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Facile Synthesis and Comprehensive Characterization of Zn-Doped CuS–TiO_2 Heterostructure for Enhanced Photocatalytic Degradation of Organic Dyes under Solar Irradiation

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ABSTRACT

A Zn-doped CuS–TiO_2 hetero-structured photocatalyst was synthesized via a simple hydrothermal approach to achieve efficient degradation of organic dyes under visible light. Structural and optical characterizations, including XRD, FTIR, UV–vis spectroscopy, SEM, and EDX, confirmed the successful formation of the heterojunction and incorporation of the dopant without secondary phases. The composite exhibited a broad absorption spectrum extending into the visible range and a high specific surface area of 82.63 m^2/g with mesoporous features, as evidenced by BET analysis. SEM revealed a heterogeneous morphology composed of nanosheets, rods, and spherical aggregates that provide enhanced surface roughness and active sites. Photocatalytic performance was evaluated using methylene blue as a model pollutant under natural sunlight, where the doped composite (30 mg) achieved nearly 98% degradation within 60 minutes. The improved activity is attributed to the synergistic effect between CuS and TiO_2, Zn-induced band gap modulation, and more effective charge separation across the heterojunction interface. Furthermore, the catalyst retained its performance over six reuse cycles, indicating good stability and reusability. These results suggest that Zn-doped CuS–TiO_2 is a promising photocatalyst for solar-driven wastewater treatment applications.

1. Introduction

Synthetic dyes are among the most pervasive and persistent pollutants discharged into the environment due to widespread use in industrial processes. They are extensively employed in textiles, paper and pulp, cosmetics, pharmaceuticals, food coloring, paints, ceramics, leather tanning, and even in photoelectrochemical devices and hair dyeing formulations [1-4]. Once released, these dyes not only deteriorate the aesthetic quality of water bodies but also pose significant ecological and health risks. Many dyes are known to be carcinogenic, mutagenic, and toxic to aquatic organisms [5]. Upon entering biological systems, they can disrupt metabolic activities, causing adverse effects such as methemoglobinemia, neurological disturbances, hypertension, skin irritation, and other health complications [6]. Owing to their ability to penetrate biological membranes and persist under varied redox and pH conditions, dyes in water can exist in multiple forms-acidic, basic, reactive, or disperse—further complicating their removal [7-8].

The increasing presence of dyes in surface water, groundwater, and soils represents a severe environmental threat. Therefore, developing efficient, economical, and sustainable dye removal technologies is imperative. Among various treatment methods, photocatalysis has emerged as a green and promising approach due to its ability to harness light energy to degrade pollutants without generating harmful byproducts [9,10]. Semiconductor-based photocatalysts such as TiO_2, ZnO, Fe_2O_3, CdS, ZnS, and Cu_2O—have shown significant potential for dye degradation. These materials, upon UV or visible light exposure, generate electron–hole pairs that initiate redox reactions, leading to the formation of reactive oxygen species (ROS) like $O_2^{\bullet-}$, H_2O_2, and $OH^{\bullet}$. These ROS are primarily responsible for breaking down complex organic molecules in water. Among them, TiO_2 (especially in its anatase form) is widely studied due to its high photoactivity, chemical stability, non-toxicity, and low cost [11,12]. With a band gap of ~3.2 eV, TiO_2 can efficiently generate charge carriers under UV light, and its redox potential aligns well with the oxidation of water and organic pollutants. Moreover, TiO_2 is self-regenerating and reusable, making it attractive for long-term environmental applications [13].

However, the practical application of pure TiO_2 is limited by its wide band gap and rapid recombination of charge carriers. To address these limitations, researchers have explored various modifications such as doping and heterojunction formation. For instance, coupling TiO_2 with narrow-bandgap semiconductors like CuS can extend its light absorption into the visible range and enhance charge separation efficiency [14]. CuS, a p-type semiconductor with a band gap of approximately 2.1 eV, exhibits excellent photostability and visible-light response. It has been explored in various fields including photocatalysis, solar energy harvesting, and batteries [13,15]. Several studies have demonstrated that CuS–TiO_2 composites significantly enhance photocatalytic performance in the degradation of pollutants such as methylene blue, 4-chlorophenol, oxytetracycline, and Cr(VI) ions [16]. These improvements are typically attributed to the formation of heterojunctions that facilitate directional charge transfer and suppress recombination. Additionally, studies have demonstrated that incorporating plasmonic metals or forming ternary heterojunctions can further enhance activity toward the degradation of various contaminants such as methylene blue, 4-chlorophenol, oxytetracycline, hexavalent chromium, and toluene [17-19].

