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Research Article

Photooxidation Contribution Study on the Decomposition of Azo Dyes in Aqueous Solutions by VUV-Based AOPs

Chih-Ming Ma,¹ Gui-Bing Hong,¹ Hua-Wei Chen,¹ Nguyen-Thi Hang,²
and Yung-Shuen Shen³

¹Department of Cosmetic Application and Management, St. Mary's Medicine Nursing and Management College, No. 100, Lane 265, San-Shing Road, Sec. 2, San-Shing Shiang, Yi-Lan 266, Taiwan

²Department of Environmental Engineering, Da-Yeh University, No. 168, University Road, Dacun, Changhua 512, Taiwan

³Holistic Education Center, Mackay Medical College, No. 46, Sec. 3, Jhong-Jheng Road, San-Jhih, Taipei City 252, Taiwan

Correspondence should be addressed to Yung-Shuen Shen, ysshenn@mmc.edu.tw

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The effects of pH value, VUV intensity, initial dye concentration, initial H₂O₂ concentration, and TiO₂ loading dose on the degradation of three azo dyes: acid Orange 8, acid Blue 29, and acid Blue 113 were studied to explore and compare the treatment efficiencies among the adopted AOPs. It was found that pH played an important role in the degradation of dyes using VUV irradiation. For VUV/H₂O₂, VUV/TiO₂, and VUV/TiO₂/H₂O₂ processes, the decoloration rates of the three azo dyes were more efficient under acidic conditions relative to alkaline conditions. The degradation rates of dyes increased with increasing concentrations of H₂O₂, but reaction rates were retarded at high concentrations of H₂O₂ because the H₂O₂ compound acted as a scavenger of the hydroxyl radical. In this paper, three azo dyes were decomposed efficiently by VUV irradiation only demonstrating the effectiveness of VUV direct photolysis.

1. Introduction

Environmental protection has been a globally important issue for the past few decades. Environmental data show that industrial wastewater mainly consists of heavy metals, volatile organic compounds (VOCs), cyanide, COD, oil, suspended solids, and sulfides. Dyes are one of the major sources of pollutants and extensively used in the textile industry, photocatalytic industry, and in photochemical applications [1]. Treatment of colored wastewaters produced by textile and other industries is a serious problem that has attracted the attention of many researchers during last few decades. In general, treatment methods of wastewater containing dyes can be divided into two main groups: (1) chemical or physical dye removal methods, which refer to the process called decoloration and (2) dye removal by means of biodegradation. Physical methods of decoloration include different precipitation methods (coagulation, flocculation, and sedimentation), adsorption (on activated carbon, biological

sludge, and silica gel), filtration, reverse, and osmosis. Chemical processes involved in chemical dye removal methods include reduction, oxidation, complexometric methods, ion exchange, and neutralization. Biological treatment can be conducted aerobically or anaerobically.

Practically speaking, each dye removal method has disadvantages. Physical methods are usually nondestructive, requiring expensive posttreatment processing. Chemical treatment methods using strong oxidants such as chlorine or ozone successfully treat dye wastes, but are also expensive due to high costs associated with the chemicals. Biological treatment methods do not effectively treat synthetic dyes because synthetic dyes are highly resistant to aerobic degradation. A chemical method has been applied and has produced interesting results in the treatment of wastewater containing dye stuffs called advanced oxidation processes (AOPs). In recent years, the use of AOPs technology to remove organic pollutants from air or wastewater has received great attention [2–8]. These methods are an application of UV radiation

