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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF Z_nO AND Al DOPED Z_nO THIN FILMS BY SOL-GEL METHOD AND ITS PHOTOCATALYTIC ACTIVITY

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ABSTRACT

In this study, ZnO and Al doped ZnO films were produced by Sol-Gel spin coating method. The films were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and UV-vis spectroscopy. Their photocatalytic activity was evaluated by the decomposition of the methyl orange dye using direct sunlight. The ZnO film was able to degrade the test pollutant (methyl orange) under sunlight was 55% after 180 min of irradiation of sun light. However, the Al doped ZnO film presented a very high degradation rate under sunlight (95% degradation). The high activity achieved by the Al doped ZnO film under natural conditions can be potentially applied to water treatment processes.

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INTRODUCTION

Zinc oxide is an important wide band gap semiconductor material, which has been used for several applications such as transparent conductors, solar cell windows, gas sensors and photovoltaic devices (Pearton *et al*., 2005; Markoc and Ôzgûr, 2009). Recently, thin films has attracted much attention as photocatalyst for photodegradation of pollutants in water (Yang *et al*., 2004; Ali *et al*., 2010). Photocatalytic materials have become a promising alternative for environmental remediation(Hoffman *et al*., 1995) because they are able to degrade several organic compounds in a more efficient way than other processes, such as biodegradation techniques (Gogate *et al*., 2004); thus, they constitute an alternative to eliminate pollutants in water and air (Comninellis *et al*., 2008). Particularly, the water pollution problem needs urgent attention, because excessive waste water from industrial use contains toxic compounds which are not biodegradable, like the dyes used in the textile industry (Robinson *et al*., 2001). The interest in ZnO is because it is a low cost alternative photo catalyst with photo degradation capacity.

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It has been reported that controllable n-type doping is easily achieved by substituting Zn with group-III elements such as aluminium. This kind of doping has been applied for the production of transparent conducting oxide films. However, Al doped ZnO has not yet been studied extensively as a photocatalyst. In this work we have studied the structural and photocataytic properties of Al doped ZnO thin films prepared by sol-gel spin-coating process.

MATERIALS AND METHODS

ZnO thin films were prepared by the sol-gel process. As a starting material, zinc acetate dihydrate ($Zn(CH_3COO)_2\ 2H_2O$) was dissolved in a mixture of absolute ethanol and monoethanolamine (MEA) yielding to a precursor concentration of 0.75 mol L^{-1}. MEA acts, at the same time, as a base and a complexing agent and the MEA to zinc acetate molar ratio was set to 2. For doped films, aluminum nitrate nonahydrate ($Al(NO_3)_3\ 9H_2O$) was added to the mixture with an atomic percentage fixed at 2 at.% Al. The precursor solution was deposited on glass substrates by spin-coating (3000 rpm, 30 s). Synthesized films, doped and undoped, were preheated at 300 °C for 10 min after each coating. This procedure was repeated seven times to increase the thickness. The films were subsequently heated up to 550 °C for 2 h in order to obtain crystallized ZnO.

