

Optimization of Photocatalytic Degradation of Phenol Using Simple Photocatalytic Reactor

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Abstract

The phenol photocatalytic degradation was investigated using heterogeneous catalyst Ag-doped ZnO nanowires under UV irradiation. Ag-ZnO nanowires were immobilized on borosilicate glass via a simple hydrothermal technique. Preliminary photodegradation studies were performed with Ag-ZnO nanowires at various concentrations of phenol (10 - 60 mg/L) at undiluted pH. After determination of the optimal initial concentration (30 mg/L), additional parameters including pH and light intensity were investigated to optimize photodegradation of phenol for large-scale application. The experimental results illustrate that the kinetics of degradation of phenol are pseudo-first order. Based on the relationship, experimental model and empirical correlation were generated and compared for validity. The experimental data were found to fit a cubic model (linear in UV irradiation intensity, I , and cubic in pH), over ranges of 10 - 60 W (UV lamp power) and 2.7 - 11.0 (pH) with a coefficient of determination (R^2) of 0.9934. This model, of the form $K(I, \text{pH}) = c_{00} + c_{10}I + c_{01}\text{pH} + c_{11}I\text{pH} + c_{02}\text{pH}^2 + c_{12}I\text{pH}^2 + c_{03}\text{pH}^3$ was found to yield a better fit than simpler (quadratic) or more complex (quartic) polynomial-based models considered. The model parameters c_{ij} and corresponding 95% confidence intervals were obtained.

Keywords

Ag-ZnO Nanowires, Photocatalyst, Photodegradation, Phenol, Model

1. Introduction

Phenols are among the numerous organic pollutants and toxic chemicals discharged into the aquatic environment that cause negative flavor and unpleasant odors in portable water. Phenols are generally bio-recalcitrant and stem from industrial sources, including pesticide, paint, petroleum and petrochemical industries, to mention a few [1]-[3]. Despite their low dosage, these pollutants may pose a major health hazard due to their mutagenicity and genotoxicity. Phenols are mutagenic and may be absorbed through the skin and harmful even at low dosage [4] [5]. Hence, degradation of phenol is important. A large body of researchers have worked on a number of remediation processes and came up with several chemical [6]-[8], biological [9], and physical [10] [11] processes. Since the application of these processes involved high costs and could possibly introduce more contaminants into the environment, developing energy efficient and “green” technologies to minimize these limitations while producing the desired complete mineralization of organic contaminants is of particular importance. Advanced oxidation processes (AOP)—a process of mineralization of organic compounds into simple products such CO₂ and water, have emerged as the most promising technology in the last few decades [12]-[14]. Among the semi-conductors for AOPs, TiO₂ and ZnO are the most often studied materials and employed due to their ability to produce hydroxyl radicals to destroy numerous types of organic contaminants [15]. Moreover, they are non-toxic, cheap and abundant.

Researchers and scientists have investigated the photodegradation of phenol using metal-doped photocatalysts, for example, Grabowska *et al.* [1] reported a three times higher photodegradation with 3% W-TiO₂ compared to TiO₂ nanoparticles under irradiation of visible light. In the presence of W-TiO₂ under visible light phenol was degraded to catechol and muconicaldehyde. Devi and Rajashekhar [16] doped TiO₂ with N₂ gas or the mineralized phenol under visible and UV light irradiation. Higher photodegradation of phenol was achieved with 0.15% dopant concentration compared to sol-gel TiO₂ under UV/solar irradiation than using hydrogen peroxide and ammonium peroxydisulfate as acceptors. Furthermore, Kavitha and Palanivelu [17] conducted a batch study using phenol as a model pollutant in industrial water to optimize parameters such as pH, hydrogen peroxide and ferrous oxide concentrations affecting Fenton-related oxidation reaction. The highest photodegradation efficiencies were Fenton (41%), solar (96%) and UV-Fenton (97%) processes.

The optimization of photodegradation of phenol with immobilized ZnO-Ag nanowires using a simple photocatalytic reactor under UV irradiation has not been shown in literature.

2. Experimental

2.1. Materials

The analytical grades chemicals are used without further purification process. Silver nitrate (AgNO₃, 99.9%) was purchased from Sigma-Aldrich and used to provide doping, zinc acetate (Zn(CH₃COO)₂, 99.9%) was procured from Fisher Scientific. Sodium hydroxide (NaOH, 44%) and hydrochloric acid (37%) were also obtained from Fisher Scientific. The double-distilled water was employed to prepare the desired phenol concentration.