Building upon these developments, this study introduces Zn doping into the CuS–TiO_2 heterostructure as a strategic approach to further enhance its photocatalytic performance. Zinc (Zn^{2+}), with an ionic radius comparable to Ti^{4+}, can partially substitute into the TiO_2 lattice, leading to the creation of oxygen vacancies and lattice distortions. These defects serve as shallow trap sites that suppress electron–hole recombination by prolonging charge carrier lifetimes [20,21]. Furthermore, Zn doping introduces localized electronic states within the bandgap, effectively narrowing the bandgap and extending the light absorption range into the visible spectrum. Zn^{2+} ions may also improve interfacial charge transfer between CuS and TiO_2 by serving as electron mediators, promoting the separation and migration of charge carriers across the heterojunction. Additionally, Zn incorporation enhances the surface acidity and active site density, which can improve dye adsorption and catalytic degradation of dye [22-24].

Although CuS–TiO_2 composites have been studied before, the specific role of Zn doping in enhancing their photocatalytic performance hasn't been explored yet. This study is the first to look into how incorporating Zn influences the overall efficiency and charge dynamics of the CuS–TiO_2 heterostructure [22-25]. Thus, the synergistic combination of CuS's visible light response, TiO_2's structural stability, and Zn's dopant effect is

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expected to yield a highly efficient, visible-light-active photocatalyst with superior photodegradation capability and reusability. In this work, we report the facile hydrothermal synthesis of Zn-doped CuS–TiO_2 nanocomposites and evaluate their structural, optical, and photocatalytic properties using methylene blue as a model pollutant under natural sunlight irradiation [26,27].

2. Experimental Methods

2.1 Preparation of Zn-Doped CuS-TiO_2 Heterostructure

To synthesize the Zn-doped CuS–TiO_2 heterostructure, 2 mmol of copper (II) chloride ($CuCl_2 \cdot 2H_2O$) was dissolved in 5 mL of ammonia, followed by the addition of 70 mL of deionized water. The solution was stirred for 5 minutes before adding 20 mL of thiourea, after which stirring continued for 30 minutes. In a separate beaker, titanium isopropoxide was added to a mixture of distilled water and KOH and stirred for 30 minutes to ensure homogeneity. Zinc precursor (1%) was added to it and stirred for 30 minutes to achieve homogenous mixing [27]. Both solutions were then transferred into a Teflon-lined stainless-steel autoclave and subjected to hydrothermal treatment at 180 °C for 24 hours. The obtained precipitate was washed thoroughly with deionized water and ethanol, centrifuged, and oven-dried at 60 °C. For comparison, pure TiO_2 and CuS and heterostructures were prepared under identical conditions [28,29].

2.2 Characterization

A Bruker D-8 diffractometer was used to examine the crystal structure and phase identification of the produced MoS_2 nanostructure material using powder X-ray diffraction (XRD), scanning at a rate of 0.3° per second over a 2θ range of 10–80°. This method assisted in verifying the material's phase purity and crystallinity. A Bruker Hyperion spectrometer was used to perform fourier transform infrared spectroscopy (FTIR) in the 500–4000 cm^{-1} range in order to identify functional groups and molecular interactions. With the use of a Shimadzu spectrophotometer, the optical properties of the material were investigated using uv-visible absorbance spectroscopy in the 300–800 nm range, which revealed information on its light absorption qualities. Field emission scanning electron microscopy (FE-SEM) and energy dispersive X-ray spectroscopy (EDX) with a TESCAN Magna microscope were used to analyze the materials' surface morphology and elemental makeup. A thorough examination of particle size, shape, and elemental distribution was made possible by these methods.

