

Kinetics and Equilibrium Studies for Removal of Reactive Red 198 From Aqueous Solutions Using Zero Valent Iron powder

Sudabeh Pourfadakari^{1,2}; Amir Hossein Mahvi^{3,4,5,*}

¹Department of Environmental Health Engineering, School of Health, Shiraz University of Medical Sciences, Shiraz, IR Iran

²Student Research Committee, Shiraz University of Medical Sciences, Shiraz, IR Iran

³Department of Environmental Health Engineering, School of Public Health, Tehran University of Medical Sciences, Tehran, IR Iran

⁴National Institute of Health Research, Tehran University of Medical Sciences, Tehran, IR Iran

⁵Center for Solid Waste Research, Institute for Environmental Research, Tehran University of Medical Sciences, Tehran, IR Iran

*Corresponding author: Amir Hossein Mahvi, Department of Environmental Health Engineering, School of Public Health, Tehran University of Medical Sciences, Tehran, IR Iran. Tel: +98-2188954914, Fax: +98-2188950188, E-mail: ahmahvi@yahoo.com

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Background: The complex aromatic structures of dyes make them more stable and more difficult to remove from aqueous solutions. Thus, it is essential to remove dyes from wastewater before discharging them into environment.

Objectives: This study aimed to investigate the efficiency of zero valent iron powder (ZVI) in removing reactive red 198 (RR-198) from aqueous solutions and analyze adsorption isotherms and kinetics.

Materials and Methods: In this laboratory study, all experiments were performed in batch systems. This study investigated the effect of various factors, such as initial dye concentration, contact time, iron powder dose, and pH, on dye removal. The adsorption parameters were determined based on Langmuir, Freundlich and Temkin isotherms, while the kinetic models were used to establish the adsorption mechanism.

Results: The results of this research showed pH = 3 and contact time = 120 minutes, increasing the ZVI dose from 200 to 5000 mg/L in 100 mg/L dye concentration, increased the adsorption efficiency from 36.78% to 97.57%. RR-198 removal followed the Freundlich isotherm ($R^2 = 0.996$ at 25°C) and the pseudo-second-order kinetic model.

Conclusions: Considering the simplicity and efficiency of zero valent iron powder, this method is recommended for removing azo dyes from aqueous environments.

Keywords: Adsorption; Equilibrium; reactive red 198

1. Background

Synthetic dyes are widely used by various industries, such as textile, tanning, leather, paper, and pulp mills. Textile industry wastewater is the main source of environmental pollution as dyes are resistant to degradation (1, 2). Synthetic dyes are generally classified into reactive, acidic, basic, vat, dispersing, direct, sulfur, etc. among which, reactive and acidic dyes are the most widely used (2, 3). More than 700000 tons of dye are produced around the world annually, and it is estimated that 2% of dyes produced may enter into wastewater (4, 5). In textile industries, almost 1% to 20% of dyes enter wastewater due to the impaired performance of the dyeing unit (6). Synthetic colors usually have aromatic molecular structures similar to those of benzene, naphthalene, anthracene, toluene and xylene, making them stable in the environment and more resistant to decomposition. Most of these dyes are toxic and might even be carcinogenic (5). Furthermore, reactive dyes are widely used in the textile industry due to their ease of use, which simply contaminates water because they are highly soluble. The

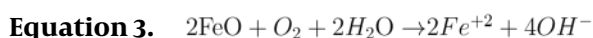
existence of a reactive dye in aquatic environments can decrease self-purification capacity as well as the photo-synthesis processes. For this reason, dyes are needed to be removed from wastewater before being discharged into the environment. Reactive red 198 (RR-198) is among this group of dyes and it was selected as the model reactive dye for this study (7). Contaminated waste water is normally treated using biological, physical, and chemical methods. Because of their complex structures they are hard to degrade utilizing biological methods and are generally treated by physical and chemical methods (8) including oxidation, coagulation and flocculation, adsorption, electrochemical, electrolysis, precipitation and membrane filtration. Most of these methods are expensive while not effective for a wide range of dyes (9-11). Of late, zero valent iron (ZVI) has been used to remove contaminants from water and wastewater due to its low toxicity, low cost, and high efficiency (2). Zero-iron powder is a strong reducing agent. Iron particles can easily be recycled through a magnet. It can be used for a wide

Implication for health policy/practice/research/medical education:

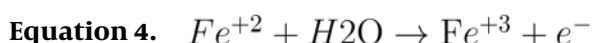
Iron powder is easy to use and can be continuously utilized due to its recyclability. Therefore, it is highly economical to replace azo dyes with iron powder.

