

Effective Photocatalytic Degradation of Methyl Orange Using $V_2O_5@ZnO$ Nanocomposite under UV and Visible Irradiations

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Today, reclamation and reuse of wastewater from textile, food and pharmaceutical industries are the big challenges for researchers to decrease the diseases and pollution in the world. Thus, this study was conducted for the design of $V_2O_5@ZnO$ nanocomposite and its application as a photocatalyst for degradation of methyl orange under UV and visible irradiations. The $V_2O_5@ZnO$ nanocomposite was synthesized with a homogeneous combination of hydrothermal synthesized ZnO nanorods and V_2O_5 nanoparticles. The morphological, structural, optical, electrochemical and photocatalytic degradation characterizations of prepared nanocomposite and its components were studied. Optical result showed the red shift in edge of absorption spectra of $V_2O_5@ZnO$ nanocomposite toward the ZnO nanorods film which indicate the transferring enhancement of photo-generated carriers between two-components of nanocomposites. It can decrease the recombination rate of photo-excited charges due to interface effect through creation of the separation channel of electrons and holes. Electrochemical impedance spectroscopy analysis displayed lower charge transfer resistance and effective separation of photo-excited charges on $V_2O_5@ZnO$ nanocomposite surface under visible light irradiation. Photocatalytic measurements showed 60% enhancement in the degradation efficiency for $V_2O_5@ZnO$ nanocomposite under 3 hours irradiation of visible light toward the UV light. It can be attributed to the high effective surface area of homogeneously combining V_2O_5 nanoparticles and ZnO nanorods and narrowing the band gap of prepared nanocomposite toward the ZnO film. Furthermore, this finding showed the stable activity for degradation of methyl orange on the $V_2O_5@ZnO$ nanocomposite film surface. These results indicate that the prepared $V_2O_5@ZnO$ nanocomposite can be applied for high efficiency degradation of methyl orange under irradiation of visible light.

Keywords: Degradation efficiency; Methyl orange; $V_2O_5@ZnO$ nanocomposite; Light irradiation; recombination rate; Electrochemical impedance spectroscopy

1. INTRODUCTION

Industrialization has shown many advantages in human's style life, which contain the development in medicine, facility in communication, promotion in agriculture efficiency and growth of economy [1, 2]. Nevertheless, the industrial revolution has also many disadvantages such as threats to plant life, climate change, deforestation, global warming and pollution of air, soil and water, which causes extinctions of many species of plants and animals, respiratory diseases, cancers and harm fetal health [3, 4]. Therefore, many researches have been focused on reclamation and reuse water that provide a unique and viable opportunity to augment traditional water supplies. Organic dyes are one of pollutants groups, which are generated from printing, photography, painting, leather, and textile industrial wastewater [5, 6].

The methyl orange ($C_{14}H_{14}N_3NaO_3S$) is one of most applicant azo dyes in the textile, food and pharmaceutical industries. Studies showed it could be used to determine hydrogen gas and hydrochlorides [7, 8]. It is also well known as a pH indicator. Therefore, application and introduction of methyl orange could make a great impact on the water quality and its toxicity might reveal carcinogenic effects [9-11]. Thus, efficient and selective determination and degradation techniques for methyl orange is essential.

For many decades, numerous techniques such as adsorption, oxidation, reduction, reverse osmosis, segregation, coagulation, micro and nano-filtration, anaerobic, aerobic, flocculation, ion exchange, complexometric, electrochemical and photocatalytic degradation have been used for removal of the dye from industrial wastewaters [12-14]. Among these techniques, photocatalytic degradation have been known as a most interest techniques to remove the organic dye from polluted waters because of high efficiency, low cost and simplicity of the dye degradation process through irradiation sun and UV lights in semiconductors such as TiO_2 , SiO_2 , WO_3 , ZnO , Fe_2O_3 , SnO_2 , CdS , SnO_2 , ZnS , etc [15, 16].

Moreover, photocatalytic degradation process can be improved by optimization the photocatalyst component and surface with different nano-scale morphology such as nanoparticles, nanowires, nanofibers, dendrites, nanoplates, nanotubes and quantum dots [17, 18]. Accordingly, designing binary semiconductors or nanocomposites from different nanostructures can promote the effective surface area of photocatalyst and create more charge carriers in the photocatalytic process [15, 17]. For example, Kamil et al. [19] synthesized carbon nanotube/titanium dioxide nanocomposites and applied as high efficiency photocatalyst to berberine photodegradation. Nanakkal et al. [20, 21] also showed that photocatalytic activity graphene- ZnO nanocomposite was more than ZnO .

