Modelling and Optimization of Photocatalytic Degradation of Naphthalene Using ZnO-ZnFe$_2$O$_4$ Composite

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P A P E R   I N F O

A B S T R A C T

ZnO-ZnFe$_2$O$_4$ composite was applied for photocatalytic degradation of naphthalene in fresh and saline waters under visible light irradiation. The effects of initial concentration of naphthalene, ZnO-ZnFe$_2$O$_4$ dosage and salinity on photocatalytic degradation of naphthalene were investigated using the central composite design. Statistically significant model quadratic equation was developed for photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite. The most significant parameter in the photocatalytic degradation is the ZnO-ZnFe$_2$O$_4$ dosage followed by the initial concentration of naphthalene and then salinity. The highest photocatalytic degradation of naphthalene was achieved at salinity of zero (that is in fresh water). The predicted optimum conditions for photocatalytic degradation of naphthalene using the ZnO-ZnFe$_2$O$_4$ composite are: initial naphthalene concentration of 16.8 mg/l, ZnO-ZnFe$_2$O$_4$ dosage of 0.50 g/l, and salinity of 0 ppt. The model quadratic equation was validated by performing experiments under the predicted optimum values. The experimental and the predicted values of naphthalene degradation under the predicted optimum values were 99 and 98.8 %, respectively. Hence, the developed quadratic model is reliable for predicting photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite.

INTRODUCTION

Polycyclic aromatic hydrocarbons are persistent organic pollutants that are often toxic, carcinogenic and mutagenic. Polycyclic aromatic hydrocarbon enter the environment through various anthropogenic and natural events [1]. Most Polycyclic aromatic hydrocarbon are highly stable to light and various microorganisms in the environment [2]. Naphthalene is one of the 16 toxic polycyclic aromatic hydrocarbon on the list of the Environmental Protection Agency of the United States of America and the European Union [3]. Even though most polycyclic aromatic hydrocarbons are non-polar molecules, naphthalene is sparingly soluble in water [4]. Naphthalene is frequently found in oil produced water and oil spills [5].

Photocatalysis is a process during which a semiconductor interacts with electromagnetic radiation of sufficient energy to produce reactive oxidizing species which can lead to chemical transformation of various organic and inorganic pollutants. During photocatalysis, reactive hydroxyl radicals oxidize hydrocarbons to yield carbon dioxide and water [6]. Therefore, photocatalysis has huge potential for treating oil spillage because the carbon dioxide produced may be used by sea plants, and the water produced will increase the amount of water in the sea. Consequently, the removal of spilled oil

on water can be achieved with the aid of photocatalysts [7, 8]. Moreover, photocatalysis is an environment friendly process that can be driven by solar energy [9]. In recent times, there has been a lot of research interest in photocatalytic degradation of naphthalene in water. The mechanisms of photocatalytic degradation of naphthalene using TiO$_2$ [10], Ti/ZnO–Cr$_2$O$_3$ composite [11], La co-doped TiO$_2$ [12], and Co$_3$O$_4$/Bi$_2$O$_2$CO$_3$ [13], TiO$_2$/NiO [14]. In all the reported cases naphthalene is finally mineralized to yield carbon dioxide and water during photocatalysis.

Present work focuses on ZnO-ZnFe$_2$O$_4$ composite that has a band gap of about 2.2 eV [15]. Hence, ZnO-ZnFe$_2$O$_4$ composite is capable of absorbing visible light which constitutes about 46 % of the solar spectrum [16]. The magnetic property of ZnO-ZnFe$_2$O$_4$ composite facilitates its physical separation after photocatalytic treatment of contaminated water using magnets [15-20]. In recent times, several researchers have synthesized ZnO-ZnFe$_2$O$_4$ composites and applied them for photocatalytic treatment of various water pollutants [15-23]. Recently, we synthesized ZnO-ZnFe$_2$O$_4$ composite via microwave combustion method [24]. The results of the control experiment indicate that 46.6 % of naphthalene was removed via adsorption on the surface of the ZnO-ZnFe$_2$O$_4$ composite in the dark within one hour.

