

RESEARCH PAPER

Ag₂O/Zn_{1.8}Cd_{0.2}SnO₄ Nanocomposite as a Highly Efficient Visible-light Photocatalyst for Degradation of Methyl Orange

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ABSTRACT

A silver oxide (Ag₂O) based nanocomposite (Ag₂O/Zn_{1.8}Cd_{0.2}SnO₄) was synthesized as an efficient photocatalyst operating under visible light irradiation. A facile hydrothermal route was chosen to prepare the nanocomposite. Determining the optical band gap (2.59 eV) established that the nanocomposite has the capability as a visible-light photocatalyst. The photocatalytic activity was tested for degradation of methyl orange (MO) under 150 min illumination by a visible light source. More than 93% of MO was removed using 0.05 g of the nanocomposite. To further enhance performance, the effects of solution pH and H₂O₂ concentration were investigated. The MO degradation efficiency increased to around 95% and 98% by adjusting the pH to 3 and addition of 1 mL of 0.01 mM of H₂O₂, respectively. The excellent photocatalytic activity of the nanocomposite is truly attributed to the retarded recombination of the photo-generated charge carriers, as the result of the facilitated electronic transitions across the energy levels of the Ag₂O and Zn_{1.8}Cd_{0.2}SnO₄. Furthermore, the nanocomposite photocatalytic performance was investigated over 5 successive reaction cycles. It was revealed that the synthesized nanocomposite can repeatedly degrade the MO solution, without showing significant loss of the photocatalytic efficiency.

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INTRODUCTION

Nanotechnology has been providing practical and viable solutions to many challenges faced in recent decades [1-3]. Medical, agricultural, and industrial any various technical fields can greatly benefit from nanotechnology [4, 5]. One of the most concerning issue for scientists worldwide is

environmental pollution, which can be significantly reduced through nanotechnological approaches [6, 7]. Pollutants originating from facilities, power plants, farms, and cities are destroying our planet. Aquatic life is extremely affected by these pollutants, and now many of microorganisms, fish,

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and even coral reefs are becoming endangered or extinct. Because, seafood constitutes a major part of the diet of people in many societies, the poisoned fish can ultimately harm consumers. Consequently, dangerous diseases such as cancer, and immune disorders are developing [8, 9].

In this context, nano-photocatalysis has motivated researchers to adopt green and environmentally-friendly methods for removing pollution from soil, air, and water [10]. Photocatalytic purification relies on an efficient photocatalyst that can operate under visible region of light to produce powerful radical for oxidizing pollutants [11, 12]. However, single-component photocatalyst materials (such as TiO_2 or ZnO) rarely provide satisfying results, due to premature recombination of charge carriers (electrons and holes) formed upon illumination of photocatalysts [13, 14]. Nanocomposites composed of different metal oxides can address this requirement for synthesizing an efficient photocatalyst [15, 16]. Transition of charges between conduction bands and valence bands of different oxide materials can effectively reduce the recombination of charges and in turn enhance the photocatalytic activity of nanocomposite [17, 18].

Silver oxide (Ag_2O) is regarded as a great visible-light-sensitizer for photocatalytic applications, owing to its narrow band gap, high visible light activity, high surface area providing abundant active sites, and efficient charge transfer ability [19-22]. Additionally, it has been suggested that the partial reduction of Ag^+ to Ag^0 forms plasmonic states on the surface of Ag_2O particles, which enhance the separation of photo-generated charge carriers [23, 24]. As a result, the photocatalytic activity of the Ag_2O -based compounds increases, for example: Patel et al. synthesized $\text{In}_2\text{O}_3/\text{Ag}/\text{Ag}_2\text{O}$ photocatalyst for degradation of ciprofloxacin and 4-nitrophenol [25]. Also, photocatalytic degradation of antibiotics has been reported using plasmonic effect of $\text{Ag}/\text{Ag}_2\text{O}/\text{C}_3\text{N}_4$ photocatalyst [26]. Mohamed et al. designed an $\text{Ag}_2\text{O}/\text{LaTiO}_3$ heterojunction photocatalyst for desulfurization of thiophene under visible light irradiation [27].