2.3 Photocatalytic Degradation of Methylene Blue

The photocatalytic efficiency of the synthesized Zn-doped CuS-TiO_2 heterostructure would be rigorously evaluated through the degradation of methylene blue (MB), under solar irradiation. The experimental setup would involve dispersing a known quantity of the prepared heterostructure catalyst in an aqueous solution containing a specific concentration of MB. Prior to irradiation, the suspension would be stirred in the dark for a period to establish adsorption-desorption equilibrium between the catalyst and the dye. Subsequently, the mixture would be exposed to the light source, and at predetermined time intervals, aliquots of the solution would be withdrawn, centrifuged to remove the catalyst, and analyzed using UV-vis spectroscopy to monitor the decrease in MB concentration by observing the characteristic absorption peak [24-28].

3. Results and Discussion

3.1 Morphological Characterization

The surface morphology of the synthesized CuS-TiO_2 heterostructure was investigated using SEM at a magnification of 50,000×. Scanning electron microscopic image of the prepared Zn-doped CuS-TiO_2 heterostructure is given in Fig. 1. The micrograph reveals a dense and compact arrangement of nanostructured particles with non-uniform grain sizes ranging from approximately 30 to 80 nm. The particles appear to be aggregated, forming a porous network that is favorable for catalytic applications. The formation of a heterostructured composite is evident from the irregular, rough surface morphology, which indicates the successful integration of CuS nanoparticles with the TiO_2 matrix. This coupling likely enhances surface roughness and increases the number of active sites, which are critical for photocatalytic performance. The high surface area and porous structure suggest that the heterojunction provides improved interfacial contact between CuS and TiO_2, facilitating efficient charge transfer and separation of photogenerated electron-hole pairs.

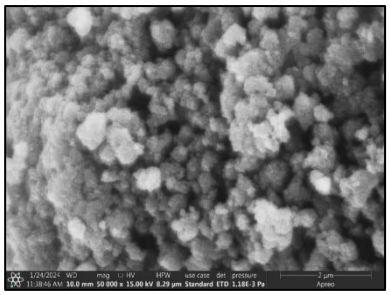
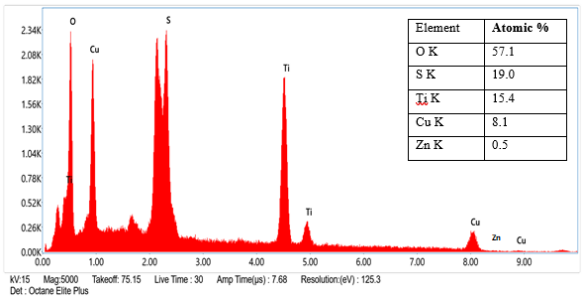


Fig. 1 Scanning electron microscopic image of the prepared Zn-doped CuS-TiO_2 heterostructure

3.2 Structural Characterization

EDX spectra of the prepared heterostructure along with the composition is shown in Fig. 2. Atomic percentage of different elements suggests that the prepared compound contains only Cu, S, Ti and O elements. It confirms that there is no impurity in the compound produced. The peaks appearing at around ~0.5 keV corresponds to oxygen, copper is appearing at ~0.9 keV, ~8.0 keV, and ~9.0 keV, sulfur near ~2.3 keV, titanium around ~4.5 keV and ~4.9 keV and zinc around ~8.6 keV. This spectrum indicates the presence of Cu, S, Ti, O and Zn in the sample, which is consistent with Zn-doped CuS@TiO_2 composite nanomaterial. As shown in the EDX spectrum the ratio of CuS to TiO_2 is close to the atomic percentage taken for both.



