Combustion synthesis and characterization of visible-light-driven Tm-doped ZnO nanoparticles used for photodegradation of methylene blue

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Tm-doped ZnO nanoparticles were synthesized by a tartaric acid-assisted combustion method and followed by calcination at 600 °C for 2 h. Phase and morphology of assynthesized ZnO with and without Tm dopant were characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM). The analytical results certified that Tm³⁺ ions were incorporated in hexagonal wurtzite ZnO structure. The particle size of ZnO was decreased with increasing in the weight content of Tm³⁺ dopant because the grain boundary growth of ZnO was hindered by Tm³⁺ dopant. The particle sizes of ZnO, 1% Tm-doped ZnO, 3% Tm-doped ZnO and 5% Tm-doped ZnO nanoparticles are 72.09 \pm 17.37 nm, 32.77 \pm 11.08 nm, 23.08 \pm 4.61 nm and 25.27 \pm 6.03 nm, respectively. The photocatalytic activity of as-synthesized ZnO with and without Tm dopant was evaluated through the degradation of methylene blue (MB) under visible light irradiation. In this research, 3% Tm-doped ZnO nanoparticles have the highest photocatalytic activity of 97.75% under visible light irradiation within 100 min. Active spices used for degradation of MB photocatalyzed by 3% Tm-doped ZnO nanoparticles were also investigated.

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1. Introduction

Growing of textile and printing industrialization can cause numerous harmful to human health and organic contaminants in water which cannot be easily treated by tradition method [1-5]. The semiconductor photocatalysts have been reported to have high effective degradation of organic pollutant in wastewater because of cost effectiveness, nontoxicity, stability, high activity and complete mineralization of organic dyes by transforming them into H₂O and CO₂ without secondary pollution problems [1-3, 6]. Particularly, zinc oxide (ZnO), a semiconductor substance is able to be used in various applications such as transparent conductive oxide, UV-light emitters, optoelectronic devices, solar cells, photocatalyst, antibacterial oxide and gas sensors because of its direct wide band gap of 3.37 eV and strong exciton binding energy [1, 4, 5, 7]. The applicability of ZnO is important for the degradation of organic pollutants because ZnO is very abundance on earth, high photosensitive, low cost, non-toxic, and physical and chemical stable [2, 4, 5, 7]. Unfortunately, the photocatalytic performance of ZnO as photocatalyst is limited due to low separation efficiency of photogenerated electron-hole pairs and active only ultraviolet light [2-5, 8]. Therefore, doping material is a method to enhance the performance of the oxide compound to improve photocatalytic properties of ZnO and harvest visible light region [1-3, 5, 9]. The corporation of rare earth ions such as Ce [10, 11], Eu [12, 13], Gd [14, 15], La [16] and Tm [17-19] in the crystal lattice is an effective way to improve visible light-harvesting and charge

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separation properties [10-19]. Thus, Tm-doped metal oxide materials can lead to improve their photocatalytic activity because of the special intra-4f electronic structure of Tm and accelerating photogenerated carrier separation [17-19].

Different synthetic methods such as precipitation [1, 2, 20], sol-gel [3, 5, 6], microwaveassisted solution method [7, 21], combustion [9, 22], hydrothermal method [23, 24], etc were used to synthesize large quantity of ZnO nanostructure materials. Among them, the combustion method is exothermic reaction between metal precursor and organic fuel for the synthesis of metal oxide such as ZnO [22, 25, 26], Fe₂O₃ [27, 28], SnO₂ [29, 30] and TiO₂ [31, 32] because the method is environmentally friendly, low decomposition temperature and simple, and can lead to high purified products and fast production rate [22, 25, 26].

In the present work, the synthesis of Tm-doped ZnO as visible-light-driven photocatalyst was prepared by tartaric acid-assisted combustion method. Phase, structure, morphology and chemical state of elements of Tm-doped ZnO with different contents of Tm dopant were investigated. The photocatalytic properties of Tm-doped ZnO nanoparticles were monitored through the degradation of methylene blue (MB) illuminated by visible light.