Herein, a superior visible light activated nanocomposite was synthesized by mixing Ag_2O and $\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$. A facile and straightforward method was employed to synthesize the $\text{Ag}_2\text{O}/\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$ ($\text{Ag}_2\text{O}/\text{ZCSO}$) nanocomposite. According to the results provided in literature, Ag_2O strongly induces the visible light sensitization

of photocatalysts. Therefore, it is expected that the coupling Ag_2O with ZCSO will result in significantly enhanced photoactivity for degradation of organic pollutants.

In this work, an aqueous solution of methyl orange (MO) was used as a model of organic pollutant. Methyl orange, widely used in dyeing industry, causes severe environmental damage due to its refractory nature [28, 29]. Furthermore, it is well known that methyl orange has the potential health risks for human [30]. The visible-light photocatalytic degradation of MO was evaluated by systematically examining the influence of key parameters, including amount of loaded nanocomposite, pH of MO solution, and H_2O_2 concentration.

MATERIALS AND METHODS

Synthesis of Ag_2O nanoparticles

Ag_2O nanoparticles were synthesized under simple co-precipitation reaction. 1.0 mmol of AgNO_3 was dissolved into 50 mL of deionized water in the presence of 0.05 g of sodium dodecyl sulfate (SDS) as surfactant. Then, the Ag_2O nanoparticles was precipitate using addition of NaOH (0.5 M). The obtained dark brown solid was separated using centrifugation, rinsed using deionized water, and heated at 120 °C overnight.

Synthesis of $\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$ nanoparticles

To synthesize the $\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$ nanoparticles, metal precursors including $\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, and $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$ were dissolved into a 50 mL deionized water containing 0.1 g polyethylene glycol, under vigorous stirring for 30 min. After that, to adjust the pH of solution to around 9, NaOH solution (0.5 M) was added dropwise. Next, the formed solid were separated using centrifugation and washed three times using ethanol solution (20% V/V). The solid was dried at 120 °C in an oven overnight and finally calcined at 700 °C for 6 h.

Synthesis of $\text{Ag}_2\text{O}/\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$ nanocomposite

In order to couple the two components of nanocomposite, Ag_2O and $\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$, equal amounts of the Ag_2O and $\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$ (1:1) were dispersed into 50 mL of deionized water and continuously stirred for 1 h. Then, the mixture was transferred to an autoclave and subjected to hydrothermal treatment at 180 °C for a duration of 8 h. Finally, the light brown nanocomposite was

separated using centrifugation and dried at 90°C for 5 h.

Photocatalytic degradation procedure

The degradation of an aqueous solution of methyl orange (MO) was performed as follows: a specific amount of the nanocomposite (0.02, 0.03, 0.04, 0.05, and 0.06 g) was dispersed into the constant concentration of MO solution (50 mg/L). The mixture was magnetically stirred for 30 min in darkness in order to adsorb the MO molecules on the surface active sites of nanocomposite. Then, illumination was carried out using an Osram lamp (100 W) from a fixed distance of 40 cm for 150 min. The degradation efficiency, expressed as C/C_0 , where C_0 and C are concentration of MO solution before and after illuminating, respectively, was determined by UV/Vis spectroscopy at characteristic maximum absorption wavelength of methyl orange (500 nm). During the photocatalytic experiment, 5

mL aliquots of the dye solution were withdrawn at 30-min intervals. The collected samples were subsequently centrifuged at 6000 rpm for 10 min to remove the nanocomposite particles, and then the absorption of photocatalytically treated MO solution was assessed.

Characterization

Crystallinity and phase structure of the prepared $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite were characterized using X-ray diffraction (XRD) by Rigaku Ultima IV diffractometer. Morphology of the nanocomposite was investigated using field emission electron microscopy (TESCAN mira 3). Optical characteristics of the $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite were assessed using diffuse reflectance spectroscopy (DRS) by JASCO UV/Vis spectrophotometer.

