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Degradation of Acid Orange 8 through Photocatalysis in the Presence of ZnO/polyaniline Nanocomposite

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The current study focused on the photocatalytic degradation of acid orange 8 (AO8) from textile wastewater in the presence of a ZnO/polyaniline (ZnO/PANi) nanocomposite. A ZnO/PANi nanocomposite was created using the sol gel method. The loading of ZnO nanosheets on the surface of the PANi layer was studied in terms of morphology and the structure of nanocomposite. The optical band gap of PANi, ZnO, and ZnO/PANi nanocomposite were estimated to be 2.39, 2.62, and 3.07 eV, respectively, indicating that the optical band gap of ZnO/PANi nanocomposite decreased toward ZnO due to the introduction of PANi. EIS studies revealed that the charge transfer resistance of ZnO/PANi nanocomposite was significantly lower than that of pure PANi and ZnO. The photocatalytic treatment of 200 ml of 10 mg/L AO8 solution revealed that total AO8 degradation occurred after 60, 55, and 45 minutes of UV light illumination, and complete AO8 degradation occurred after 70, 60, and 35 minutes of visible light illumination using PANi, ZnO, andZnO/PANi nanocomposite, respectively. Because of its narrow band gap and photoactivation under visible light, the degradation efficiency of ZnO/PANi nanocomposite under visible light illumination was higher than under UV light illumination. The photodegradation performance of PANi, ZnO, and ZnO/PANi nanocomposite for degradation of AO8 solution from real samples (textile wastewater) was investigated, and the results showed that ZnO/PANi nanocomposite has an efficient photodegradation performance for treatment of AO8 in textile wastewater.

Keywords: Photocatalyst; Optical band gap; ZnO/polyaniline nanocomposite; Acid orange 8; Textile wastewater

1. INTRODUCTION

Textile industries pollute the environment by releasing organic and inorganic pollutants into water sources and soils [1, 2]. Pollutants such as dyes, dissolved solids, suspended solids, and toxic

metals abound in textile effluent [3, 4]. It is estimated that dyeing and finishing products used in textile production contribute to global clean water pollution [5, 6]. Washing synthetics releases an estimated 0.5 million tons of microfibers into the ocean each year, causing an increase in mutagenic, carcinogenic, allergic, and cytotoxic agents that endanger all life forms [7, 8].

Many researchers have been looking for an appropriate treatment method to remove dye contaminates from wastewater [9]. The dye removal techniques are adsorption [10], photocatalysis [11, 12], sonication [13], coagulation/flocculation [14, 15], chemical oxidation [16], and ultra-filtration [17]. One issue with these removal techniques is that they perform incompletely destructively and transfer contamination from one phase to the next [18, 19]. As a result, a new and distinct type of pollution has emerged, necessitating additional treatments [20, 21]. Photocatalytic degradation, for example, is a high-quality treatment procedure for removing dissolved organic pollutants like dyes from industrial waste water [22, 23].

Acid orange 8 (AO8: sodium;4-[(2-hydroxynaphthalen-1-yl) diazenyl)-3methylbenzenesulfonate) is an azo dye of the single azo class. It's used to dye wool, silk, cotton, vinegar, polyamide fiber, and fiber, as well as paper, leather dyeing, and organic pigments. Because AO8 is a toxic azo dye with potential toxicity such as carcinogenicity, mutagenicity, and allergies, the waste produced by industries that use it requires extensive research for effluent remediation. As a result, this study focuses on the photocatalytic degradation of AO8 from textile wastewater in the presence of a ZnO/polyaniline nanocomposite.

2. EXPERIMENT

2.1. Synthesis ZnO/PANi nanocomposite

ZnO nanoparticles were synthesized using the sol-gel technique [24]. In a volume-ratio of 5:1, 0.2 M zinc acetate (99.99%, Sigma-Aldrich) was added to methanol (98%, Sigma-Aldrich). To achieve a transparent solution, the mixture was stirred for 30 minutes. The pH of the transparent solution was then adjusted to 9.0 using 1 M NaOH (97.0%, Sigma-Aldrich) aqueous solution, which was stirred for one hour at room temperature to obtain the sol. The sol was then centrifuged for 10 minutes at 1000 rpm to obtain a white precipitate, which was then dried at 90 °C for 120 minutes to yield ZnO nanoparticles. 4g of polyaniline (PANi) powder was ultrasonically dissolved in 100 mL of m-cresol to make the ZnO/PANi nanocomposite (99%, Sigma-Aldrich). The solution was stirred for 180 minutes and then filtered through a 0.22 µm membrane (MF-Millipore™, Merck, Germany). 50 wt% prepared ZnO nanoparticles were mixed with a filtered solution of PANi, and the mixture was stirred for 180 minutes. Finally, the product was dried at room temperature.