Element	Atomic %
O K	57.1
S K	19.0
Ti K	15.4
Cu K	8.1
Zn K	0.5

Fig. 2 EDX spectrum and elemental composition of the of prepared Zn-doped CuS-TiO_2 heterostructure

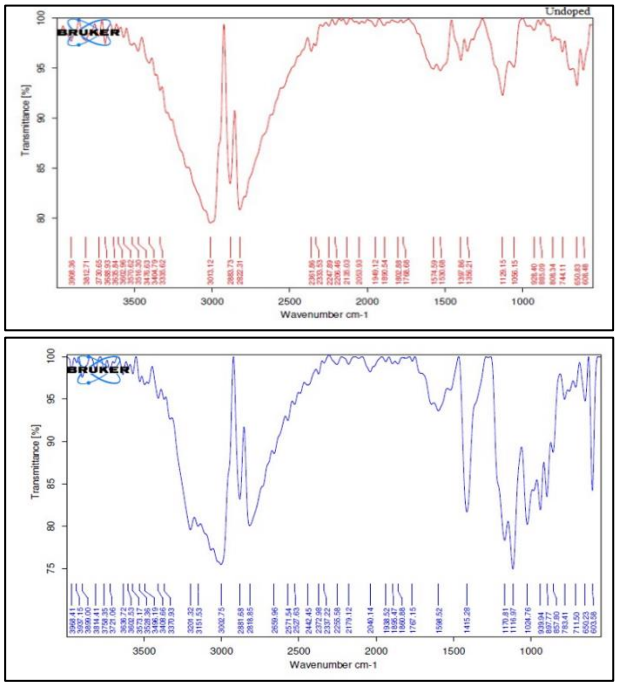


Fig. 3 FTIR spectra of undoped CuS-TiO_2 and Zn-doped CuS-TiO_2

The FTIR spectra of pure CuS, pure TiO_2, CuS–TiO_2 composite, and Zn-doped CuS–TiO_2 are shown in Fig. 3. In the vibrational spectrum of pure CuS, a characteristic peak is observed around 620 cm^{-1}, which corresponds to the stretching vibrations of Cu–S bonds. In the case of pure TiO_2, a peak appearing at 664 cm^{-1} is attributed to the O–Ti–O bending vibrations, while another peak at 1405 cm^{-1} corresponds to the Ti–O–Ti stretching mode. Upon the formation of the CuS–TiO_2 composite, notable

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changes are observed in both the peak positions and their intensities. The Cu–S stretching peak at 620 cm^{-1} in pure CuS shifts to 608 cm^{-1} in the composite, indicating a change in the local bonding environment. Similarly, the O–Ti–O vibration shifts from 664 cm^{-1} to 650 cm^{-1}, and the Ti–O–Ti peak shifts from 1405 cm^{-1} to 1397 cm^{-1}. These shifts confirm a chemical interaction between CuS and TiO_2 during composite formation, suggesting the successful formation of a heterostructure. The FTIR spectrum of doped CuS–TiO_2 further supports the presence of structural and electronic modifications introduced by the dopant, as reflected in additional changes in the vibrational modes.

The prepared Zn-doped CuS-TiO_2 heterostructure was characterized using X-ray diffraction (XRD), and the corresponding pattern is shown in Fig. 4. The diffraction peaks observed in the XRD pattern match well with the standard data for CuS and TiO_2, as referenced by JCPDS File No. 06-0664 for CuS and 01-071-1167 for TiO_2. In the CuS–TiO_2 composite, all the identified peaks can be attributed to the respective phases of CuS and TiO_2, indicating the successful formation of the heterostructure without any secondary or impurity phases. Specifically, the peaks observed at 25.5537° and 48.2140°, corresponding to the (101) and (200) crystallographic planes, confirm the presence of TiO_2 in the anatase phase. Additionally, peaks appearing at 29.5158° and 32.9370°, indexed to the (102) and (103) planes respectively, are characteristic of the hexagonal covellite phase of CuS. The crystallite size measured for CuS and TiO_2 measured using Scherrer's equation are 14.75 nm and 8.74 nm respectively. These results collectively confirm the coexistence of anatase TiO_2 and hexagonal CuS within the composite. No specific peaks corresponding to the formation of any compound of zinc has been found. Furthermore, the sharp and well-defined peaks suggest good crystallinity and the successful integration of both components into the heterostructured material.