Although nano-scale semiconductors have been proven able to enhance the photocatalytic degradation of organic compounds or dyes, the effect of $V_2O_5@ZnO$ nanocomposite on the photocatalytic activities had not been previously reported. Therefore, this study has revealed the design of nanocomposites based on two nano-scale semiconductors. The $V_2O_5@ZnO$ nanocomposite was fabricated with a combination of hydrothermal synthesized ZnO nanorods and V_2O_5 nanoparticles and applied as photocatalyst to degradation of methyl orange under UV and visible irradiations.

2. EXPERIMENTAL

2.1. Synthesize the $V_2O_5@ZnO$ nanocomposite

In order to prepare the $V_2O_5@ZnO$ nanocomposite from synthesized ZnO nanorods and V_2O_5 nanoparticles, in the first step, the ZnO nanorods were grown through a hydrothermal method. Therefore, the mixture of 1M Zinc nitrate hexahydrate (98%, Merck, Germany), 2M sodium hydroxide ($\geq 97.0\%$, Merck, Germany) and 0.1M cetrimonium bromide ($\geq 98.0\%$, Sigma-Aldrich, UK) with the volume ratios of 1:2:1 was stirred under 45°C for 2 hours. Then, the stirred mixture was transferred to 50 ml PTFE/PPL lined stainless steel in an autoclave under 150°C for 10 hours.

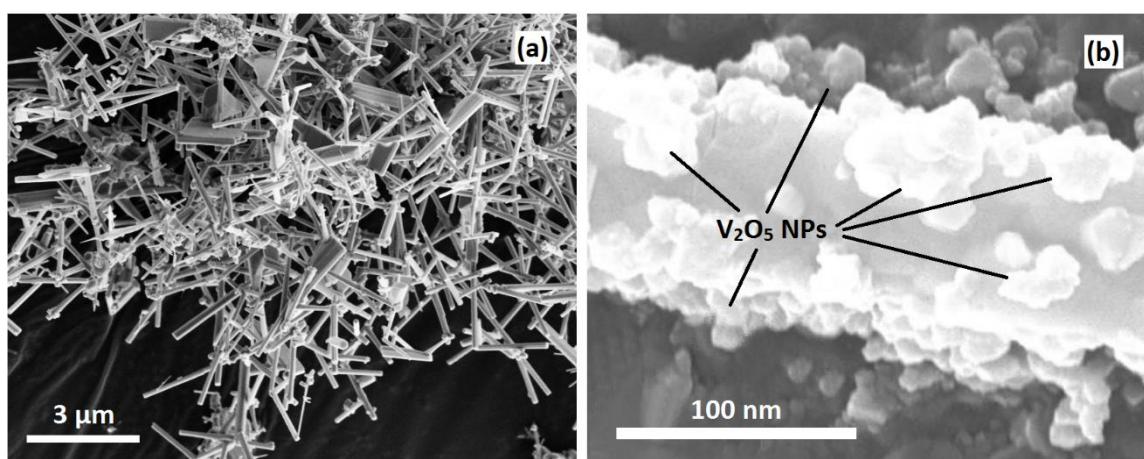
In next step, for synthesize the V_2O_5 nanoparticles through hydrothermal method, 1M ammonium metavanadate ($\geq 99.0\%$, Sigma-Aldrich, UK) was mixed to 0.1 M sulphuric acid (98%, Shijiazhuang Xinlongwei Chemical Co., Ltd., China) with the volume ratios of 1:1 under magnetic stirring at 30°C for 1 hour and then transferred to 50 ml PTFE/PPL lined stainless steel in autoclave under 200°C for 10 hours.

The hydrothermal synthesized ZnO nanorods and V_2O_5 nanoparticles powders were added to graphene powder (99wt% Single Layer, Shanghai Xinglu Chemical Technology Co., Ltd., China) in weight ratio of 5:5:0.5 and ultrasonicated for 1 hour in methanol (99.9%, Hubei Aoks Bio-Tech Co., Ltd., China). The obtained suspension was dried at room temperature.