2. Experimental procedure

Zinc nitrate hexahydrate $(Zn(NO_3)_2 \cdot 6H_2O)$, thulium nitrate hexahydrate $(Tm(NO_3)_3 \cdot 6H_2O)$, tartaric acid $(C_4H_6O_6)$, ethanol (C_2H_5OH) and sodium hydroxide (NaOH) were used without further purification. Each 0.01 mol $Zn(NO_3)_2 \cdot 6H_2O$ and 0–5% $Tm(NO_3)_3 \cdot 6H_2O$ by weight were weighted and dissolved in 50 ml C_2H_5OH solutions under continued stirring. Subsequently, 0.01 mol tartaric acid in 50 ml C_2H_5OH solution was added in these solutions to form gel precursors. Then, 1 M 20 ml NaOH solution was also added to them with continued stirring for 30 min. The as-synthesized gel precursors were filtered, washed with distilled water, and dried and further calcined in air at 600 °C for 2 h.

Thermal analysis of dried gel was analyzed by thermogravimetric analysis (STA 8000 Simultaneous Thermal Analyzer (TGA), Perkin Elmer) in N₂ atmosphere with 10 °C.min⁻¹ heating rate at T_R–800 °C. Phase of products was characterized by X-ray diffraction (XRD) on a Philips X'Pert MPD X-ray diffractometer equipped with Cu K_a radiation ranging from 10° to 80° at a scanning rate of 0.005 deg/s. The morphology of products was characterized by transmission electron microscopy (TEM) and selected area electron diffraction (SAED) taken on a JEOL, JEM 2010 with an acceleration voltage of 200 kV. The molecular vibrational modes were measured by Fourier transform infrared spectroscopy (FTIR) and were recorded by a Bruker Tensor 27 FTIR spectrometer with KBr as a diluting agent and operated in the range of 400–4,000 cm⁻¹ with 4 cm⁻¹ resolution. X-ray photoelectron spectroscopy (XPS) was carried out using an Axis Ultra DLD | Kratos–Kratos Analytical with a monochromated Al K_a radiation (1486.6 eV) as a providing source. All the XPS spectra were calibrated relative to the C 1s electron peak at 285.0 eV.

The photocatalytic activities of the as-synthesized samples were determined by measuring the degradation of methylene blue (MB) solutions under visible light irradiation from a xenon lamp. Each 200 mg of the photocatalysts was added in 200 ml 10^{-5} M MB aqueous solutions. Prior to the test, the suspensions were magnetically stirred in the dark for 30 min to establish an adsorption/desorption equilibrium of MB on surfaces of the photocatalysts. During photocatalytic test, 5 ml solution was withdrawn every defined period of time and centrifuged to remove the photocatalytic powder. The concentration of MB solution was analyzed by a Perkin Elmer Lambda 25 UV-visible spectrometer at 664 nm wavelength. The decolorization efficiency was calculated by the following.

Decolorization efficiency (%) =
$$\frac{C_o - C_t}{C_o} \times 100 \frac{C_0 - C}{C_0} \times 100$$
 (1)

, where C_o and C_t are the concentrations of MB before and after visible light irradiation for a period of time (t).

3. Results and discussion

The TGA/DTA graphs of the dried precursor synthesized by combustion method using tartaric acid as fuel were analyzed and shown in Fig. 1. The Tm-doped ZnO-tartaric acid precursor with 5% Tm loaded dopant shows two steps of weight loss at T_R -580 °C which are related to the evaporation of crystalline water, combustion of tartaric acid and the crystallization process of Tm-doped ZnO [9, 14, 22, 25, 33, 34]. The first weight loss at T_R -200 °C is associated to the evaporation of moisture and hydration of water molecules in Tm-doped ZnO-tartaric acid precursor about 8 % [9, 14, 22, 25, 33, 34]. The second weight loss of Tm-doped ZnO-tartaric acid precursor shows the rapid weight loss at 200-580 °C which is related to the decomposition of tartaric acid and a small amount of nitrate with weight loss of 47% [9, 14, 22, 25, 34]. The Tm-doped ZnO-tartaric acid precursor is attributed to be stable at 580 °C and above. Thus, the precursor was completely changed to crystalline product and the calcination temperature was 600 °C in this research. According to DTA result, the exothermic peak was detected at 341 °C due to the transformation of Tm-doped ZnO-tartaric acid precursor into the Tm-doped ZnO crystal [25, 26, 30, 33].



Fig. 1. TGA and DTA graphs of 5% Tm-doped ZnO-tartaric acid precursor at T_R -800 °C in N_2 atmosphere.

