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Abstract

We prepared a series of ZnO and ZnO/PVA catalysts by sol-gel method. The wurtzite ZnO phase was confirmed by X-ray diffraction analysis (XRD). Forming spherical shaped ZnO and ZnO/PVA nanoparticles was proved by Scanning electron microscope (SEM) images. Decomposition of Methylene Blue (MB) and methyl orange (MO) by synthesized pure ZnO and ZnO/PVA nanoparticles were studied in the UV–Vis region. ZnO/5PVA was the most active in decomposition of MB, while ZnO/7PVA was the most active in decomposition of MO. By studying biological activity of pure ZnO and ZnO/PVA in diabetic rats shows that ZnO/5PVA was the most active in lowering glucose level by different percentages.

Keywords

Zinc oxide, photo degradation, biological activity, nanoparticles.

1. Introduction

Nanocomposites are a special type of materials having unique properties and different application potentials in diverse areas [1-4]. The development of nanopowders was achieved due to introduction of nanotechnology. Successfully joined features of parent constituents in a single material can lead to novel properties of nanocomposites. Recently,

ZnO which is a well-known and an important semiconductor has been received more attention from researchers because of its wide potential applications in photo catalytic oxidation of organic compounds [5], solar cells [6] and sensors [7] etc. Both photo catalytic metal oxides ZnO and TiO₂ have been focused in literatures in degradation of organic pollutants in water and air due to their unique ability in the environmental detoxification [8-15]. ZnO with band gap = 3.37 Ev was the most suitable in photo catalysis due to its high stability and photosensitivity and absorption over a larger fraction of the solar spectrum than TiO₂ [16].

Many chemical and physical methods of synthesis have been pursued to improve the photo catalytic activity of ZnO nanoparticles [17-19]. In this study, we synthesized an eco-friendly and affordable photo catalyst (ZnO) using sol-gel method. Calcination of the synthesized samples occurred at various temperatures and characterization was identified by several approaches. The synthesized ZnO catalyst was used to investigate the removal of MO and MB dyes as a well-known water pollutants because the presence of these dyes in water causes some diseases for humans and animals [20].

In addition, the biological activity of ZnO in diabetic rats was evaluated. The use of poly vinyl alcohol (PVA) as matrix for this work due to its very unique and significant physical and mechanical characteristics. It possess high optical transparency, dielectric strength, solubility in water, nontoxicity, biodegradability, biocompatibility, excellent film forming properties, good gas barrier properties against ambient gases and doping dependent electrical and optical properties. Which arise from presence of OH groups and formation of hydrogen bond in it [21, 22].

The target of our work involves improving the photocatalytic and biological properties of ZnO by loading the PVA onto its surface.

2. Experimental

2.1. Materials

Zn-acetate dihydrate $\text{Zn}(\text{CO}_2\text{CH}_3)_2(\text{H}_2\text{O})_2$ (98%), oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) (99.5%) and ethanol ($\text{C}_2\text{H}_6\text{O}$) (99%) used in this paper are analytical grade and used without purification. MB and MO were from Aldrich chemicals, Poly vinyl alcohol (PVA) (Sigma Aldrich, $M_w=86000\text{g/mol}$) was used as the surface modificant. Double distilled water was used in preparing aqueous solutions.

2.2. Preparation of the catalyst

2.2.1. Preparation of ZnO nanoparticles

Sol-gel method was utilized in the synthesis of ZnO nanoparticles. Initially, a solution of 10.99 g Zinc acetate in 300 ml EtOH was stirred vigorously for 1 hr. In another beaker, 17.71 g oxalic acid was mixed with 200 ml ethanol, stirred for 1 hr. at 50 °C and added to the previous solution slowly. In addition, the obtained white gelatinous precipitate was dried under vacuum at 90 °C for 2 hrs., then calcined at different temperatures (300 °C, 400 °C and 500 °C).

2.2.2. Preparation of ZnO/PVA nanoparticles

To prepare 5, 7 and 10 % of ZnOP nanoparticles, a solution of PVA (0.25, 0.35 and 0.5 g) in 35 ml ethanol, respectively was added to zinc acetate solution (10.99 g in 300 ml ethanol) as shown in fig. 1, and the final precipitate was obtained using oxalic acid solution as previously mentioned. The final powder was resulted by vacuum drying followed by calcination at the same temperatures.