2.2. Characterizations and measurements

The morphology of synthesized ZnO nanorods and V_2O_5 nanoparticles and $V_2O_5@ZnO$ nanocomposite was characterized by field-emission scanning electron microscopy (FESEM, Hitachi, Chiyoda-ku, Tokyo, Japan). The crystal structures of synthesized samples were studied by X-ray diffraction (XRD, D/MAX-3C diffractometer Rigaku, Tokyo, Japan) which was operating at power of 35 kV and 20 mA in wavelength of $\text{CuK}\alpha$ ($\lambda=1.5418 \text{ \AA}$). The optical absorption spectra prepared samples were studied using a UV-VIS spectrophotometer. EIS experiments were carried out in frequency range from 10^{-3} to 10^5 Hz at a potentiostatic signal amplitude of 5 mV using potentiostat equipment (CHI 660E, CH Instrument Company, Shanghai, China) in a standard three-electrode electrochemical cell. The electrochemical cell contained Ag/AgCl electrode as the reference electrode, Pt wire as the counter electrode and the prepared samples (ZnO nanorods and $V_2O_5@ZnO$ nanocomposite) as working electrode. The electrolyte of EIS measurement was 0.1M Na_2SO_4 (99%, Jinan Resi Pharmaceutical Technology Co., Ltd., China) solution.

A photoelectrocatalytic study was conducted in a test chamber that contained a photocatalyst and the methyl orange (Jinan Boss Chemical Industry Co., Ltd., China) solution which was prepared with deionized water. In order to establish an adsorption-desorption equilibrium between the surface of photocatalyst and dye solution, prior to the photocatalytic measurement the prepared dye solution was maintained in darkness for 30 minutes. Optical absorbance spectra of samples was obtained using a UV-Vis diffuse reflectance spectrometer (Perkin-Elmer Lambda 750, USA). 500 W parallel xenon lamp (Xi'an Toption Instrument Co., Ltd., China) and Uvc Lamp (254nm, T8 G13 Uv, Haining



90°, 49.79°, 52.02°, 52.72°, 55.79°, 56.49°, 58.61°, 59.19°, 61.17°, 62.31° and 64.88° which indicating the orthorhombic growth of V_2O_5 nanoparticles with (301), (011), (111), (310) (211), (401), (002), (102), (411), (600), (302), (021), (112), (020), (601), (220), (121), (611), (701), (321), (402) and (512) planes (JCPDS card No. 00-041-1426), respectively. Mixture of planes of both of hexagonal wurtzite of ZnO and orthorhombic phase of V_2O_5 are observed for $V_2O_5@ZnO$ nanocomposite XRD pattern which exhibiting the combination of V_2O_5 with ZnO structure.

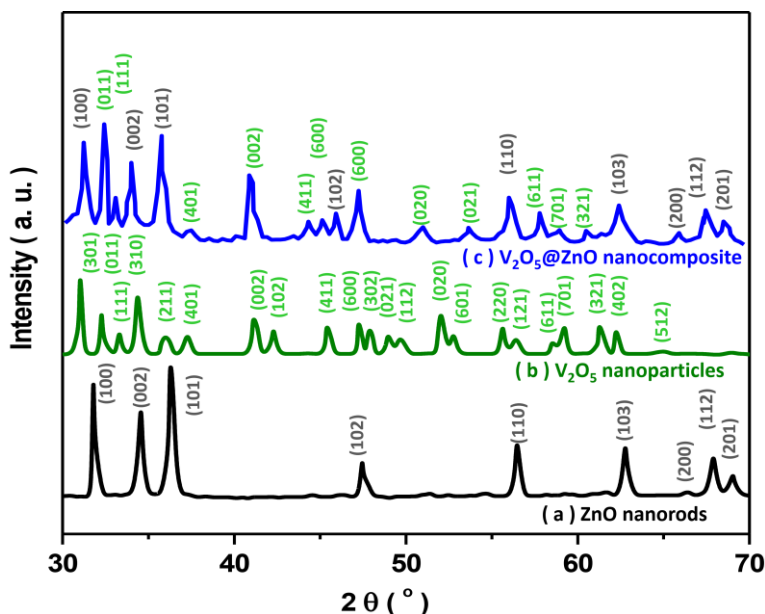


Figure 2. The XRD patterns of (a) ZnO nanorods, (b) V_2O_5 nanoparticles and (c) $V_2O_5@ZnO$ nanocomposite.