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Moreover, when the same experiment was carried out under visible light irradiation, 99% of naphthalene was degraded via photocatalysis within one hour [24]. The effects of process parameters on photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite were not reported. In this paper, the effects of three process parameters (initial concentration of naphthalene, ZnO-ZnFe$_2$O$_4$ dosage and salinity) on photocatalytic degradation of naphthalene were investigated using the statistical central composite design.

MATERIALS AND METHODS

The procedure for microwave preparation and characterization of the ZnO-ZnFe$_2$O$_4$ composite has been reported previously [24].

Design of photocatalytic experiments
The central composite design (CCD) of the response surface methodology as implemented in Design Expert 6.0.6 software package was applied to investigate the effect of initial concentration of naphthalene (A), ZnO-ZnFe$_2$O$_4$ dosage (B) and salinity (C) on the photocatalytic degradation of naphthalene using the ZnO-ZnFe$_2$O$_4$ composite. The response was chosen to be the percentage photocatalytic degradation of naphthalene after 60 min of photocatalytic treatment. The ranges and coding of the process variables used are presented in Table 1. Twenty experiments were designed. The designed experiments consist of 8 factorial points, 6 axial points, and 6 replications at the center points in order to ascertain reproducibility of the results [25]. The experimental data were subjected to analysis of variance (ANOVA) in order to determine the significance of the model equation and the process parameters. An empirical model equation was developed for predicting the photocatalytic degradation of naphthalene and determination of the optimum conditions for photocatalytic degradation of naphthalene using the ZnO-ZnFe$_2$O$_4$ composite.

Photocatalytic experiments
A 500 W tungsten–halogen lamp was used as the source of the visible light irradiation. The lamp is frequently used in photocatalytic experiments to simulate sunlight [26]. Aqueous solutions of naphthalene of varying concentrations were prepared by serial dilution method. To study the effect of salinity on the photocatalytic process, appropriate amounts of NaCl were added to aqueous solutions of naphthalene.

A pre-determined amount of the ZnO-ZnFe$_2$O$_4$ powders were dispersed into 100 ml of naphthalene solution in the photoreactor at room temperature. Before irradiation, the suspensions were stirred for 60 min in the dark to establish adsorption/desorption equilibrium of the naphthalene molecules on the photocatalyst surface. The stirred suspensions were then continuously irradiated for one hour,

after which, ZnO-ZnFe$_2$O$_4$ particles were separated. The residual concentration of naphthalene in the treated water was measured using Jenway UV–vis spectrophotometer at 275 nm (the maximum absorption wavelength of naphthalene). The percentage photocatalytic degradation of naphthalene was calculated using Equation 1.

$$Degradation = \frac{(A_0 - A_t)}{A_0} \times 100\% \qquad (1)$$

where A$_t$ is the absorbance of naphthalene remaining in the treated water after irradiation time, t, and A$_0$ is the absorbance of naphthalene solution before irradiation.

RESULTS AND DISCUSSION
Modelling of photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite
The effect of three process parameters (ZnO-ZnFe$_2$O$_4$ dosage, initial concentration of naphthalene and salinity) on the percentage photocatalytic degradation of naphthalene were analyzed using the central composite design. The design matrix consisting of the three process variables, experimental and predicted values of the percentage degradation of naphthalene is presented in Table 2. The experimental responses were used to develop a model quadratic equation that describes photocatalytic degradation of naphthalene using the ZnO-ZnFe$_2$O$_4$ composite (Equation 2).

$Degradation\ (\%) = 103.128 + 0.112*A - 0.084*B - 0.175*C$ (2)
$- 0.008*A^2 - 0.001*B^2 - 0.008*C^2 + 0.003*A*B - 0.002*A*C + 0.002*B*C$