2.2. Instruments and characterizations

The X-ray diffraction (XRD) patterns of samples were obtained using a Bruker D8 127 diffraction analyzer (operating at 30 kV and 30 mA, using CuK radiation (λ = 0.15418 nm), D8

advance, USA). Field emission scanning electron microscopy (FESEM) was utilized to survey the morphology of nanostructures. UV–Visible spectroscopy was used to record the UV–Vis absorption aspectrum (Shimadzu UV-1800, Kyoto, Japan). Electrochemical impedance spectroscopy (EIS) measurements were performed on a working electrode (photocatalysts modified Indium tin oxide (ITO) coated glass), a counter electrode (Pt plate), and a reference electrode (Ag/AgCl) using an electrochemical setup (CHI 660E, USA). EIS measurements were taken in a 0.5 M Na₂SO₄ (99%, Merck, Germany) solution over a frequency range of 10^{-1} – 10^{5} Hz with a 5 mV amplitude.

2.3. photocatalytic degradation of AO8

A Pyrex Glass Batch Reactor was used for the photocatalytic degradation experiments. The visible and UV light sources were a xenon lamp (PEAK Instruments (Shanghai) Co., Ltd., China) and a Mercury Curing Lamp (Zhuozhou Xupurui Lighting Source Manufacture Co., Ltd., China). Under magnetic stirring, 20 mg of photocatalysts were dispersed in 200 ml of AO8 aqueous solution for each experiment. The AO8 aqueous solution was made with deionized water for the control samples and textile wastewater for the real sample. The mixtures were magnetically stirred for one hour in the dark before degradation measurements to achieve the adsorption-desorption equilibrium between the photocatalyst particles and AO8 molecules. After certain intervals of irradiation, a given volume of the suspension was filtered through 0.22 µm Millipore membranes to remove the photocatalyst particles, then centrifuged at 10000 rpm for 10 minutes. The irradiated AO8 solution was analyzed by a spectrophotometer (Shimadzu UV-1800, Kyoto, Japan) to evaluate the degradation efficiency of the photocatalyst using the following equation:

Degradation efficiency (%) =
$$\frac{I_0 - I_t}{I_0} \times 100 = \frac{C_0 - C_t}{C_0} \times 100$$
 (1)

Where I_o and I_t are the absorbance intensity of primary and irradiated AO8 solutions, respectively, and C_o (mg/L) is the primary concentration of AO8 dye whereas C_t (mg/L) is the concentration of irradiated AO8 dye.

3. RESULT AND DISCUSSION

3.1. Study of morphology and structure of nanocatalysts

Figure 1 depicts the XRD pattern of PANi, ZnO, and ZnO/PANi nanocomposite powders. The characteristic peaks of PANi at $2 = 15.23^{\circ}$, 20.22° , and 26.17° correspond to the (011), (020), and (200) crystal planes of PANi, respectively, confirming the formation of PANi polymer (JCPDS card no.06-0464) [25, 26]. Diffraction peaks at 31.62° , 34.19° , 35.97° , 47.48° , 56.27° , 63.09° , and 67.61° , respectively, correspond to the typical hexagonal wurtzite structure with (100), (002), (101), (102), (110), (103), and (112) planes, according to the XRD pattern of ZnO. (JCPDS card no.36-1451) [27-29]. XRD patterns of powders of ZnO/PANi nanocomposite shows only three peaks of (100), (002) and (101) plane of ZnO and the lower crystallinity of ZnO compared to pure ZnO sample, indicating

the amorphous nature of PANi may inhibit crystallization of ZnO [30], and successfully synthesized ZnO/PANi nanocomposite by sol-gel method.



Figure 1. XRD patterns of powders of PANi, ZnO and ZnO/PANi nanocomposite.

Figure 2 indicates SEM micrographs of PANi, ZnO, and ZnO/PANi nanocomposite. As can be seen in the SEM micrograph of PANi, it has a well-defined fibrous structure with clusters. The SEM micrograph of ZnO nanoparticles reveals a uniform sheet-shaped nanomaterial with a 100nm average size. The SEM micrograph of the ZnO/PANi nanocomposite shows that ZnO nanoparticles are uniformly distributed across the entire surface of PANi, reducing clusters and pores in the nanocomposite and demonstrating the loading of ZnO nanosheets on the PANi layer's surface.



