

Synthesis and Photocatalytic Performance of CuV_2O_6 for Environmental Remediation

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Abstract

In this study, CuV_2O_6 nanoparticles were successfully synthesized via a sol–gel method and systematically investigated for their structural, morphological and photocatalytic properties. X-ray diffraction analysis confirmed the formation of a well-crystallized anorthic phase with high structural purity. Field emission scanning electron microscopy revealed a stacked, sheet-like morphology forming a hierarchical structure, which provides an increased surface area and abundant active sites for photocatalytic reactions. Energy-dispersive spectroscopy verified the presence of Cu, V and O elements with atomic percentages. The bandgap of 2.14 eV is calculate through UV-DRS analysis. The photocatalytic performance of the synthesized nanoparticles was evaluated through the degradation of methylene blue under UV irradiation, achieving a degradation efficiency MB by around 75% and BG by about 78%. These findings demonstrate that sol–gel synthesized CuV_2O_6 nanoparticles are promising candidates for environmental remediation, particularly for the removal of organic pollutants from wastewater.

Keywords: CuV_2O_6 nanoparticles, sheet like morphology, methylene blue

1. Introduction

Metal oxide semiconductors have emerged as one of the most promising classes of materials for photocatalytic applications owing to their excellent physicochemical stability, low cost, abundance and environmentally benign nature. These materials possess suitable band gap energies that enable the generation of electron–hole pairs under light irradiation, which subsequently participate in redox reactions for the degradation of organic and inorganic pollutants. Semiconductor photocatalysis has been widely applied in wastewater treatment, hydrogen production, carbon dioxide reduction and self-cleaning surfaces. The fundamental mechanism involves light-induced excitation of electrons from the valence band to the

conduction band, leaving behind holes that contribute to oxidative and reductive reactions at the catalyst surface [1,2]. Among various metal oxides, copper (II) oxide (CuO) has attracted considerable attention due to its narrow band gap (approximately 1.2–1.9 eV), which allows strong absorption in the visible light region. CuO is a p-type semiconductor with good electrical conductivity and high catalytic activity. Its ability to efficiently absorb visible light makes it particularly suitable for solar-driven photocatalytic applications. Furthermore, CuO exhibits good chemical stability and can generate reactive oxygen species such as hydroxyl radicals ($\bullet\text{OH}$) and superoxide radicals ($\text{O}_2^{\bullet-}$), which play a crucial role in the degradation of organic dyes and toxic contaminants in water. Because of these properties, CuO-based nanostructures have been extensively studied for environmental remediation and energy-related applications [2,3].

In recent years, mixed metal oxides such as copper vanadates have gained increasing interest due to their enhanced photocatalytic performance compared to single-component oxides. CuV_2O_6 , a copper vanadate semiconductor, exhibits favorable band alignment and improved charge carrier separation resulting from the synergistic interaction between Cu^{2+} and V^{5+} ions. The incorporation of vanadium introduces multiple oxidation states, which facilitate electron transport and reduce the recombination rate of photogenerated electron–hole pairs. This improved charge separation significantly enhances photocatalytic efficiency. Additionally, CuV_2O_6 possesses suitable redox potentials that enable effective degradation of organic pollutants under UV or visible light irradiation. Structural modifications and controlled morphologies, such as nanosheets, nanorods, or hierarchical assemblies, can further improve surface area and active site availability, thereby enhancing catalytic activity [4,5].

The synthesis method plays a crucial role in determining the structural, morphological, and optical properties of photocatalysts. Among various preparation techniques, the sol–gel method has been widely adopted for synthesizing metal oxide nanomaterials due to its simplicity, cost-effectiveness and excellent compositional control. The sol–gel process involves the transition of a system from a colloidal solution (sol) into a three-dimensional network (gel), allowing homogeneous mixing of metal precursors at the molecular level. This technique offers several advantages, including precise stoichiometric control, lower processing temperatures, improved purity and the ability to tailor particle size and morphology. These characteristics are particularly important for CuV_2O_6 synthesis, as uniform distribution of copper and vanadium ions enhances crystallinity and photocatalytic performance [6,7].