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range of contaminants, including chlorinated organic compounds such as phenol, aromatic nitro compounds, nitrate, herbicides and heavy metal ions. Dyes are also oxidized by zero valent iron (12). Metallic iron reacts in aquatic environments as follows (Equations 1, 2, 3) (2, 13):



In the above mentioned reactions, two free electrons are produced and used as the recovery agent. Moreover, divalent iron in aquatic environments is oxidized as follows and creates sediments. One electron is also released during this reaction (Equation 4).



Three electrons are released during oxidation of zero valent to trivalent iron, replacing the ions of organic compounds and leading to their decomposition.

2. Objectives

Considering the advantages mentioned for ZVI, the present study aims: 1) to investigate the efficiency of zero valent iron powder in removing reactive red 198 (RR-198) from aqueous solutions, 2) to investigate the influence of

other variables, including contact time, pH of solution, ZVI dose and initial dye concentration and 3) to analyze adsorption of isotherms and kinetics.

3. Materials and Methods

Iron powder exceeding 98% purity and effective particle size of 150 μ was purchased from Merck Company, Germany and used without additional treatment. In addition, reactive red 198 with 95% purity was purchased from Alvan Sabet Company, Hamedan, Iran and employed without additional treatment. The features of the utilized dye are presented in Table 1. Other chemicals with laboratory purity, used in the study, were also prepared by Merck Company, Germany.

3.1. Methods

The present applied study was conducted to assess adsorption of RR-198 by zero valent iron particles in batch systems. To perform the experiments 250 mL laboratory jars were used. Besides, HCl and NaOH 1N were used in order to adjust the pH level. The effect of different parameters, including reaction time (5 - 150 minutes), initial dye concentration (20, 50, 100 and 200 mg/L), pH (3 - 10) and (ZVI) powder (200, 500, 1000, 2500 and 5000 mg/L) on the dye removal process were assessed. Mixing was performed by a shaker (rotator R430) with 150 rpm. At the end of the equilibrium time, in order to separate the zero valent iron powder from the dye solution, the samples were centrifuged at 4000 rpm for 10 minutes and passed through 0.2 μ filter paper (12, 14, 15). Finally, the remaining dye concentration in the solution was analyzed by UV/Vis spectrophotometer (Hatch-DR5000, Germany)

Table 1. Features of Dye Used in the Study

Parameter	Characteristic
Type color	Anionic
Symbol	RR198
Chemical formula	C ₂₇ H ₁₈ ClN ₇ Na ₄ O ₁₅ S ₅
Molecular weight, g/mol	968/21
Wave length of maximum absorption, nm	518
Chemical structure of color	

at a wave length of 518 nm according to the standard methods and efficiency was measured (16). In this study, using each factor method independently, each parameter was separately optimized and sample size was determined. The sample size of this study was 101. The amount of dye adsorbed by the adsorbent (mg/g) and adsorption efficiency (%) were obtained using Equations 6 and 7 (17).

$$\text{Equation 5.} \quad q_e = \frac{(C_o - C_t)V}{M}$$

$$\text{Equation 6.} \quad \text{Re (\%)} = \frac{(C_o - C_t)}{C_o} \times 100$$

Where q_e is the amount of adsorbed dye (mg/g), C_o is the initial dye concentration in the solution (mg/L), C_t is the remaining dye concentration in the solution (mg/L), V is the volume (L) and M is the adsorption dose (g).