2.3. Characterization

2.3.1. X-ray diffraction (XRD)

XRD patterns was evaluated using PW 150 (Philips) using Ni-filtered Cu K α radiation in the 2 θ range of 10°-80° to identify the formation of ZnO, ZnO/PVA nanoparticles phases and crystal size as a non-destructive method [23].

Fig. 1 Reaction scheme of ZnO/PVA nanocomposites

2.3.2. UV–Vis Spectrophotometer (Band gab)

Using UV–Vis reflectance Spectrophotometer, the obtained wavelength and absorbance values for the prepared photocatalysts were utilized in determination of band gap values. From tacu`s equation, by plotting $(\alpha h\nu)^{1/2}$ versus $(h\nu)$, we obtained the band gab values of the prepared photocatalysts [24, 25].

$\alpha h\nu = A(h\nu - E_g)^n$	
α	coefficient of absorption
A	Constant
$h\nu$	energy of photon
E_g	optical energy
n	n = 2 for an indirect band gab & n = 1/2 for a direct band gap

2.3.3. ZnO and ZnO/PVA particle size

ZnO and ZnO/PVA particle size was obtained by SEM analyses using a JSM-5900 LV with an accelerating voltage of 20 kV [26].

2.3.4. Bronsted and lewis acid sites of ZnO and ZnO/PVA

Were investigated by FT-IR analysis that was done on Bruker spectrophotometer.

2.3.5. Potentiometric titration

Non-aqueous potentiometric titration was used to determine total acid sites of the samples [27]. Firstly, 0.1 g of catalyst was activated by heating under vacuum for 2 hrs., then added to 10 ml of acetonitrile for 3 hrs. to be adsorbed on the active sites followed by the titration of this suspension against 0.01 N n-butyl amine (0.05 ml/min). Orion 420 digital model device was utilized to determine the variation of electrode potential.

2.3.6. The degradation of MB and MO in water using UV/Vis light

The photo catalyst activities of ZnO and ZnO/PVA nanoparticles were estimated using degradation at 20 °C with external lamp (Halogen Mercury lamp, 400 w UV/Vis lamps).

2.3.7. Antidiabetic activity

Before the beginning of the experiment, a number of 12 albino rats (100-120 g) were kept for adaptation under normal laboratory conditions for 7 days. All rats were fed on balanced basal diet and allowed free access of water.

2.3.7.1 Induced of diabetes mellitus:

The experimental rats (12 rats) were divided randomly into two major groups:

Group 1 for 30 days without any treatment, 4 rats received a normal diet (represent the control diabetic rats).

Group 2 for 24 hrs., 8 rats were fasted, then intraperitoneal injected by streptozocin (STZ) freshly prepared in 0.1 M citrate buffer, pH 4.5 at a dose of 4.5 mg/100g of body weight to induced diabetes mellitus according to the reported method [42]. Serum glucose level of rats was determined 72 hrs., post-injection rats were fasted for 18 hrs. before determination. Rats with serum glucose levels over 250 mg/dl were considered sufficiently as streptozocin diabetic rats and ready for treated with ZnO and ZnO/PVA.

Group 2 (8 diabetic rats) were divided randomly to 2 series (4 rats for each) as follows:

Series 1: were treated with ZnO, ZnO/5PVA, ZnO/7PVA and ZnO/10PVA with dose 3.5 mg/Kg of body weight.

Series 2: were treated with ZnO, ZnO/5PVA, ZnO/7PVA and ZnO/10PVA with dose 7 mg/Kg of body weight.

By heparinized tubes blood samples were collected from the eye canthus every 5 days after the beginning of extracts administration. Then, each blood sample was centrifuged to obtain clear serum where serum glucose levels for fasting animals were determined immediately.