Fig. 2 shows XRD patterns of the as-synthesized ZnO and Tm-doped ZnO with different weight contents of Tm dopant combined with calcination at 600 °C for 2 h. XRD pattern of pure ZnO phase without Tm dopant was indexed to hexagonal wurtzite ZnO structure comparing to the JCPDS No. 36-1451 [35].



Fig. 2. XRD patterns of 0-5% Tm-doped ZnO samples synthesized by tartaric acid-assisted combustion method over the 2θ range of (a) $10^{\circ}-80^{\circ}$ and (b) $30^{\circ}-38^{\circ}$.

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XRD patterns of Tm-doped ZnO samples are still in the same phase as that of pure wurtzite ZnO structure. They indicated that the Tm dopant did not have the influence to change the structure of hexagonal wurtzite ZnO structure. Moreover, the main diffraction peaks at $30^{\circ}-38^{\circ}$ of pure ZnO sample were shifted towards the lower angle after being doped by Tm. Clearly, Tm ions were successfully incorporated into ZnO lattice and Tm³⁺ ions substituted for Zn²⁺ ions in ZnO lattice [2, 9, 10, 13, 14, 15]. This emphasize can be explained by the size difference of Tm³⁺ ions (radius = 0.87 Å [36-38]) and Zn²⁺ ions (radius = 0.74 Å [2, 13, 15, 16]). The lattice parameters of ZnO and Tm-doped ZnO were determined using the below equations.

$$\frac{1}{d_{hkl}^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$$
(2)

$$\lambda = 2d_{(hkl)}\sin\theta \tag{3}$$

, where (hkl) is the Miller index, d_{hkl} is the interplanar space for the (hkl) plane at 20 Bragg angle, λ is the X-ray wavelength of Cu K_{α} (1.54056 Å), and a and c are the lattice constant of the hexagonal structure [9, 16, 20]. The a and c lattice parameters of ZnO were 3.2427 Å and 5.1977 Å for ZnO, and 3.2532 Å and 5.2095 Å for 5% Tm-doped ZnO, respectively. The crystallite sizes of as-prepared ZnO and Tm-doped ZnO samples were calculated by Debye–Scherer formula.

$$\mathbf{D} = k\lambda/\beta_{\rm hkl}\cos\theta \tag{4}$$

, where k = 0.89 for spherical shape, λ is the wavelength of Cu K_a radiation (0.154056 nm), θ is the Bragg angle of the (101) X-ray diffraction peak and β is the full width at half maximum (FWHM) of the diffraction peak [3, 5, 6, 15, 16]. The crystallite sizes of ZnO, 1% Tm-doped ZnO, 3% Tm-doped ZnO and 5% Tm-doped ZnO are 58, 35, 28 and 29 nm, respectively. The particle sizes of ZnO were decreased with increasing in the weight content of Tm³⁺ dopant because the grain growth of ZnO was increasingly hindered.

Fig. 3 shows the FTIR spectra of precursors with and without calcination at 600 °C for 2 h in ambient atmosphere. The 5% Tm-doped ZnO-tartaric acid precursor shows the broad band at 3200-3400 cm⁻¹ which are assigned to the stretching vibration of OH group of adsorbed water [7, 9, 11, 14, 15]. The characteristic stretching vibration modes of C=O of metal-tartaric acid precursors show the vibration peaks at 1724 and 1611 cm⁻¹ [9, 39-41]. The peak located at 1374 cm⁻¹ was attributed to the C-H deformation [42]. After calcination at 600 °C for 2h, the vibration of characteristic metal-tartaric acid precursor was disappeared. This indicates that the tartaric acid was completely decomposed. The pure ZnO, 3% Tm-doped ZnO and 5% Tm-doped ZnO samples show the FTIR peaks at 418, 427 and 429 cm⁻¹ which are attributed to the vibration of Zn–O bond in ZnO lattice [1, 7, 9, 11, 14, 15]. The vibrational frequency of the Tm-doped ZnO sample is red shift which is ascribed to the substitution of Zn²⁺ ions by Tm³⁺ ions [1, 7, 9, 11, 15]. The broad band at 3200–3550 cm⁻¹ was detected due to O–H stretching vibration of H₂O adsorbed on the sample surface [7, 9, 11, 15].



Fig. 3. FTIR spectra of 5% Tm-doped ZnO-tartaric acid precursor and 0–5% Tm-doped ZnO samples synthesized by tartaric acid-assisted combustion method.