Therefore, the development of CuV_2O_6 photocatalysts, particularly through the sol–gel route, represents an effective strategy for designing efficient and stable materials for

environmental remediation. The synergistic electronic interaction in CuV_2O_6 combined with the controlled nanostructure obtained via sol–gel synthesis is expected to improve light absorption, promote charge carrier separation and ultimately enhance photocatalytic degradation efficiency.

2. Materials and method

2.1 Materials

All analytical reagent (AR) grade chemicals were obtained from Sigma-Aldrich and used as received without any additional purification. The precursor materials included copper(II) nitrate hexahydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), ammonium metavanadate (NH_4VO_3) and citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$).

2.2 Method

CuV_2O_6 nanoparticles were prepared via a sol–gel method. In a typical synthesis, 0.1 M copper(II) nitrate hexahydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and 0.2 M ammonium metavanadate (NH_4VO_3) were individually dissolved in 20 mL of distilled water. The two solutions were then combined and magnetically stirred for 15 minutes to ensure homogeneity. Afterward, 0.1 M citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) was added as a complexing agent. The resulting mixture was maintained at 60 °C under continuous stirring for 3 hours until a gel was formed. The formed gel was dried and subsequently calcined at 600 °C for 2 hours. The final brown-colored product was thoroughly ground into a fine powder and preserved for further characterization studies.

2.3 Photocatalyst experiment

The photocatalytic activity of CuV_2O_6 nanoparticles was assessed through the degradation of methylene blue (MB) and brilliant green (BG) dye under ultraviolet irradiation using an 8 W mercury UV lamp. A stock solution of MB (1×10^{-4} M) was prepared in distilled water. For experimental run, 100 mL of this dye solution was placed in a photoreactor, followed by the addition of 10 mg of the CuV_2O_6 catalyst.

Before exposure to UV light, the suspension was magnetically stirred in the dark for 30 minutes to attain adsorption–desorption equilibrium between the dye molecules and the catalyst surface. After establishing equilibrium, irradiation was initiated. At regular intervals, 5 mL aliquots were withdrawn from the reaction system to monitor the degradation process.

The concentration changes of MB and BG were analyzed using a UV–visible spectrophotometer over the wavelength range of 200–800 nm, by recording the absorbance at the dye's maximum absorption wavelength. The percentage degradation efficiency ($\eta\%$) was calculated according to the following expression:

$$\eta (\%) = \left(1 - \frac{C}{C_0}\right) \times 100 \quad (1)$$

where C_0 denotes the initial absorbance of the dye solution and C corresponds to the absorbance measured at a given irradiation time.

3. Results and Discussion

3.1 Structural analysis (XRD)

The X-ray diffraction (XRD) pattern of CuV_2O_6 was recorded to examine its phase purity and crystal structure. The observed diffraction (Fig. 1) peaks were well matched with the standard data of JCPDS card No. 30-0513, confirming the formation of single-phase monoclinic CuV_2O_6 without any detectable secondary phases or impurities. The 2θ and hkl plane values are 26.9° , 27.8° , 29.7° , 31.9° , 33.9° , 45° and 51.7° ($-1\ 1\ 0$), $(1\ 1\ 0)$, $(-3\ 0\ 1)$, $(1\ -1\ 1)$, $(1\ 1\ 1)$, $(-3\ 1\ 0)$, $(-3\ -1\ 1)$, $(0\ 0\ 3)$ and $(0\ 2\ 0)$). The close agreement between the experimental peak positions and the standard JCPDS data confirms the successful synthesis of highly crystalline CuV_2O_6 . The average crystallite size was estimated using the Scherrer equation [8],

$$D = k\lambda / (\beta \cos\theta) \quad (2)$$

where λ is the wavelength of X-ray radiation, β is the full width at half maximum (FWHM), and θ is the Bragg angle. The calculated crystallite size was found to be 44 nm, indicating the nanocrystalline nature of the prepared sample. The sharp and well-defined diffraction peaks further suggest good crystallinity of the material.