- All experiments were performed in agreement with the regulations of ‘the Institutional Animal Ethics Committee of Mansoura University, Mansoura, Egypt’, which are in accordance with the ‘Guide for the Care and Use of Laboratory Animals published by ‘the National Academy of Sciences’.
- The study was carried out in compliance with the ARRIVE guidelines

3. Results and Discussions

3.1. X-ray Analysis

The effect of temperatures of calcination and PVA concentrations on the crystal phase and the crystal size of the synthesized samples were determined using patterns of XRD fig. 2.1, 2.2. We found that all samples are typical hexagonal structure (JCPDS card No.36-1451) [28]. No phases for PVA were observed. In fig. 2.1 the intensity of (101), (002) and (100) peaks at $2\theta = (36.1, 34.3 \text{ and } 31.6)$ respectively decreased with increasing PVA content as a result of the increase in crystal deformation. The ZnO peak of diffraction (101) at 2θ (36.1) was used to measure the crystal sizes of the ZnO/PVA samples. By increasing the quantity of PVA from 35-18 nm to 21-18 nm, a decreasing in the crystallinity and the crystal sizes of ZnO was observed. In addition, increasing PVA content led to a decrease in the mean grain sizes because PVA covered the crystalline surface of ZnO, so intensity decreased. Subsequently, reduction in crystals size increases when PVA substitution with Zn^{2+} ions increases [29, 30]. Fig. 2.2 showed that at 300°C the formed peaks were at $2\theta = 25.1$ zinc oxalate hydrate with monoclinic structure and at $2\theta = 37.7$ zinc oxalate with monoclinic structure. By increasing temperature of ZnO/5PVA to 400°C and 500°C, the intensity of (101), (002) and (100) peaks at $2\theta = (36.1, 34.3 \text{ and } 31.6)$ with hexagonal structure respectively increases because the PVA content decreases by increasing temperature and ZnO phase only still.

Fig. 2.1 XRD pattern for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 400 °C

Fig. 2.2 XRD pattern for ZnO/5PVA at calcination a) 300, b) 400 and c) 500 °C

3.2. UV-Vis absorption spectra

Fig. 3 shows the variation of Band gap energy. The values of absorbance were replaced in Kubelka-Munk equation to obtain the $(\alpha h\nu)^2$ values [31]. Extrapolation of linear region of $(\alpha h\nu)^2$ versus $(h\nu)$ for ZnO/PVA films showed that increasing the quantity of ZnO in ZnO/PVA, slightly decreases the band gap of the nanocomposites. The energy of band gap for ZnO (3.25 eV) and unfilled PVA film (4.04 eV) was calculated. The range of band gap energy for ZnO/PVA nanocomposite

films was 3.95 eV to 3.2 eV. The decreasing of band gap energy caused the red shift in the peak of absorption with the increase in ZnO amount. This decrease in band gap energy may be due to charge transfer complex (CTC) formation as trap levels between (HOMO) and (LUMO) bands of PVA, which promotes the lower energy transitions leading to the observed change in band gap [7]. The E_g value of ZnO is (3.27 e.V) and ZnOP is (3.55 e.V). The values of band gap for samples containing PVA were between 3.10 and 3.16 e.V and were less than those of pure oxides.

Fig. 3 Band gap for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 300 °C

3.3. Scanning electron microscope (SEM)

Fig. 4 illustrates SEM analysis of nominated samples of ZnO and ZnO/PVA photo catalysts with various content from PVA (5, 7 and 10 %) which reveals the agglomeration of all samples to some extent. The difference in the particle sizes shown by XRD and SEM analysis for ZnO and ZnO/PVA could be due to particles aggregation [32]. SEM images revealed that increasing PVA content, increases hexagonal phase. The small crystals with various phases were interwoven with each other creating strongly bound nanoclusters [33].

Fig. 4 SEM images for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 400 °C

SEM analysis was also used to ensure distribution and size of ZnO in polymeric matrix. ZnO appeared as white spots. It is obvious that regardless of type of ZnO are well spread in polymeric matrix. This revealed that good adhesion between the surface of the ZnO nanoparticles and the polymeric matrix was demonstrated by modifying the organic surface of the ZnO nanoparticles.