Fig. 4. TEM images and SAED patterns of (a) 0%, (b) 1%, (c) 3% and (d) 5% Tm-doped ZnO samples synthesized by tartaric acid-assisted combustion method.



Fig. 5. Particle size distribution graphs of (a) 0%, (b) 1%, (c) 3% and (d) 5% Tm-doped ZnO samples synthesized by tartaric acid-assisted combustion method.

The morphology and particle size distribution of the ZnO and Tm-doped ZnO samples were investigated by TEM (Fig. 4). The TEM images show that all samples are composed of spherical nanoparticles with different sizes. When the weight content of Tm dopant was increased, the particle size was decreased. These results are in accordance with the above XRD analysis.

The particle size distribution histograms of the samples determined from TEM images (Fig. 5) evaluated for 200 nanoparticles show the variation within different size ranges of 20-140 nm, 10-70 nm, 10-45 nm and 10-40 nm for 0% Tm-doped ZnO, 1% Tm-doped ZnO, 3% Tm-doped ZnO and 5% Tm-doped ZnO samples, respectively. The frequency distribution histogram shows that almost 80% of the nanoparticles are in the 40-110 nm, 20-50 nm, 15-35 nm and 15-35 nm for 0% Tm-doped ZnO, 1% Tm-doped ZnO, 3% Tm-doped ZnO and 5% Tm-doped ZnO, 1% Tm-doped ZnO, 3% Tm-doped ZnO and 5% Tm-doped ZnO samples, respectively. The particle sizes of ZnO, 1% Tm-doped ZnO, 3% Tm-doped ZnO and 5% Tm-doped ZnO and 5% Tm-doped ZnO samples are 72.09 \pm 17.37 nm, 32.77 \pm 11.08 nm, 23.08 \pm 4.61 nm and 25.27 \pm 6.03 nm, respectively. The selected area electron diffraction (SAED) patterns of ZnO and Tm-doped ZnO samples (inserted in Fig. 4) show bright concentric rings of electron diffraction, indicating that all samples are polycrystal. These patterns were indexed to the (100), (002), (101), (102) and (110) planes of wurtzite hexagonal ZnO structure.



Fig. 6. (a) XPS survey spectrum and high-resolution XPS spectra for (b) Tm 4d, (c) Zn 2p and (d) O 1s of 3% Tm-doped ZnO nanoparticles synthesized by tartaric acid-assisted combustion method.

The content and the chemical state of Tm-doped ZnO nanoparticles were studied by XPS. Fig. 6a shows the XPS survey spectra of 3% Tm-doped ZnO sample which demonstrates the presence of Tm, Zn and O elements. The high resolution XPS spectrum of Tm 4d containing in 3% Tm-doped ZnO sample (Fig. 6b) shows the symmetric binding energy peak at 176.2 eV of Tm 4d_{5/2}, indicating the typical characteristic Tm ions containing in ZnO lattice is trivalent [17, 18, 43, 44]. Fig. 6c shows the binding energies of Zn 2p core level at 1021.28 and 1044.26 eV for 3% Tm-doped ZnO which are attributed to the Zn $2p_{3/2}$ and Zn $2p_{1/2}$ orbital levels, respectively [2, 4, 8, 9, 15]. The binding energy of O 1s core level of 3% Tm-doped ZnO sample was fitted with the Gaussian analysis (Fig. 6d). The fitted XPS spectrum of O1s was deconvoluted into three symmetric binding energy peaks at 530.01, 531.08 and 531.93 eV. The binding energies of O 1s at

530.01 and 531.08 eV are related to the Zn-O bonding and O vacancy in the hexagonal ZnO lattice [2, 4, 8, 9, 15]. The other binding energy of O 1s in 3% Tm-doped ZnO sample is specified as the chemisorbed oxygen species of H_2O and CO_2 on the surface of sample [8, 9, 15, 45, 46]. In this research, the very high intensity ratio of chemisorbed oxygen species of H_2O of 3% Tm-doped ZnO nanoparticles can lead to enhance the photocatalytic rate.