3.2 Optical absorption spectra of the prepared ZnO nanorods, V_2O_5 nanoparticles and $V_2O_5@ZnO$ nanocomposite films

In order to evaluate the photocatalytic activities, optical absorption spectra of the prepared ZnO nanorods, V_2O_5 nanoparticles and $V_2O_5@ZnO$ nanocomposite films on glass substrates were recorded at wavelength from 300 to 1000 nm. As seen in Figure 3, there is a strong near-band-edge absorption peak at 375 nm for ZnO nanorods film because of it's the bandgap value of ~ 3.29 eV [24, 25]. The recorded UV-vis absorption spectrum of V_2O_5 nanoparticles film exhibits a sharp absorption peak in the UV region that it extended to the visible region (absorption edge at ~ 475 nm) which attributing to it's the bandgap value of ~ 2.60 eV and the charge transfer transition from the O 2p valence band to the empty V 3d orbitals [26, 27]. For $V_2O_5@ZnO$ nanocomposite film, the ambiguous and weak absorption edge is observed. It can be found that the absorption edge extended to a visible region (~ 467 nm) compared to that of ZnO nanorods film. It is referred to as the band gap value of $V_2O_5@ZnO$ nanocomposite film as 2.64 eV [28, 29] which indicates the great absorption ability in the visible range. Therefore, the prepared nanocomposite can be conducted for photocatalytic application under irradiation of visible light. The red shift in absorption edge can be described with interface effect which can enhance the transferring of photo-generated carriers between two-components of nanocomposites [30, 31]. Accordingly, the interface effect of $V_2O_5@ZnO$ nanocomposite film can

decrease the recombination rate of photo-excited charges through creation of the separation channel of electrons and holes (h^+) [32, 33].

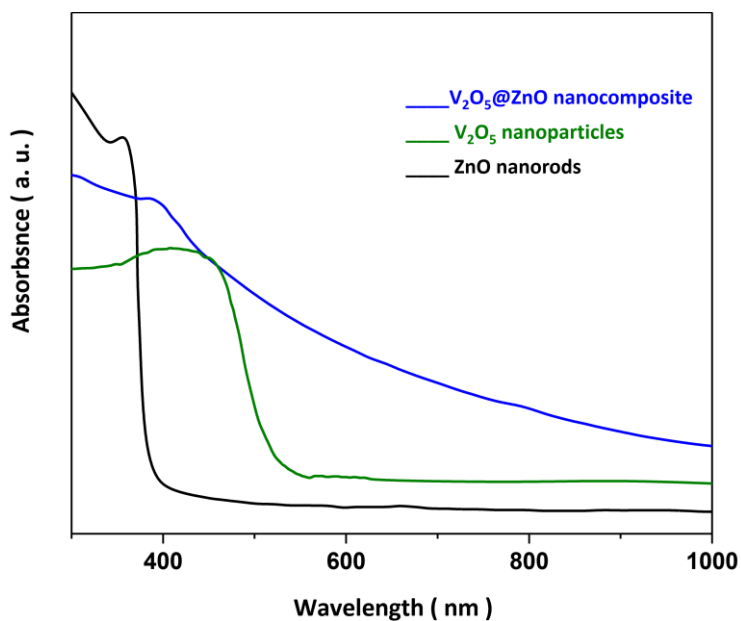


Figure 3. Optical absorption spectra of the prepared ZnO nanorods, V₂O₅ nanoparticles and V₂O₅@ZnO nanocomposite film on glass substrates at wavelength from 300 to 1000 nm.

3.3 Photocatalytic activity of ZnO nanorods and V₂O₅@ZnO nanocomposite films for degradation of methyl orange

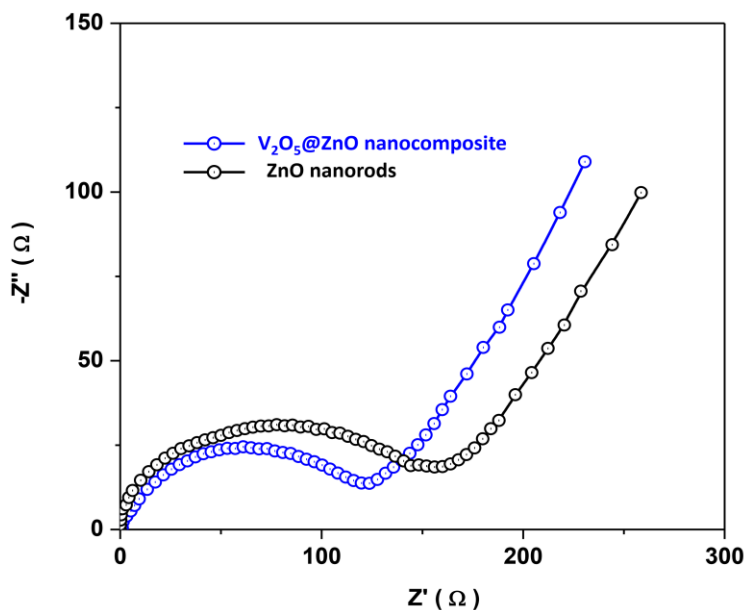


Figure 4. EIS Nyquist plots of ZnO nanorods and V₂O₅@ZnO nanocomposite films under visible light irradiation.