2.2. Photoreactor

The various experiments were carried out in a laboratory fabricated aluminum photoreactor with dimension of 38.1 cm (15 in) long by 5.1 cm (2 in) wide by 2.5 cm (1 in) high. Aluminum sheet of 5.6 cm was used to build this reactor. A peristaltic pump (Masterflex L/S, 160 ml/min flow) was connected and pumped water from the glass beaker to the reactor. The water circulated through the reactor and returned to the beaker continuously during the experiment. The reactor system contained a lid, a UVA transparent glass, which was employed to prevent evaporation. The vessel top contains a light source. A lamp box which consisted of two Southern New England Ultraviolet Company Rayonet RPR-3500 Å UVA lamps (30.5 cm long) was used; the lamps provided irradiation in the UVA range of 350 - 400 nm. Other information about the photoreactor can be found at Udom *et al.* [18]. The light intensity at the surface of the reactor in the photoreactor was measured by an LI-COR pyranometer.

2.3. Preparation of Ag-Zn NWs

A seed solution of equimolar ratio (5 mM) of AgNO₃ and Zn(CH₃COO)₂ in ethanol was prepared; a 1.25 ml aliquot of this solution was placed on a substrate. An aliquot of 0.5 ml seed particle solution was placed on the

substrate (borosilicate glass) and randomly tilted in different directions to ensure uniform distribution. The seeded substrate was dried in an oven at 100°C for 10 min to remove moisture and another 30 min at 350°C to remove unreacted organic matter. After cooling, the substrate was placed in a reactor containing the growth solution, and stirred for 2 hours at 90°C. After this step, the substrate was rinsed with water and oven dried at 100°C for 10 min. and 350°C for 30 min.

2.4. Procedure and Analysis

The reaction mixture was placed for 30 min in the dark to establish adsorption equilibrium of phenol solution on the photocatalyst and then UV irradiated for 150 min. At certain interval, aliquot (5 ml) were collected and analyzed to assess photodegradation. A gas chromatograph (GC) with flame ionization detector (FID) via the head space method was used for phenol analysis. The GC used was SRI8600 with a 30 m DB-624 capillary column. To evaluate the impact of initial concentration and catalyst dosage on photocatalytic efficiency, experiments were performed at initial phenol concentration from 10 - 60 mg/L and catalyst loading 250 - 7500 mg/L. After determination of the optimal values, additional parameters including pH and light intensity were investigated for optimization and a model was established. Diluted NaOH and HCl were used to adjust pH within range 2.7 to 11 and was measured using Orion 5 Star ThermoScientific. The unadjusted initial phenol solution measured pH 8.2.

3. Result and Discussion

3.1. Effect of Initial Phenol Concentration

The effect of initial phenol concentration to the removal rate equation is vital for the optimization of the various concentration of phenol and development of a model. The reaction rate constant, K , decreases as the initial concentration increases while holding other parameters constant. This could be due to saturation of active sites on the photocatalyst by intermediates, hereby creating fewer sites for adsorption and creation of hydroxyl ions. Unlike other contaminants, the transmittance of UV light through the aqueous solution of phenol, due to its transparency, does not contribute to the decrease in rate constant. As observed in the [Figure 1](#), the empirical first-order relationship of different initial concentrations of phenol to the reaction rate constant is illustrated. Shukla *et al.* [19] examined the role of initial concentration (12.5 - 37.5 ppm) in the photodegradation of phenol. Optimum value was achieved at 12.5 ppm, under the condition studied ($\text{ZnO} = 0.4 \text{ g/L}$, persulfate = 2 g/L ad power = 330 W). Pardeshi and Patil have shown a decrease in phenol for the increase in concentration range from 25 - 300 mg/L [20]. However, less number of OH and O_2 radicals are available due to the constant reaction condition where more substrates molecules are adsorbed on the surface on the photocatalyst.

