BENI-SUEF UNIVERSITY JOURNAL OF BASIC AND APPLIED SCIENCES 5 (2016) 277–283

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Full Length Article

Characterization and photocatalytic properties of cotton fibers modified with ZnO nanoparticles using sol–gel spin coating technique

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ARTICLE INFO

Article history:
Received 23 April 2016
Received in revised form 24 August 2016
Accepted 30 August 2016

Keywords:
ZnO nanoparticles
Cotton
Sol–gel method
Photocatalysis

ABSTRACT

Zinc oxide nanoparticles (ZnO NPs) were prepared using the sol–gel method. Cotton fibers were loaded with ZnO nanoparticles using sol–gel spin coating technique. The prepared ZnO NPs and ZnO-coated cotton were characterized by scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX). The self-cleaning property of ZnO-coated cotton and the photocatalytic removal of methyl orange dye from the contaminated water and cotton fibers were studied by measuring the optical absorbance after exposure to sunlight and Philips 200W lamp illumination. The results showed that the cotton loaded with ZnO nanoparticles could efficiently decompose 73% of methyl orange dye in the sunlight and 30.7% in the lamp illumination after 12 hours. ZnO nanoparticles decomposed methyl orange dye by 92.7% in the sunlight and 26.4% in the lamp illumination after 7 hours.

1. Introduction

Cotton fabrics are widely applied in our daily life because of their excellent properties such as softness, affinity to skin, bio-degradability, and regeneration property (Zhang et al., 2013). The abundant water-absorbing hydroxyl groups on the cotton surface make the fiber absorbent and easily stained by the liquids. Also, environmental and health problems associated with hazardous wastes and toxic water pollutants have attracted much attention (Baruah and Dutta, 2009; Gopal et al., 2007; Sadiq and Rodriguez, 2004). Especially, organic dyes are one of the major groups of pollutants in waste waters that are released from textile and other industrial processes. Dyes and organic chemicals that are emitted from various sources cause considerable problems for

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http://dx.doi.org/10.1016/j.bjbas.2016.08.003

microorganisms, aquatic environments, and human beings, and could be converted into dangerous byproducts through oxidation, hydrolysis, or other chemical reactions occurring in the wastewater. Also, the increased awareness of consumers toward health and hygiene makes it important to find new strategies to remove the hazard dyes from waste water and produce self-cleaning textiles.

During the last decades different physical and biological techniques have been used to remove the hazard dyes, such as adsorption or coagulation, chlorination, sedimentation, filtration and membrane technologies (Ibrahim and Halim, 2008; Padmanabhan et al., 2006; Robinson et al., 2001; Yang and Cheng, 2007). These techniques are of high costs, generate mutagenic and carcinogenic by-products, and merely transfer the non-biodegradable matter into sludge, giving rise to another phase of pollution. So a further treatment of the new pollution phase is needed (Arslan et al., 2000). Recently, there has been considerable interest in the utilization of advanced oxidation processes (AOPs) for the complete destruction of dyes. Heterogeneous photocatalysis is one of the advanced oxidation processes (AOPs) that is very effective in the purification of toxic pollutants presented in the industrial waste water because of its ability to complete mineralization of these pollutants (Kato et al., 2005; Ollis and Turchi, 1990). Advantages of the photocatalytic process include its inexpensive cost; it depends on non-toxic substances (semiconductors) which provide a high surface area, and it can be powered by available light source (sunlight).