3.2. Adsorption Isotherm and Kinetics

In this study, the equilibrium of adsorption data was investigated using Langmuir, Ferundlich and Temkin isotherm models. The experiments were performed by changing the initial dye concentration from 20 to 200 mg/L with a zero valent iron powder dose of 5000 mg/L, at a temperature of 25°C and the equilibrium time of 120 minutes. The Langmuir isotherm is the most applicable adsorption isotherm. Saturated monolayer adsorption is presented by Equation 8 (18):

$$\text{Equation 7.} \quad q_e = \frac{Q_m K_L C_e}{1 + K_L C_e}$$

The linear form of the Langmuir equation is presented as follows (Equation 9):

$$\text{Equation 8.} \quad \frac{1}{q_e} = \frac{1}{Q_m} + \left(\frac{1}{K_L Q_m} \right) \frac{1}{C_e}$$

Where q_e is the quantity of adsorbed dye (mg/g), C_e is the equilibrium concentration of the dye in the solution (mg/g), K_L is the Langmuir constant (L/mg) and Q_m is the theoretical monolayer saturation capacity (mg/g). The Freundlich isotherm describes equilibrium on heterogeneous surfaces and hence does not assume monolayer capacity. The Ferundlich isotherm equation is as follows:

$$\text{Equation 9.} \quad q_e = K_f C_e^{\frac{1}{n}}$$

The linear form of the Ferundlich equation is also presented as follows (Equation 11):

$$\text{Equation 10.} \quad \log q_e = \log K_f + \left(\frac{1}{n} \right) \log C_e$$

Where, q_e is the quantity of adsorbed dye per unit mass of adsorbent (mg/g), C_e is RR-198 concentration in the solution at equilibrium (mg/L), and K_f and n are the Ferunlich constants which are appropriate to the adsorption capacity and the adsorbent adsorption intensity, respectively. The values of K_f and n can be obtained from the intercept and slope of the linear plot of $\log q_e$ vs. $\log C_e$. The Temkin isotherm equation is presented as follows (Equation 12)(19):

$$\text{Equation 11.} \quad q_e = B_1 L_n (K_t C_e)$$

The linear form of the Temkin equation is presented as follows (Equation 13):

$$\text{Equation 12.} \quad q_e = B_1 \ln K_T + B_1 \ln C_e$$

In this study, K_T and $B_1 = RT/b$ are constant, R is the gas constant (8.314 J/mol k), $T(k)$ is the absolute temperature, K_T is the equilibrium bond constant (L/mg) related to the maximum bond energy and B_1 is related to adsorption heat. Moreover, in order to assess the adsorption process of RR-198 on zero valent iron powder, pseudo-first-order, pseudo-second-order and intra-particle diffusion kinetic models were investigated. These models describe the adsorption of dye of RR198, on the ZVI. The kinetics equations are shown in Table 2.

4. Results

4.1. Effect of Contact Time

Dye removal was investigated at the contact time of 5 to 150 minutes, with initial dye concentration of, 100 mg/L, pH = 3 and 5000 mg/L dose of zero valent iron powder. The effect of contact time on the dye removal percentage is shown in Figure 1. The results indicated that as the time increased, dye removal efficiency increased. Adsorption speed was fast and contaminants removal accelerated during the first 60 minutes; thereafter, it followed a constant slope and reached equilibrium after 120 minutes.

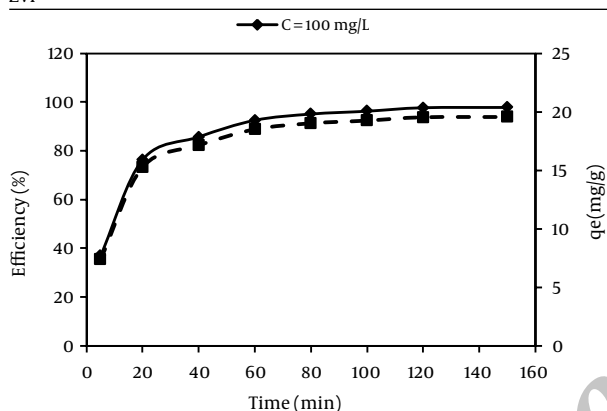
4.2. Effect of pH

In order to assess the effect of pH on RR-198 removal, the experiments were performed at the pH ranges of 3 to 10, initial dye concentration of 100 mg/L, contact time of 5 - 120 minutes and a 5000 A constant slope mg/L dose of zero valent iron powder. The results are presented in Figure 2. Figure 2 indicates that as pH level increased from 3 to 10, dye removal efficiency reduced from 97.34% -84.68%. By decreasing pH level from 10 to 3, adsorption capacity increased from 16.93 to 19.46 mg/g.