In order to study the excitation and photo-generated carrier transfer in ZnO nanorods and $V_2O_5@ZnO$ nanocomposite films, the EIS measurements were recorded. Figure 4 displays the EIS Nyquist plots of ZnO nanorods and $V_2O_5@ZnO$ nanocomposite films under visible light irradiation which implying the diameter of the Nyquist semicircle of $V_2O_5@ZnO$ nanocomposite is smaller than that of ZnO nanorods film. It can disclose lower charge transfer resistance of $V_2O_5@ZnO$ nanocomposite than ZnO nanorods film [34, 35]. Moreover, it can refer to interface effect and effective separation of photo-generated charge on $V_2O_5@ZnO$ nanocomposite [34, 36].

To evaluate the photocatalytic activity of ZnO nanorods and $V_2O_5@ZnO$ nanocomposite films for the degradation of methyl orange, degradation measurements were recorded in 5 mM of methyl orange aqueous solution under UV and visible lights irradiation. Figures 5 and 6 show the degradation of methyl orange in three conditions without catalyst as control the experiment (blank) and with photocatalyst of ZnO nanorods and $V_2O_5@ZnO$ nanocomposite films. As observed, the blank measurement exhibits 20% and 22% degradation efficiency after 3 hours irradiation of UV and visible lights, respectively. As seen, 42% and 40% degradation efficiency belonging to ZnO nanorods film under 3 hours irradiation of UV and visible lights, respectively that the slightly increase photo-degradation activity under UV irradiation due to the band gap value and photo-excited activity of ZnO in UV region. It is shown 60% promotion in the degradation efficiency for $V_2O_5@ZnO$ nanocomposite under 3 hours irradiation of visible light due to high effective surface area of combination of V_2O_5 nanoparticles and ZnO nanorods [37, 38]. Accordance the optical absorption study, interface effect of V_2O_5 nanoparticles and ZnO nanorods can improve the transferring of photo-generated carriers between two semiconductors of nanocomposites and decrease the recombination rate of photo-excited charges [30, 32]. Moreover, the band edge potential levels of two compounds is an important factor to determine the flowchart of photo-generated carriers in the composites. The positions of edge of valence band (E_{VB}) and the conduction band (E_{CB}) in $V_2O_5@ZnO$ composite can be determined from the equations (2) and (3) [39]:

$$E_{CB} = X - E_e - 0.5 E_g \quad (2)$$

$$E_{VB} = X - E_e + 0.5 E_g \quad (3)$$

Where E_g is the band gap energy of component of composite (2.60 eV for V_2O_5 and 3.29 eV for ZnO), and X is the electronegativity (6.10 eV for V_2O_5 and 5.79 eV for ZnO). E_e is the energy of free electrons on the hydrogen scale (~ 4.5 eV) [40]. Therefore, E_{CB} and E_{VB} of V_2O_5 are 0.3 eV and 2.9 eV, respectively. -0.35 eV and 2.93 eV are obtained for E_{CB} and E_{VB} of ZnO, respectively. Photocatalytic activity is based on generation of electron-hole pairs through the excitation in a bandgap. With visible light irradiation of $V_2O_5@ZnO$ nanocomposite, V_2O_5 could be considered as a sensitive semiconductor to absorb the visible light. Afterwards, the absorbed photon can create the electron-holes pair or can excite the electrons of the valence band to the conduction band. These photo-excited electrons can be trapped through joining to electrons in the ZnO conduction band because of vicinity of E_{VB} of V_2O_5 and ZnO which reduces the recombination rate of photo-excited electrons [41, 42]. In addition, photo-generated holes stay in the V_2O_5 surface. Therefore, the photo-generated

electrons and holes can create the hydroxyl radicals ($\text{OH}^\bullet$) which can oxidize the adsorbed methyl orange molecules on the photocatalyst surface [43].