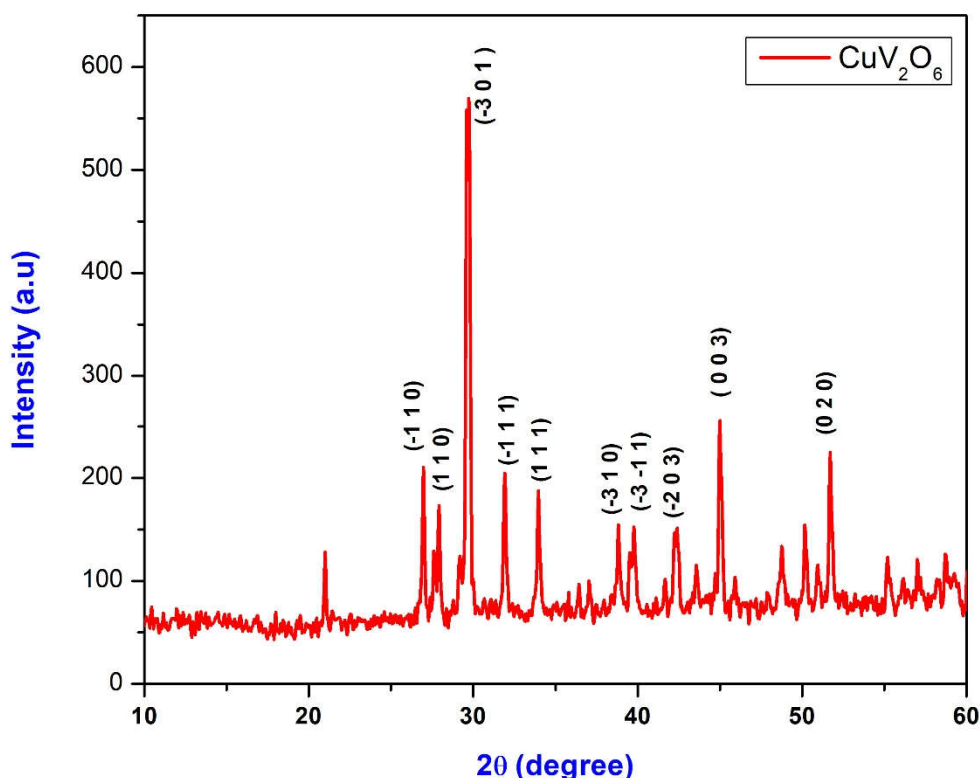


Figure 1. X-ray diffraction pattern of CuV_2O_6 calcined at 600 °C.

3.2 Surface Morphology and Elemental Composition Analysis

The surface morphology of the synthesized sample was examined by field emission scanning electron microscopy (FESEM). At low magnification of 1 μm (Fig. 2(a)), particles appear to be densely stacked, producing a layered and sheet-like morphology. At higher magnification of 100 nm (Fig. 2(b)), the stacked structures are more clearly distinguished, exhibiting well-arranged stacked sheet formations with a relatively compact and ordered architecture. The sheets are closely packed and interconnected, forming a hierarchical structure.

The layered arrangement enhances the effective surface area and provides abundant active sites for dye adsorption. Moreover, the inter-sheet spacing and interconnected framework facilitate efficient light harvesting and improve charge carrier transport, thereby reducing electron–hole recombination [9]. This structural configuration is expected to contribute significantly to the improved photocatalytic degradation performance of the material. EDAX analysis was performed to confirm the elemental composition of the prepared sample. The EDAX spectrum (Fig. 3(a)) indicates the exclusive presence of copper (Cu), vanadium (V), and oxygen (O) without detectable impurities. The atomic percentages shown in the insert table image (Fig.

3(b)) were found to be approximately 17% Cu, 36 % V and 45 % O. The uniform distribution of these elements further supports the successful synthesis of the targeted compound and its suitability for photocatalytic applications [10].