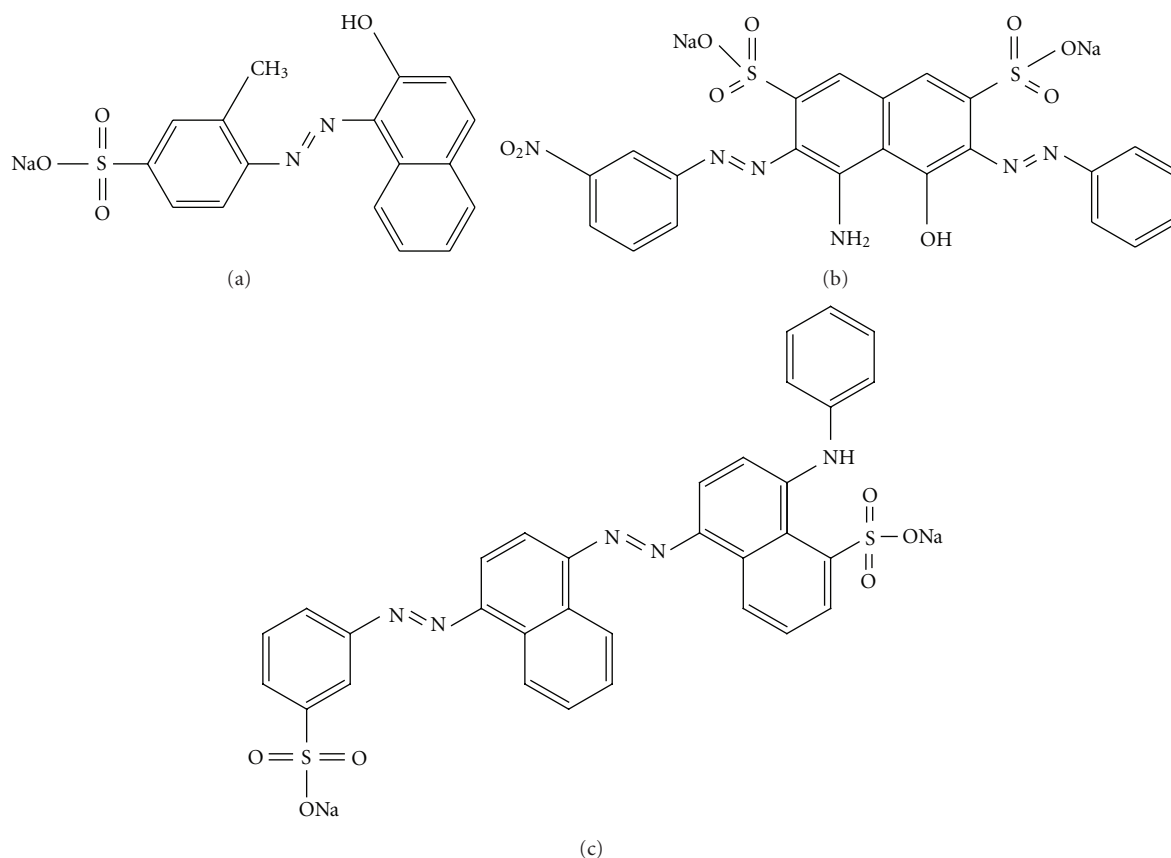
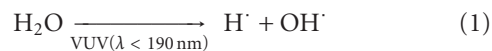


FIGURE 1: Chemical structure of (a) acid Orange 8; (b) acid Blue 29; (c) Blue 113.

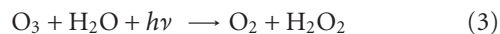
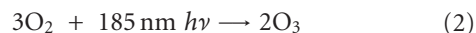
in the presence of some oxidants which can generate strong oxidable radicals to decompose pollutants in the solution.

During the last decade, chemical organic wastewater decomposition techniques have been developed. Among these techniques, UV irradiation in combination with powerful oxidants (e.g., O_3 and H_2O_2) may be a promising method for effectively removing color and refractory organics from effluent. This method does not generate chemical sludge, and the toxicity of wastewater is generally reduced. A considerable disadvantage to the degradation processes introduced above is the need to add external agents into the aqueous medium. In such situations, the effectiveness of the processes relies on solid-liquid and gas-liquid mass transfer that sometimes limits the degradation processes. The development of novel vacuum light (VUV) sources over the last few years has opened up new possibilities for in-situ generation of hydroxyl radicals (OH^\cdot) [9–11]. Hence, the vacuum-UV photolysis of water (H_2O -VUV) is still a field of active research compared to other AOPs [12]. As exhibited in (1), the special requirements of the VUV photolysis of water are related to the formation of a high local concentration of hydroxyl radicals (OH^\cdot) and a series of other species within a photochemical reaction zone of less than 0.1 mm [13, 14]. The high energy UV light causes the homolysis of water into hydroxyl radicals and hydrogen molecules. Light sources that can support this process include “ozone-producing” low-pressure mercury lamps (emitting at 185 nm) and the

Xe excimer lamp (emitting at 172 nm). The depth of this reaction volume is defined by the high absorption cross-section of water at the wavelength