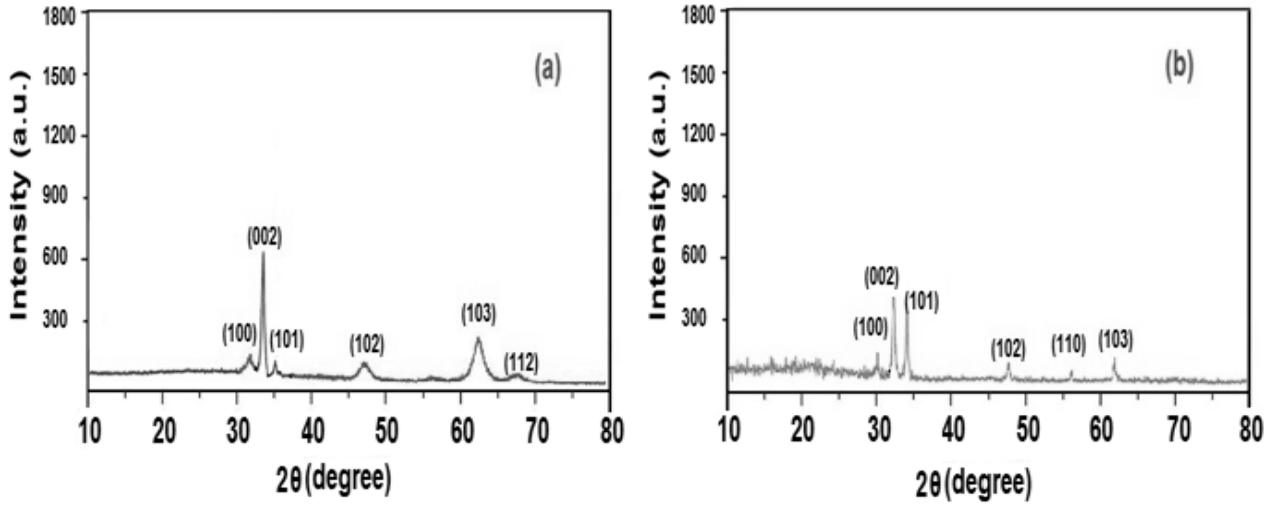

Figure 1. XRD pattern of (a) pure ZnO (b) Al doped ZnO thin films

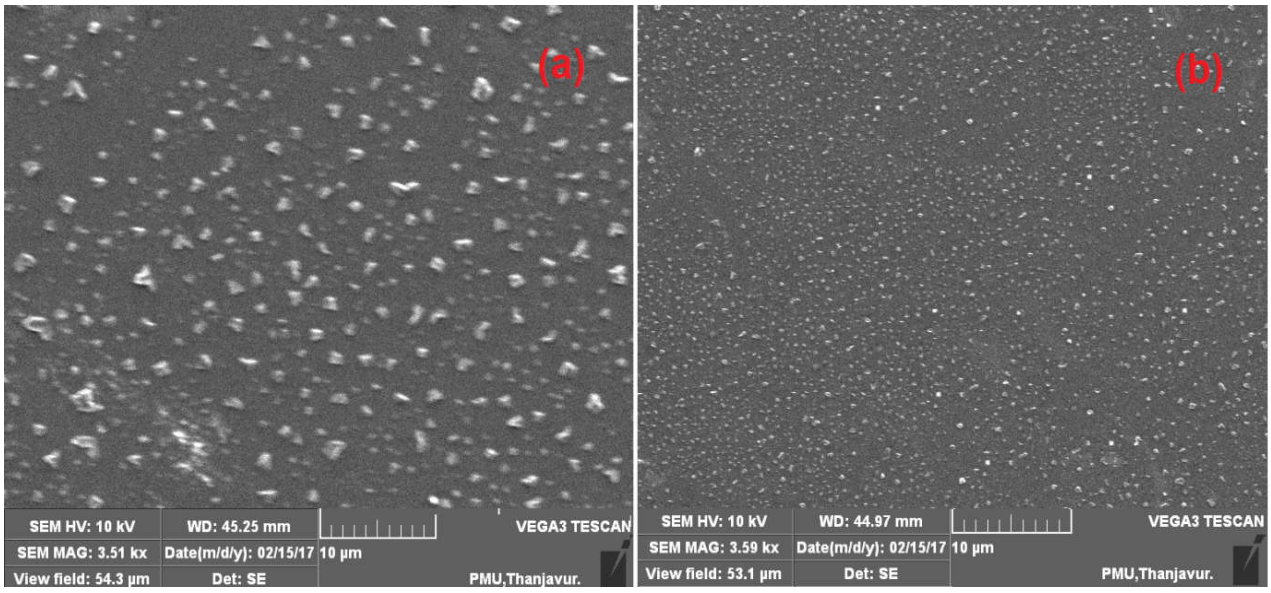

Figure 2. SEM images of (a) pure ZnO (b) ZnO/Al thin films

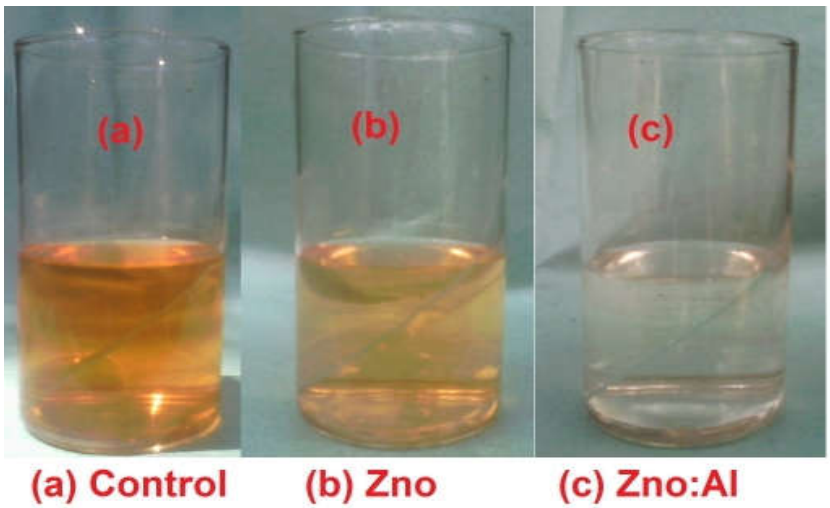

Figure 3. The degradation of the methyl orange solution was measured using the pure and doped ZnO films under the direct sun light