Table 2. Kinetic Equations and Linear Forms (20)

Kinetics	Equation	Linear Form
Pseudo-first-order	$\frac{dq_t}{dt} = K_1 (q_e - q_t)$	$\log (q_e - q_t) = \log (q_e) - \frac{K_1}{2.303} t$
Pseudo-second-order	$\frac{dq_t}{dt} = K_2 (q_e - q_t)^2$	$\frac{t}{q_t} = \left(\frac{1}{K_2 q_e^2} \right) + \left(\frac{1}{q_e} \right) t$
Intra-particle diffusion		$q_t = K_{\text{dif}} t^{0.5} + C$

Figure 1. Effect of Equilibrium Time on RR-198 Adsorption Process Using ZVI



pH = 3, adsorbent dose of 5000 mg/L.

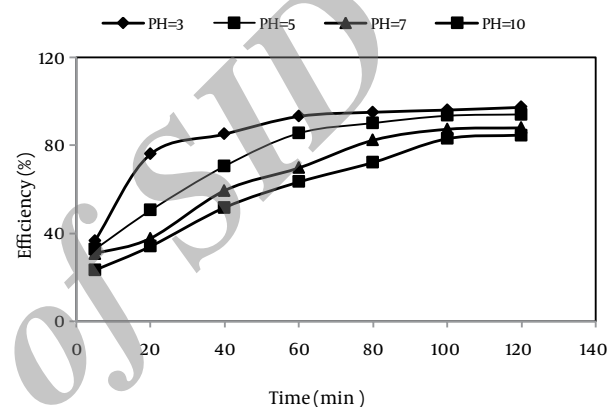


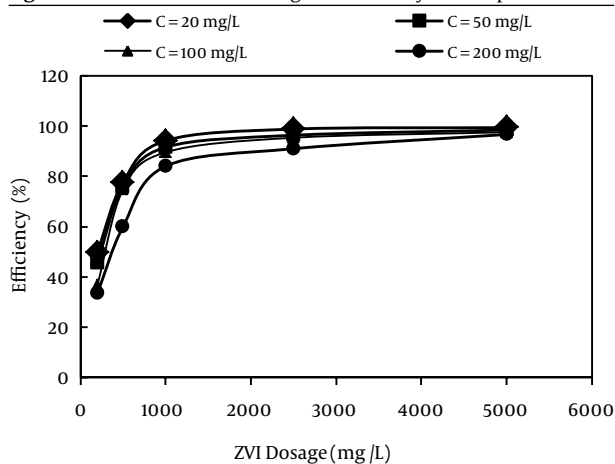
Figure 2. Effect of pH on RR-198 Adsorption Process Using 5000 mg/L of ZVI

4.3. The Effect of Adsorbent Dose and Initial Dye Concentration

The effect of zero valent iron powder dosage on the dye removal percentage was investigated by adding various dosages of iron powder (200 - 5000 mg/L) to different dye concentrations (20, 50, 100 and 200 mg/L) at contact time of 120 minutes and pH = 3. The effect of zero valent iron powder on dye adsorption is presented in Figure 3.

According to the results, by increasing the dose of iron powder from 200 to 5000 mg/L at dye concentration of 100 mg/L, removal efficiency increased from 36.78% to 97.57%, while adsorption capacity was reduced. In order for better understanding the effect of the iron powder dose on dye removal efficiency, a linear regression was drawn using MATLAB software version 7.8 (Figure 4) and the best qualification was related to the linear regression coefficient of $R^2 > 0.9278$. The effect of initial dye concentration is presented in Figure 5. By increasing dye concentration from 20 to 200 mg/L in the iron powder dose of 5000 mg/L, the dye removal efficiency decreased from 99.41% to 96.85% although dye adsorption capacity increased from 3.97 mg/g to 38.78 mg/g.