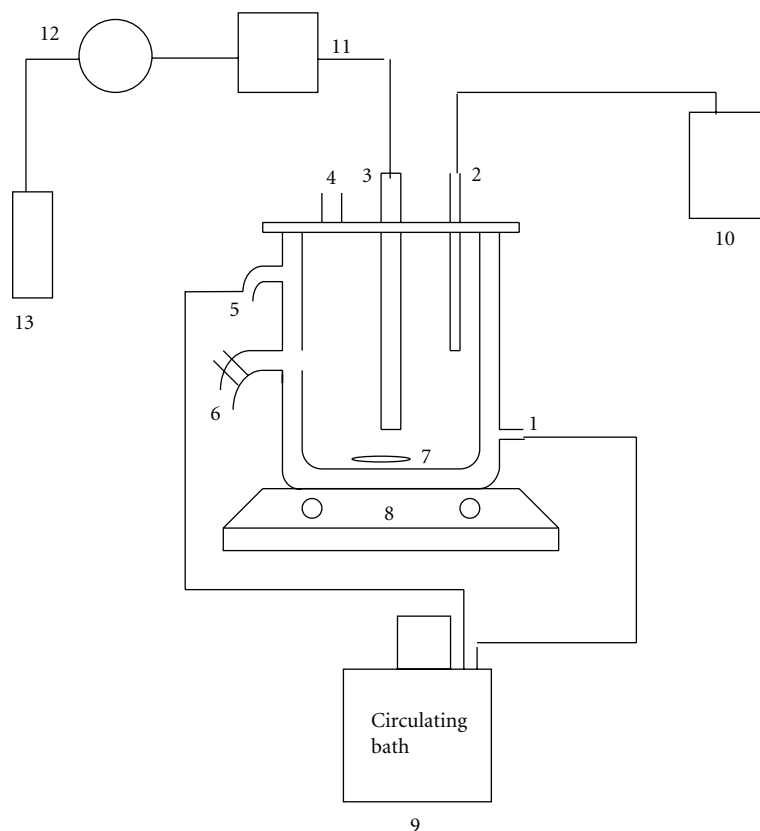


Oxygen molecules in an aqueous solutions can be excited by VUV using a wavelength ranging between 140 nm to 190 nm to generate hydroxyl radicals as shown bellow [15]:



The VUV irradiation of contaminated water provides a simple technique for allowing the oxidation and mineralization of water contaminants without the addition of supplementary oxidants. For example, in the semiconductor industry, VUV irradiation has been used to treat ultrapure water. However, research is still necessary to fully understand principles associated with the VUV-induced water treatment photochemical process.

Moreover, many researchers agree that titanium dioxide, at present, is one of the better photocatalysts for environmental applications. Relative to other photocatalysts, titanium dioxide has advantages such as a high activity high



- | | |
|--|-----------------------------------|
| (1) Input of water to control temperature | (8) Magnetic stirrer |
| (2) pH controller | (9) Circulating bath |
| (3) VUV lamp | (10) pH meter |
| (4) Input of pH adjusting solution | (11) Pen ray power supply |
| (5) Output of water to control temperature | (12) Variable voltage transformer |
| (6) Sampling port | (13) Electricity |
| (7) Stir bar | |

FIGURE 2: The schematic diagram of the reactor system.

stability to light illumination, low cost and nontoxicity [16]. For these reasons, there are much application and development of TiO_2 photocatalytic technique [17, 18]. In this study, TiO_2 is used to treat azo dye compounds in aqueous solutions in this paper. The aim of this paper is to investigate reaction behaviors associated with the decomposition of dye-containing wastewaters by VUV-based advanced oxidation processes (AOPs). The effects of pH value, VUV intensity, initial dye concentration, initial H_2O_2 concentration, and TiO_2 loading dose on the degradation of three azo dyes: acid Orange 8, acid Blue 29, and acid Blue 113 were studied to explore and compare the treatment efficiencies among the adopted AOPs.

2. Methods and Materials

To differentiate the effects of the VUV intensity, initial dye concentration, initial H_2O_2 concentration, and TiO_2 loading

dose on the degradation of three azo dyes, experiments were conducted and studied in the following systems: (1) 185 nm VUV direct photolysis system, (2) 185 nm VUV/ H_2O_2 system, (3) 185 nm VUV/ TiO_2 system. Each system employed in this research contained one batch annular photoreactor. The outer tube of the annular photoreactor was made entirely of Pyrex glass with an effective volume of 2.0 L and was water-jacketed to maintain constant solution temperature at 25°C . The low-pressure mercury 185 nm vacuum-UV lamp was inserted directly into the reactor at the center. The light intensity of the UV lamp was kept constant with approximately 12 watts maximum output. The solution pH value was kept manually constant at desired levels using NaOH and H_2SO_4 solutions. The dyes and H_2O_2 and other chemicals used were reagent grade, and all experimental solutions were prepared with deionized water. In this study, three azo dye compounds were used and molecular structure is shown in Figure 1.