The quadratic model was subjected to analysis of variance (ANOVA). From the ANOVA results presented in Table 3; the statistical significance of the model and each model term as well as the quality of the fit of the quadratic model were determined. The F values (Fisher variation ratio) and values of p-value (probability) were used to check the significance of the model and model terms. The p-value of the quadratic model (<0.0001) is less than 0.0500; this indicates that the quadratic model is significant. The p-value of the Lack-of-Fit of the model (0.083 > 0.0500) is an indication that the Lack-of-Fit is insignificant relative to the pure error. The high F-value (333.26) of quadratic model also indicates that the model is significant [27]. The trend in F values of the model terms suggests that ZnO-ZnFe$_2$O$_4$ dosage has the largest effect on the photocatalytic degradation process followed by the initial concentration of naphthalene and then salinity. The linear and quadratic relations between ZnO-ZnFe$_2$O$_4$ dosage and initial concentration of naphthalene, as well as the linear and quadratic relations between ZnO-ZnFe$_2$O$_4$ dosage and salinity are also highly significant with $p < 0.0500$. Adeq Precision of 122.795 indicates an adequate signal because the mimimum desired Adeq Precision is 4. Hence, the developed model can be used to navigate the design space.

TABLE 1. Ranges of the actual values of the studied process variables for photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite

Variable	Code	Low actual	High actual
Initial concentration of naphthalene (mg/l)	A	5.00	25.00
ZnO-ZnFe$_2$O$_4$ dosage (g/l)	B	0.50	2.50
Salinity (ppt)	C	0.00	35.00

TABLE 2. Design matrix, experimental and predicted responses for photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite

Run	Independent variables			Degradation, (%)	
	A	B	C	Experimental	Predicted
1	1.8	150.0	17.5	52.6	51.2
2	15.0	18.2	17.5	94.6	94.5
3	5.0	50.0	0.0	76.9	79.9
4	15.0	150.0	17.5	52.2	53.2
5	5.0	50.0	35.0	83.6	86.1
6	15.0	150.0	17.5	52.3	53.2
7	15.0	150.0	46.9	52.8	58.5
8	15.0	150.0	11.9	52.3	52.2
9	25.0	50.0	0.0	85.5	82.9
10	15.0	150.0	17.5	52.7	53.2
11	15.0	150.0	17.5	53.5	53.2
12	15.0	150.0	17.5	49.2	53.2
13	31.8	150.0	17.5	50.7	55.7
14	5.0	250.0	0.0	21.0	17.2
15	25.0	250.0	0.0	18.7	20.3
16	15.0	150.0	17.5	49.6	53.2
17	15.0	318.2	17.5	58.3	58.5
18	25.0	250.0	35.0	21.3	26.5
19	5.0	250.0	35.0	22.5	23.5
20	25.0	50.0	35.0	68.9	89.2

TABLE 3. Analysis of variance (ANOVA) of the developed model quadratic equation for photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	5313.37	9	590.37	1176.69	< 0.0001
A	67.42	1	67.42	134.38	< 0.0001
B	3559.61	1	3559.61	7094.77	< 0.0001
C	1.73	1	1.73	3.45	0.1056
A^2	8.42	1	8.42	16.77	0.0046
B^2	489.78	1	489.78	976.21	< 0.0001
C^2	3.95	1	3.95	7.87	0.0263
AB	33.31	1	33.31	66.39	< 0.0001
AC	0.69	1	0.69	1.37	0.2798
BC	29.57	1	29.57	58.95	0.0001
Residual	3.51	7	0.50		
Lack of Fit	2.22	2	1.11	4.29	0.0823

$R^2 = 0.9993$; $R^2_{adj} = $ of 0.9985

The closeness of the predited R^2_{pred} (0.9993) and R^2adj (0.9985) also indicates excellent predictability of the model. As apparent in Table 2, the experimental and predicted values of the percentage degradation of naphthalene are very close. This can also be seen from the plot of actual versus predicted values displayed in Figure 1a. Therefore, the model quadratic equation perfectly explains the experimental range studied. The model diagnostic plots displayed in Figure 1 were used to check whether the model satisfies the assumptions of the ANOVA. Figure 1b shows normal plot of residuals, from where it is seen that all the errors encountered during the experiment follow a particular pattern in line with the normal probability plot of residuals theory [27]. The plot of residual versus run number displayed in Figure 1c does not follow a particular trend. This indicates that the quadratic model equation is good and should not be discarded. The plot of residuals against predicted is used in diagnosing nonlinearity or non-constant error variance. The plot is often used to finding outliers. A good predicted result shows a random scatter with all the data points between -3 and +3 as shown in Figure 1d [27, 28].