Figure 2. SEM micrographs of (a) PANi, (b) ZnO and (c) ZnO/PANi nanocomposite

3.2. Optical study of photocatalysts

As shown in Figure 3a, UV-vis absorption spectroscopy was used to investigate the optical properties of the PANi, ZnO, and ZnO/PANi nanocomposite. As observed, the light absorption intensity of PANi toward ZnO is slightly reduced in the UV region but significantly increased in the visible region. Furthermore, the antibonding polaron state (type π - π *) of the benzenoid units is

associated with a specific absorption band at 355 nm in the PANi absorption spectra [31, 32]. The light absorption intensity of ZnO/PANi nanocomposite is significantly increased in both the UV and visible regions compared to ZnO. Furthermore, the absorption edge of the ZnO/PANi nanocomposite is slightly red-shifted, which may be explained by the fact that PANi is a conductive polymer, and the addition of PANi to the ZnO structure introduces an intermediate energy level, increasing the energy of absorbed light [33]. Moreover, the introduction of introduction PANi in nanocomposite causes an increase in the charge carrier concentration, and the intermediate energy gap region between the conduction band and the valence band becomes more active [34, 35]. As consequence, the free electrons can disintegrate easily from the interface of the ZnO/PANi nanocomposite because of the creation of hydrogen bonding between the PANi chain and ZnO molecules. This leads to more contributed in absorption light [36, 37]. Therefore, in the ZnO/PANi nanocomposite under visible light irradiation, the electrons can be transferred to the conductivity band of ZnO nanoparticles from the lowest unoccupied molecular orbital (LUMO) band of PANi molecules [31]. Consequently, ZnO nanoparticles are photoactivated under UV light, and ZnO/PANi nanocomposite is photoactivated under both of UV and visible light irradiation. The optical band-gap (E) of samples may be measured using Kubelka's formula as the following equation [38, 39]:

 $(\alpha hv)^{1/2} = A(hv-E)$ (2)

Where α presents absorption coefficient, hv is photon energy and A is proportionality constant. The optical band-gap can be determined by Tauc plot using the intercepts of the extrapolated linear part plots of $(\alpha h\nu)^{1/2}$ versus photon energy (h ν) as depicted in Figure 3b [38]. The optical band gaps of PANi, ZnO, and ZnO/PANi nanocomposite are estimated to be 2.39, 2.62, and 3.07 eV, respectively, as shown in Figure 3b. Because of the addition of PANi, the optical band gap of the ZnO/PANi nanocomposite decreases toward ZnO, improving the conductivity of the nanostructure and forming new intermediate energy-levels between the valence and conduction bands [40, 41]. It causes the high separation efficiency of photogenerated electron–hole pairs at the ZnO/PANi interface [40, 42].



Figure 3. (a) UV-vis absorption spectra and (b) related Tauc plots of PANi, ZnO and ZnO/PANi nanocomposite.

3.3. Electrochemical study of photocatalysts

The photoelectrocatalytic processes at the photocatalyst/electrolyte interfaces have been better understood using EIS studies. The Nyquist and Bode plots of PANi, ZnO, and ZnO/PANi nanocomposite are shown in Figures 4a and 4b. Figure 5 also shows the equivalent circuit that was used to fit the EIS spectra in order to obtain the physical parameters listed in Table 1. The series impedance (R_s) is related to the nonzero intersection of the semicircle with the real axis at the high frequency side, and ZN is the Nernst diffusion impedance. C_{dl} is the chemical capacitance at the photocatalyst/electrolyte interface. Rct stands for charge transfer resistance and indicates how easily electrons transfer at the electrode/solution interface in the middle frequency range [43-45]. The charge transfer resistance of the ZnO/PANi nanocomposite is significantly lower than that of pure PANi and ZnO, indicating that the loading of ZnO nanosheets on the surface of the PANi layer promotes electron transfer at the photocatalyst/electrolyte interface, resulting in an increase in the photocatalytic reaction rate [46-48]. Additionally, ZnO/PANi nanocomposite exhibits the lower Nernst diffusion impedance, implying good diffusion ability in the electrolyte. The displayed Bode plots in Figures 4b also demonstrate the peak frequency (f_{max}) of ZnO/PANi nanocomposite toward pure PANi and ZnO is shifted to the low frequency.



Figure 4. (a) Nyquist and (b) Bode plots of PANi, ZnO and ZnO/PANi nanocomposite.