Nanostructured materials are attracting significant attention in the textile field and water treatment because of their higher surface area and unique physical and chemical properties compared to bulk materials. Hence, they are suitable for preparing hygienic surfaces and can be used in various applications (Chen and Chiang, 2008; Selvam and Sundrarajan, 2012). Metal oxide semiconductor nanoparticles, as heterogeneous photocatalysts, have attracted much interest due to their size-tunable physical and chemical properties. These materials have longer lifetimes and are chemically stable under extreme conditions such as high temperature or pressure (Martins et al., 2013). As one of the most important semiconductor photocatalysts, nanostructure ZnO has attracted more interests because of its considerable photocatalytic efficiency and good stability (Kalyani et al., 2006; Matei et al., 2008). It has a wide band gap of 3.37 eV with a large exciton binding energy of 60 meV (Zhang et al., 2013). Moreover, ZnO is bio-safe and biocompatible for medical applications (Asif et al., 2011; Broasca et al., 2013). Several works reported the synthesis and high photocatalytic efficiencies of ZnO nanoparticles (Chakrabarti and Dutta, 2004; Curridal et al., 2003; Fotou and Pratsinis, 1996; Hong et al., 2006). The sol–gel method has been receiving significant attention since it enables us to develop low-cost and simple deposition procedure to obtain large-area and high-quality ZnO nanostructures for technological applications.

In this paper, ZnO nanoparticles prepared by sol–gel method are loaded on the cotton fabrics using a spin-coating technique to produce self-cleaning ZnO-coated cotton. The chemical composition, morphological and photocatalytic properties of the fabricated ZnO NPs and ZnO-coated cotton are investigated. Also, the photocatalytic mechanism is discussed.

2. Experimental details

2.1. *Preparation of ZnO nanopowder*

0.2 M of zinc acetate dihydrate was dissolved in 50 ml of methanol and stirred for 30 min to prepare solution A. Sodium hydroxide (0.1 M) was dissolved in 50 ml of methanol and stirred for 1 h to prepare solution B. Solution B was added to solution A dropwise under constant stirring until the pH of the solution reaches 9 after 2 h. Then, the mixture was filtered and dried at 50 °C for further half an hour. Subsequently, the powder was annealed for half an hour at 150 °C.

2.2. *Preparation of cotton coated with ZnO*

2.2.1. *Washing of the cotton*

The cellulosic cotton was washed first with water and detergent at 80 °C for 30 min to remove the impurities such as wax and fat. Then it was washed several times with a large amount of deionized water. They were further cleaned in acetone then in ethanol for 30 min at 60 °C and dried at room temperature for 24 h.

2.2.2. *Preparation of ZnO solution*

The used substances are zinc acetate dihydrate, 2-methoxyethanol, and monoethanolamine as a precursor, solvent, and stabilizer, respectively. The molar ratio of MEA to zinc acetate dihydrate was maintained at 1:1. A clear and homogeneous solution was obtained after stirring at 60 °C for 2 h. The solution was aged for 24 hours at room temperature before the coating process.

2.2.3. *Coating the cotton with ZnO*

For coating the cotton with ZnO, the ZnO NPs solution was spin-coated into cotton fabrics at 1100 rpm for 60 s. Then, ZnO-coated cotton was dried at 50 °C for 30 min in a drier to get rid of the excess 2-methoxyethanol and monoethanol amine residuals. These coating steps were repeated for ten times, and after this process the obtained samples were annealed in a furnace at 150 °C in air for 2 h.

2.3. *Photocatalysis experiment*

The photocatalytic ability of ZnO NPs and the ZnO-coated cotton was tested by decomposition of methyl orange (MO). A solution of 10 ppm methyl orange dye was prepared then divided into 6 beakers, and each beaker contains 100 ml of the dye. The beakers were divided into two categories – one for sunlight illumination and the second for a Philips 200W lamp illumination – and the distance between the lamp and reactor was kept at 30 cm. For each group, three photocatalysts were used for comparison: unloaded cotton (0.4 gram), 0.1 g of ZnO nanoparticles, and ZnO-loaded cotton (0.4 gram). The absorbance of the dye was measured every 1 hour.

2.4. *Samples characterization*

The morphology and chemical composition of ZnO NPs-coated cotton fabrics and the prepared ZnO nanoparticles were

studied by a field emission-scanning electron microscope (FE-SEM, model: Quanta 250) attached with energy dispersive X-ray (EDX) unit. The elemental analysis and quantification of cotton loaded with ZnO nanoparticles were done with EDX. The photocatalytic activity was followed by measuring the absorbance of the dye at 464 nm by UV-visible spectrophotometer (Jenway 6305).

