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Methyl Red Dye-Sensitized Zinc Oxide as Photocatalyst for Phenol Degradation Under Visible Light

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Abstract. Zinc oxide (ZnO) is one of the semiconductor photocatalysts having large band gap energy, which is usually enough for the degradation of organic pollutant such as phenol. However, large band gap energy also means that ZnO is mostly active under UV light only. In this study, ZnO was sensitized using a methyl red dye and the improved photocatalytic activity was confirmed for degradation of phenol under visible light irradiation. The ZnO was first prepared by a hydrothermal method, then was sensitized with methyl red (MR) dye through an impregnation method with various contents of MR (1-3 wt%). Fourier transform infrared spectra indicated a change in methyl red structure after impregnation on ZnO due to the basicity of ZnO. While the MR addition did not affect the band gap energy of ZnO, it improved the optical properties of ZnO in the visible light region. Fluorescence spectra revealed that the addition of 1 and 2 wt% MR slightly red-shifted the excitation maxima of ZnO, while 3 wt% addition resulted in a blue-shift. Furthermore, it was shown that ZnO has an oxygen vacancy with emission at 558 nm and the presence of methyl red quenched the emission intensity. The photocatalytic activity of ZnO increased after dye sensitizing, where the best performance was obtained on the ZnO with 2 wt% of MR addition. The better photocatalytic activity of ZnO after dye sensitization could be proposed due to the additional electron transfer from MR to ZnO generated under visible light irradiation.

INTRODUCTION

Phenol is an organic solvent and a synthesis precursor with a wide range of usage in industry. With its high toxicity and solubility in water [1], phenol is easily carried into water bodies and into the human body. Various methods, including distillation [2], extraction [3], and photochemical processes [4] have been used to treat phenol. Another promising method is through photocatalytic degradation. Photocatalytic degradation has been used as an environmentally safe for various organic pollutants. Advantages of this method include the complete mineralization of various organic compounds and solar light usage potential [5, 6]. The photocatalysts used generally are semiconductors with wide band gap energy such as titanium dioxide (TiO₂) [7-9] or zinc oxide (ZnO) [10-14].

ZnO is a semiconductor with a wide band gap energy enough for photomineralization of organic waste. However, due to its large band gap, ZnO has a low photocatalytic activity under visible light irradiation [10], limiting its use as photocatalyst when solar radiation is taken into account. One approach to improve the absorption of the ZnO would be via dye sensitization. Dye sensitization is a method extensively studied for photosensitization of semiconductors that could be applied for solar cell [15-17] and photocatalyst [18-20].

Currently, various types of dyes have been employed as sensitizer agent for ZnO photocatalysts. Natural dyes such as porphyrin [21], carotenoid [22], and betacyanin [23] were reported to show good activity, but the bulkiness of the dye lowered the amount of dye that could be immobilized onto ZnO surface [24]. The synthetic dyes to sensitize ZnO included eosin yellow [23], rhodamine B [25], methylene blue, and resorcinol [26]. However, the

meta- and ortho- substitution in these dyes lowered sensitizer concentration and decreased the donor-acceptor distance, which increased the chance of severe charge recombination in dye-semiconductor system [24].

On the other hand, methyl red (MR) is an azo dye with a vibrant red color that has been employed as a dye sensitizer in TiO₂-ZnO as a dye mixture [27]. The existence of carboxyl groups in the dye allows sensitization of MR on various materials including ZnO due to its well-known anchoring function [28]. In addition, having *para*-substituted phenyl ring could potentially cause the MR acting as an efficient sensitizer since the *para*-dyes have been reported to be better sensitizers due to their perpendicular anchoring on semiconductors and sufficient donor-acceptor distance to prevent severe recombination [24]. In this study, the effect of MR sensitization on ZnO towards its photocatalytic activity under visible light irradiation was investigated for the first time for phenol degradation.

EXPERIMENTAL

The chemicals were commercially purchased and used without further treatments. The synthesis of ZnO was carried out similarly to the previously reported method [29]. The zinc acetate dihydrate (Zn(CO₂CH₃)₂·2H₂O, 99.5–101%, Emsure) and sodium hydroxide (NaOH, 99%, Emsure) were dissolved separately in 75 mL of distilled water at the concentration of 0.1 and 0.2 M, respectively. The Zn(CO₂CH₃)₂·2H₂O solution was then added dropwise to the NaOH solution, continued by stirring at 700 rpm for an hour. The mixture was then poured into a Teflon lined autoclave and heated using an oven for 6 hours at 150 °C. The obtained ZnO suspension was then separated through decantation. In order to get rid of leftover precursor and byproducts, the ZnO slurry was then washed repeatedly using distilled water until the suspension pH reached 7 before the drying process in the oven at 60 °C overnight.