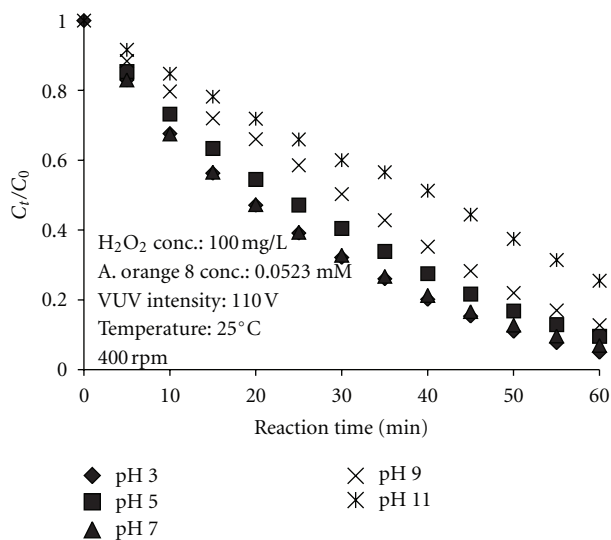


FIGURE 3: The degradation of acid Orange 8 at different pH in aqueous solutions by VUV/ H_2O_2 process.

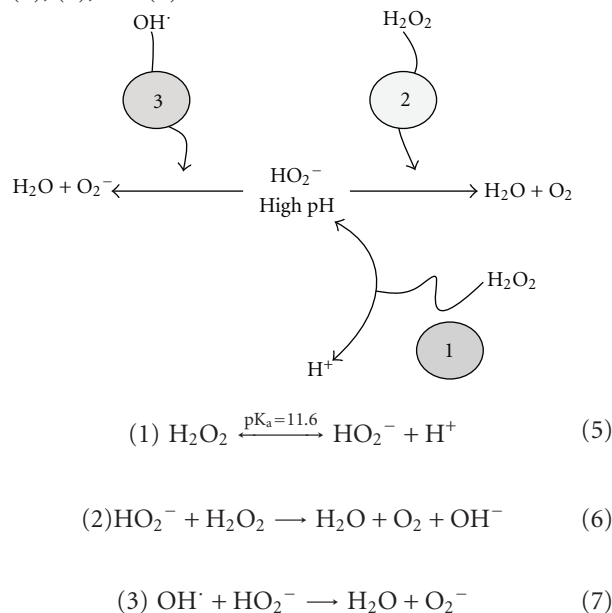
The solution of dyes was added to the reactor with a predetermined amount of H_2O_2 solution. Typical reaction runs lasted 60 min. At desired time intervals, aliquots of solution were withdrawn from the sampling port, which was located at the bottom of the reactor, and analyzed for dyes and H_2O_2 concentrations. Total sample volumes were kept below 2% of the total reactor volume. Each run of the experiments in this work was replicated twice. The standard deviation of the concentration of dyes was analyzed to be ± 0.1 mg/L, respectively. The concentration of H_2O_2 in the aqueous solution was determined by the KI titration method. The UV light absorbances of reacting solutions were detected by a HITACH U-2000 UV/Visible spectrophotometer.

All experiments considering pH as an effect lasted in 60 minutes for photodestruction of three azo dyes in various pH. The solutions of dye were adjusted to the desired pH by adding H_2SO_4 or NaOH. Additions of H_2SO_4 and NaOH were minimized to avoid changing the volume of the reaction mixture. All runs were carried under the same conditions: 0.0523 mM of initial dye concentration, 100 mg/L of H_2O_2 concentration, 25°C of reaction temperature, and 400 rpm of stirring speed. Reactions were assessed at five pH levels: 3, 5, 7, 9, and 11. In VUV/ TiO_2 or VUV/ TiO_2 / H_2O_2 experiments, the dye solution was placed in the reactor and mixed with desired concentration of TiO_2 powder such as 0.5 g/L by the magnetic stirrer in 10 min. The schematic diagram of the reactor system employed in this research is shown in Figure 2.