Effects of the process variables on photocatalytic degradation of naphthalene using ZnO-ZnFe₂O₄ composite

The response surface and contour plots for the effect of each pair of the studied variables are shown in Figure 2. As shown in Figures 2a and 2c, photocatalytic degradation of naphthalene decreases with increase in ZnO-ZnFe₂O₄ dosage. This observation can be rationalized as follows: the number of active sites of photocatalysts increases with increase in photocatalyst dosage. However, light penetration is reduced at high photocatalyst dosage due to increased opacity and increased light reflectance. Moreover, deactivation of the activated molecules by collision with ground state molecules and agglomeration of the photocatalyst particles may also contribute to low percentage of degradation at high photocatalyst dosage used. Similar observations have been reported for other organic substrates [29, 30]. Figures 2a and 2b show that photocatalytic degradation of naphthalene increases with increase in the initial concentration of naphthalene. It has been observed that when initial concentration of a substrate is increased, more molecules of the substrate are adsorbed onto the photocatalyst surface, thereby improving photocatalytic degradation of the substrate [31, 32]. As shown in Figures 2b and 2c, increase in salinity leads to slight decrease in the percentage photocatalytic degradation of naphthalene. Indeed, it has been reported that salinity impedes photolytic degradation of naphthalene due to free-radical scavengers and photon competitors [32].

As summarized in Table 3, there are significant interactions between the initial concentration of naphthalene and ZnO-ZnFe₂O₄ dosage (p-value < 0.0001) and between ZnO-ZnFe₂O₄ dosage and salinity (p-value = 0.0001). Thus,

Figure 2a shows that the percentage degradation of naphthalene is more sensitive to changes in the initial concentration of naphthalene at high ZnO-ZnFe₂O₄ dosage than at low ZnO-ZnFe₂O₄ dosage. Similarly, Figure 2c shows that the percentage degradation of naphthalene is more sensitive to changes in the salinity at high ZnO-ZnFe₂O₄ dosage than at low ZnO-ZnFe₂O₄ dosage. Highest percentage degradation of naphthalene was achieved at the highest initial concentration of naphthalene, lowest ZnO-ZnFe₂O₄ dosage and lowest salinity.

Optimization of photocatalytic degradation of naphthalene using ZnO-ZnFe₂O₄ composite