RESULTS AND DISCUSSION

Structural properties

Fig. 1 shows the XRD pattern for the synthesized

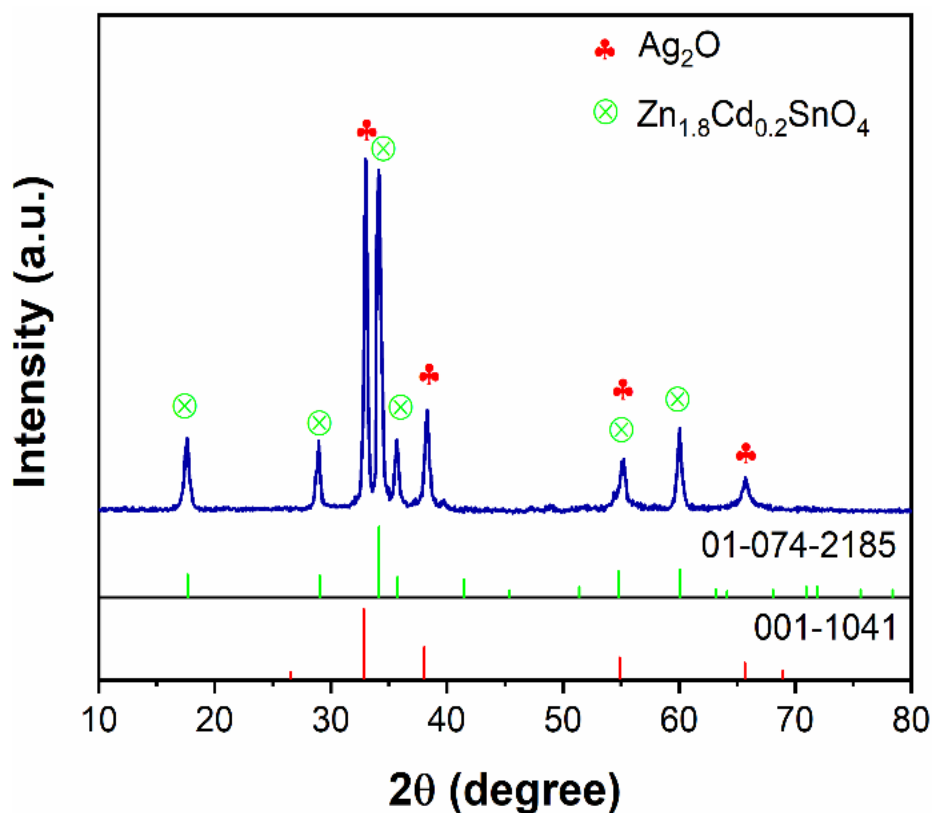


Fig. 1. XRD pattern for the $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite.

nanocomposite, representing the several diffraction peaks at the different angles associated with the two coupled Ag₂O and Zn_{1.8}Cd_{0.2}SnO₄ components. According to suggested JCPDS codes, the monoclinic phase of Ag₂O (00-1041, marked with ♣) is identified by the peaks at $2\theta = 32.9^\circ$, 38.1° , 54.7° , and 65.7° , while the cubic phase of Zn_{1.8}Cd_{0.2}SnO₄ (01-074-2185, marked with ⊗) is observed at $2\theta = 17.6^\circ$, 28.9° , 34.1° , 54.7° , and 60.1° . The crystallite size of the nanocomposite was calculated using the Scherrer method, which revealed that the nanocomposite consists of the crystallites with an average size of approximately 27.4 nm.

Surface properties and compositional analysis

FESEM image for the synthesized Ag₂O/ZCSO nanocomposite, Fig. 2a and b, exhibits the semi-spherical ZCSO nanoparticle with the size range below 100 nm, anchoring onto the larger Ag₂O particles. Moreover, Fig. 2c shows the EDX spectrum, which reveals the microanalysis result of the nanocomposite. As seen, the nanocomposite consists of Ag (47.22%), Zn (16.98%), Cd (2.71%), Sn (18.15%), O (14.94%), and no impurities are observed.