The MR sensitization on the synthesized ZnO was carried out by adding 0.5 g of ZnO and 1, 2, and 3 wt% of MR (C₁₅H₁₅N₃O₂, ACS reagent, crystalline, Sigma-Aldrich) into 100 mL of ethanol (99.5%, Emplura, Merck). The solution was stirred at 700 rpm for 24 hours. Similar steps to those mentioned in the synthesis of ZnO were then used to wash and dry the MR-sensitized photocatalysts. The obtained samples were labeled as MR(x%)/ZnO, where x shows the amount of added MR.

The synthesized ZnO and MR(x%)/ZnO samples were subjected to several characterizations. Fourier-transform infrared spectroscopy (FTIR; JASCO FT/IR-6800), diffuse reflectance ultraviolet-visible (DR UV-vis) spectrophotometer (JASCO V-760), and spectrofluorometer (JASCO FP-8500) were employed to study the optical, fluorescence properties, and functional groups in the prepared samples, respectively. All spectra were recorded at room temperature. As for the FTIR spectra measurement, all the samples were prepared in a pellet form with the addition of potassium bromide (KBr, spectroscopy grade).

In the photocatalytic activity test, phenol (C₆H₅OH, 99–100.5%) was used as the organic pollutant model. The light source employed was a halogen lamp (Dolan-Jenner MI-157 150W) with the main wavelength of 410–800 nm. The photocatalyst (50 mg) was suspended into the phenol solution (25 mL, 50 ppm). The mixture was then stirred for 2 hours under the dark condition to achieve the adsorption-desorption equilibrium, followed by irradiation using the halogen lamp for 3 hours. The suspension was filtered to remove the solid photocatalyst using a filter membrane prior to the measurement of phenol content using high-performance liquid chromatography (HPLC; Shimadzu, LC-20AT) with C-18 column (YMC-Triart, S-5 μm, 12 nm). The percentage of degradation of phenol was calculated from the percentage ratio of degraded phenol concentration to the initial concentration of phenol.

RESULTS AND DISCUSSION

Functional Group Analysis

To determine the functional groups in the synthesized ZnO and MR-sensitized ZnO samples, the samples were measured using the FTIR spectrophotometer. Figure 1 shows the FT-IR spectra of synthesized materials. As depicted in Fig. 1(a), the ZnO showed the absorption peaks at 3400 and 1639 cm⁻¹ corresponding to the stretching and bending vibrations of O-H and H-O-H of water [30], while the stretching vibration of Zn-O could be detected at 423 cm⁻¹ [31]. The MR(x%)/ZnO samples also exhibited the characteristic peaks of the ZnO, but with several new absorption peaks that were originated from the MR (Figs. 1(b)-(d)). As can be seen in Fig. 1(e), the MR gave O-H stretching vibration peak was detected at 3400 cm⁻¹ [30], stretching vibrations of -C=O bond from its carboxyl group at 1699 cm⁻¹, -N=N- bond from its azo group at 1600 cm⁻¹, C=C bond from its aromatic rings at 1364 cm⁻¹, and C-O bond from its carboxyl group at 1146 cm⁻¹, as well as the vibrational peak of

its disubstituted benzene at 827 cm^{-1} [32]. The FTIR spectra revealed that the MR has been successfully impregnated onto the ZnO without changing the functional groups of the ZnO.