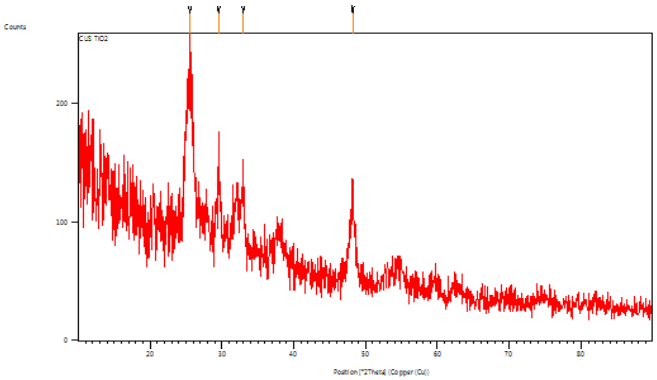

Fig. 4 XRD pattern of Zn-doped CuS-TiO_2 heterostructure

The UV–visible absorption spectra of the undoped and Zn-doped CuS–TiO_2 composites are presented in Fig. 5, within the wavelength range of 250–800 nm. Both the undoped and doped samples exhibit extended absorption toward longer wavelengths, indicating enhanced visible light harvesting capabilities. This red-shifted absorption is attributed to free carrier intra band absorption, which becomes more pronounced upon doping. In the spectra, the maximum absorption for TiO_2 is observed at approximately 330 nm, corresponding to its intrinsic band gap transition, while a slight shift to 327 nm is noted for the doped composite. Similarly, CuS shows a broad absorption band in the visible region, with a characteristic peak around 462 nm, which shifts to 430 nm in the doped sample. These shifts and enhancements in absorption suggest strong interfacial interaction in the composite system and the successful incorporation of Zn dopant, which contributes to improved light utilization in the visible region-favorable for enhanced photocatalytic performance.

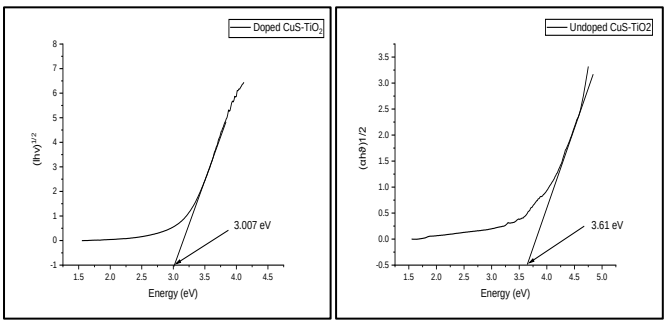

Fig. 5 UV-Vis spectra of undoped CuS-TiO_2 heterostructure and Zn doped CuS-TiO_2 heterostructure

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The Brunauer–Emmett–Teller (BET) specific surface area of the synthesized CuS–TiO_2 nanocomposite was evaluated using nitrogen adsorption–desorption isotherms. The analysis yielded a high correlation coefficient (R^2) of 0.999218, indicating excellent linearity and reliability of the BET plot. The measured The Brunauer–Emmett–Teller (BET) specific surface area of the CuS–TiO_2 was found to be 82.63 m^2/g, which is notably high and can be attributed to the small crystallite size and the pronounced aggregation of nanoparticles within the composite. This suggests that the nanostructured morphology plays a crucial role in enhancing the surface area [29-31].

Additionally, the material exhibited a total pore volume of 0.19166 cm^3/g and an average pore radius of 3.88 nm, indicating the presence of mesoporous structures. These characteristics reflect a well-developed porous network, which is beneficial for improving photocatalytic performance due to increased active sites and enhanced diffusion of reactant molecules. The significantly increased specific surface area and porosity of the CuS–TiO_2 composite are likely the result of a well-controlled synthesis strategy, which promotes uniform nucleation and growth of nanocrystallites. Such morphological control contributes not only to improved structural stability but also to enhanced light absorption and charge separation—factors that are essential for high-efficiency photocatalytic applications.

