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Synthesis and Characterization of Ag-Doped ZnO Nanoparticles and Their Photocatalytic Degradation Activity

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Abstract

Nanocrystallines Ag-doped ZnO (1, 3, 5, 7 and 9 wt% of AgNO3) were synthesized using coprecipitation method and were characterized using X-ray diffractometry (XRD) and scanning electron microscope-energy dispersive spectroscopy (SEM-EDS). This result was supported by XRD diffractogram which showed that the peak intensity of 9 wt% was five times higher than that of 1 wt% and the average size of ZnO-Ag nanocrystal was around 20-60 nm. The increasing concentration of Ag, which was confirmed by EDS, resulted in the morphology change of ZnO nanoparticles and in the increasing photocatalytic activity of Ag-ZnO in degrading remazol yellow (RY).

Keywords: *Ag-doped ZnO Nanoparticles, coprecipitation method, photocatalytic, remazol yellow*

1. Introduction

The technology for handling waste problems has been done in several ways. One such technology is photocatalysts that use semiconductors, which are very effective and can eliminate harmful substances in polluted water so that they have become the focus of research by several researchers. [1,2] Among various semiconductors, ZnO has been considered as an excellent photocatalyst candidate because it has high photosensitivity, non-toxic, abundant availability, low cost, and others.[3,4] ZnO has an energy band gap of 3.37 eV and an excitation binding energy of 60 meV at room temperature.[5,6] Therefore, ZnO is an important material for UV lasers and optoelectronic devices, and also ZnO has electrical and optical properties so it is widely used as a photoconductor, integrated sensor.[7] However, the practical application of ZnO materials still faces two challenges, namely the limited absorption spectrum range in UV light (λ <387 nm) [8] and the relatively low reuse after used. [9]

So far, various strategies have been explored to overcome these weaknesses, including manipulating the surface, [10] forming heterostructures [11,12] or nanocomposites, [13,14] modified with polymers [15] or metals, [16,17] and others. Many researchers have demonstrated a significantly expanded light absorption range and extended the life of the charge carrier, which has been excited through the decoration of noble metal nanoparticles to ZnO. [18-20] This is mainly due to the respective effects of surface plasmon resonance (SPR) and the formation of the Schottky barrier at the metal-ZnO interface.

Among the precious metals that have been investigated, silver nanoparticles (Ag) play an important role in improving photocatalytic performance for the degradation of organic dyes.[19,22] Dopant Ag acceptor is a good candidate for ZnO. Chen et al [23] reported the microstructure and optical properties of Ag-doped ZnO nanoparticles, which were synthesized through the wet oxidation-doping process method. The results showed that doping with this method can improve the optical properties of ZnO nanoparticles. It was found that Ag-doped ZnO nanoparticles were more efficient than ZnO which was not doped in photocatalytic degradation of Acid Red 88 (AR.88).

As the process of making nanoparticles is easy, it requires an economical and simple method that attracts many researchers. The wet chemical method in making nanoparticles is an alternative method compared to conventional ceramic methods. Various types of wet chemical synthesis methods include coprecipitation method. [24] Coprecipitation is a promising method because the process uses low temperatures and is easy to control particle size so the time required is relatively shorter. [25] Some substances commonly used as precipitating agents in coprecipitation are hydroxides, carbonates, sulfates and oxalates. The results from this method are expected to have smaller and more homogeneous particle sizes.

Therefore, this study tries to synthesize Ag doped ZnO nanoparticles using the coprecipitation method. The nanoparticles produced will be analyzed by X-Ray Diffractometry (XRD) and Scanning Electron Microscope (SEM) and their activity is tested as a photocatalyst in degrading synthetic remazol yellow (RY) dyes.

2. Experiment

2.1 Materials

Zinc nitrate tetrahydrate ($Zn(NO_3)_2.4H_2O$) (Sigma-Aldrich), silver nitrate ($AgNO_3$) (Sigma-Aldrich), and sodium hydroxide (NaOH) (Sigma-Aldrich) were used as the precursors for synthesis and remazol yellow (RY) (Sigma-Aldrich) was used as the model dye for photocatalytic studies. All the reagents were of analytical grade and used without any further purification.

2.2 Synthesis of ZnO Nanoparticles

Synthesis of ZnO nanoparticles was performed using previously described method. [26] ZnO Nanoparticles were synthesized by coprecipitation method using zinc nitrate tetrahydrate ($Zn(NO_3)_2.4H_2O$) precursor as Zn source. A solution of 100 mL ($Zn(NO_3)_2.4H_2O$) 0.2 M was stirred for 30 minutes and a solution of NaOH 1 M was slowly added until pH 13 was reached and a precipitate was formed. The mixture was filtered and rinsed with distilled water until pH 7 was reached. The precipitate was dried in an oven at 80 °C for 6 hours and calcined at 400 °C to produce ZnO.