Figure 5. The equivalent circuit.

Sample	$R_{s}(\Omega)$	$R_{ct}(\Omega)$	$Z_{N}(\Omega)$
PANi	4.38	1.65	15.59
ZnO	4.37	1.27	12.22
ZnO/PANi	4.28	1.19	6.09

Table 1. The obtained parameters by fitting the EIS spectra with the equivalent circuit.

Electron lifetime (τ_n) can be determined as $\tau_n = 1/(2\pi f_{max})$ [49-51], which implies that f_{max} is inversely associated with the lifetime of an electron. Thus, the lower f_{max} of ZnO/PANi nanocomposite is indicative of a longer electron lifetime and a decrease in photogenerated charges recombination [49, 52, 53]. The EIS results support the optical findings that ZnO/PANi nanocomposite has lower charge resistance and lower electron recombination rate, resulting in higher photodegradation efficiency [54].

4.4. Photocatalytic treatment of AO8

Figure 6a and 6b show the results of a photocatalytic treatment of 200 mL of 10 mg/L AO8 solution using PANi, ZnO, and ZnO/PANi nanocomposite under visible and UV-light illuminations, which were compared to the degradation in the control sample (without photocatalyst). The degradation efficiencies for the control sample are 0.5 percent and 0.1 percent after 90 minutes of UV and visible light illumination, respectively. Meanwhile, after 10 minutes of UV and visible light illumination, all photocatalysts show a significant increase in degradation efficiency. Figure 6a shows that after 10 minutes, PANi, ZnO, and ZnO/PANi nanocomposite show degradation efficiencies of 13%, 22%, and 35%, respectively, and total degradation of AO8 is obtained after 60, 55, and 45 minutes of UV light illumination.

Figure 6b shows that after 10 minutes of visible light illumination, PANi, ZnO, and ZnO/PANi nanocomposite have degradation efficiencies of 12%, 15%, and 45%, respectively, and total degradation of AO8 is obtained after 70, 60, and 35 minutes, respectively. These findings suggest that the ZnO/PANi nanocomposite has a high photocatalytic activity under UV and visible light illuminations. It is related to AO8's faster degradation rate, efficient photogenerated electron-hole separation, and quick charge transfer compared to PANi and ZnO, according to EIS and optical analyses. Furthermore, under visible light illumination, the degradation efficiency of ZnO/PANi nanocomposite is higher than under UV light illumination, which is due to its narrow band gap and photoactivation under visible light.



Figure 6. The results of study of photocatalytic treatment 200 ml of 10 mg/L AO8 solution using PANi, ZnO and ZnO/PANi nanocomposite under (a) UV and (b) visible light illuminations.

Photocatalytic reaction mechanisms for degradation AO8 under UV and visible light illuminations is associated with the conduction band of ZnO which is lower than the highest occupied molecular orbital (HOMO) of PANi [40]. Thereby, PANi could act as an acceptor for photogenerated holes in the nanocomposite photocatalyst [55]. The ZnO conduction band position is lower than LUMO of PANi, so ZnO could act as a sink for the photogenerated electrons in the nanocomposite photocatalysts [40, 42]. Light can excite both ZnO and PANi, resulting in photogenerated carriers and excited electrons, respectively. When PANi is exposed to visible light, a large number of photogenerated electrons may be injected into the conduction band of ZnO. The injected electrons in ZnO's conduction band will then transfer to the photocatalysts' surfaces, where they will react with water and oxygen to produce hydroxyl and superoxide radicals, which are good oxidants for AO8 [56, 57].

The photocatalytic performance of ZnO/PANi nanocomposite for the degradation of AO8 solution with different intimal concentrations was investigated under visible light illumination because of the importance of pollutant concentration in all water treatment processes. Figure 7 shows that as the initial AO8 concentration increases, the photocatalytic degradation efficiency decreases. After 35, 55, 70, and 90 minutes of visible light illumination, complete degradation of 10, 30, 50, and 100 mg/L of AO8 occurs. Table 2 summarizes the photocatalytic performance of PANi, ZnO and ZnO/PANi nanocomposite for AO8 solution degradation and various published photocatalysts for AO8 treatment. As shown in Table 2, ZnO/PANi nanocomposite has a higher photocatalytic efficiency than ZnO/PANi nanocomposite due to efficient photogenerated electron-hole separation and quick charge transfer [58]. Moreover, Table 2 shows the comparison between photocatalytic degradation of AO8 to the other methods (sonochemical, adsorption and Fenton) which indicates the sonochemical method is efficient for high concentrations of AO8 solution, and photocatalytic degradation based on ZnO/PANi nanocomposite shows better efficiency than adsorption and Fenton methods.