3. Results and Discussion

3.1. pH Effect on the Degradation of Three Azo Dyes in Aqueous Solution by VUV/ H_2O_2 Process. The decomposition of acid Orange 8 by VUV/ H_2O_2 process at different pH values is shown in Figure 3. Results indicated that the photolytic dye degradation appeared to be best at acidic conditions for acid

Orange 8. This results match findings of Shen and Lin [19]. Photolytic dye degradation of acid Blue 29 and acid Blue 113 was best at a neutral pH, which matches findings of AlHamedi et al. [20]. According to the results of Alhamedi et al. [20] and Daneshvar et al. [21], hydrogen peroxide will be deprotonated with the formation of H_2O_2/HO_2^- equilibrium under alkaline conditions. The HO_2^- species reacts with nondissociated molecules of H_2O_2 according to (6), which leads to oxygen and water, instead of producing hydroxyl radicals under VUV irradiation. Therefore, the instantaneous concentration of OH^\cdot is lower than expected: see (5), (6), and (7)



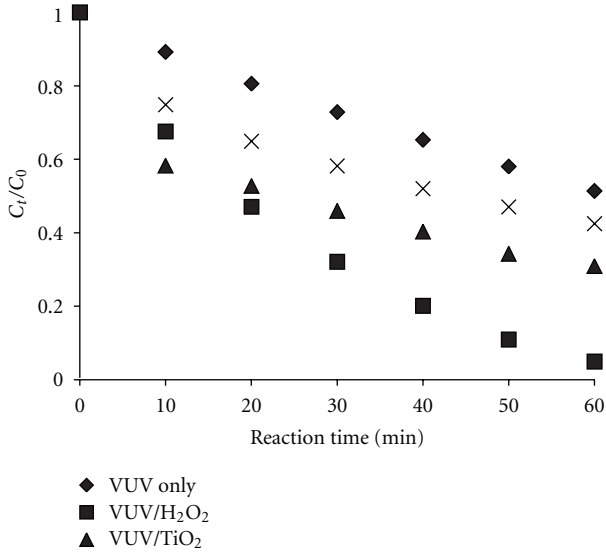
The observed first-order reaction k values at different pH in the degradation of three azo dyes by VUV/ H_2O_2 process are given in Table 1.

Based on the above findings, it was recognized that at the same dye concentration of 0.0523 mM, acid Orange 8 was decomposed more rapidly relative to acid Blue 29 and acid Blue 113. Decomposition reached approximately 90% after 60 min of irradiation time, whereas only approximately 50% of acid Blue 29 and 60% of acid Blue 113 were decomposed in the same irradiation time. In terms of reaction rate, the results are ordered as follows: acid Orange 8 > acid Blue 29 > acid Blue 113 at pH 3, acid Orange 8 > acid Blue 113 > acid Blue 29 at pH 7 and pH 11.

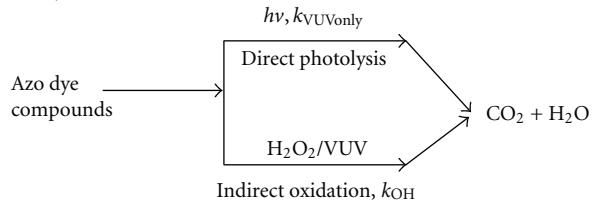
Azo dye compounds can be decomposed simultaneously by VUV direct photolysis and hydroxyl radical indirect oxidation in aqueous solutions by VUV/ H_2O_2 process to generate some organic intermediates. The final products of carbon dioxide and water were formed. Decomposition of pollutants in aqueous solutions by the VUV/ H_2O_2 process was primarily attributed to the competing reaction, direct photolysis, and the hydroxyl radical attack. The position pathway of the three azo dyes by the VUV/ H_2O_2 process is similar to results of Shen and Lin [19]. Assuming that both direct photolysis and indirect oxidation of the three azo dyes were pseudo-first order, the removal rate constant for free radical destruction (k_{OH^\cdot}) can be determined by

TABLE 1: k values at different pH in the degradation of three azo dyes by VUV/ H_2O_2 process.

pH	Acid Orange 8		Acid Blue 29		Acid Blue 113	
	k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2
3	0.0474	0.9779	0.0102	0.9993	0.0081	0.9977
5	0.0377	0.9822	0.0090	0.9962	0.0133	0.9978
7	0.0430	0.9907	0.0124	0.9985	0.0175	0.9994
9	0.0331	0.9619	0.0144	0.9986	0.0104	0.9928
11	0.0213	0.9659	0.0097	0.9751	0.0168	0.9883