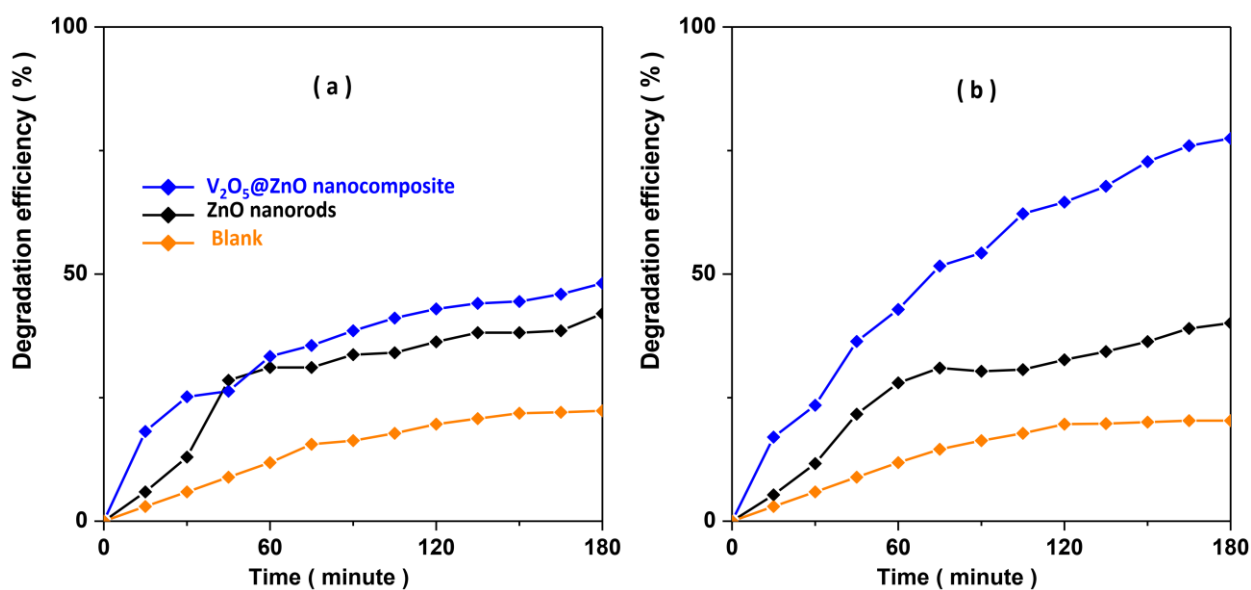


Figure 5. Degradation efficiency of ZnO nanorods and $\text{V}_2\text{O}_5@\text{ZnO}$ nanocomposite films for methyl orange under irradiation (a) UV (b) visible lights.

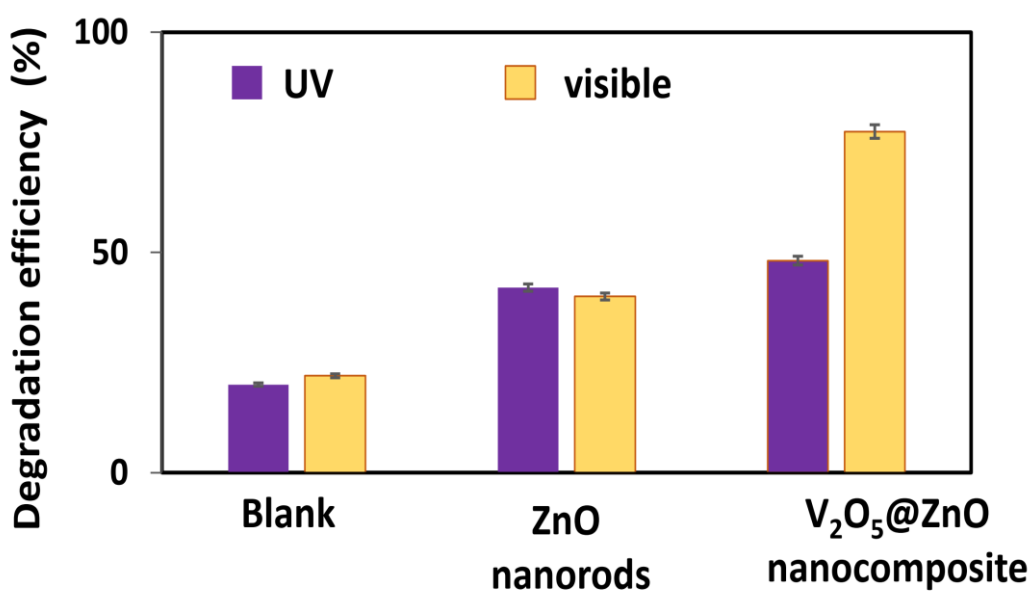


Figure 6. Comparison of photocatalysts efficiency of ZnO nanorods and $\text{V}_2\text{O}_5@\text{ZnO}$ nanocomposite films for methyl orange after 3 hours irradiation of UV and visible lights.