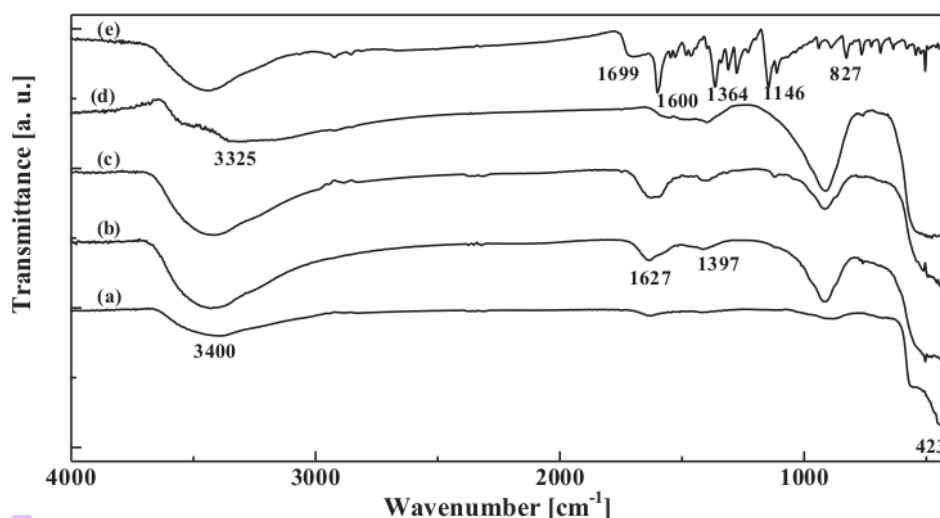


FIGURE 1. FTIR spectra of (a) ZnO, (b) MR(1%)/ZnO, (c) MR(2%)/ZnO, (d) MR(3%)/ZnO, and (e) MR samples

As mentioned above, several new absorption peaks could be observed in the MR(x%)/ZnO samples due to the presence of the MR. However, compared to the absorption peaks of the MR, it could be observed that these absorption peaks were shifted to higher or lower wavenumber. The vibrational peak at 3400 cm^{-1} was shifted to 3325 cm^{-1} due to hydrogen bond interactions between MR compounds [33], which was observed when the amount of MR was high (3%). On the other hand, the vibrational peaks of -N=N- shifted from 1600 to 1627 cm^{-1} due to the pH-induced structural change in the azo bond of the MR as illustrated in Fig. 2 in good agreement with other literature [34]. This could occur due to the basicity of the ZnO. The aromatic C=C vibrational peak at 1364 cm^{-1} was also shifted to 1397 cm^{-1} due to $\pi\text{...}\pi$ stacking interactions between the MR compounds [33].

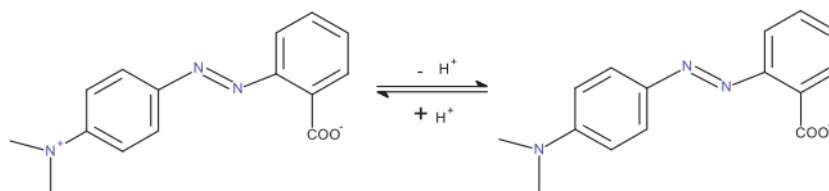


FIGURE 2. Structural change in MR due to the changes in acidity

Optical Properties

In order to determine the optical characteristic changes of ZnO after MR sensitization, the ZnO and the MR(x%)/ZnO samples were measured using DR UV-vis spectrophotometer and spectrofluorometer. The DR UV-vis spectra of the materials are shown in Fig. 3(a) as plots of Kubelka-Munk (KM) function vs wavelength. The ZnO and MR(x%)/ZnO samples were shown to have strong absorption in the UV region with a maximum peak at 316 nm . The presence of MR did not affect the absorption of the maximum peak, however, the addition of 1 and 2 wt% MR gave a red shift on the ZnO band edge while 3 wt% addition gave a new peak at 544 nm . The red shift and new peak appearance suggested the successful impregnation of MR on ZnO. In order to determine the band gap energy of the pure ZnO and MR-sensitized ZnO samples, Tauc plot was used as shown in Fig. 3(b). The band gap energies

of synthesized samples were estimated to be in the range of 3.28–3.29 eV, indicating that the MR addition did not affect much the band gap energy of the ZnO, in agreement to its function as a dye sensitizer.