3. Results and discussion

3.1. *Microscopic characterization (SEM images)*

The morphology of ZnO NPs, unloaded cotton, and ZnO-loaded cotton was studied by FE-SEM. The FE-SEM images of ZnO NPs (Fig. 1a,b) showed that they consist of homogeneous and spherical-like shape nanoparticles. ZnO NPs have a diameter that ranges from 12.4 nm to 16 nm, with an average value of about 14.2 nm. This small diameter results in increasing the surface area and hence improves the photocatalytic activity. Fig. 1c illustrated the FE-SEM image of cotton fabric coated with ZnO nanoparticles. ZnO was loaded upon the cotton homogeneously and attached well to the cotton. The good adhesion between ZnO and the fabric is due to the hydroxyl (OH) group presented in the chemical formula of the cotton (inset of Fig. 1c). So ZnO nanoparticles can easily bind to hydroxyl groups through possibly hydrogen bonding (Gardner et al., 2008). The cotton was fully coated with ZnO NPs, which formed a shell around the cotton fibril as compared with the uncoated cotton which has a smooth surface with no particles on its surface as shown in Fig. 1d,e. Furthermore, it could be seen from Fig. 1e that ZnO nanoparticles show a narrow size distribution and the density of particles per unit area is very high. Then these nanoparticles work as a shell around the cotton fiber.