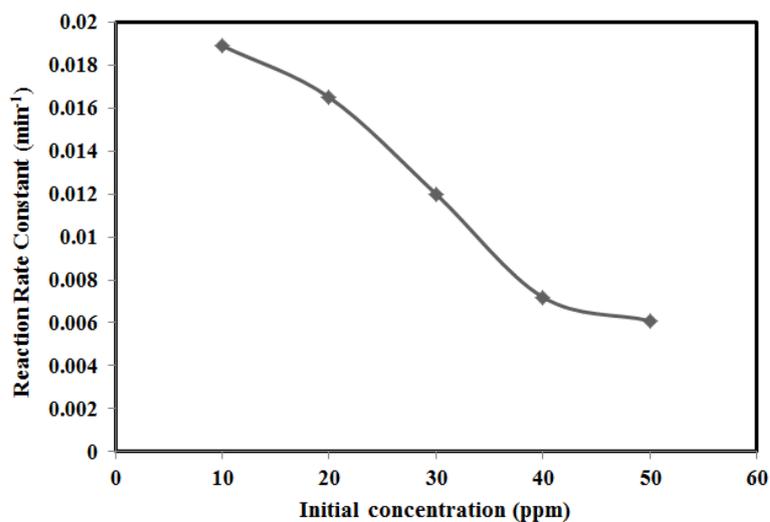


Figure 1. The effect of the initial concentration of phenol on reaction rate constant. Ag-ZnO NW = 500 mg/L, $T = 295 \pm 2 \text{ K}$, pH = 8.7, Light intensity = 60 W/m^2 .

3.2. Effect of UV Intensity

The photocatalytic degradation of organic compound is dependent effect of UV light intensity where the UV irradiation creates the photons needed to move electrons in the bands (valence band to conduction band) of the photocatalyst semiconductor. The irradiation produces the energy to drives the overall reaction, thus the reaction rate constant depends on the intensity of the irradiation. The rate of photocatalytic mineralization of phenol increases as more photons (increasing radiation) of sufficient energy reach the surface of the catalyst. The light intensity plays a key role in photochemical reaction for initiating the electron-hole formation. Chiou *et al.* [21] examined the UV light intensity (20 - 40 W) effect on the phenol degradation. Under UV irradiation over TiO₂ photocatalyst, the reaction rate constant was 0.0083, 0.012 and 0.031 min⁻¹ with light intensity of 20, 100 and 400 W, respectively. A more or less linear correlation was established between the rate constants and light intensity. Ollis *et al.* [22] have studied the degradation of organic pollutants (benzene and perchloroethylene) in a system of UV/TiO₂ at intensity ≤ 25 mW/cm² and further, shown that the reaction rate was independent of the light intensity for higher intensity.

In this study, varying intensities of UV radiation (10, 30, and 60 W) were used with Ag-ZnO NWs to catalyze the degradation of phenol in water at varying pH (2.7, 5.0, 8.1, and 11.0). The data are plotted in **Figure 2** as reaction rate constant versus UV lamp intensity. Approximate linearity with respect to lamp intensity is observed at each pH level considered.

3.3. Effect of pH

The role of pH on the efficiency of photocatalytic degradation of phenol was investigated in the pH range 2.7 - 11; the results are plotted in **Figure 3**. The three sets of data, reaction rate versus pH level, correspond to data collected at constant UV lamp intensity (10, 30, or 60 W). While the choice of interpolation curve is somewhat arbitrary (for each set of data, a cubic polynomial is fit to the data points), each curve suggests a maximum reaction rate at a pH level of approximately 5.5 - 6.0. At pH (5 - 6), most of the phenol molecules are adsorbed on the surface of the photocatalyst due to the undissociated nature of the phenol thereby producing higher photocatalytic efficiency. The surface of the photocatalyst is negatively charged, at higher pH (higher alkalinity), phenolate intermediates may be repelled away from the catalyst surface thereby opposing adsorption of contaminant molecules. Thus, lower degradation of phenol is observed in alkaline environments. Similar results were reported by Pardeshi and Patil [20] and Lathasree [23]. Akbar and Onar [24] studied the effect of pH on the photodegradation of phenol at the pH range 3 - 8. At mildly acidic (pH 5) condition, the highest photocatalytic activity was observed, while addition of an oxidant/electron acceptor (H₂O₂) to the system increased phenol degradation to 99.2%.

3.4. Empirical Correlation

In formulation of a complete model equation for the degradation of phenol by Ag-ZnO nanowires at varying pH and UV intensities, the authors initially examined the data and their dependence on each variable separately, as depicted in **Figure 2** and **Figure 3**. As can be seen in **Figure 2**, the dependence on UV lamp intensity is adequately modeled by a linear equation; that is:

$$K(I) = a_1 I + a_0$$

here, I is the UV lamp intensity, in W, and a_i , with units of W⁻ⁱ min⁻¹, represents the constants that fit the empirical model to the collected data. Similarly, the data suggest that the pH dependence can be captured by a polynomial, albeit with more terms. In other words:

$$K(\text{pH}) = \sum_{i=0} b_i \text{pH}^i$$

For the case of a cubic polynomial, for instance:

$$K(\text{pH}) = b_3 \text{pH}^3 + b_2 \text{pH}^2 + b_1 \text{pH} + b_0$$

here, b_i (units of min⁻¹) likewise represents the constants of the empirical model.