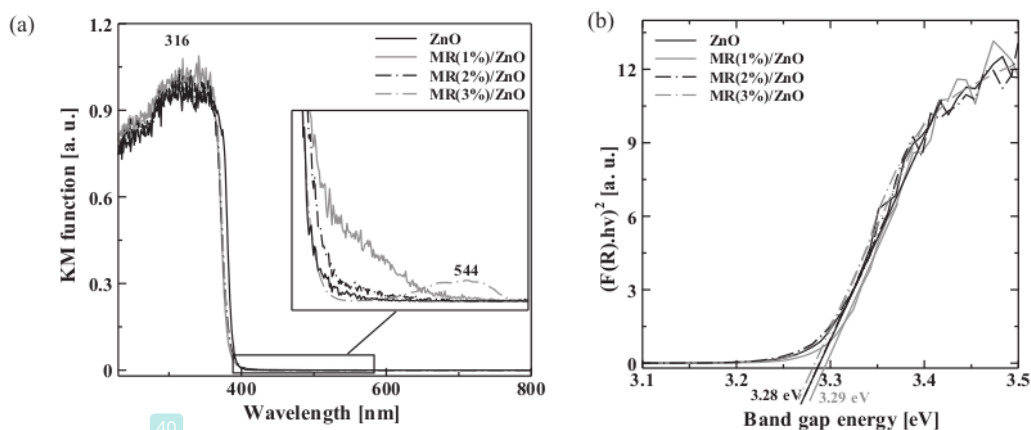


FIGURE 3. (a) DR UV-vis spectra and (b) Tauc plots of ZnO and MR(x%)/ZnO samples

Fluorescence spectra of the ZnO and MR(x%)/ZnO samples are shown in Fig. 4. The excitation and emission spectra were recorded to investigate the aggregation of MR and the electron-hole recombination on the ZnO before and after addition of MR, respectively. The excitation spectra were measured at an emission wavelength of 558 nm, while the emission spectra were measured at excitation wavelengths of 340–390 nm, depending on each respective excitation peak, which was different with the amount of added MR. There were two clear maximum peaks in Fig. 4(a). The first peak was observed around 275 nm, which was not changed with the changes in the added amount of MR. On the other hand, the second maximum excitation wavelength showed a red shift from 372 to 374 and 377 nm after 1 and 2 wt% MR addition, respectively, while 3 wt% addition resulted in the blue shift of the excitation wavelength to 347 nm. This result aligned with the reported computational study of MR aggregation on TiO₂ [32], where π ... π stacking interactions could induce the red shift, while the hydrogen bond formation could lead to the blue shift. The emission spectra of the samples are shown in Fig. 4(b). The ZnO was shown to give a yellow emission at 558 nm, which was attributed to the electron transition from the conduction band (CB) of ZnO to the oxygen vacancy (V_o) [34]. This strong emission band indicated the presence of an oxygen vacancy defect in the synthesized ZnO. The addition of MR caused the emission intensity to be reduced, suggesting a lower charge recombination rate could be achieved in the MR(x%)/ZnO samples than in the bare ZnO.

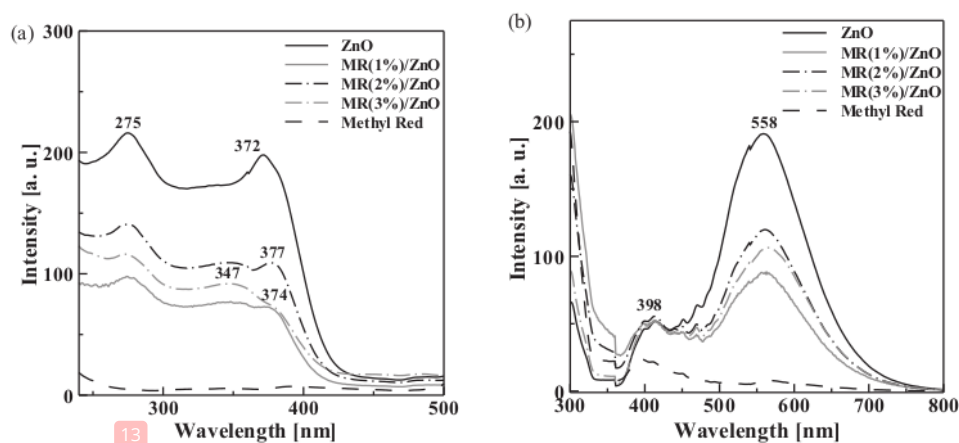


FIGURE 4. (a) Excitation and (b) emission spectra of ZnOMR(x%)/ZnO, and MR samples

Photocatalytic Activity

The ZnO and MR-sensitized ZnO samples were tested for their photocatalytic phenol degradation under visible light irradiation for 3 hours and the results are depicted in Fig. 5. As expected, ZnO showed a negligible activity under visible light irradiation (0.1%) due to the inability of ZnO to absorb the visible light. In contrast, the photocatalytic activity enhancement of ZnO could be achieved on all MR(x%)/ZnO samples. The photocatalytic activity of ZnO increased from 0.1% phenol degradation to 4.1, 6.8, and 6.4% phenol degradation with the addition of 1%, 2%, and 3% of MR, respectively. This result clearly revealed that the MR acted as a dye sensitizer for the ZnO so that photocatalytic activity enhancement of *ca.* 68 times could be achieved on the best photocatalyst, which was the MR(2%)/ZnO. Such enhancement could be realized due to the efficient electron transfer from the MR to the ZnO.