For further study of photocatalyst activity, the stability of $\text{V}_2\text{O}_5@\text{ZnO}$ nanocomposite films for degradation of methyl orange (10 mM) was studied under visible light irradiation. Figure 7 shows that the photo-degradation efficiency decreases after 60 minutes and 120 minutes irradiation. Moreover,

after 180 minutes of visible light irradiation periods, the degradation efficiency shows stable activity for up to five cycles.

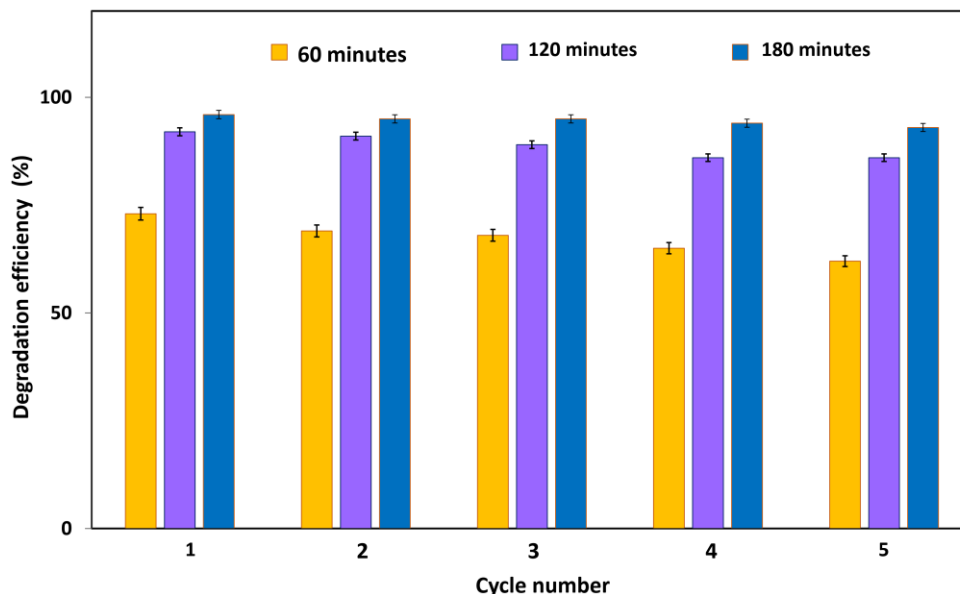


Figure 7. Cycling stability degradation experiments of $V_2O_5@ZnO$ nanocomposite film for degradation 10 mM methyl orange under visible light irradiation for 60, 120 and 180 minutes.

Table 1 illustrates a comparison between the synthesized $V_2O_5@ZnO$ nanocomposite film in this study and other reported literature based on ZnO photocatalyst for degradation of methyl orange. By considering the degradation time, the synthesized $V_2O_5@ZnO$ nanocomposite film displays better or comparable degradation efficiency. It is a remarkable point that many studies have been conducted on the degradation of methyl orange under UV irradiation, while, in this study photodegradation is performed under simulated sunlight.

Table 1. Comparison of between the degradation performances of photocatalyst based on ZnO for degradation of methyl orange.

Catalyst	Light source	Degradation efficiency (%)	Degradation time (minute)	ref
Fe-doped ZnO nanoparticles	UV	83	120	[34]
ZnO nano-mushrooms	UV	92	210	[36]
ZnO nanoneedle	UV	95.4	140	[44]
Graphene/ZnO nanorods	Uv-vis light by Xe lamp	89	240	[45]
$V_2O_5@ZnO$ nanocomposite film	Simulated sunlight	98	180	This work

In the photocatalytic oxidation mechanism of organic dye molecules, photo-induced active species such as $OH^\bullet$, h^+ , photo-generated electrons (e^-) and superoxide radical anions ($O_2^{\bullet-}$) can oxidize the dye molecules which are generated in photo-excitation and separation of electron-hole pairs in semiconductors [41, 43]. For identify the main photo-induced active specie in degradation process, four scavengers were added before the photo-degradation experiments that these include 0.1 mM solution of benzoquinone (BQ) as trapping agent of $O_2^{\bullet-}$, $AgNO_3$ as trapping agent of e^- ,

ammonium oxalate (AO) as trapping agent of h^+ and isopropyl alcohol (IPA) as trapping agent of $OH^\bullet$ [46]. Figure 8 shows the methyl orange degradation efficiency in the present and absent of the scavengers. Results show that the degradation efficiency of methyl orange is decreased in the presence of BQ, $AgNO_3$, AO and IPA because of the inhibition of the active species. As observed, degradation efficiency of methyl orange shows drastically drop in present of BQ which revealing that $O_2^{\bullet-}$ plays a main role in photo-degradation of methyl orange in the $V_2O_5@ZnO$ nanocomposite film surface.