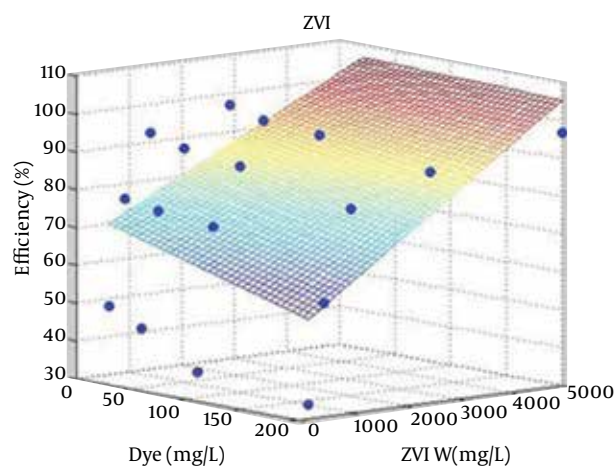
Figure 3. Effect of Various ZVI Dosages on Efficiency of Adsorption Process



pH = 3, contact time of 120 minutes and various dye concentrations (mg/L).

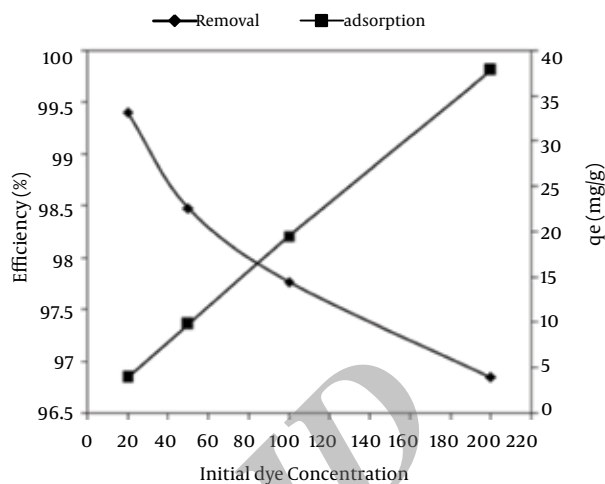
The decolorization efficiency of dyes reported in different articles has been summarized in Table 3.

Figure 4. Multivariate Linear Regression Modeling of RR-198 Using ZVI Method



pH = 3, contact time of 120 minutes, various dye concentrations and various dosages of zero valent iron powder.

Figure 5. Effect of Initial Dye Concentration on Adsorption Efficiency



pH = 3, Contact Time of 120 Minutes.

Table 3. Decolorization of Azo Dyes by Different Methods (21-25)

Dye	Decolorization methods	Conditions	Decolorization Efficiency, %	References
Reactive red 198	By <i>Aspergillus parasiticus</i> fungal biosorbent	Time = 50 min, temperature 50°C, pH = 2	98.57	(21)
Reactive red 198	UV/TiO ₂ /H ₂ O ₂	Time = 35 min, (dye 50 mg/L, H ₂ O ₂ = 450 mg/L	100	(22)
Reactive red 198, reactive black 5	nanoscale zerovalent iron	Time = 90 min, pH = 3	70	(23)
Reactive red 198	ozonation	Time = 30 min	96	(24)
Reactive red 198	electrochemical coagulation process	Time = 30 min	99	(25)
Reactive red 198	zero valent iron	Time = 120 min, pH = 3	97.57	current study

Table 4. Results of Adsorption Isotherm Calculation

Types of Isotherm	Temperature, 25°C
Freundlich	
N	1.524
K _f	3.491
R ²	0.996
Langmuir	
Q _m	32.258
K _l	0.111
R ²	0.990
K _T	2.129
Temkin	
B _T	8.738
R ²	0.923