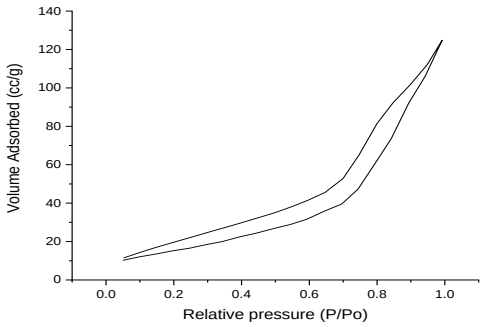

Fig. 6 Nitrogen adsorption–desorption isotherms and BJH pore size distribution of Zn-doped CuS–TiO_2 nanocomposite

3.3 Photocatalytic Degradation of Dye

Photocatalytic degradation efficiency of the nanomaterial was studied by the change in the concentration of the dye methylene blue with the addition of the composite to the dye solution. It was well established that when aqueous Zn doped CuS-TiO_2 suspension was irradiated with light energy greater than its band gap energy, the conduction band electrons (e^-) and valence band holes (h^+) were generated. The photogenerated electrons could reduce the dye or react with electron acceptors such as O_2 adsorbed on the Zn doped CuS-TiO_2 When light interacts with a photocatalyst like Zn-doped CuS-TiO_2, it kickstarts a chain of reactions that effectively break down organic molecules, such as dyes. This process begins with the light exciting electrons within the photocatalyst, causing them to jump to a higher energy level. These energized electrons can then react with oxygen present on the surface or dissolved in water, forming superoxide radical anions ($O_2\bullet^-$). Simultaneously, holes are created where the electrons originated. These positively charged holes have two main pathways: they can directly oxidize the organic dye molecule, breaking it down into a positively charged fragment (R^+), or they can react with hydroxide ions (OH^-) or water (H_2O) to generate highly reactive hydroxyl radicals (OH•). Together, these potent species superoxide radical anions, hydroxyl radicals, and other peroxide radicals act as powerful oxidizing agents, collaborating to efficiently decompose and degrade the organic dye molecules right on the semiconductor's surface. Essentially, the photocatalyst leverages light energy to produce these highly reactive oxygen species and directly oxidize the dye, leading to its degradation.

Zn-CuS-TiO_2 (hVB^+) + H_2O	→	Zn-CuS-TiO_2 + H^+ + $OH^\bullet$
Zn-CuS-TiO_2 + OH^-	→	Zn-CuS-TiO_2 + $OH^\bullet$
Zn-CuS-TiO_2 + O_2	→	Zn-CuS-TiO_2 + $O_2^{\bullet-}$
$O_2^{\bullet-}$ + H^+	→	$HO_2^\bullet$
Dye + $OH^\bullet$	→	degradation products
Dye + h VB^+	→	oxidation products
Dye + e CB^-	→	reduction products
Dye + $O_2^{\bullet-}$	→	degradation products

However, identifying the major contributor between ROS species is very critical. The generation of ROS and their environment in the oxidation of methylene blue primarily relies on the appropriate potential of the band edges inherent in a photocatalyst.

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To evaluate the photochemical degradation performance, a fixed amount of nanomaterial was added to a predetermined volume of dye solution. Prior to the experiment, the pH of the solution was adjusted to the desired value. The resulting suspension was then kept in the dark for 30 minutes to achieve adsorption–desorption equilibrium between the photocatalyst and the dye molecules. Following the dark equilibration period, the suspension was exposed to sunlight irradiation for time intervals of 30, 60, 90, 120, and 180 minutes, initiating the photocatalytic degradation process. At each time interval, aliquots were withdrawn from the reaction mixture, and the suspended photocatalyst particles were separated via centrifugation to obtain a clear supernatant [32]. The transparent solutions were analyzed using UV–Visible spectroscopy to monitor the degradation of dye by measuring the decrease in absorbance at the characteristic wavelength. The degradation performance under different experimental conditions is illustrated through the absorbance–time curves shown in Fig. 7.

This Fig. 7(a) shows the variation of the removal efficiency with time. On increasing the exposure time of the sun light the degradation efficiency of the composite increases due to more degradation. The graph explores how different catalyst dosages (10 mg, 20 mg and 30 mg) impact methylene blue degradation. On moving from 10 mg to 30 mg the efficiency increases with the catalyst dosage, indicating better photocatalytic activity. The highest efficiency (98 %) is observed at 30 mg after 1 hours. The variation in methylene blue degradation efficiency with different catalyst dosages is influenced by the availability of active sites for the reaction. Increasing the dosage to 10 mg to 30 mg increased photocatalytic activity due to an increase in the number of active sites, leading to increased degradation efficiency.