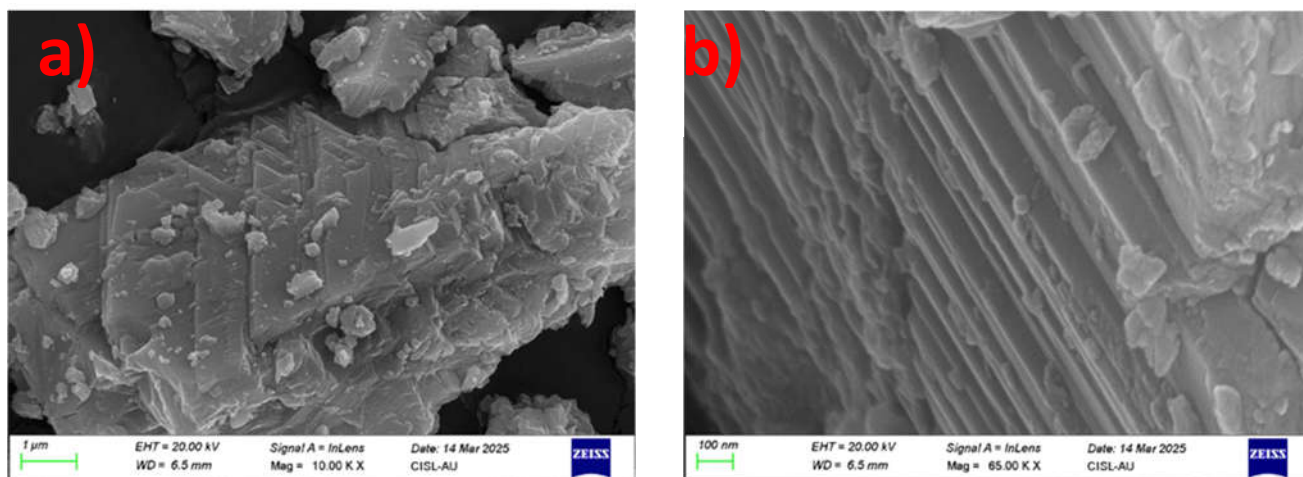


Figure 2. FESEM images of CuV_2O_6 nanoparticles (a) stacked micro-sized particles, (b) sheet-like morphology at 100 nm

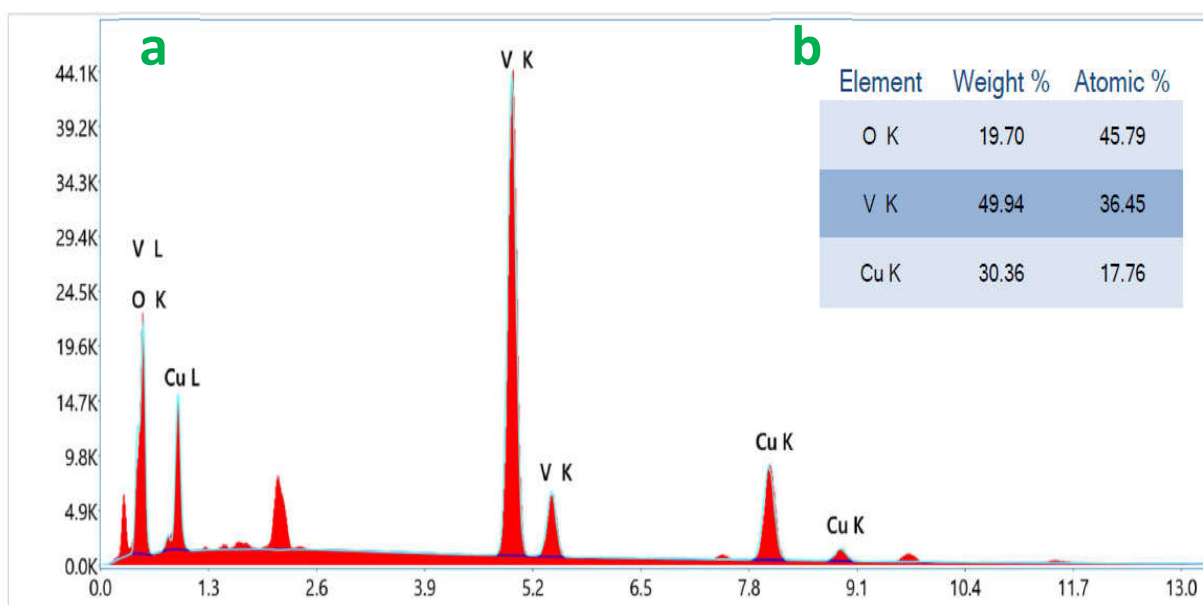


Figure 3. Elemental analysis of CuV_2O_6 nanoparticles (a) EDAX spectrum confirming the presence of Cu, V and O, and (b) corresponding atomic percentage