Figure 7. Photocatalytic degradation efficiency of ZnO/PANi nanocomposite at various initial concentrations (10, 30, 50 and 100 mg/L) of AO8 under visible light illumination.

Table 2. Comparison the photocatalytic activity of photocatalyst in this study with that of various reported photocatalysts in the literature for the treatment of AO8.

Degradation method	catalyst	AO8 (mg/L)	Light source	Degradation time(min)	Degradation efficiency(%)	Ref.
Photocatalysis	PANi	10	UV	60	100	This
			visible	70	100	work
Photocatalysis	ZnO	10	UV	55	100	
			visible	60	100	
Photocatalysis ZnO/PANi nanocomposite		10	UV	45	100	
		10	visible	35	100	
		30		55	100	
		50		70	100	
		100		90	100	
Photocatalysis	TiO ₂ -P25	91	UV	90	75	[59]
Photocatalysis	Hydrogel/Fe ₂ O ₃	50	UV	60	62.50	[60]
	composite					
Photocatalysis	Immobilized TiO ₂ on	30	UV	210	100	[61]
	glass plates					
Photocatalysis	TiO ₂	19	UV	60	95	[62]
Photocatalysis	ZnO	10	Solar	120	70	[63]
Sonochemical	CCl ₄ and t-butyl alcohol	1000		30	99	[64]
Adsorption	Carbon	500		1440	60	[65]
Fenton	Fe ²⁺ and H ₂ O ₂	9.62		180	98	[66]

The photodegradation performance of PANi, ZnO, and ZnO/PANi nanocomposite for AO8 solution degradation from real sample (textile wastewater) was investigated. The degradation efficacy of 200 mL of 10 mg/L AO8 solution made of real textile wastewater is shown in Figure 8. Total AO8 treatment of a real sample requires 85, 70, and 48 minutes of visible light illumination, respectively, using PANi, ZnO, and ZnO/PANi nanocomposite. When these results are compared to the degradation efficiency in Figure 5b, it is clear that total AO8 treatment takes longer in textile wastewater samples.

It has something to do with other organic and dye pollutants found in textile wastewater. Meanwhile, the results show that the ZnO/PANi nanocomposite is effective at photodegrading AO8 in textile wastewater.



Figure 8. The degradation efficacy of PANi, ZnO and ZnO/PANi nanocomposite for treatment of 200 mL of 10 mg/L AO8 solution made from actual sample under visible light illumination.

4. CONCLUSION

The degradation of AO8 from textile wastewater using photocatalysis in the presence of a ZnO/polyaniline (ZnO/PANi) nanocomposite was presented in this study. The ZnO/PANi nanocomposite was made using the sol gel method. The loading of ZnO nanosheets on the surface of the PANi layer was demonstrated by the morphology and structure of the nanocomposite, and the ZnO/PANi nanocomposite was successfully synthesized using sol-gel technique. As a result of the optical study, the optical band gaps of PANi, ZnO, and ZnO/PANi nanocomposite were estimated to be 2.39, 2.62, and 3.07 eV, respectively. Because of the addition of PANi, the optical band gap of the ZnO/PANi nanocomposite decreased toward ZnO, improving the conductivity of the nanostructure and forming new intermediate energy-levels between the valence and conduction bands. It causes the high separation efficiency of photogenerated electron-hole pairs at the ZnO/PANi interface. The charge transfer resistance of the ZnO/PANi nanocomposite was significantly lower than that of pure PANi and ZnO, indicating that the loading of ZnO nanosheets on the surface of the PANi layer promotes electron transfer at the photocatalyst and electrolyte interface, resulting in an increase in the photocatalytic reaction rate. Total AO8 degradation was obtained after 60, 55, and 45 minutes of UV light illumination, and complete AO8 degradation was obtained after 70, 60, and 35 minutes of visible light illumination using PANi, ZnO, and ZnO/PANi nanocomposite, respectively. Because of its narrow band gap and photoactivation under visible light, the degradation efficiency of ZnO/PANi nanocomposite under visible light illumination was higher than UV light illumination. The photodegradation performance of PANi, ZnO, and ZnO/PANi nanocomposite for degradation of AO8 solution from a real sample (textile wastewater) was investigated, and the results revealed that ZnO/PANi nanocomposite is effective for AO8 treatment in textile wastewater.

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