In order to determine the optimum values of the three process variables that will lead to the highest photocatalytic degradation of naphthalene, optimization of the variables was performed using Design expert 6.0.6 software. The goal was to achieve maximum photocatalytic degradation of naphthalene when the three process variables are within the range presented in Table 1. The results of the numerical optimization show that maximum percentage degradation of 98.8% can be achieved under the following predicted optimum conditions: initial naphthalene concentration of 16.8mg/l, ZnO-ZnFe₂O₄ dosage of 0.50 g/l, and salinity of 0 ppt. To validate that the developed quadratic model for photocatalytic degradation of naphthalene, experiments were performed under the predicted optimum conditions listed. The experimental degradation (99%) is in excellent agreement with the predicted degradation (98.8 %). This validates the developed quadratic model. Therefore, the developed quadratic model is reliable for predicting photocatalytic degradation of naphthalene.
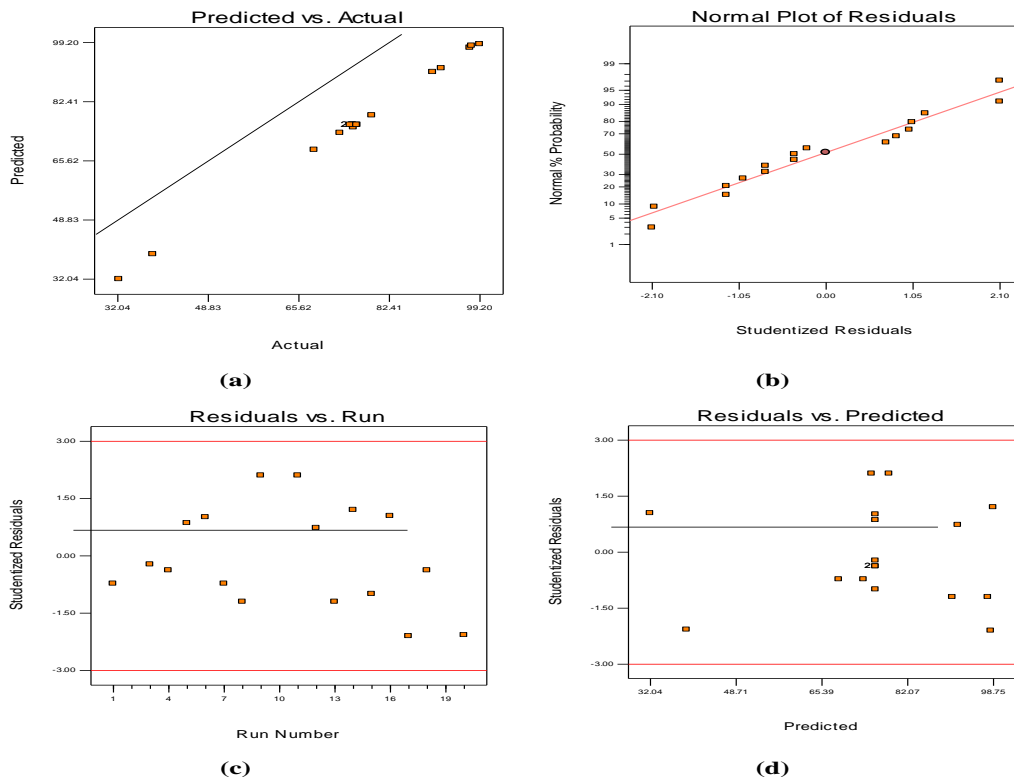

Figure 1. Diagnostic plots for photocatalytic degradation of naphthalene using ZnO-ZnFe₂O₄ composite: (a) Plot of predicted against actual values, (b) Normal plot of residuals, (c) Residual versus run plot, and (d) Residual versus predicted plot
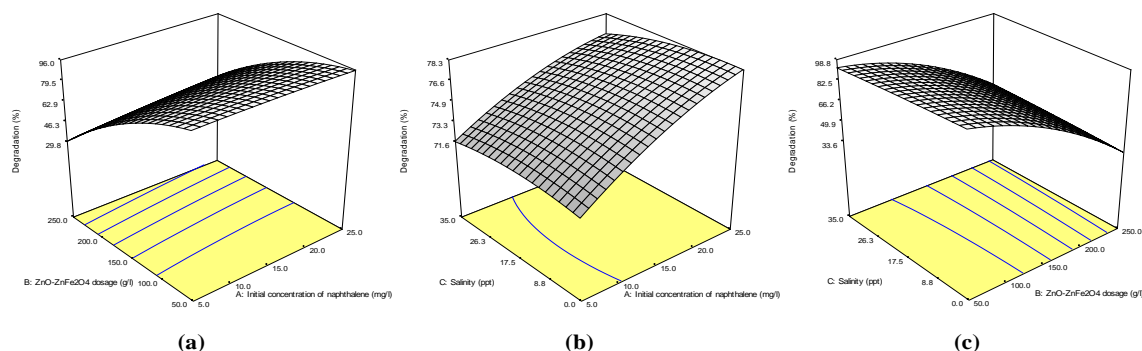
Figure 2. Response surface for simultaneous influence of (a) initial concentration of naphthalene and ZnO-ZnFe$_2$O$_4$ dosage, (b) initial concentration of naphthalene and salinity, (c) ZnO-ZnFe$_2$O$_4$ dosage and salinity