FIGURE 4: The degradation of acid Orange 8 under VUV irradiation at acidic condition, $[Dye] = 0.0523$ mM, $[H_2O_2] = 100$ mg/L, $[TiO_2] = 0.5$ g/L, VUV intensity = 110 V, pH 3, 25°C, 400 rpm.

difference between the rate constant of VUV/ H_2O_2 treatment (k_{VUV/H_2O_2}) and the rate constant of VUV system alone ($k_{VUV\text{only}}$) ((8) and (9)).



$$-r_{VUV/H_2O_2} = -\frac{dC}{dt} = k_{VUV/H_2O_2}C = k_{VUV\text{only}}C + k_{OH} \cdot C \quad (8)$$

$$k_{OH} = k_{VUV/H_2O_2} - k_{VUV\text{only}} \quad (9)$$

The pseudo-first-order decomposition rate constants of Acid Orange 8 by direct photolysis and the VUV/ H_2O_2 process are summarized in Table 2. The contribution to the decomposition of Acid Orange 8 by hydroxyl radical destruction was clearly decreased with increasing solution pH values. At acidic and neutral conditions, the contribution to the decomposition of the three azo dyes was higher

than at the alkaline indirect oxidation process. Acidic and alkaline conditions failed to create significant differences in the indirect oxidation of acid Blue 113. The photon absorptions of acid Blue 113 ($\epsilon_{\text{Blue113}}$) at acidic, neutral, and alkaline condition are close together: 0.0563, 0.0593, and 0.040, respectively. On the other hand, direct photolysis was the most important contribution for the removal of azo dyes in high-alkaline solutions.

3.2. Comparison of the Degradation of Azo Dyes in Aqueous Solution by Various AOPs.

The degradation of Acid Orange 8 by the four processes is shown in Figure 4. In terms of decomposition rate efficiency, the Acid orange 8 degradation processes are ordered as follows: VUV/ H_2O_2 > VUV/ TiO_2 > VUV/ TiO_2 / H_2O_2 > VUV only process. No significant decomposition was observed when adding H_2O_2 into VUV/ TiO_2 process or adding TiO_2 into VUV/ H_2O_2 process under acidic conditions. The highest treatment efficiency of Acid Orange 8 was approximately 95% after 60 min using a VUV/ H_2O_2 process. Treatment efficiency associated with the VUV/ TiO_2 / H_2O_2 process was the lowest. This result may possibly be due to a slowing in the generation of hydroxyl radicals due to effects associated with excess amounts of TiO_2 and H_2O_2 . The pseudo-first-order rate constants (k_{app} , min^{-1}) of the decomposition of dyes at various pH values by VUV/ TiO_2 and VUV/ TiO_2 / H_2O_2 process are determined and summarized in Table 3. Table 4 displays the summarized results for comparison among treatment efficiencies of dyes by various AOPs and illustrates differences of decomposition rates associated with the three dyes and AOPs. To evaluate oxidation efficiencies of the H_2O_2 and TiO_2 oxidants used in this study, the ratio between reaction rate constants (k) at different pH levels with doses of H_2O_2 and TiO_2 was calculated, as presented in Table 5. The ratio $k/[H_2O_2]$ was always larger than the ratio $k/[TiO_2]$. It was found that the oxidation efficiency associated with H_2O_2 processes was larger than those with TiO_2 processes and that the AOP decomposition rate of dyes in H_2O_2 processes was faster than those using TiO_2 (i.e., $k/[H_2O_2] > k/[TiO_2]$). According to Liu et al. [22], these azo dyes contain the sodium ion which is easily dissociated in water and leaves the dye molecule with negative charges, so it makes the system easier to be adsorbed on the surface of the catalyst to proceed reaction at lower pH value. Keiichi et al. [23] also found that these dyes are negatively charged due to sulfonate group and hydroxyl group; thus positively charged TiO_2 surface adsorbed more azo dyes at lower pH value, and more decomposition was

TABLE 2: The summary of the pseudo-first-order decomposition rate constant, k (min^{-1}), of Acid Orange 8 by direct photolysis and the VUV/ H_2O_2 process.

pH	Direct photolysis			Indirect oxidation		$k_{\text{VUV}/\text{H}_2\text{O}_2}/k_{\text{VUVonly}}$ (%)
	$k_{\text{VUV}/\text{H}_2\text{O}_2}$	k_{VUVonly}	Contribution (%)	k_{OH^\cdot}	Contribution	
3	0.0474	0.0108	22.78	0.0366	77.22	4.39
5	0.0377	0.0106	28.12	0.0271	71.88	3.56
7	0.0430	0.0135	31.40	0.0295	68.60	3.19
9	0.0331	0.0119	35.95	0.0212	64.05	2.78
11	0.0213	0.0146	68.55	0.0067	31.45	1.46