Optical properties

DRS spectrum represents the absorption

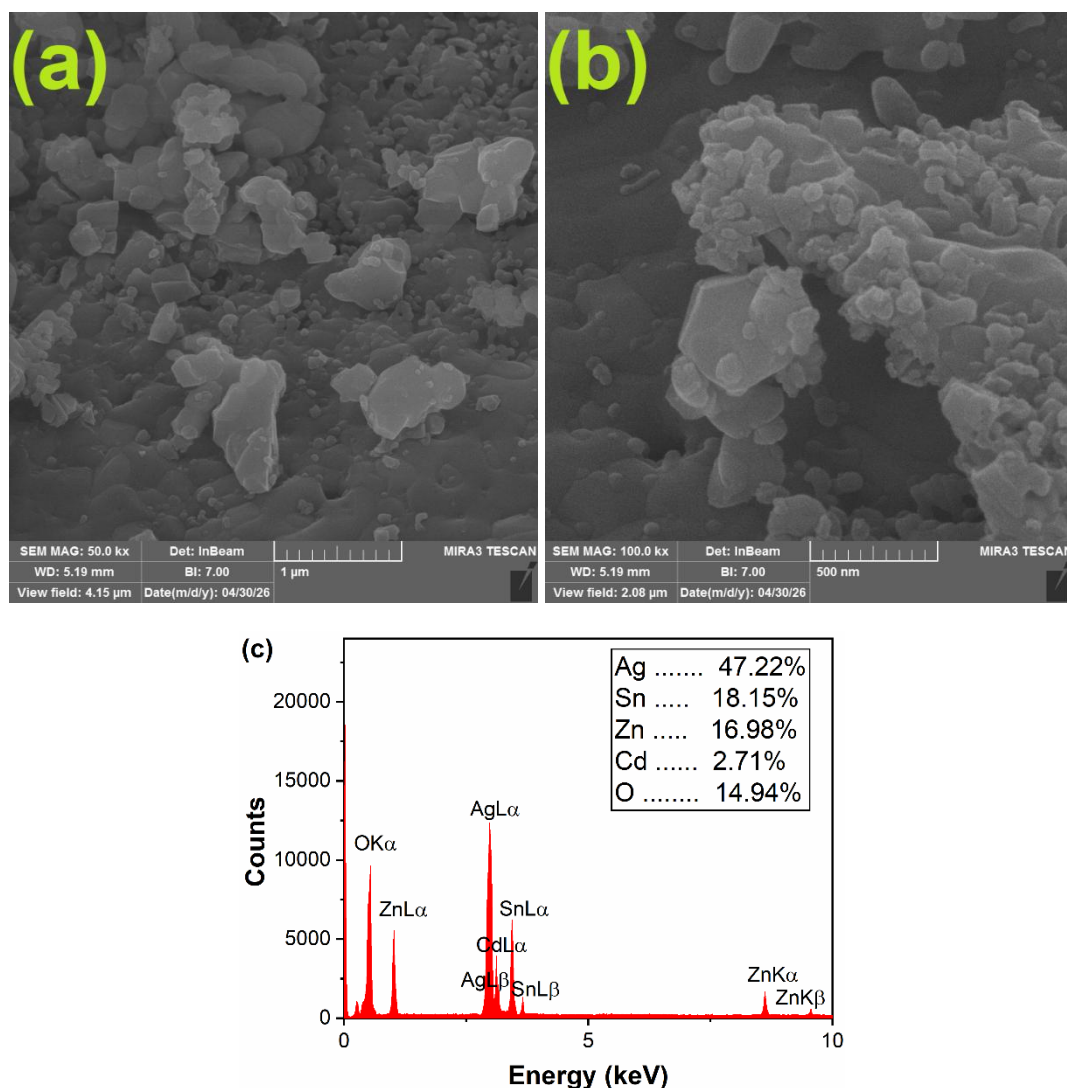


Fig. 2. FESEM images (a, b) and EDX spectrum (c) for the synthesized Ag₂O/ZCSO nanocomposite.