Also, graph (Fig. 7(b)) shows the degradation of methylene blue at different pH levels. The degradation of methylene blue varies with pH, with the highest removal efficiency observed at pH 10.54. pH influences the charge interactions between dye molecules and the adsorbent or catalyst, with neutral pH likely providing optimal conditions for effective degradation. In basic environments, repulsion between dye molecules and the catalyst reduces efficiency. Over time, hydroxyl radicals enhance degradation, though alkalinity may introduce side reactions that enhance efficiency compared to pH 7. In acidic conditions (pH 3), more dye molecules interact with active sites, but reduce the active site can't bind to dye molecule leading to decreased removal efficiency due to ongoing adsorption and degradation reactions, which often follow pseudo-first or pseudo-second-order kinetics.

The variation of degradation efficiency of the doped CuS-TiO_2 composite with time is shown in Fig. 7(c). Thus, removal efficiency increases with time as reaction kinetics and prolonged interactions facilitate greater methylene blue degradation [10-15, 25-30]. After around 60 minutes saturation stage appears and not much increase in the degradation efficiency was observed.

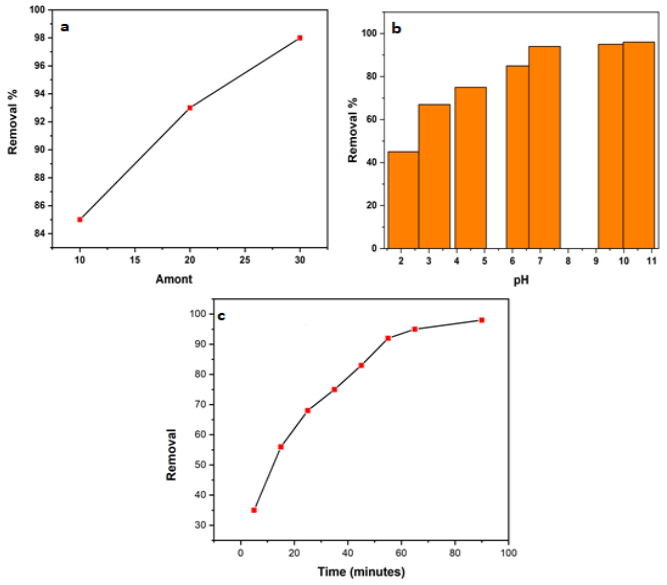

Fig. 7 Variation of removal efficiency of the prepared composite with amount of the composite (a); pH of the dye solution (b); time for the photocatalytic degradation (c)

The enhanced photocatalytic performance of the CuS-TiO_2 heterostructure is anticipated due to the synergistic effects between CuS and TiO_2. Specifically, CuS, with its narrow band gap, can absorb a significant portion of visible light, generating electron-hole pairs. These photogenerated electrons are then efficiently transferred to the conduction band of TiO_2, while holes remain in the valence band of CuS or are transferred to TiO_2, thereby promoting effective charge separation and significantly suppressing electron-hole recombination. This improved charge separation and extended light absorption range are crucial for generating more reactive oxygen species (such as ·OH and $\cdot O_2^-$), which are responsible for the oxidative degradation of methylene blue into less harmful compounds.

4. Conclusion

The study clearly highlights the enhanced photocatalytic behavior of CuS-TiO_2 in comparison to the commercially obtained TiO_2 for photocatalytic degradation of methylene blue. CuS-TiO_2 and Zn doped CuS-TiO_2 nanoparticles were effectively synthesized using the hydrothermal method. The SEM images revealed the spherical and regular shape of the nanoparticles and the atomic percentage of oxygen and sulfur were found to be 57.1% and 19% respectively using EDX spectrum. IR spectra showed the presence of CuS and TiO_2 nanoparticles and present the shifting of peaks in doped and undoped composite due to interaction of compound. The crystalline nature of the nanoparticles was confirmed and Debye – Scherrer formula was used to find the range of crystallite size, which was found to be 11 nm using by XRD. A depletion region is formed at the interface of Zn-doped CuS-TiO_2 composite and improved the photocatalytic performance of CuS-TiO_2 under the solar light irradiation. Under optimized conditions maximum degradation efficiency observed is around 98%.

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