3.3 Optical analysis (UV-DRS)

UV-Diffuse Reflectance Spectroscopy (UV-DRS) was used to examine the optical characteristics of the formed CuV_2O_6 - 600 °C compound in order to assess its band gap energy.

The Tauc relation was used to determine CuV_2O_6 optical band gap energy (E_g):

$$\alpha h\nu = A(h\nu - E_g)^n \quad (3)$$

Where α is a constant, h is the photon energy, α is the absorption coefficient, and $n = 1/2$ denotes a straight permitted transition. The band gap was determined by projecting the linear part of the curve to the energy axis in a plot of $(\alpha h\nu)^2$ against photon energy ($h\nu$). The estimated band gap energy (Fig. 4) of CuV_2O_6 was determined to be about 2.14 eV which is advantageous for photocatalytic applications.

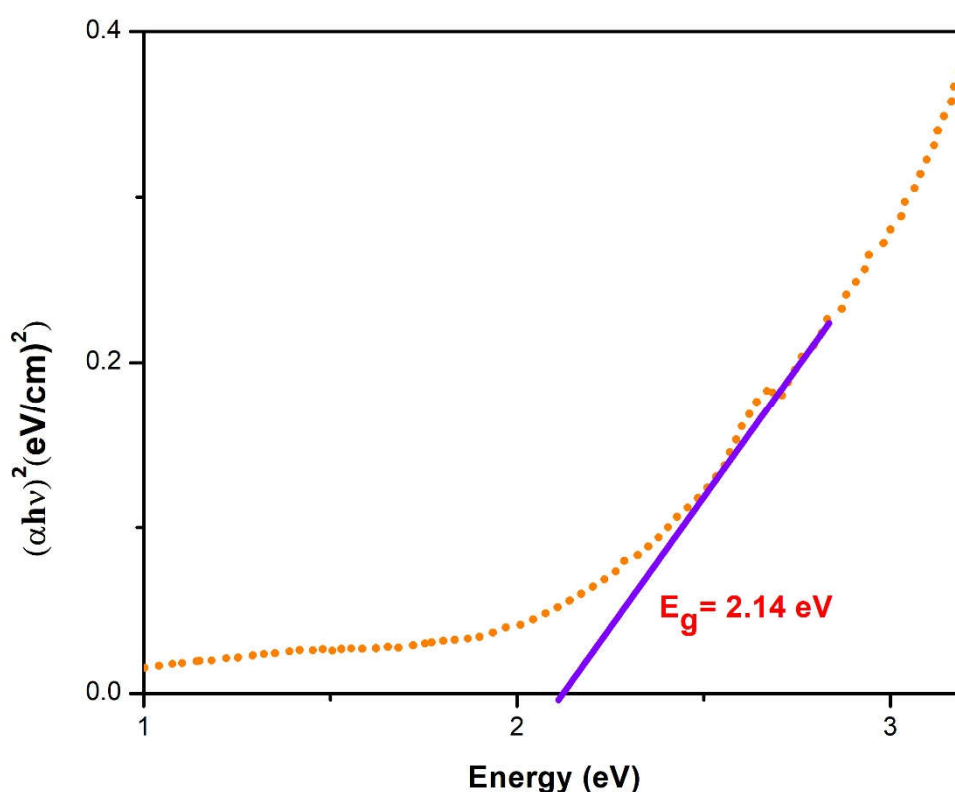


Figure 4 Tauc plot for optical band gap estimation of CuV_2O_6

4. Photocatalytic analysis

Methylene blue (MB) and brilliant green (BG) dyes were degraded under ultraviolet (UV) light irradiation in a photodegradation chamber in order to assess the photocatalytic