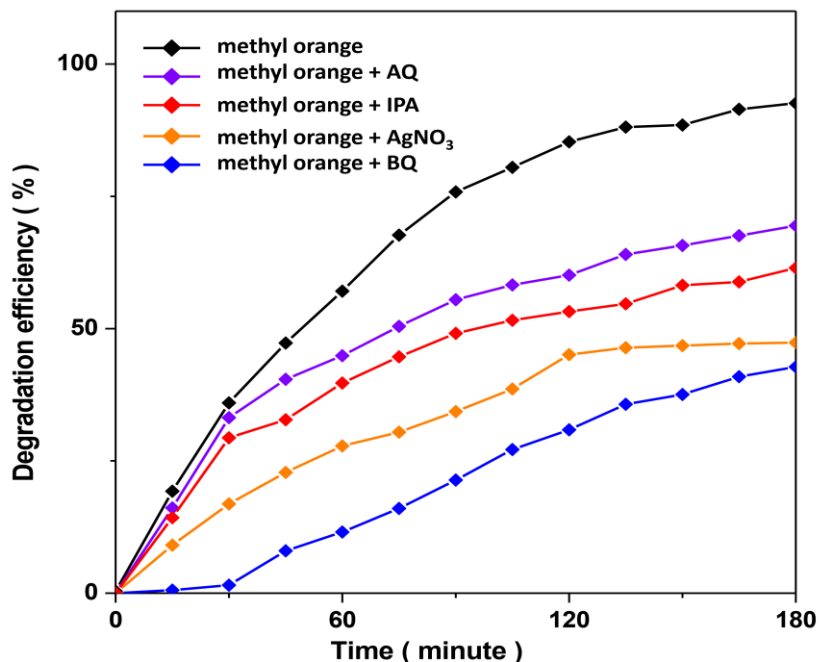


Figure 8. The methyl orange degradation efficiency on $V_2O_5@ZnO$ nanocomposite film surface in present and absent of the scavengers (BQ, $AgNO_3$, AO and IPA).

4. CONCLUSION

This study was carried out for the fabrication of $V_2O_5@ZnO$ nanocomposite and application as photocatalyst in degradation of methyl orange under UV and visible irradiations. Therefore, both of ZnO nanorods and V_2O_5 nanoparticles were synthesized with a hydrothermal method and then the $V_2O_5@ZnO$ nanocomposite was synthesized. The morphological and structural characterizations of synthesized samples were analyzed using FESEM and XRD analyses, which demonstrated the high effective surface area of $V_2O_5@ZnO$ nanocomposite. Studying on the optical properties of samples showed the red shift in edge of absorption spectra of $V_2O_5@ZnO$ nanocomposite toward the ZnO nanorods film which indicate the enhancement the transferring of photo-generated carriers between two-component of nanocomposites and decrease the recombination rate of photo-excited carriers due to creation the separation channel of electrons and holes in visible region. EIS results displayed lower charge transfer resistance and effective separation of electrons and holes on $V_2O_5@ZnO$ nanocomposite surface under visible light irradiation. Results of photocatalytic measurements showed that the degradation efficiency of ZnO nanorods film under 3 hours irradiation of UV and visible lights

were 42% and 40%, respectively which illustrate the slightly enhancement in photo-degradation activity under UV irradiation due to the band gap value and photo-excited activity of ZnO in UV region. Moreover, 60% enhancement in the degradation efficiency for V₂O₅@ZnO nanocomposite under 3 hours irradiation of visible light was observed due to high effective surface area of combination of V₂O₅ nanoparticles and ZnO nanorods. Result showed the stable activity for degradation of methyl orange in the V₂O₅@ZnO nanocomposite film surface. Results also displayed the photo-induced active of O₂^{•-} played a major role in photo-degradation of methyl orange on V₂O₅@ZnO nanocomposite photocatalyst surface. The prepared nanocomposite can be used for high efficiency photocatalytic application under irradiation of visible light.

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