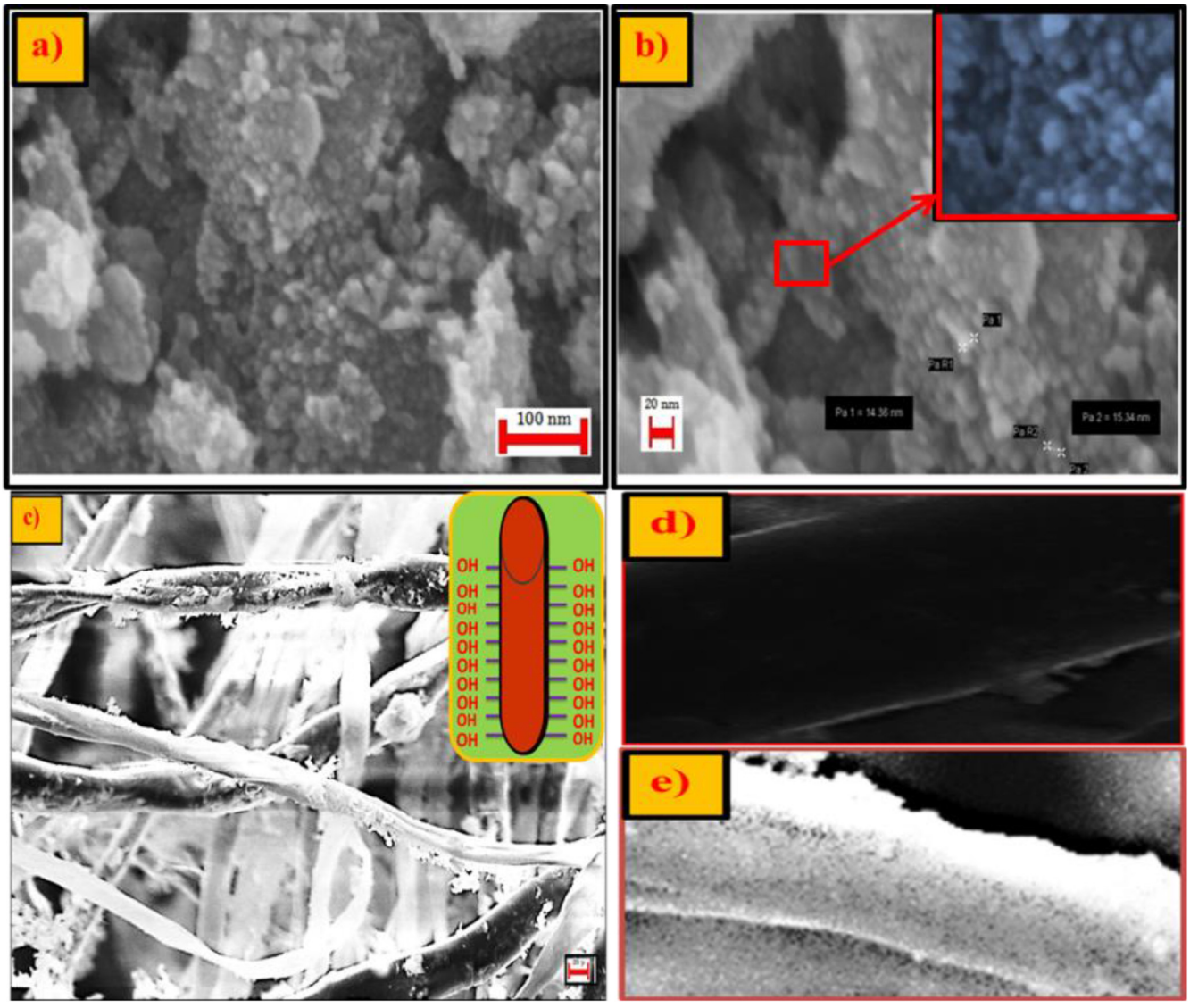

Fig. 1 – FE-SEM images of (a) and (b) ZnO NPs at two different magnifications, (c) and (e) cotton coated with ZnO NPs at two different magnifications, and (d) uncoated cotton. The inset of (c) is a schematic formula of cotton fiber.

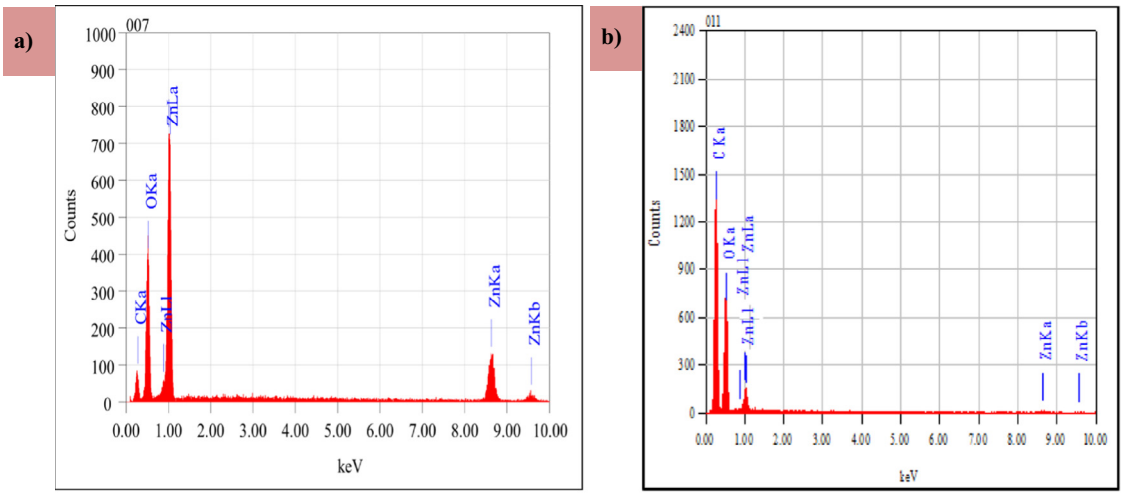

Fig. 2 – EDX spectra of (a) ZnO nanoparticles and (b) cotton loaded with ZnO nanoparticles.

3.2. *Energy dispersive X-ray analysis (EDX)*

The elemental analysis and quantification of ZnO nanopowder and ZnO-coated cotton were investigated by energy dispersive X-ray microanalysis (EDX) as illustrated in Fig. 2. Fig. 2a showed EDX spectrum of ZnO nanoparticles. The result indicates that the powder contains strong zinc and oxygen signals with a weak signal for carbon (C), which may be presented through zinc acetate dihydrate precursor ($Zn(CH_3COO)_2.2H_2O$). There are four peaks relevant to Zn at 0.9, 1.0, 8.6 and 9.6 KeV, and one peak for oxygen at 0.5 KeV. Also, the signal at 0.3 KeV is relevant to carbon. These results imply the high purity of the synthesized Zn NPs.