It is instructive to compare the present results with some recent literature reports on modelling and optimization of photocatalytic degradation of naphthalene using various photocatalysts. Mahmoodi and Sargolzaei [33] applied the response surface methodology to model and optimize photocatalytic degradation of naphthalene using TiO$_2$ nanoparticles as the photocatalyst. They developed a quadratic model that described the effect of the four independent parameters (agitation speed, air flow rate, photocatalyst dosage, and UV light intensity) on the degradation of naphthalene. Their optimization results showed that maximum photocatalytic degradation of naphthalene of 66.3 % can be achieved at the following optimum conditions: agitation speed of 150 rpm, air flow rate of 5 L/h, TiO$_2$ dosage of 2.17 g/l, and UV light power of 18.67 W [33]. Zhou *et al*. [34] also used response surface methodology to investigate photocatalytic degradation of naphthalene using TiO$_2$/Fe$_3$O$_4$-SiO$_2$ as the photocatalyst under UV irradiation. Their results showed that irradiation time, pH, and the photocatalyst loading had significant influence on photocatalytic degradation of naphthalene, and the maximum photocatalytic degradation of 97.39% was predicted at optimum irradiation time of 97.1 min, pH of 2.1, and TiO$_2$/Fe$_3$O$_4$-SiO$_2$ dosage of 0.962 g/l, respectively [34]. In the present work, the optimum ZnO-ZnFe$_2$O$_4$ dosage (which afforded 99.04% degradation of naphthalene under visible light irradiation) is only 0.5 g/l. The high photocatalytic activity of ZnO-ZnFe$_2$O$_4$ under visible light irradiation makes it a very promising photocatalyst for solar driven photocatalytic processes because 46% of the solar spectrum is visible light; UV constitutes less than 5% of the solar spectrum [16].

CONCLUSION

Photocatalytic degradation of naphthalene using ZnO-ZnFe$_2$O$_4$ composite is described by a quadratic model equation. ZnO-ZnFe$_2$O$_4$ dosage and initial concentration of naphthalene have the largest effect on the degradation process. The highest photocatalytic degradation of naphthalene was achieved at salinity of zero (that is in fresh water). The predicted optimum conditions for photocatalytic degradation of naphthalene using the ZnO-ZnFe$_2$O$_4$ composites are: initial naphthalene concentration of 16.8

mg/l, ZnO-ZnFe$_2$O$_4$ dosage of 0.50 g/l, and salinity of 0 ppt. The experimental and the predicted values of naphthalene degradation under the predicted optimum values are 99 and 98.8 %, respectively. Hence, the developed quadratic model is reliable for predicting photocatalytic degradation of naphthalene.

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Persian Abstract

چکیده

کامپوزیت ZnO-ZnFe2O4 برای تخریب فوتوکاتالیستی نفتالین در آب تازه و شور در معرض تابش نور قابل مشاهده است. اثر غلظت اولیه نفتالین، دوز ZnO-ZnFe2O4 و شوري بر تخریب فوتوکاتالیستی نفتالین با استفاده از طراحی کامپوزیت مرکزی مورد بررسی قرار گرفت. معادله درجه دوم معنی‌داری برای تخریب فتوکاتالیستی نفتالین با استفاده از کامپوزیت ZnO-ZnFe2O4 ایجاد شد. مهم‌ترین پارامتر در تجزیه فوتوکاتالیستی، دوز ZnO-ZnFe2O4 و سپس غلظت اولیه نفتالین و نهایت شوری است. بیشترین تخریب فوتوکاتالیستی نفتالین در شوری صفر (یعنی در آب شیرین) حاصل شد. شرایط مطلوب پیش‌بینی شده برای تخریب فوتوکاتالیستی نفتالین با استفاده از ترکیبات ZnO-ZnFe2O4 عبارتند از: غلظت اولیه نفتالین ۱۶/۸ میلی‌گرم بر لیتر، مقدار ZnO-ZnFe2O4 به میزان ۰/۵۰ گرم در لیتر و شوري ۰/۰ ppt. معادله درجه دوم معکوس با انجام آزمایش‌ها در مقادیر بهینه پیش‌بینی شد. مقادیر تجربی و پیش‌بینی شده تخریب نفتالین در مقادیر پیش‌بینی شده به ترتیب ۹۹ و ۹۸/۸ درصد بود. از این رو، مدل درجه دوم توسعه یافته برای پیش‌بینی تخریب فوتوکاتالیستی نفتالین با استفاده از کامپوزیت ZnO-ZnFe2O4 قابل اعتماد است.