TABLE 3: The k_{app} (min^{-1}) values at different pH in both VUV/ TiO_2 and VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$ process.

pH	Acid Orange 8		Acid Blue 29		Acid Blue 113	
	VUV/ TiO_2	VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$	VUV/ TiO_2	VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$	VUV/ TiO_2	VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$
3	0.0148	0.0120	0.0086	0.0115	0.2469	0.2729
5	0.0151	0.0108	0.0065	0.0079	0.0324	0.0248
7	0.0141	0.0103	0.0044	0.0033	0.0068	0.0064
9	0.0129	0.0091	0.0044	0.0027	0.0061	0.0065
11	0.0096	0.0075	0.0044	0.0037	0.0060	0.0062

TABLE 4: Summarized result for comparison among treatment efficiencies of dyes by various AOPs.

Items	Acid Orange 8	Acid Blue 29	Acid Blue 113
Condition	[Dye] = 0.0523 mM, [H_2O_2] = 100 mg/L, [TiO_2] = 0.5 g/L, VUV intensity = 110 V, 25°C, 400 rpm		
pH 3	VUV/ H_2O_2 > VUV/ TiO_2 > VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$ > VUV only	VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$ > VUV/ TiO_2 > VUV/ H_2O_2 > VUV only	VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$ > VUV/ TiO_2 > VUV/ H_2O_2 > VUV only
pH 7	VUV/ H_2O_2 > VUV/ TiO_2 > VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$ > VUV only		
pH 11	VUV/ H_2O_2 > VUV only > VUV/ TiO_2 > VUV/ $\text{TiO}_2/\text{H}_2\text{O}_2$		

TABLE 5: The comparison of oxidation efficiency.

Dye	pH	VUV/ H_2O_2 process		VUV/ TiO_2 process	
		k (min^{-1})	$k/[\text{H}_2\text{O}_2]$ ($\text{min}^{-1} \text{mM}^{-1}$)	k_{app} (min^{-1})	$k_{\text{app}}/[\text{TiO}_2]$ ($\text{min}^{-1} \text{mM}^{-1}$)
Acid Orange 8	3	0.0474	0.01612	0.0148	0.00236
	5	0.0377	0.01282	0.0151	0.00241
	7	0.043	0.01462	0.0141	0.00225
	9	0.0331	0.01125	0.0129	0.00206
	11	0.0213	0.00724	0.0096	0.00153
Acid Blue 29	3	0.0102	0.00347	0.0086	0.00137
	5	0.009	0.00306	0.0065	0.00104
	7	0.0124	0.00422	0.0044	0.00070
	9	0.0144	0.00490	0.0044	0.00070
	11	0.0097	0.00330	0.0044	0.00070
Acid Blue 113	3	0.0081	0.00275	0.2469	0.03945
	5	0.0133	0.00452	0.0324	0.00518
	7	0.0175	0.00595	0.0068	0.00109
	9	0.0104	0.00354	0.0061	0.00097
	11	0.0168	0.00571	0.006	0.00096