Fig. 2b showed the EDX spectrum of ZnO-coated cotton. The strong signals from C and O in Fig. 2b are due to the cotton (Xu and Cai, 2008) which consists of a long chain of glucose molecules as illustrated in the inset of Fig. 1c. The presented signals of Zn and O atoms confirm the successful deposition of ZnO in situ cotton fabric samples. There is a strong peak relevant to carbon at 0.3 KeV and O peak at 0.5 KeV due to cotton and three peaks at 0.9, 8.6 and 9.7 KeV relevant to Zn. These test results are in agreement with other research work (Lu et al., 2006).

The quantitative analysis of the EDX spectra shown in Fig. 2 is displayed in Table 1. For the pure ZnO NPs, the ratios of Zn and O signals are 63.97% and 30.3%, respectively, which confirm the formation of ZnO, as shown in Table 1. The presence of the C atom in a trace amount (5.73%) is due to the zinc acetate precursor as illustrated before. In the ZnO coated cotton sample, the highest percent is C atom (93.94%) because of the cellulosic cotton. The percent of Zn (4.19%) and O (1.87%) is low because ZnO is loaded upon the cotton in trace amount.

3.3. *Photocatalytic activity*

The photocatalytic efficiency was expressed in terms of percent of degradation from Eq. (1).

$$\text{Percent of degradation} = \frac{C_0 - C}{C_0} \times 100 \quad (1)$$

where C_0 represents the initial concentration of the dye, C is the final concentration after illumination, A_0 is the initial absorbance, and A is the variable absorbance (An et al., 2014; Fallah et al., 2011). The photocatalytic properties of ZnO NPs, the pristine and ZnO coated cotton were measured through the photodegradation of methyl orange dye under the illumination of 400 W lamp or the direct sunlight. The degradation rate (C/C_0) of MO versus the exposure time in an hour is shown in Fig. 3.

Fig. 3a,b shows the degradation rate versus the exposure time under (a) lamp and (b) sunlight illumination. It can be seen that ZnO nanoparticles showed a photocatalytic activity in the sunlight of 92.7% after 7 hours, which is much higher than their activity under the lamp illumination (26.4%) after the same period. These results may be attributed to that the high band gap energy of ZnO that makes it very efficient in the pres-

Table 1 – Quantitative analysis (chemical composition) of EDX spectra of ZnO and ZnO-coated cotton.

ZnO NPs			ZnO/Cotton		
Element	(keV)	Mass %	Element	(keV)	Mass %
O	0.525	30.3	O	0.525	1.87
C K	0.277	5.73	CK	0.277	93.94
Zn K	8.630	63.97	ZnK	8.630	4.19
Total		100.00	Total		100.00