In fitting the multivariate model, the most obvious choice for a mathematical description of the data would be a similar polynomial, which can be formulated as follows.

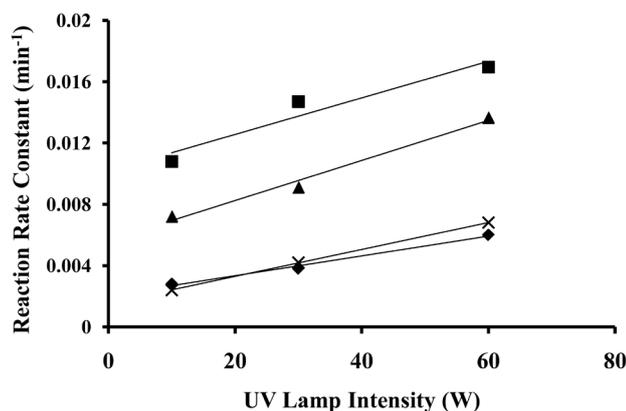


Figure 2. The effect of the UV light intensity on reaction rate constant; Ag-ZnO NW = 500 mg/L, $T = 295 \pm 2$ K, initial concentration = 30 ppm, and pH = 2.7 (◆), 5.0 (■), 8.7 (▲), and 11 (×).

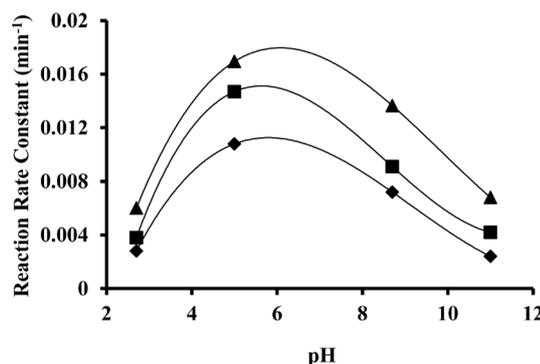


Figure 3. The effect of pH on reaction rate constant; Ag-ZnO NW = 500 mg/L, $T = 295 \pm 2$ K, initial concentration = 30 ppm, and UV lamp intensity = 10 W (◆), 30 W (■), and 60 W (▲).

$$K(I, \text{pH}) = \sum_{i=0} \sum_{j=0} c_{ij} I^i \text{pH}^j$$

In this case, c_{ij} (units of $\text{W}^{-i} \text{min}^{-1}$) represents the constants of the multivariate model. In fleshing out the form of the final model, it is useful to recall the linearity of the data with regard to varied UV lamp intensity; hence, in this study, we neglected any terms of quadratic or higher order in intensity (*i.e.*, $i \leq 1$). Similarly, we recognize that, given that cubic polynomials capture the pH dependence quite well, we can neglect quartic and higher order terms in pH (*i.e.*, $j \leq 3$).

The data obtained from experiment were fitted to potential polynomial models using least-squares regression. The results of the analysis of various potential models are tabulated as **Table 1**. For each equation, derived constants and the corresponding coefficient of determination (R^2) are given.

It can be seen that on the basis of the coefficient of determination, the best fit is given by the following equation:

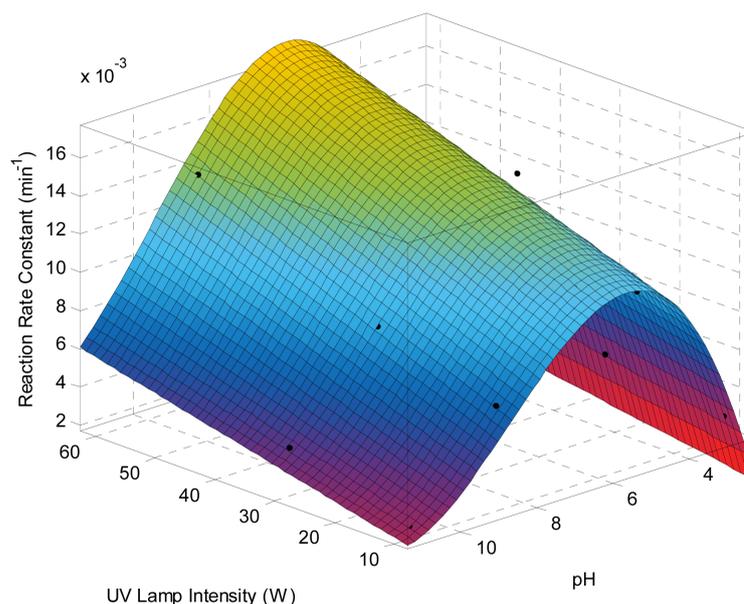
$$K(I, \text{pH}) = c_{00} + c_{10}I + c_{01}\text{pH} + c_{11}I\text{pH} + c_{02}\text{pH}^2 + c_{12}I\text{pH}^2 + c_{03}\text{pH}^3$$