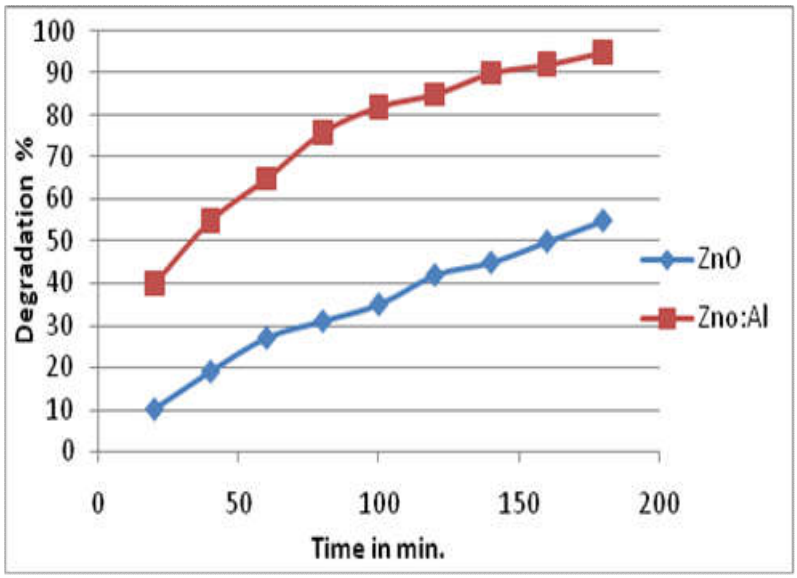

Figure 4. Degradation percentage of pure Zno and Al doped ZnO

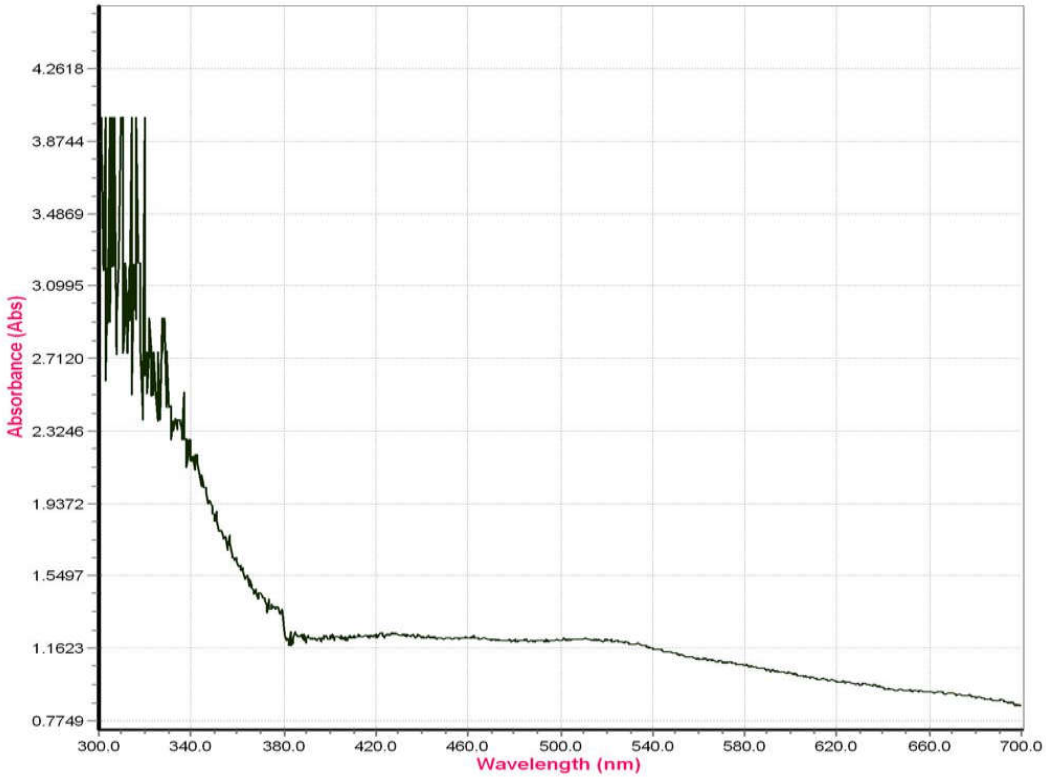

Figure 5. The UV-Visible spectrum of the methyl orange solution (Al doped ZnO) after 24 hrs

