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Removal of Reactive Black 5 by zero-valent iron modified with various surfactants

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ABSTRACT

Zero-valent iron (ZVI) particles were synthesized by the aqueous phase borohydride reduction method, and the synthesized ZVI particles were used for the reductive degradation of Reactive Black 5 (RB5) dye in aqueous solution. The sizes of the synthesized ZVI particles were in the microscale range, with an average diameter of 13.57 μm. To study the efficiency of surfactant-treated ZVI particles for the decolorization of RB5 solution, three different surfactants, triton X-100 (TX100, non-ionic surfactant), cetyl trimethyl ammonium bromide (CTAB, cationic surfactant), and sodium dodecyl sulfate (SDS, anionic surfactant) were selected for the treatment of ZVI. The normalized residual concentration after decolorization of 500 mg/l RB5 by ZVI for 3 h was 0.236, while ZVI particles treated with TX100 (0.5 g/l), CTAB (1.0 g/l), and SDS (2.5 g/l) exhibited normalized residual concentration of 0.172, 0.154, and 0.393, respectively, after 3 h. The color removal efficiency was found to be increased with decrease in initial pH of dye solution, and ZVI exhibited good color removal efficiency at acidic pH. Decolorization kinetics by pseudo-first-order rate equation showed that removal rate was increased after treatment with TX100 as well as CTAB, while that was reduced after SDS treatment.

1. Introduction

The effluents discharged from textile, paper, printing, and leather industries contain a large variety of reactive dyes, and these dyes are of great environmental concern due to their huge applications and toxic nature [1]. Azo reactive dyes represent about 50% of all reactive dyes, and azo dyes are known to be toxic, carcinogenic and mutagenic. Their release in the environment can cause non-aesthetic pollution, and moreover, azo dyes are not degraded by conventional aerobic wastewater treatment due to their recalcitrance [2]. Thus, effluents containing dye molecules must be treated before their discharge into water bodies to minimize the threat to the environment [3]. Many processes are employed to remove dye molecules from colored effluents, and the treatment methods can be divided into three categories: (i) physical methods such as adsorption [4–6], membrane filtration [7], and ion exchange [8]; (ii) chemical methods such as chemical oxidation [9], electrochemical degradation [10], and ozonation [11]; and (iii) biological degradation [12]. The main disadvantage of physical methods is that they simply transfer the dye molecules to another phase rather than destroying them, and they are effective only when the effluent volume is small [13]. Chemical methods with high cost are rarely used in the actual treatment process, and the disposal of sludge containing chemicals at the end of treatment requires further use of chemicals [1,3]. Biological degradation processes suffer from low degradation efficiency or even no degradation for some dyes [14].

A great deal of attention has been paid to develop new treatment strategies to degrade the dye molecules. Zero-valent iron (ZVI) particles are inexpensive, environmentally friendly strong reducing agents and can donate two electrons to many environmental contaminants [15]:

$$Fe^0_{(S)} \rightarrow Fe^{2+} + 2e^- \qquad (1)$$

Due to its effective electron donating capacity, ZVI particles have been studied in recent years for the treatment of wastewater contaminated with chlorinated compounds [16,17], nitro aromatic compounds [18], nitrates [19], heavy metals [20], organochlorine pesticides [21], and dyes [22,23]. The reaction between Fe^0 and H_2O or H^+ can generate H atoms, which induce the cleavage of the azo bond (–N=N–), thus damaging the chromophore group and conjugated system of azo dyes [24]. ZVI particles convert azo dye into some products that are more susceptible to biological degradation processes [25]. The other advantages of using ZVI particles for the decolorization process include a low iron concentration remaining in the sludge, no requirement for further treatment of effluents, and easy recycling of the spent iron powder by magnetism [23].

To enhance the reducing effect of ZVI particles, some strategies, like coating iron particles with a second catalytic metal such as Pd, Pt, Ag or Ni [26] and modification with surfactants [27–29], have been practiced. Surfactants, an important group of amphiphiles,

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Table 1
Physico-chemical properties of different surfactants and RB5.