behavior of the $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite, shown in Fig. 3. As expected, coupling Ag_2O and $\text{Zn}_{1.8}\text{Cd}_{0.2}\text{SnO}_4$ nanoparticles has substantial benefit for the visible-light activity of the nanocomposite. The coupling of Ag_2O with ZCSO results in the formation of a heterojunction, which modifies the electronic band structure and effectively redshifts absorption edge of the nanocomposite toward

the visible light region. Obviously, compared to the pure ZCSO, the nanocomposite exhibits the enhanced light absorption, which implies that the synthesized nanocomposite can efficiently operate under the visible light irradiation for degrading the organic pollutants. Using Tauc method [31] (inset to Fig. 3), the optical band gap value for the $\text{Ag}_2\text{O}/\text{ZCSO}$ was estimated to be 2.59 eV. In comparison,

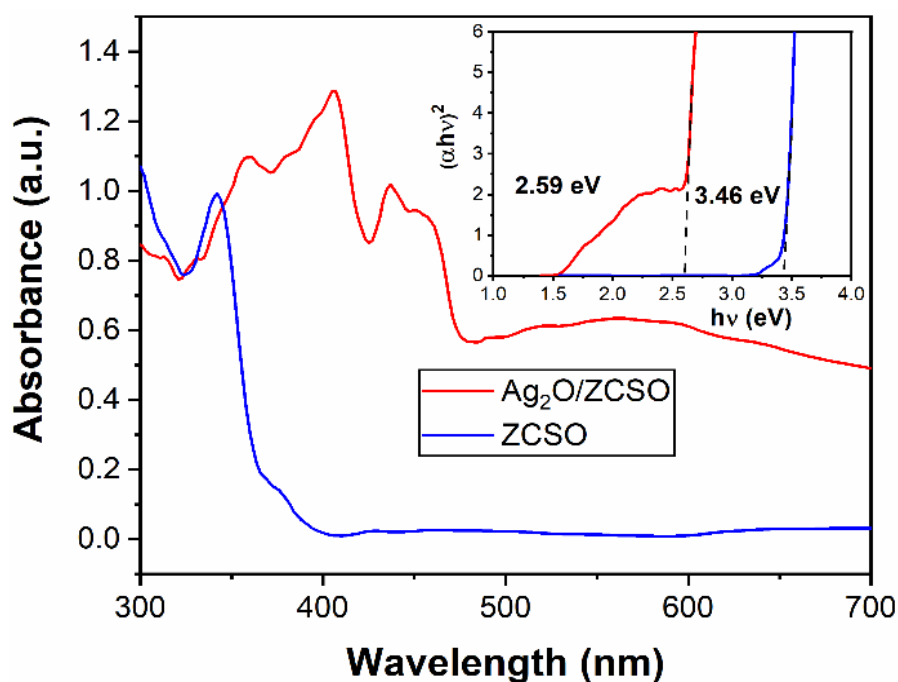


Fig. 3. DRS spectra and Tauc plots for $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite and pure ZCSO nanoparticles.

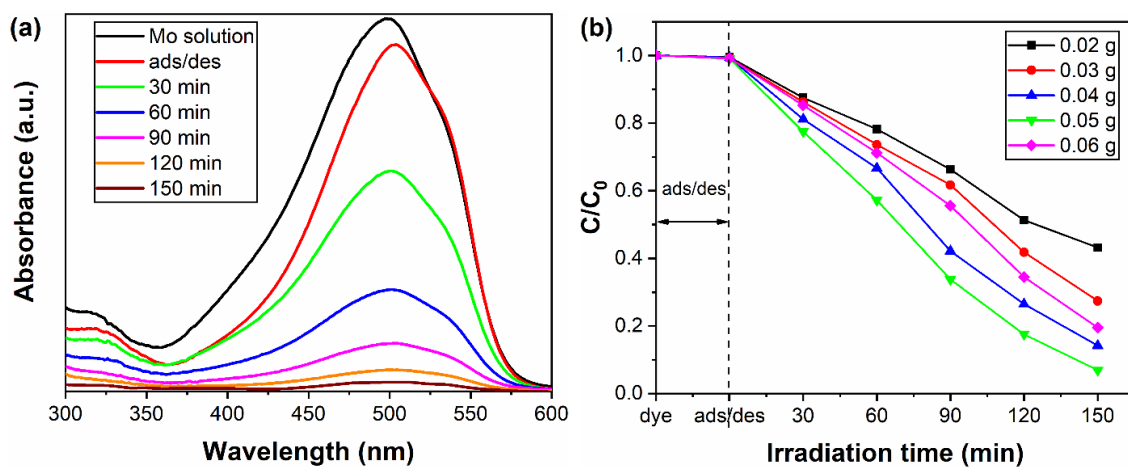


Fig. 4. UV/Vis spectra (a) and degradation efficiency (b) of photocatalytically treated MO using $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite.