3.4. Acidity Test

Potentiometric titration was utilized to estimate the surface acid sites of the solid catalysts [34, 35]. Neutralization of the surface acid sites is conducted by the volume added of n-butyl amine/g. Evaluation of the electrode potential (mV) as a function of the progressive increasing of the concentration of n-butylamine (mmol / g catalyst) [36]. In fig. 5 by increasing PVA content up to 10%, the total number of acid sites increase. The amount of n-butyl amine/g used for neutralization of the surface acid sites of solid catalysts as a function of PVA content at 500°C and the total number of acid sites/g were tabulated in table 1.

Fig. 5 Acidity curve for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 400 °C

Table 1. PVA content, Volume of n-butyl amine/g and total number of acid sites/g

Sample	ml added	Total № of acid sites/g
ZnO	0.31	1.867×10^{21}
ZnO/5PVA	0.42	2.529×10^{21}
ZnO/7PVA	0.49	2.951×10^{21}
ZnO/10PVA	0.92	5.541×10^{21}

The total number of acid sites of solid catalyst /g was evaluated from the following equation:

Total number of acid sites/g = (ml equivalent/g) $\times$ N $\times$ 1000	
N	Avogadro's number

3.5. Photocatalytic activity

The photo catalyst activity of the prepared ZnO and ZnO/PVA catalysts were evaluated on the degradation of (MB) and (MO) below UV/vis. light [37].

At the beginning, a dry and clean bottle contains 50 mL dye solution (10 ppm) and 0.1 mg/L of photo catalyst was stirred for 30 mins. in the dark. Then the bottle was irradiated by UV/visible light for a period and 1 mL of the mixture was taken out then diluted 10 times and were centrifuged for 15 mins. to settle down the catalyst. The dye concentration was measured using UV/vis. spectrophotometer at certain wavelength according to $\lambda_{\max}$ for each dye (MB = 666 nm & MO = 464 nm). Then the percentage of removal was evaluated from:

$$\% \text{ removal} = \frac{C_0 - C_t}{C_0} * 100$$

C_0 : concentration of dye (mg/L) at the beginning

C_t : concentration of dye (mg/L) at any time (mins.)

3.5.1. Action of PVA content on MB degradation

Fig. 6 Photo degradation of a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 300 °C of MB

Improvement of the activity of a large band gap photo catalyst such as ZnO depends on doping technique as an effective method. Table 1 reveals that ZnO has a lower photocatalytic activity and highest number of acid site than ZnO/PVA. For MB cationic dye the low photocatalytic activity of ZnO probably due to (1) fast electrons and holes recombination (2) participation of small part of these electrons and holes in the photo catalyst reaction [38]. Among ZnO/PVA, ZnO/5PVA is the best doping level to obtain the maximum photo catalyst activity and this refers to the influence of PVA in decreasing the recombination rate and creating a new energy level in ZnO [39]. Photocatalytic activity decreases

by the increase in PVA content beyond 5% as shown in fig. 6. This is due to the action of PVA high content as centers of recombination [40, 41].

3.5.2. Action of temperature of calcination on MB degradation

SEM, XRD and UV–Vis analyses proved that temperature of calcination plays an important function in the band gap and the structure, so the variation of photocatalytic activity is attributed to a variation in calcination temperature. Fig. 7 reveals the photocatalytic activity of ZnO/5PVA of MB cationic dye photo degradation at (300, 400 and 500 °C) calcination. At 300 °C calcination, ZnO/5PVA shows the best photocatalyst activity, SEM and XRD analyses confirmed this result.

Fig. 7 Photo degradation of MB by ZnO/5PVA calcined at a) 300, b) 400 and c) 500 °C

3.5.3 Effect of PVA concentration on MO degradation

Fig. 8 Photo degradation for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 300 °C of MO

Fig. 8 reveals the activity of ZnO with PVA concentrations photocatalyst calcined at 300 °C for MO anionic dye. Among ZnO/PVA, ZnO/7PVA is the best doping level to obtain the maximum photo catalyst activity. Photocatalytic activity decreases by the increase in PVA content beyond 7%.

3.5.4. Action of temperature of calcination on MO degradation

Fig. 9 Photo degradation of MO by ZnO/7PVA calcined at a) 300, b) 400 and c) 500 °C

Fig. 9 reveals the photo catalyst activity of ZnO/7PVA of MO anionic dye photo degradation calcined at 300 °C shows the best photocatalytic activity.