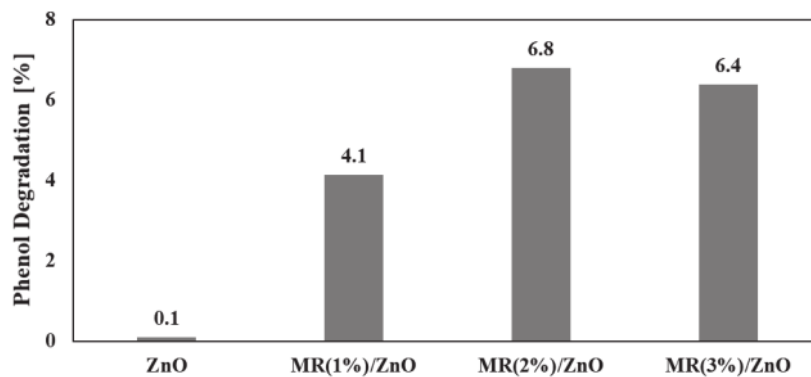


FIGURE 5. Photocatalytic phenol degradation on the ZnO and MR(x%)/ZnO samples under visible light irradiation

An analogy to the computational calculation of aggregated MR on the TiO₂ [33], the effect of MR amount to the aggregation of MR on the ZnO as also supported by excitation spectra above could be explained similarly, which was strongly related to the photocatalytic activity results. It has been reported that the ideal distance of donor-acceptor or highest occupied molecular orbital (HOMO)-lowest unoccupied molecular orbital (LUMO) should not be too close as to result in severe recombination, yet should not be too far as to result in inefficient electron transfer due to the distance [27]. At 1 wt% MR addition, only one layer of MR was deposited on the ZnO surface, allowing normal electron transfer due to MR's *para*-substitution having sufficient donor-acceptor distance. At 2 wt% MR addition, a dimer could be deposited on ZnO through $\pi\cdots\pi$ stacking interaction between two MR compounds. The HOMO was relocated to the outermost MR, increasing the distance between electron donor-acceptor. However, the electron transfer efficiency improved due to the $\pi\cdots\pi$ stacking interaction between the MR's aromatic groups. In the $\pi\cdots\pi$ stacking interactions, the orbitals of both aromatic groups tend to overlap and the electrons tend to be delocalized [35]. This delocalization led to the ease of the electron flow from outer MR to inner MR and further to the semiconductor, hence increasing the photocatalytic activity of 2 wt% MR-sensitized ZnO. At 3 wt% MR addition, hydrogen bonds could form between the carboxyl groups of second and outermost MR, which was in good agreement with the FTIR spectrum shown in Fig. 1(d). In addition to the increase in the donor-acceptor distance, potential wells would be also formed, which would localize electrons in the bond's orbital [36], hence limiting electron mobility from outermost MR to second MR. This, in turn, would decrease the electron transfer rate from the MR to the ZnO, resulting in a decreased photocatalytic activity after 3 wt% addition.

In addition, the reduced emission intensity after the MR was added onto the ZnO suggested that the added MR could reduce the oxygen vacancy in the ZnO and/or trap electrons if the composite was excited under UV light. The latter would also suggest that the addition of MR resulted in a lower electron-hole recombination on the ZnO, which could give benefits for reactions under UV light. However, since the reaction was carried out under visible light irradiation, the low emission intensity after excitation under UV light would suggest the possible pathway that the electron excitation could occur from the valence band of the ZnO to the MR, which make the ZnO gave good activity under visible light after MR addition.

CONCLUSION

The MR-sensitized ZnO photocatalysts have been successfully synthesized by impregnation of the MR onto the ZnO photocatalyst. FTIR revealed the presence of characteristic absorption peaks of both MR and ZnO. The shifting of the vibration peaks observed on the MR-sensitized samples would be caused by the structural changes due to the basicity of the ZnO, the $\pi\cdots\pi$ stacking interactions and the hydrogen bond formation. The last two interactions would be related to the added amount of the MR. In good agreement with the FTIR spectra, the $\pi\cdots\pi$ stacking interactions was confirmed to induce the red shift, while the hydrogen bond formation could lead to the blue shift in the excitation spectra. Emission spectra showed that charge recombination could be reduced with the presence of the MR. Photocatalytic tests showed that the best sample with the highest photocatalytic activity for phenol removal under visible light was MR(2%)/ZnO. Under visible light for 3 hours, the activity of unmodified ZnO increased close to sixty eight times from 0.1% to 6.8%. This enhanced photocatalytic activity was contributed from the efficient electron transfer from the MR to the ZnO, suggesting that the MR could be used as an efficient dye sensitizer of the ZnO photocatalyst.

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