efficacy of the produced CuV_2O_6 photocatalyst. As shown in Fig. 5 & 6 (a) the UV–Vis absorption spectra shows the characteristic dye peaks with respect to absorbance vs wavelength. Before being exposed to UV light, 0.05 g of CuV_2O_6 was added to the dye solution and mixed in the dark to create equilibrium between adsorption and desorption. By detecting the decrease in the distinctive absorption peaks of MB and BG at 664 and 624 nm, respectively, UV–visible spectroscopy was used to track the degradation behaviour. The steady deterioration of the dye molecules was confirmed by a continual drop in the absorbance intensity at the appropriate wavelengths under UV irradiation [11]. Fig. 5 & 6 (b) shows changes in absorbance as a function of irradiation time. The CuV_2O_6 photocatalyst degraded MB by around 75% and BG by about 78% after the 60-minute interval. Fig. 5 & 6 (c) shows the degradation efficiency of MB and BG. Because of its molecular structure, which promotes greater adsorption on the CuV_2O_6 surface and improved interaction with reactive oxygen species produced during the photocatalytic process, BG has slightly higher degradation efficiency [12].

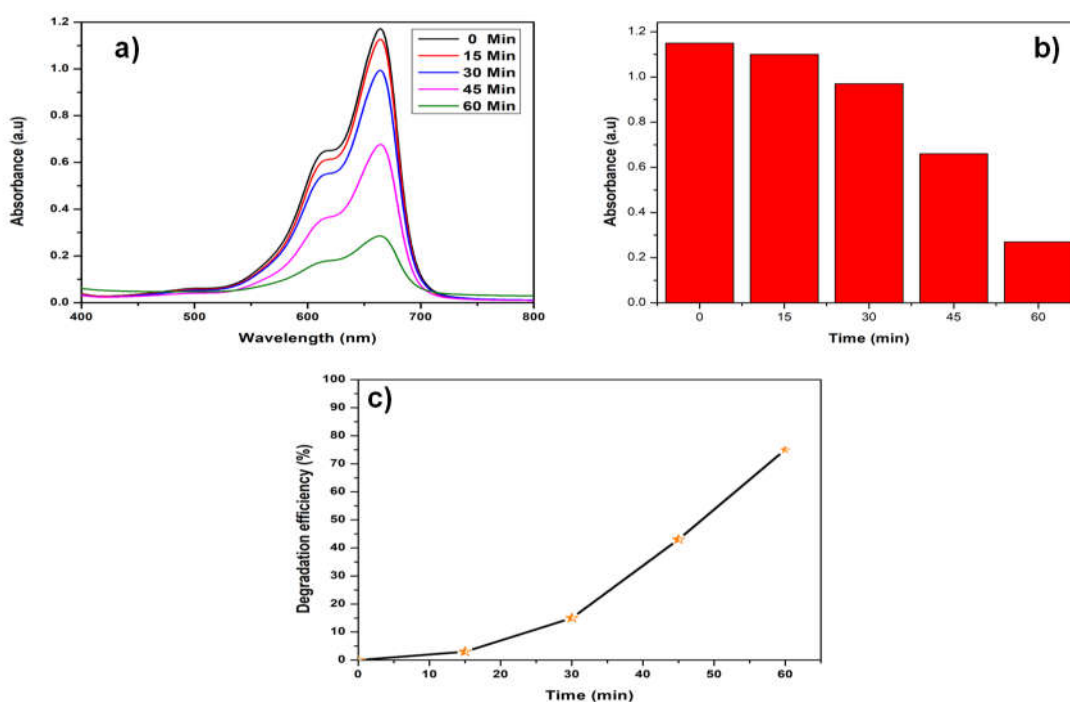


Figure. 5 (a) UV–visible absorption spectra recorded at different irradiation intervals, (b) change in absorbance intensity with respect to exposure time, and (c) corresponding degradation.