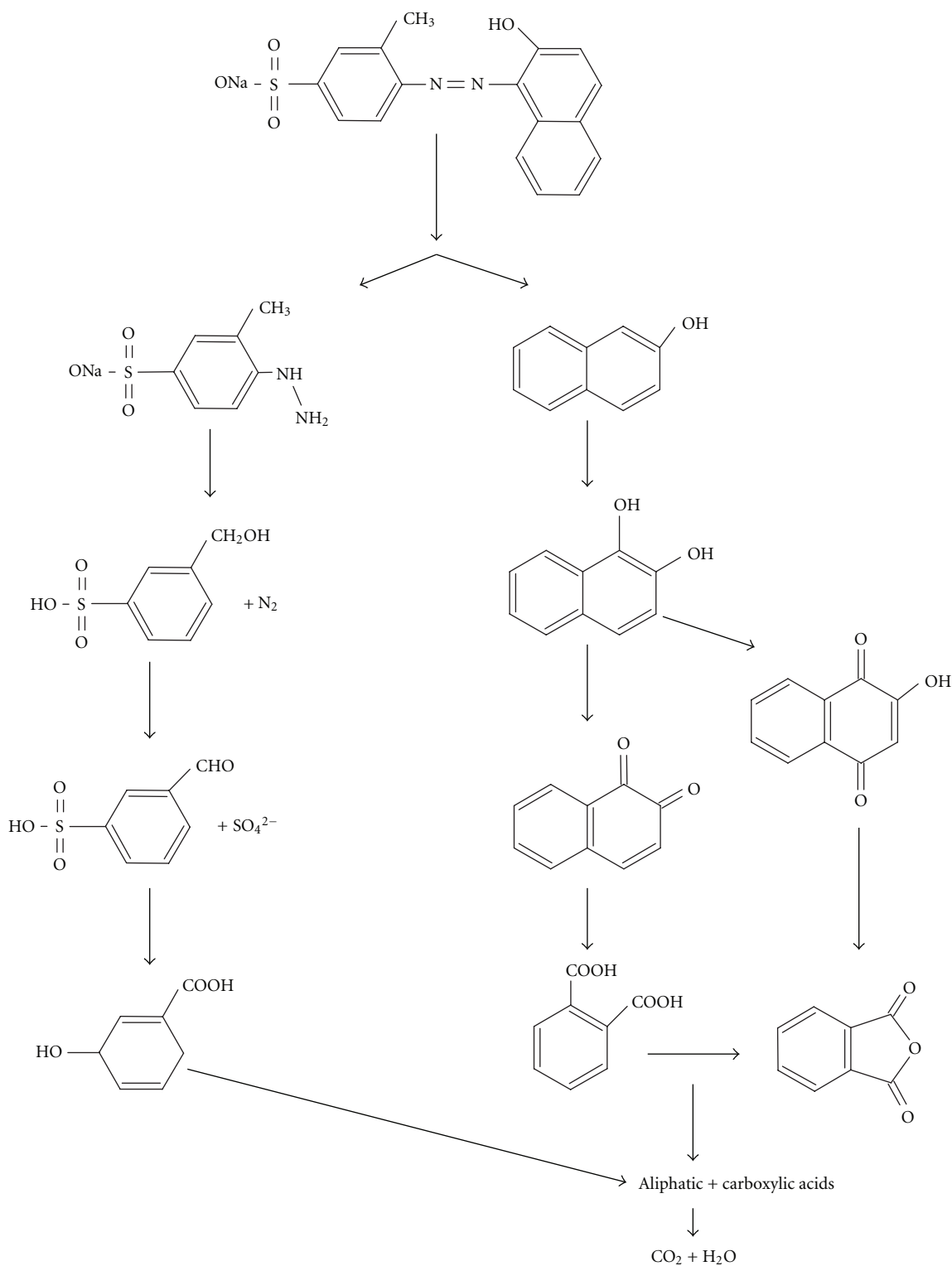


FIGURE 5: Reaction pathway of photocatalytic degradation of acid Orange 8.

achieved. By using the pathway modeling of acid Orange 7 in the research of Konstantinou [16], the photodegradation mechanism of acid orange 52 in the research of Galindo et al. [24], and the degradation mechanism of Reactive Orange 4 in the research of Muruganandham et al. [25], the

degradation pathway of acid Orange 8 was tried to model based on above models, as shown in Figure 5. However, the model for pathway degradation of acid Blue 29 and acid Blue 113 was not shown in this study because of their complicated mechanism and structure.

4. Conclusions

The development of novel vacuum light sources over the last few years has opened up new possibilities for in situ generation of hydroxyl radicals. Hence, the vacuum-UV photolysis of water is still a field of active research compared to other AOPs. In this study, the results obtained have shown that the VUV photolysis, VUV/TiO₂, and VUV/H₂O₂ processes were capable of efficiently decomposing dye wastewaters. For VUV-based AOPs, the decomposition rates of dyes were significantly raised compared with those by VUV direct photolysis. The individual contribution to the decomposition of dyes by direct photolysis and free hydroxyl radicals destruction generated from the excitement of O₂, H₂O, and H₂O₂, respectively were differentiated by the proposed conceptual model in the VUV/O₂ and VUV/H₂O₂ systems. The relative contribution to the decomposition of dyes by OH[·] indirect oxidation generated from the photolysis of various oxidants (O₂, H₂O, and H₂O₂) was found to be dependent on the solution pH values. The decomposing rates of dyes decreased by increasing the molecular size of dyes for all of the VUV-based AOPs.

Acknowledgment

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