pure ZCSO nanoparticles have no significant the visible-light activity, with the corresponding band gap of 3.46 eV.

Photocatalytic experiments

The $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite was examined as the visible-light photocatalyst for the removal of MO. Fig. 4a shows the UV/Vis spectra of the MO solution during 150 min the photocatalytic reaction. As seen, a continuous reduction in the absorption intensity of MO was observed throughout the irradiation period, confirming the progressive decomposition of Mo molecules in the presence of the $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite. The effect of the nanocomposite dosage on the MO removal is further illustrated in Fig. 4b. The degradation of the MO solution approached the highest level when 0.05 g of the nanocomposite was loaded (93.1%). However, when the amount of the nanocomposite was further increased to 0.06 g, a decrease in the degradation activity was obtained. Accordingly, the photocatalyst dosage of 0.05 g can be considered the optimum loading for achieving the maximum photocatalytic removal of MO. To reason this observation, the excessive

catalyst loading of the nanocomposite hinders the light beams from passing through the MO solution due to scattering, thereby leading to the lower light activation of the nanocomposite [32].

Investigation of pH effect

Fig. 5 shows the efficacy of the $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite to degrade the MO solution at different pH values. To this end, the degradation reaction was performed using the MO solution at various pH levels (4, 7, and 10). At acidic solution (pH = 4), the MO degradation increased to 95.2%, while it decreased to 69.9% in alkaline condition (pH = 10). Because of anionic nature of methyl orange [33], the positively charged surface of the nanocomposite helps to adsorb more dye molecules on the active sites thereon [34]. As a result, the photocatalytic activity increases under acidic conditions which grants positive charges on the nanocomposite surface.

Investigation of oxidant effect

In order to promote the photocatalytic reaction, H_2O_2 was used as an oxidant agent. Fig. 6 shows the effect of different concentrations (0.01, 0.02,