2.3 Synthesis of ZnO-Ag Nanoparticles

Synthesis of ZnO-Ag nanoparticles was performed using previously described method by Thaweesang *et al*. (2013) [26] and silver metal. $Zn(NO_3)_2.4H_2O$ and $AgNO_3$ were used as the sources of ZnO and Ag, respectively. 5.229 g of $Zn(NO_3)_2.4H_2O$ and $AgNO_3$ (1, 3, 5, 7, and 9 wt% relative to zinc nitrate) were added to 100 mL distilled water and the mixture was stirred for 30 minutes. After that, a solution of NaOH 1 M was slowly added to the mixture until pH 13 was reached, and a precipitate was formed. The mixture was filtered and rinsed with distilled water until pH 7 was reached. The precipitate was dried in an oven at 80 °C for 6 hours and calcined at 400 °C to produce ZnO-Ag. The size of crystallite obtained was calculated using Scherrer equation: [27]

$$d = \frac{k\lambda}{\beta \, Cos\, \theta} \quad (1)$$

where β is FWHM (*Full Width at Half Maximum*) of diffraction line at 2θ scale, λ is wavelength used in XRD, that is 0.15406 Å, and k is Scherrer constant 0.94.

2.4 Characterization

The morphologies of the ZnO and ZnO-Ag nanoparticles were examined using an X-ray diffraction (XRD) (Rigaku SmartLab 3 kV) and *scanning electron microscope – energy dispersive spectroscopy* (SEM-EDS) (JEOL-JSM-6510 LA).

2.5 Photocatalytic measurements

Photocatalytic activity was assessed following the procedure described by Labhane *et al*. (2015). [28] 20 mg ZnO and ZnO-Ag were each mixed with 20 mL RY 20 ppm in glass vials and were irradiated using a UV lamp in a reactor for 3 hours. Subsequently, the mixtures were centrifuged for 30 min and the dyes remained in the RY solutions were determined using a UV-vis spectrophotometer. The amount of RY degraded was calculated using the following equation:

$$\%Degradation = \left(\frac{C_o - C_t}{C_o}\right) x\ 100\ \% \quad \text{.......................................}(2)$$

where C_o is RY initial concentration and C_t is concentration of RY remained.

In an experiment to explore the effect of irradiation time, some 20 mg ZnO and ZnO-Ag catalysts were each mixed with 20 mL RY 20 ppm in glass vials and were irradiated using UV lamp for 20, 40, 60, 80, 100, 120, and 140 min. Then, the mixtures were centrifuged for 30 min and the dyes remained in the RY solutions were determined using a UV-vis spectrophotometer. The amount of RY degraded was calculated using equation (1).

3. Results and Discussion

3.1. XRD Pattern Analysis of Ag-ZnO Nanoparticles

Figure 1 shows XRD patterns of ZnO and ZnO-Ag nanoparticles synthesized in our work. By comparing the patterns and the 2θ values of diffraction peaks to JCPDS database no.36-1451, it is clear that the ZnO particles in our samples are in hexagonal wurtzite phase at 2θ values of 31.759°, 34.448°, 36.232°, 47.518°, 56.575°, 62.845°, 66.40°, 67.912° and 69.063°. On the other hand, Ag particles are in face-centered cubic (fcc) phase at 2θ values of 38.13°, 44.282°, and 64.390°. [29]

These diffraction peaks show similar diffraction patterns, except for the concentration of Ag precursor. At $AgNO_3$ 1%, the diffraction peak of Ag crystals at 2θ = 38.13° shows very low intensity. At $AgNO_3$ 9%, the peak diffraction shows five times higher intensity than the previous one. In addition, the peak diffraction at other values of 2θ show similar intensity for all samples.

Crystallite size of pure ZnO calculated by Scherrer equation is 28.5 nm and the size is decreased when Ag is added in the material (Table 1). Ag metals incorporated into the structure of ZnO inhibit the growth of ZnO particles and, in consequence, reduce the ZnO crystallite size, as reported by Labhane *et al*. (2015). [28] The size of ZnO-Ag (9 wt%), however, is smaller than that of ZnO nanoparticles without Ag dopant. Furthermore, there is no regular pattern in the size of Ag crystallite with the increasing concentration of Ag precursor.

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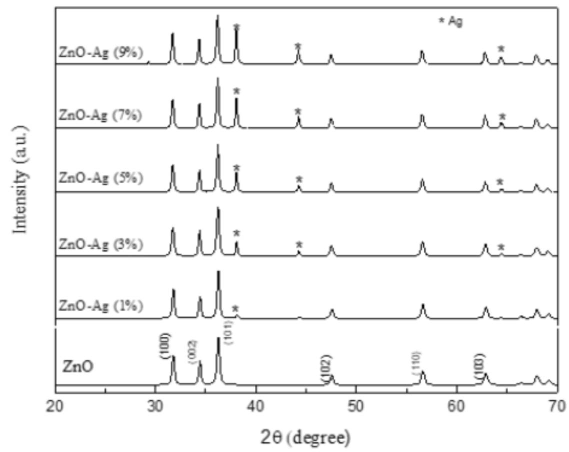

Figure 1. X-ray diffraction pattern of ZnO and ZnO-Ag nanoparticles

Table 1. The effect of Ag concentration on ZnO crystallite size

Ag concentration (%)	d_{ZnO} ***(nm)***	d_{Ag} ***(nm)***
0%	28.54	-
1%	25.22	38.84
3%	24.17	58.13
5%	27.25	49.75
7%	28.16	58.26
9%	28.36	51.09

3.2. Morphology and elemental composition of ZnO and ZnO-Ag

Observation on morphology of ZnO particles synthesized was performed using *Scanning Electron Microscope* (SEM) with 20,000 X and 40,000 X magnification and is presented in Figure 2.