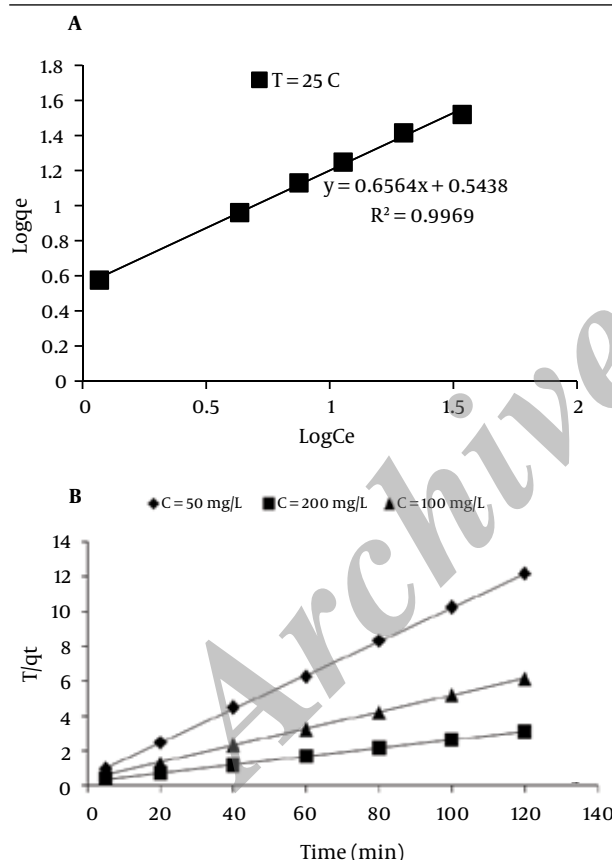
4.4. Adsorption Isotherm and Kinetics

The results and parameters of the three isotherms and their correlation coefficients are shown in Table 4.

The study results indicated that in comparison to other isotherms, The Freundlich isotherm model had a better correlation in dye removal by ZVI ($R^2 = 0.996$ for 25°C). Besides, by increasing the dye equilibrium concentration, the adsorption equilibrium capacity increased and maximum adsorption capacity of ZVI was obtained as 32.258 mg/g at 25°C. According to the results, the pseudo-second-order kinetic model was the most effective with $R^2 = 0.999$ for dye concentrations of 50, 100 and 200 mg/L. The results of the kinetics equations are shown in Table 5. The second-order rate constant k_2 and q_e are determined from the intercept and slope of the plot obtained by plotting t/q_t vs. t . Figure 6 shows the Freundlich adsorption isotherm and pseudo-second-order Kinetic model for RR-198 dye adsorption by ZVI.

Table 5. Results of the Kinetics Study

Kinetics	Parameters	Concentration, mg/L		
		50	100	200
Pseudo-first-order	k_1	0.036	0.036	0.034
	q_{ecal}	4.591	11.402	24.888
	R^2	0.969	0.985	0.988
Pseudo-second-order	k_2	0.017	0.005	0.002
	q_{ecal}	10.416	21.276	43.478
	h	1.848	2.506	3.984
	R^2	0.999	0.999	0.999
Intra-particle diffusion	K_{dif}	0.503	1.25	2.695
	C	5.035	7.536	12.48
	R^2	0.821	0.809	0.845

**Figure 6.** Modeling A) Freundlich Adsorption Isotherm and B) Pseudo-second-order Kinetic Model at different Dye Concentrations for RR-198 Adsorption Using ZVI

5. Discussion

5.1. Contact Time

According to the results, increasing the contact time in this system led to greater utilization of the iron powder active surface eventually resulting in production of

more free electrons, owing to higher quantities of Fe²⁺ ions in solution in acidic conditions. Therefore, the oxidation rate of RR-198 in the ZVI reaction is higher (26). The results of the study by Chang et al. (15) showed that at 2 g/L dose of zero valent iron, dye removal percentage reached its maximum range in the first 15 minutes, while no change was observed in dye removal after 120 minutes. The results of the study conducted by Rahmani et al. (2) on removing the two reactive dyes - reactive black 5 and acid orange 7 - using the Fenton-like mechanism showed that in Fe⁰ and Fe⁰/UV systems the maximum dye removal was obtained at pH = 3 and an equilibrium time of 120 minutes.

5.2. pH

Considering the fact that dye removal efficiency is maximized in acidic solutions, pH = 3 was used as the optimum pH level in further experiments. In general, more H⁺ exists in acidic solutions compared to basic solutions and consequently the reaction between the dye and the zero valent iron is stronger at basic pH levels. On the other hand, due to existing hydroxyl ions, Fe³⁺ is converted to Fe(OH)₃. This is mainly because of the hydrolyzation of iron ions and deposition of iron oxide on the metallic iron surface, which leads to occupation of active sites, slows the reaction speed, and eventually stops the reaction (27, 28). In the study by Satapanajaru et al. (29) on removing reactive red 198 and reactive black 5 dyes using iron nanoparticles, reduction of pH from 9 to 3 increased the kinetic speed of dye removal reaction. For both dyes, the removal efficiency reached 100% after 120 minutes. In the study conducted by Gomathi et al. (30) on methyl orange dye removal using the advanced Fenton process zero valent metallic iron, it was found that by decreasing pH from 9 to 2, the efficiency of dye removal increased, and optimal standard condition was found to be in pH = 3.