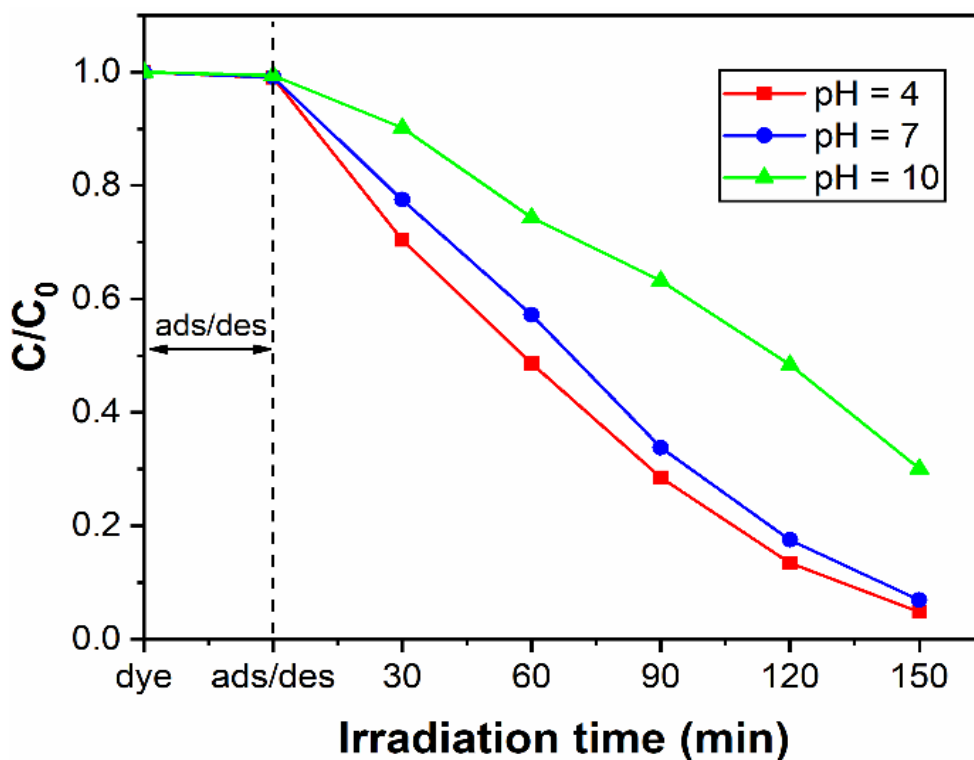


Fig. 5. Degradation efficiency of the MO solution under different pH conditions.

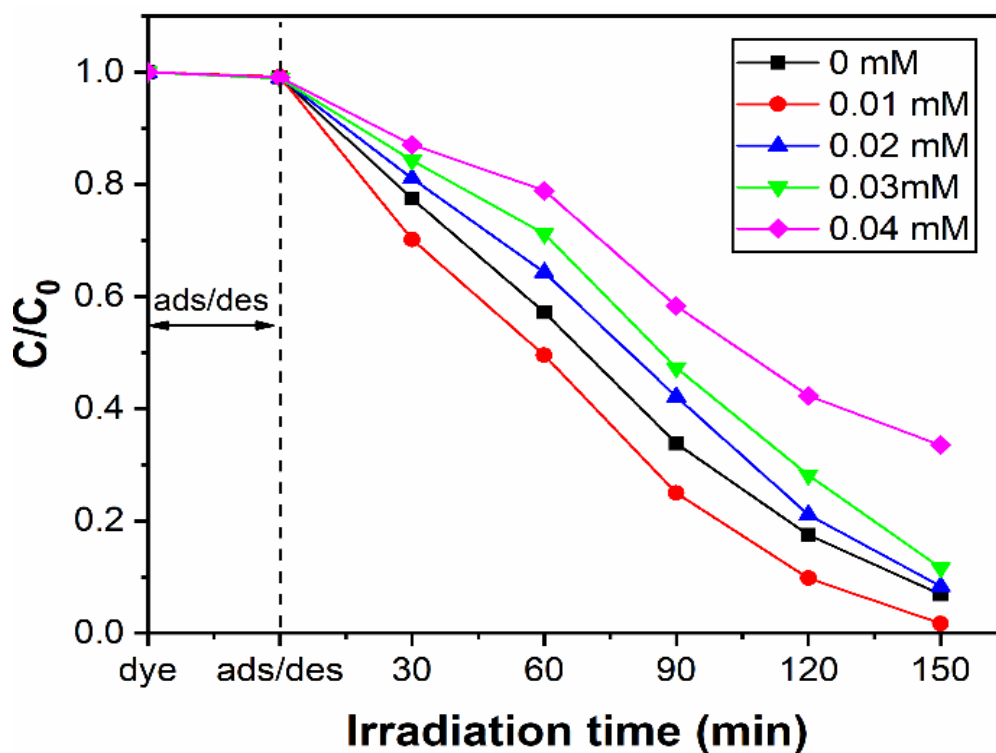


Fig. 6. Degradation efficiency of the MO solution under different concentrations of H_2O_2 .