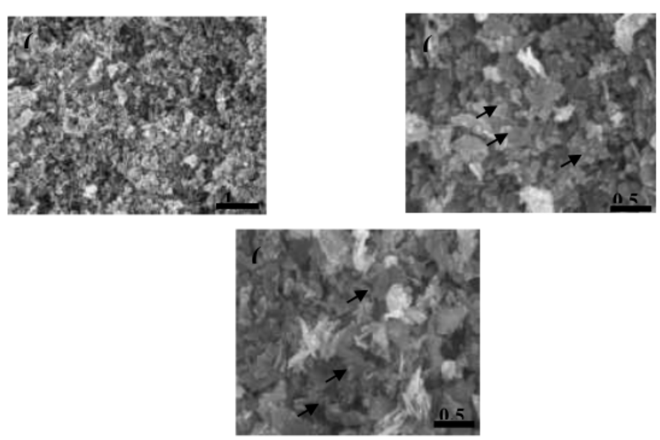

Figure 2 . SEM images of Ag-doped ZnO. (a) ZnO, (b) ZnO-Ag (1%) and (c) ZnO-Ag (9%)

Surface morphology observation of the particles shows the existence of round granules which confirm the formation of ZnO particles (Figure 2a). Indistinct round granules, however, are observed in Ag-doped ZnO because of the agglomeration (Figure 2b and 2c). Irregular forms of rod, sheet, and needle shape, which indicate the existence of Ag are observed, and the round granules of ZnO are located on their surfaces (indicated by arrows). Moreover, the figures show that more round-shaped ZnO is observed in ZnO-Ag (1 wt%) than in ZnO-Ag (9 wt%) which dominated by irregular forms of Ag. The change in surface morphology of ZnO and ZnO-Ag observed in SEM images is consistent with elemental composition of the particles analyzed by EDS (Table 2) showing that the higher the precursor concentration, the higher the Ag concentration in the particles.

Table 2. Elemental composition in ZnO and ZnO-Ag nanoparticles.

No.	Sampel	Element	Mass (%)
1	ZnO	O	20.34
		Zn	79.66
2	ZnO-Ag (1%)	O	18.50
		Zn	79.55
		Ag	1.95
3	ZnO-Ag (3%)	O	20.14
		Zn	73.97
		Ag	5.89
4	ZnO-Ag (5%)	O	21.08
		Zn	72.82
		Ag	6.10
5	ZnO-Ag (7%)	O	16.35
		Zn	72.93
		Ag	10.72
6	ZnO-Ag (9%)	O	24.43
		Zn	62.25
		Ag	13.33

3.3. Photocatalytic Performance of **ZnO-Ag** *Composite Catalysts*

RY dye was used to evaluate the effect of UV irradiation time on photodegradation activities of ZnO and ZnO-Ag nanoparticles (Figure 3).

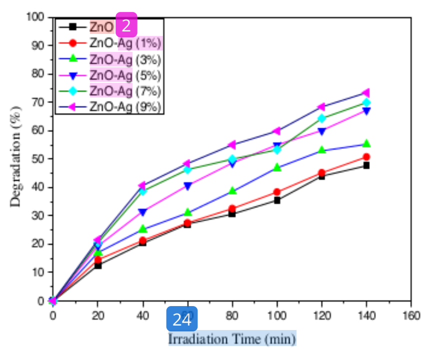

Figure 3. The effect of irradiation time on the amount of RY degraded by Ag-doped ZnO

Figure 3 shows the increasing activity of the photocatalysts with the increasing irradiation time in degrading RY under UV light. It is also shown that the higher the amount of Ag in Ag-doped ZnO, the higher the activity and ZnO-Ag (9 wt%) has the highest ability in degrading RY. It is clear that Ag could increase the rate of photocatalysis and, in turn, increase the amount of dye degraded. The photodegradation mechanism through which ZnO act as photocatalyst was described by Chong *et al.* [30] that used Ni-doped ZnO. The initial step was a migration of electrons in the surface of Ag-doped ZnO in which the electrons were excited from the valence to the conduction band on the surface of the semiconductor and, then, migrated to Ag. This step created positive holes in the surface of ZnO semiconductor at which target substances were oxidized. The longer the period of UV irradiation, the more hydroxyl radical (·OH) created and, then, the radicals produced under UV irradiation attack and photodegrade the dye.

4. Conclusion

ZnO-Ag nanoparticle was synthesized by coprecipitation method, and the average size of ZnO-Ag nanoparticle synthesized was reduced by the increasing concentration of Ag. The higher the concentration of Ag, the higher the ability of ZnO-Ag photocatalyst in degrading RY.

5. Acknowledgments

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