Name	Type	Chemical formula	MW (g/mol)	CMC (g/l)
TX100	Non-ionic surfactant	$C_{14}H_{22}O(C_2H_4O)_{9.5}$	625	0.11
CTAB	Cationic surfactant	$C_{19}H_{42}BrN$	365	0.34
SDS	Anionic surfactant	$NaC_{12}H_{25}SO_4$	288	2.33
RB5	Anionic reactive azo dye	$C_{26}H_{25}Na_4N_5O_{19}S_6.4Na$	991.82	–

MW: molecular weight; CMC: critical micelle concentration of surfactant in aqueous solution.

show affinity for hydrophobic contaminants and zero-valent iron (ZVI) surfaces. It has been reported that the modification of ZVI particles with surfactants may affect the degradation rate of chlorinated compounds, either favorably or adversely depending on the type of surfactant [30]. The alteration in the degradation rate of chlorinated compounds by ZVI particles in the presence of surfactants might be due to: (i) enhanced solubilization in the surfactant micelles in the solution phase, (ii) an enhanced surface concentration of contaminants on the sorbed surfactant micelles, and (iii) competitive sorption between the contaminant and surfactant [30–32].

The purpose of this study was to compare the efficiency of surfactant-treated ZVI with normal ZVI particles for the decolorization of a dye solution. Three different surfactants, triton X-100 (TX100, non-ionic surfactant), cetyl trimethyl ammonium bromide (CTAB, cationic surfactant) and sodium dodecyl sulfate (SDS, anionic surfactant) were selected for modification of ZVI particles. Reactive Black 5 (RB5) was selected as a model anionic dye, and it belongs to the class of azo reactive dyes, which are abundantly used in textile industries for dyeing. In this study, the surfactant concentrations were varied from below to above the critical micelle concentration (CMC). Different parameters, such as the initial solution pH, initial concentration of dye and contact time were also investigated in this study.

2. Materials and methods

2.1. Materials

Ferrous sulfate heptahydrate ($FeSO_4{\cdot}7H_2O$) was purchased from Samchun Pure Chemical Co. Ltd., Korea. Sodium borohydride ($NaBH_4$), RB5, and TX100 [octylphenolpoly (ethylene glycol ether)$_{9.5}$], CTAB, and SDS, were purchased from Sigma–Aldrich, USA. Table 1 represents the physico-chemical properties of RB5 and the three different surfactants used in this study. Other analytical grade chemicals were purchased from Sigma–Aldrich, USA.

2.2. Synthesis of zero-valent iron (ZVI) particles

ZVI particles were prepared by a liquid phase reduction method, using $NaBH_4$ as the reducing agent [33]. The experiments were conducted by degassing and purging the solvents with nitrogen (N_2) before use. Twenty-five milliliters (0.22 M) aqueous $NaBH_4$ solution were added drop-wise into 100 ml $FeSO_4{\cdot}7H_2O$ (0.04 M) with mild stirring under an N_2 atmosphere. Ferrous ion (Fe^{2+}) was reduced to elemental iron according to the following reaction:

$$Fe^{2+}_{(aq)} + 2BH^{-}_{4(aq)} + 6H_2O_{(aq)} \rightarrow Fe^{0}_{(s)} + 2B(OH)_{3(aq)} + 7H_{2(g)} \qquad (2)$$

After the reaction, the solution was continuously stirred for another 20 min under an N_2 environment. The resultant black ZVI particles were separated from the solution by centrifugation at a relative centrifugal force (RCF) of 2862 × g (4000 rpm) for 5 min and washed with N_2-saturated deionized water and at least three times with 99% absolute ethanol. Finally, the ZVI particles were dried in an oven at 60 °C.

2.3. Characterization

X-ray diffraction (XRD) patterns of the ZVI solid sample were studied using D/MAX2500H (Rigaku), and the patterns were recorded over a 2-theta (2θ) range of 5–90°. The particle size distribution of the synthesized ZVI particles was determined using a Cilas 1064 laser particle size analyzer with a measurement range from 0.04 to 500 μm in wet dispersion mode. The samples for particle size analysis were prepared by dispersing the particles in ethanol using ultrasounds.