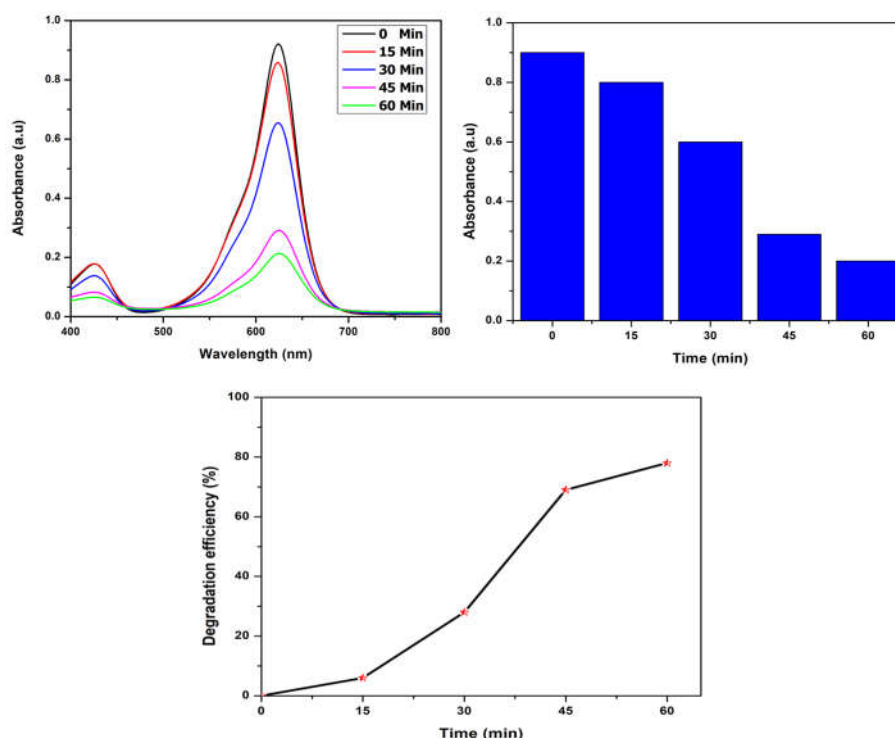


Figure.6 (a) UV–visible absorption spectra recorded at different irradiation intervals, (b) change in absorbance intensity with respect to exposure time, and (c) corresponding degradation.

Conclusion

In this study, CuV_2O_6 nanoparticles were successfully synthesized via the sol–gel method and characterized for their structural, morphological and photocatalytic properties. XRD analysis confirmed the formation of a well-crystallized anorthic phase, indicating high structural purity. FESEM images revealed that the material consists of stacked particles with a sheet-like morphology, providing a hierarchical structure that enhances surface area and active sites for photocatalytic reactions. EDAX analysis confirmed the presence of Cu, V and O with atomic percentages of the expected stoichiometry of the compound. Photocatalytic studies using methylene blue and brilliant green as a dye, demonstrates the CuV_2O_6 nanoparticles exhibit significant activity under UV irradiation, achieving a degradation efficiency MB by around 75% and BG by about 78%. The combination of favourable morphology, effective charge separation and appropriate electronic structure contributed to this enhanced performance. The results suggest that sol–gel synthesized CuV_2O_6 is a promising photocatalyst for environmental remediation applications, particularly in the degradation of organic pollutants.

Author contribution

NKS contributed to conceptualization, Resources, Writing, designing and editing original manuscript; SA contributed to writing and editing of the manuscript. SG contributed to investigation, methodology and supervision. All authors read and approved the manuscript.

Funding

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

Data availability

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Acknowledgment

The corresponding author gratefully acknowledges the Head of the Department of Physics and the Centralized Instrumentation and Service Laboratory (CISL) at Annamalai University, Annamalai Nagar, Tamil Nadu, India, for providing access to DST-FIST and RUSA supported instrumental and analytical facilities essential for this research.

Declarations**Ethics approval and consent to participate**

Not applicable.

Consent for publication

All authors agree to submit the manuscript.

Competing interests

The authors declare no competing interests.

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