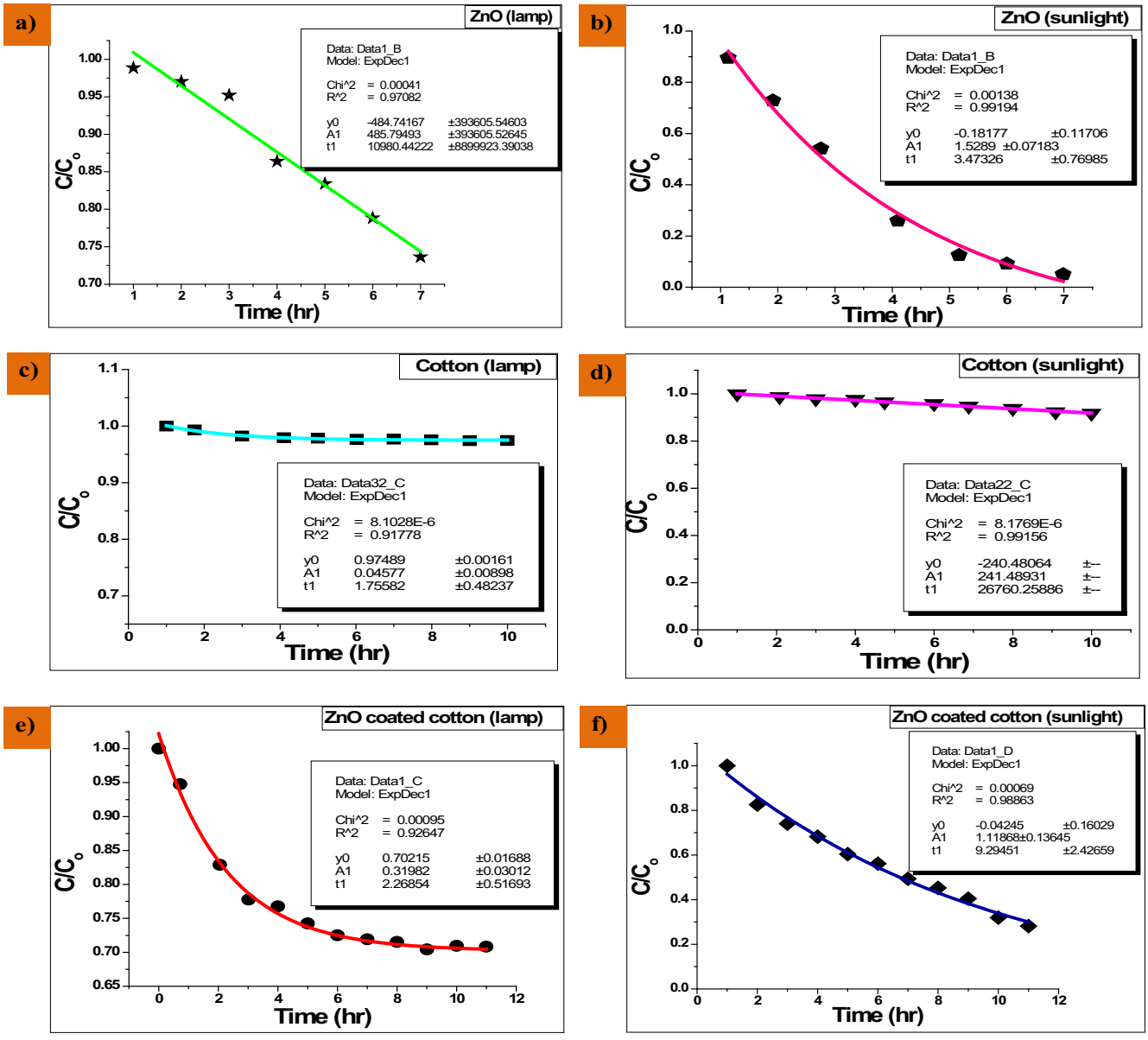

Fig. 3 – The photocatalytic activity measurements of (a, b) ZnO NPs, (c, d) uncoated cotton, and (e, f) ZnO-coated cotton under the lamp (left column) and sunlight (right column) illumination.

ence of UV-light (sunlight) more than the visible light (lamp) (Rauf et al., 2011).

Almost, the uncoated cotton has no photocatalytic activity either under the lamp or in the sunlight as illustrated in Fig. 3c,d. There was a slight difference in the photocatalytic activity after 10 hours between the sample under the lamp (2.5%) and sample in the sunlight (8.7). This very low catalytic activity of the uncoated cotton is attributed to the hydroxyl group presented in the chemical formula of the cotton (inset of Fig. 1c).

ZnO coated cotton showed higher photocatalytic activity than the uncoated cotton under the lamp illumination (30.7%), but the most effective photocatalytic activity of this sample was observed in the sunlight (73%) after the same period (12 hours) as shown in Fig. 3e,f. ZnO coated cotton was more efficient in the sunlight because of the high band gap of ZnO. These results are in agreement with other research works (Hong et al., 2006; Wu et al., 2009; Xu et al., 2010). As shown from Fig. 3, the behavior of degradation rate with exposure time is well fitted with exponential decay equation; $\frac{C}{C_o} = y_o + A_1 e^{-t/t_1}$, where the values of constants y_o, A_1, and t_1 are given inside each figure. Then the curves can be fitted well by exponential decay curves suggesting the first order kinetics. The difference between the photocatalytic activities of pristine, ZnO coated cotton and ZnO NPs is attributed to the amount of ZnO NPs presented in the samples as ZnO NPs decompose the dye through the photo-catalytic mechanism.