4. Biological activity

4.1 Study the relationship between the following factors for the two series:

Table 2. Serum glucose level at different days and % of PVA for two series.

Levels of :	Treatment period	Normal control	Diabetic control	ZnO dose	
				3.5 mg	7 mg
Serum Glucose at ZnO	Zero time	116.59±03	343.11±08	433.75	431.24
	5 days	117.44±53	365.19±04	400	390
	10 days	117.07±01	386.35±11	377.5	371.71
	20 days	117.21±00	399.63±00	300.01	276.62
Serum Glucose at ZnO/5PVA	Zero time	116.59±03	343.11±08	347.15	346.00
	5 days	117.44±53	365.19±04	320.00	311.65
	10 days	117.07±01	386.35±11	302.21	290.09
	20 days	117.21±00	399.63±00	240.00	210.88
Serum Glucose at ZnO/7PVA	Zero time	116.59±03	343.11±08	468.6	467.5
	5 days	117.44±53	365.19±04	432.2	420.7
	10 days	117.07±01	386.35±11	407.9	391.5
	20 days	117.21±00	399.63±00	324.1	284.6
Serum Glucose at ZnO/10PVA	Zero time	116.59±03	343.11±08	520.5	519.4
	5 days	117.44±53	365.19±04	480	467.3
	10 days	117.07±01	386.35±11	453.6	435
	20 days	117.21±00	399.63±00	360	315

1. Blood glucose level and days at constant PVA content

Fig. 10 Glucose level against days for ZnO, ZnO/5PVA, ZnO/7PVA and ZnO/10PVA calcined at 300 °C for two series 3.5mg and 7mg

From fig. 10 and table 2 reveal that the best result for lowest glucose level is ZnO/5PVA calcined at 300 °C for the two series.

2. Blood glucose level and PVA content at constant days

Fig. 11 Glucose level against PVA content for a) 0, b) 5, c) 10 and d) 20 days calcined at 300 °C for two series 3.5mg and 7mg

From fig. 11 reveals that the best result for lowest glucose level after 20 days is ZnO/5PVA for the two series.

4. Conclusion

ZnO and ZnO/PVA nanoparticles were prepared using sol-gel method. The formation of hexagonal phase of polycrystalline ZnO and ZnO/PVA was confirmed by (XRD). SEM analysis showed that ZnO and ZnO/PVA nanocomposites have a spherical shape. Photocatalytic experiments showed that ZnO/5PVA is the most photocatalytically active MB cationic dye while ZnO/7PVA is the most on MO anionic dye and the best calcination was 300 °C. Potentiometric titration proved that ZnO/10PVA has the highest number of total acid sites. Biological evaluation of ZnO and ZnO/PVA showed that ZnO/5PVA calcined at 300 °C is the best result for decreasing glucose level in diabetic rats. So, loading PVA onto the surface of ZnO improved the photocatalytic and biological properties of pure ZnO.

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Figures

Figure 1

Reaction scheme of ZnO/PVA nanocomposites

Figure 2

2.1 XRD pattern for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 400 °C. 2.2 XRD pattern for ZnO/5PVA at calcination a) 300, b) 400 and c) 500 °C

Figure 3

Band gap for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 300 °C

Figure 4

SEM images for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 400 °C

Figure 5

Acidity curve for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 400 °C

Figure 6

Photo degradation of a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 300 °C of MB

Figure 7

Photo degradation of MB by ZnO/5PVA calcined at a) 300, b) 400 and c) 500 °C

Figure 8

Photo degradation for a) ZnO, b) ZnO/5PVA, c) ZnO/7PVA and d) ZnO/10PVA calcined at 300 °C of MO

Figure 9

Photo degradation of MO by ZnO/7PVA calcined at a) 300, b) 400 and c) 500 °C

Figure 10

Glucose level against days for ZnO, ZnO/5PVA, ZnO/7PVA and ZnO/10PVA calcined at 300 °C for two series 3.5mg and 7mg

Figure 11

Glucose level against PVA content for a) 0, b) 5, c) 10 and d) 20 days calcined at 300 °C for two series 3.5mg and 7mg