5.3. Adsorbent Dose and Initial Dye Concentration

In the zero valent iron powder system, increasing the iron powder dose raises the level of raw materials producing free electrons. Therefore, the free electrons production escalates leading to higher efficiency. Also, by increasing the iron dosage, more active sites are created for more rapid reaction speed at the initial stages and, as a result, more iron particles contact with dye molecules eventually increasing the dye removal efficiency (29, 31). As a result, increasing iron powder has a greater effect on dye removal. Deng et al. (32) also conducted a study on reactive dye removal utilizing the UV/Fe⁰ process and showed that by increasing the iron dose from 0.5 to 4 g/L in acidic pH, the dye removal speed improved. Daud et al. (33) studied acidic dye removal with the Fenton-like process, by increasing the catalyst dose from 2 to 5 g/L with a contact time of 150 minutes, where the removal rate of acid red 1 reached 99%. The findings of the current study showed that as the dye concentration increased, the re-

moval efficiency was decreased. This might be due to the saturation of the adsorption sites by the adsorbent in lower dye concentrations (34). In the study by Chang et al. (35) on dye removal through Fe^0 air process with reactive black 4 and 5, the removal percentage decreased in higher dye concentrations where the highest removal percentage was related to the concentration of 50 mg/L with 99% removal efficiency. The results of the study conducted by He et al. (31) on removing the Reactive dyes using zero-valent iron showed that by increasing the initial dye concentration from 300 mg/L to 700 mg/L, the decolorization efficiency decreased from 98.53% to 97.89% for reactive brilliant red K-2G, and from 99.8% to 95.74% for reactive brilliant blue KN-R and from 99.64% to 99.22% for malachite green.

5.4. Adsorption Isotherm and Kinetics

As shown in Table 4, the Freundlich isotherm model had better correlation compared to other isotherms. By increasing the dye equilibrium concentration, the adsorption equilibrium capacity is increased. This is due to the accessibility of the adsorption sites at the initial moments of the process. In general, values of n in ranges of 1 to 10 illustrated that the adsorbate was favorably adsorbed by the adsorbent (36). The value of n 1.524, at 25°C was greater than 1, indicating favorable removal conditions. Qadri et al. (37) conducted a study on cridine Orange using magnetic Nanoparticles. The results showed that Freundlich isotherm and pseudo-second-order kinetic model were the best for describing the reaction of adsorption. Iram et al. (38) conducted a study on neutral red dye removal using Fe_3O_4 hollow nanospheres, and found that pseudo-second-order kinetic model was the dominant model for dye concentrations of 20, 50, 100, 150 and 200 mg/L with $R^2 > 0.999$ (Table 6).

Iron powder is easy to use and can be continuously utilized due to its recyclability. Therefore, it is highly economical in removing azo dyes. Acidic pH is one of the effective factors in increasing the efficiency of zero valent iron powder and at pH = 3, the dye removal efficiency reaches its maximum level. Moreover, maximum adsorption was

observed at 5000 mg/L dose of zero valent iron powder and contact time of 120 minutes. Freundlich isotherm and pseudo-second-order Kinetic model are the best for describing the RR-198 adsorption reaction on zero valent iron powder catalyst. However, acidification of the environment is one of the major limitations of this method.

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Authors' Contribution

The overall implementation of this study including design, experiments, data analysis and manuscript preparation were the results of joint efforts by individuals who are listed as coauthors of this paper. All authors made extensive contributions to the review and finalization of this manuscript.

Financial Disclosure

The authors declare that there were no conflicts of interests.

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Table 6. Notations Used in Kinetic Models

	Nomenclature
Pseudo first-order rate constant, 1 min	K_1
Pseudo second-order rate constant, g/mg min	K_2
Intraparticle diffusion rate constant, mg/g min 0.5	K_{dif}
Equilibrium adsorbent concentration on adsorbent, mg/g	q_e
Calculated values of q_e , mg/g	q_{ecal}
Maximum monolayer capacity, mg/g	Q_m
Correlation coefficients	R^2

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