3.3.1. *The photocatalytic mechanism*

The irradiation of ZnO particle with photons of energy equal or greater than its band-gap (3.2 eV) results in the transition

of electrons from the valence band (VB) to the conduction band (CB). The result of this process is a region of positive charge termed a hole (h^+) in the VB, and a free electron (e^-) in the CB (Eq. (2)) (Alhamed and Abdullah, 2010). At the ZnO nanoparticle surface, the holes reacted with surface hydroxyl groups (OH^-) or adsorbed H_2O molecules to form $OH^\bullet$ radicals (Eqs. (3) and (4)).

$$ZnO + photon \rightarrow ZnO\,(h^+ + e^-) \quad (2)$$

$$h^+ + OH^- \rightarrow OH^\bullet \quad (3)$$

$$h^+ + H_2O \rightarrow H^+ + OH^\bullet \quad (4)$$

In the absence of electron acceptors the electron–hole recombination is possible according to Eqs. (5) and (6):

$$OH^\bullet + H^+ + 2e^- \rightarrow H_2O \quad (5)$$

$$\tfrac{1}{2}\,O_2 + H^+ + 2e^- \rightarrow H_2O \quad (6)$$

The presence of oxygen prevents this recombination by trapping electrons and forms superoxide ions (Eq. (7)). The final product of the reduction may also be $OH^\bullet$ radical (Eq. (8)) and the hydroperoxy radical $HO_2^\bullet$. The presence of other more powerful electron acceptors than O_2, for example, the hydrogen peroxide, increases the efficiency of the oxidative reaction (Eq. (9)). Hydroxyl radicals have the power to oxidize organic dyes (Kaur and Singhal, 2014; Rincón et al., 2001).

$$e^- + O_2 \rightarrow O_2^{\bullet-} \quad (7)$$

$$2O_2^{\bullet-} + H^+ \rightarrow 2OH^\bullet + O_2 \quad (8)$$

$$H_2O_2 + photon \rightarrow OH^\bullet + OH^- \quad (9)$$

The $OH^\bullet$ is the main oxidant for the degradation of MO molecules in water. The $OH^\bullet$ attaches to the aromatic ring of MO and later finds multiple substitutions. The MO molecules could be degraded through demethylation, which is a process where the N\C bond of the amine group leads to the substitution of a methyl group with a hydrogen atom. The intermediate could be further attacked by $OH^\bullet$, and subsequently could break into smaller molecules generating CO_2 and H_2O at the end of the reaction (Eq. (10)) (Kaur and Singhal, 2014).

$$OH^\cdot + MO \rightarrow \text{degradation products}\,(CO_2 + H_2O)$$

$$h^+ + MO \rightarrow \text{degradation products}\,(CO_2 + H_2O) \quad (10)$$

4. Conclusion

ZnO nanoparticles and cotton coated with ZnO nanoparticles were successfully prepared via sol–gel method. The cotton was coated with ZnO nanoparticles by spin coating technique. The morphologies and chemical compositions of the prepared ZnO NPs and the coated cotton were investigated by SEM and EDX. SEM images showed that spherical-like ZnO NPs of average diameter 15 nm were successfully prepared, and ZnO NPs were loaded on the cotton in a homogeneous manner. The EDX spectrum of ZnO showed Zn and O signals at ratios 63.97% and 30.30%, which confirms a good purity of ZnO NPs. ZnO NPs and ZnO-coated cotton were used as photocatalysts for the removal of methyl orange dye from aqueous solutions under sunlight and a Philips 200W lamp. ZnO NPs showed a high photocatalytic activity that reached 93% using the sunlight and 26.4% using the lamp after 7 hours. The ZnO-coated cotton showed self-cleaning property against methyl orange dye and decomposed the dye by 73% and 30.7% under the sunlight and lamp illumination, respectively.

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