This equation is plotted in **Figure 4** with experimental data for comparison. It is interesting to note that the addition of the $c_{13}I\text{pH}^3$ term does nothing appreciable to improve the accuracy of the fit of the model equation. Also, the authors examined higher order polynomials with similar deficiency in accuracy: a simple quartic polynomial gives an R^2 value of 0.9866. As such, the above equation is recommended for further design calculations and scale up of the bench scale reactor.

Finally, we consider the uncertainty in the derived model equation parameters. For the chosen equation

Table 1. Derived model parameters and coefficient of determination for candidate model equations.

Model equation	c_{00}	c_{10}	c_{01}	c_{11}	c_{02}	c_{12}	c_{03}	c_{13}	R^2
$c_{00} + c_{10}I + c_{01}pH + c_{11}IpH + c_{02}pH^2$	-0.01392	8.116E-05	0.007423	2.846E-06	-5.604E-04				0.8943
$c_{00} + c_{10}I + c_{01}pH + c_{11}IpH + c_{02}pH^2 + c_{12}IpH^2$	-0.009616	-4.796E-05	0.005811	5.118E-05	-4.428E-04	-3.528E-06			0.9057
$c_{00} + c_{10}I + c_{01}pH + c_{11}IpH + c_{02}pH^2 + c_{12}IpH^2 + c_{03}pH^3$	-0.02801	-4.796E-05	0.01637	5.118E-05	-0.002168	-3.528E-06	8.393E-05		0.9934
$c_{00} + c_{10}I + c_{01}pH + c_{11}IpH + c_{02}pH^2 + c_{12}IpH^2 + c_{03}pH^3 + c_{13}IpH^3$	-0.02819	-4.257E-05	0.01648	4.808E-05	-0.002184	-3.023E-06	8.475E-05	-2.459E-08	0.9934

**Figure 4.** The recommended polynomial model equation plotted on three axes as a surface with experimental data (•) overlain.

$$\left[K(I, pH) = c_{00} + c_{10}I + c_{01}pH + c_{11}IpH + c_{02}pH^2 + c_{12}IpH^2 + c_{03}pH^3 \right],$$

95% percent confidence intervals were generated for each equation parameter; the results are presented in **Table 2**. It can be seen that while a good fit was obtained, on the basis of the coefficient of determination, there is a good degree of variability over the 95% confidence intervals for most of the model parameters. It may be beneficial as a part of future investigative work to examine operating conditions of intermediate pH and/or UV intensity to the work considered here; by considering a greater breadth of data, a greater degree of certainty regarding the equation parameters of the resultant model may be obtained.

4. Conclusion

In this work, a facile hydrothermal technique was employed for immobilization of Ag-ZnO NWs on a borosilicate glass for successful degradation of phenol. From a preliminary study, an optimal initial concentration (30 mg/L) of phenol was attained. An attempt to form an overall model for the variation in phenol degradation rate for the function of intensity of UV light and pH level of the solution was demonstrated. For constant pH, it was found that the reaction rate is approximately linear with respect to the intensity of UV light. For constant intensity of UV light, the reaction rate is approximately cubic in pH. As such, a cubic model equation was derived (see **Table 2**)

Table 2. Derived model equation with parameter values/95% confidence intervals.

Parameter	$c_{00} + c_{10}I + c_{01}pH + c_{11}IpH + c_{02}pH^2 + c_{12}IpH^2 + c_{03}pH^3$			
	Value		95% Confidence Interval	
c_{00}	-0.02801	-0.03554	-	-0.02048
c_{10}	-4.796E-05	-1.710E-04	-	7.504E-05
c_{01}	0.01637	0.01265	-	0.02009
c_{11}	5.118E-05	8.586E-06	-	9.377E-05
c_{02}	-0.002168	-0.002723	-	-0.001612
c_{12}	-3.528E-06	-6.599E-06	-	-4.564E-07
c_{03}	8.393E-05	5.752E-05	-	1.103E-04

with only linear terms in UV light intensity. The equation was found to fit the data with a coefficient of determination (R^2) of 0.9934, and 95% confidence intervals were generated for all model parameters. Additional terms to the model equation were found not to improve the overall fit to the experimental data.

Acknowledgements

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