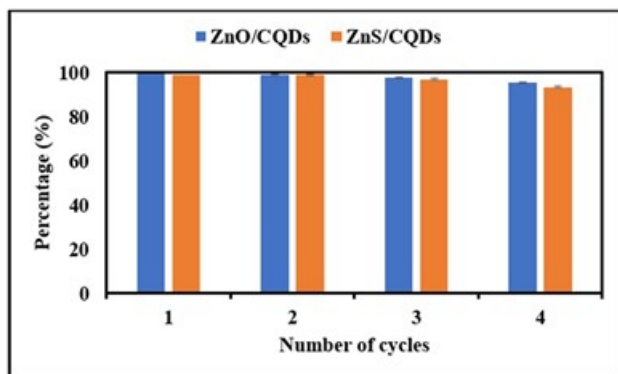


Figure 4. Study of the stability of a) ZnO/CQDs NPs and b) ZnS/CQDs in MB degradation

The results show good photocatalytic performance over the four cycles for both photocatalysts, with a slight decrease in the removal percentage. It should be noted that this decrease in removal percentage is approximately 5% (i.e., from 100% to 95%) for ZnO/CQDs and 7% (i.e., from 100% to 93%) for ZnS/CQDs nanoparticles used as photocatalysts. This decrease in catalytic activity is due to the leaching of the photocatalyst into the solution as a result of its small size [27]. This difference in mass loss between the two photocatalysts after the four cycles suggests that ZnO/CQDs is more stable than ZnS/CQDs NPs.

4. Conclusion

The objective of this study was to synthesize ZnO and ZnS based nanomaterials combined with carbon quantum dots (CQDs), and then to compare their photocatalytic performance in the degradation of organic pollutants. To this end, ZnO and ZnS NPs were synthesized using the precipitation method and then combined with CQDs by simple mixing to obtain ZnO/CQDs and ZnS/CQDs nanocomposites, followed by the application of these nanomaterials for the degradation of MB in a synthetic solution.

XRD revealed that the presence of CQDs does not affect the crystalline structure of the synthesized ZnO and ZnS NPs. Further characterization by SEM and TEM revealed a porous structure resulting from the aggregation of nanoparticles with average diameters of approximately 4 nm, 30 nm, and 39 nm for the CQDs, ZnO/CQDs, and ZnS/CQDs, respectively. Furthermore, the study of reflectance in the visible spectrum shows that the presence of CQDs results in 86% and 88% reflection of sunlight in ZnO/CQDs and ZnS/CQDs NPs, respectively.

Photocatalytic degradation tests of MB reveal that the ZnO/CQDs composite exhibited the highest efficiency, degrading 100% of MB in 80 minutes, compared to ZnS/CQDs NPs, which achieved this performance in 120 minutes. These results suggest that CQDs could be used to enhance the photocatalytic properties of semiconductors for the degradation of organic pollutants in aqueous solutions under solar irradiation.

Conflicts of Interest

The authors declare no conflicts of interest regarding this article.

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