The films were characterized by X-ray diffraction (D8 Advance Bruker) using the CuKα1wavelength (1.54056˚ A). Scanning Electronic Microscopy (SEM) analysis was done by using VEGA3 TESCAN machine, Japan. The photocatalytic efficiency of the films was evaluated by monitoring the discoloration of a methyl orange (MO) solution (Sigma-Aldrich, 85% purity) with a concentration of 10^{-5} M, at the maximum absorption wavelength using a UV-vis spectrophotometer (Sl-210 Double Beam UV Visible Spectrophotometer (Elico)). The samples (doped and undoped ZnO films) was exposed to direct sunlight. The absorption spectra of the methyl orange solution were measured after each 30 minutes of illumination up to 3 hours.

The degradation percentage of the dye was obtained with the absorbance maxima ratio of the methyl orange before (A_0) and after (A) an irradiation time, as follows (Pradhan *et al*., 2012):

Degradation % = $(1-A/A_0)$ X 100

RESULTS AND DISCUSSION

The crystal structures in thin films were identified using XRD. Figure 1 displays the X-ray diffraction patterns of the samples. For both ZnO and Al doped ZnO films the patterns corresponded to five diffraction peaks of crystalline ZnO: (100), (002), (101), (102), (103). This indicates that films had a hexagonal wurtzite structure.

The intensities of diffraction peaks of the (100), (002), (101), (102) and (103) planes tended to decrease with entry of Al^{3+} dopant. This indicates that doping with Al^{3+} decreased the crystallinity of ZnO films (Neha *et. al*., 2011). SEM results are presented in Figure 2. In SEM images it can be seen that pure ZnO film had larger grain size. But with the Al^{3+} dopant, grains were arranged more densely arranged, narrower grain size distribution has been obtained in Al doped ZnO thin film.

Photocatalytic Activity

The photocatalytic activity of the films was studied by the degradation of an aqueous solution 10^{-5} M of methyl orange dye ($C_{14}H_{14}N_3SO_3Na$, 85% from Sigma–Aldrich) with a pH of 6.8. The pH of the solutions remained constant after de degradation. The methyl orange dye (MO) was chosen as a test compound because is a typical pollutant in waste water from textile industry (Robinson *et al*., 2001) besides, the degradation of this kind of dyes is easily followed by absorption spectroscopy.

The samples were immersed in 50 mL of the dye solution contained in a glass beaker. The degradation of the methyl orange solution was measured using the pure and doped ZnO films under sun light. Figure 3 shows a picture taken the sample exposed to sunlight after 2 hours. It is also shown no degradation of the methyl orange (MO) control sample was observed (Figure 3a) , but it is clearly seen that the maximum decoloration of the dye is achieved faster for the sample doped with Al than for the pure ZnO film (Figure 3c). The ZnO film was able to degrade the methyl orange solution under direct sunlight is 55% after 180 min of irradiation. Al doped ZnO film presented a very high degradation rate under sunlight (95% degradation after the same 180 min of irradiation time, shown in Figure 4). After 24 hrs , 100% decolorization took place in methyl orange solution containing Al doped Zno . The UV-Visible absorption spectrum of the methyl orange solution (Al doped Zno) was measured after 24 hrs , shown in Figure 5.

Conclusion

Photocatalytic degradation of methyl orange under the sun light is performed to investigate the photocatalytic activity of Al-doped ZnO in comparison with that of pure ZnO. Thin films of Al-doped ZnO are synthesized via spin coating method in aqueous medium using zinc acetate and monoethanolamine (MEA) as precursors. Influence of Al doping onto structural, morphological and photocatalytic properties have been investigated. Structural analysis depicts hexagonal (wurtzite) crystal structure. The effect of photocatalytic activity of Al-doped ZnO in the degradation of methyl orange is studied and results are compared with pure ZnO.

The results show that the rate of degradation of methyl orange over Al-doped ZnO is higher as compared to that of pure ZnO. The photocatalytic ability of the Al-doped ZnO film allows the development of a low cost, high efficiency, and environmentally friendly material for water treatment applications using sunlight.

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