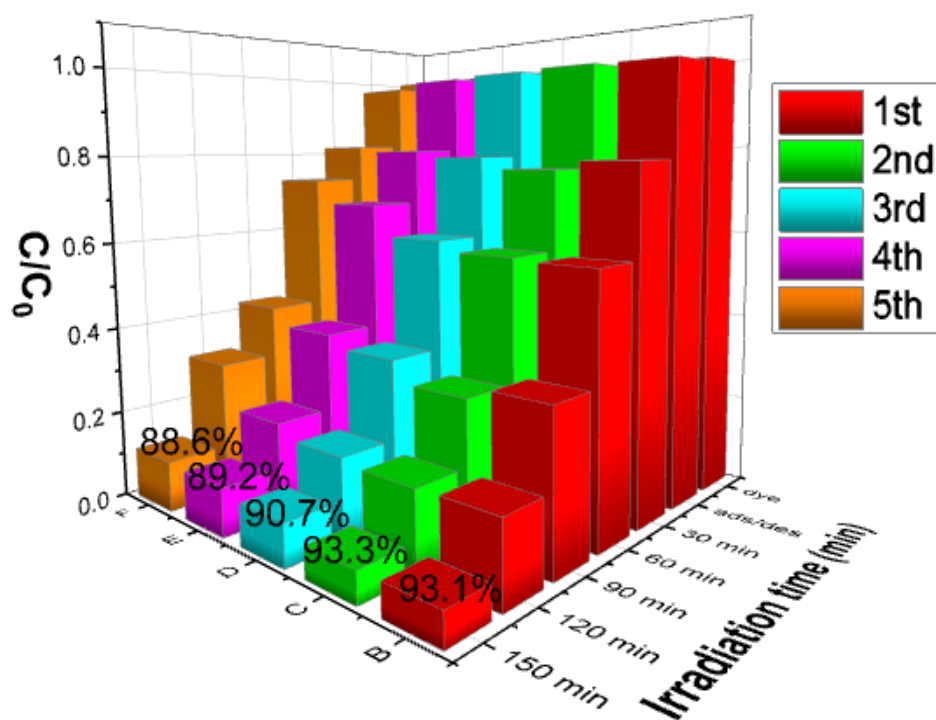


Fig. 7. Reusability experiments using $\text{Ag}_2\text{O}/\text{ZCSO}$ nanocomposite.

0.03, and 0.04 M) of H₂O₂ on the photocatalytic degradation of MO solution. As shown, when 1mL of 0.01 M of H₂O₂ was added, the degradation efficiency of MO solution peaked at 98.3%. It is well established that the addition of an appropriate amount of H₂O₂ helps to increase the degradation through formation of more oxidative radicals [35]. However, the photocatalytic activity gradually decreased as the H₂O₂ concentration was increased to 0.02, 0.03, and 0.04 M. The MO degradation results were recorded to be 91.7%, 88.4% and 66.5% using addition of 0.02, 0.03, and 0.04 M of H₂O₂, respectively. It seems that the exceeded concentration of H₂O₂ adversely affects the photocatalytic activity by hindering the formation of oxidative radicals [36].

Investigation of nanocomposite recyclability

The synthesized Ag₂O/ZCSO nanocomposite was used in 5 consecutive reaction cycles. Fig. 7 shows the MO degradation efficiency using the recycled nanocomposite. After each cycle, the nanocomposite was collected, washed, and then heated at 80 °C for 5h. Fig. 7 reveals that the synthesized nanocomposite provides superior recyclability, with the decrease in the photocatalytic activity of only 5.5% after the total of 750 min (5 cycles) of the reaction time.

CONCLUSION

To sum up, the Ag₂O/Zn_{1.8}Cd_{0.2}SnO₄ nanocomposite was successfully synthesized. First, Ag₂O and Zn_{1.8}Cd_{0.2}SnO₄ nanoparticles were synthesized using co-precipitation method. Then, they were combined as the nanocomposite through hydrothermal route. Due to the favorable low optical band gap of the nanocomposite, 2.59 eV, the photocatalytic reaction was performed under the visible light illumination for removal of the aqueous solution of methyl orange. It is indisputable that the excited electrons can be moved to the energy levels of the Zn_{1.8}Cd_{0.2}SnO₄ which decreases their recombination with holes. This phenomenon reflects itself in the reduced band gap value compared to the pure Zn_{1.8}Cd_{0.2}SnO₄. After the conducted the reaction for 150 min, the MO molecules were degraded more than 93%. In addition, the photocatalytic efficiency was increased by adjusting the pH of MO solution and addition of proper amount of H₂O₂. Also, the Ag₂O/Zn_{1.8}Cd_{0.2}SnO₄ nanocomposite is capable of utilizing under recycled use of

degradation reaction.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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