Fig. 7a shows the photodegradation of MB photocatalyzed by ZnO with and without different doped Tm contents under visible light irradiation. The photodegradation of MB photocatalyzed by ZnO nanoparticles is 17.89% within 100 min. The photocatalytic efficiencies were increased from 17.89% of ZnO to 97.75% of 3% Tm-doped ZnO nanoparticles and decreased to 88.38% of 5% Tm-doped ZnO nanoparticles under visible light irradiation within 100 min. Clearly, the photocatalytic activity of ZnO was influenced by the weight content of Tm dopant. The 3% Tm-doped ZnO nanoparticles has the highest MB degradation because the Tm dopant acted as electron acceptor to inhibit the electron–hole recombination and enhance the photocatalytic reaction [9-11, 13, 15, 16, 19]. The reaction kinetics of ZnO with and without Tm dopant under visible light irradiation was further analyzed using the Langmuir-Hinshelwood model (Fig. 7b) [2, 4, 5, 8, 9]. The plots show the linear relationship between $ln(C_o/C_t)$ versus irradiation time for photodegradation of MB photocatalyzed by ZnO and Tm-doped ZnO samples and the pseudo-first-order kinetic model with $R \rightarrow 1$ [2, 4, 5, 8, 9]. The calculated rate constants for photodegradation of MB are 1.94×10^{-3} , 0.0174, 0.0380 and $0.0121 \min^{-1}$ for ZnO, 1% Tm-doped ZnO and 5% Tm-doped ZnO, respectively.



Fig. 7. (a) Decolorization efficiency and (b) reaction kinetics for photodegradation of MB by 0-5% Tm-doped ZnO samples under visible light irradiation.

The photodegradation of MB over 3% Tm-doped ZnO nanoparticles was performed for 5 cycles under visible light irradiation. Each photocatalytic reaction, the photocatalyst was collected by centrifugation, washed with ethanol and dried for the next run. Fig. 8 shows the photodegradation of MB over 3% Tm-doped ZnO nanoparticles under visible light irradiation for five-recycled runs. In this research, the decolorization efficiency was a little decreased from 97.75% for 1st cycle to 95.30% for 5th cycle, indicating that the Tm-doped ZnO nanoparticles are very stable under visible light irradiation.



Fig. 8. Decolorization efficiencies of MB by the recycled 3% Tm-doped ZnO nanoparticles under visible light irradiation for five recycle runs.

An active radical used for the degradation of MB under visible light irradiation was investigated by adding benzoquinone (BQ), isopropanol (IPA) and ethylenediaminetetraacetic acid disodium salt (EDTA-2Na) for scavenging ${}^{\circ}O_{2}^{-}$, ${}^{\circ}OH$ and h⁺, respectively, as the results shown in Fig. 9 [2, 6, 10, 19, 23]. Clearly, the photodegradation of MB was much decreased after being added with BQ and IPA, indicating that ${}^{\circ}O_{2}^{-}$ and ${}^{\circ}OH$ radicals are the major reactive species for the degradation of MB over Tm-doped ZnO nanoparticles under visible light irradiation [2, 9-11, 13, 15, 19]. In this research, Tm³⁺ ions accepted electrons from conduction band of ZnO nanoparticles [2, 9-11, 13, 15, 19]. Electrons from Tm²⁺ ions reacted with adsorbed O₂ to produce ${}^{\circ}O_{2}^{-}$ radicals. Concurrently, holes in valance band directly reacted with H₂O/OH to produce ${}^{\circ}OH$ radicals. Both of these radicals played the role in transforming MB into CO₂ and H₂O and enhancing the photocatalytic reaction of Tm-doped ZnO photocatalyst [2, 9-11, 13, 15, 19].



Fig. 9. Effect of different scavengers in degrading of MB photocatalyzed by 3% Tm-doped ZnO nanoparticles under visible light irradiation.

4. Conclusions

In summary, Tm-doped ZnO nanoparticles containing different Tm contents were successfully synthesized by a tartaric acid-assisted combustion method and followed by calcination at 600 °C for 2 h. XRD, XPS and TEM analyses show that ZnO and Tm-doped ZnO samples are pure phase of hexagonal wurtzite ZnO nanoparticles. The 3% Tm-doped ZnO nanoparticles exhibited the highest photocatalytic activity of 97.75% within 100 min under visible light irradiation because Tm dopant played the role in accepting electrons, inhibiting electron–hole recombination, separating of electron-hole pairs and enhancing the photocatalytic reaction. In this research, ${}^{\bullet}O_{2}{}^{-}$ and ${}^{\bullet}OH$ radicals are the major reactive species used for the degradation of MB over Tm-doped ZnO nanoparticles under visible light irradiation.

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