2.4. Surfactant treatment of ZVI particles

Surfactant treatment of ZVI particles was carried out by adding 10 g/l ZVI into 100 ml surfactant solution prepurged with N_2 for 10 min. Three surfactants, such as TX100, CTAB, and SDS, were used for the treatment of ZVI particles, and the concentrations of these surfactants were varied from below to above their critical micelle concentration (CMC) as mentioned in Table 1. The TX100 concentration was varied from 0.05 to 5.0 g/l (CMC = 0.11 g/l) for the modification of ZVI particles (ZVI_{TX100}). For ZVI_{CTAB}, the CTAB concentration was varied between 0.1 and 5.0 g/l (CMC = 0.34 g/l). The SDS concentration in the solution was varied from 1.0 to 10.0 g/l (CMC = 2.33 g/l). After the treatment for 6 h under shaking conditions (200 rpm) at 30 °C and neutral pH, the resultant ZVI particles ($ZVI_{TX100,\ CTAB\ or\ SDS}$) were separated from the solution by centrifugation at an RCF of 2862 × g (4000 rpm) and washed with N_2-saturated deionized water and at least three times with 99% absolute ethanol. Finally, these ZVI particles were dried in oven at 60 °C.

2.5. Batch decolorization experiments

Batch experiments were conducted to evaluate the effect of the following parameters on the decolorization of RB5 by ZVI particles: initial solution pH, initial dye concentration and contact time. Experimental solutions of RB5 were obtained by dilution from a stock solution (1000 mg/l) into deionized water, and batch experiments were performed in 20-ml glass flat bottom vial fitted with a Teflon-lined screw cap. For each experiment, 2.5 g/l ZVI particles were added to 5 ml RB5 solution prepurged with N_2 for 5 min. All decolorization experiments were performed in triplicate and carried out in a thermostatically controlled shaker (200 rpm) at 30 °C and for 3 h, except for the decolorization kinetics studies. The initial pH value of the dye solution was adjusted to the desired levels, from pH 4 to 10, using HCl (0.5 M) or NaOH (0.5 M). The extent of decolorization as a function of the initial RB5 concentration was studied at concentrations of 10, 25, 50, 100, 250, 500 and 1000 mg/l. The decolorization kinetics experiments were performed for different time intervals up to 4 h. The initial RB5 concentration for all experiments was 500 mg/l, except for in the experiments testing the effect of the initial RB5 concentration on the decolorization of RB5 by ZVI particles. After a preselected time of decolorization, the samples were collected by filtration through a 0.45-μm PVDF membrane filter, and the residual RB5 concentration in the experimental solution (mg/l) was analyzed

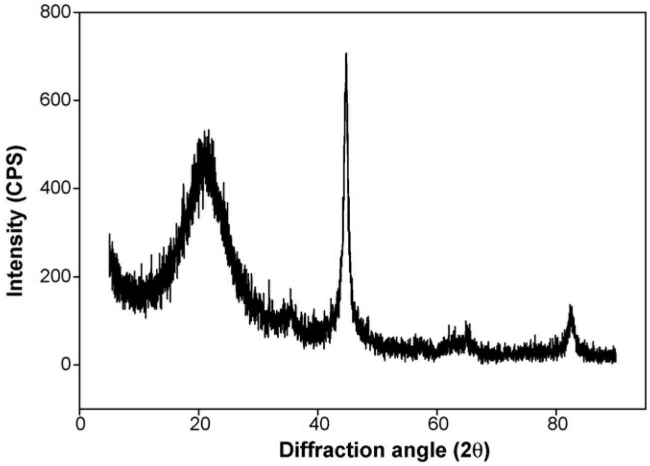

Fig. 1. X-ray diffraction patterns of the synthesized ZVI particles.

using a spectrophotometer (HACH DR-5000, USA) at a λ_{max} of 597 nm.

3. Results and discussion

3.1. Characterization

Fig. 1 shows that the XRD diffractogram of the synthesized ZVI particle had only a characteristic 2θ value of 44.7°, and no signals for iron oxides with a 2θ value of 36° were found. These diffraction patterns indicate that the iron present in the sample is mainly in its zero-valent state, and all zero-valent irons are in a single-phase cubic closet-packed structure.

Fig. 2 shows the particle size distribution (PSD) of the synthesized ZVI particles and their diameter ranges from 0.01 to 45.0 μm with a mean diameter of 13.57 μm. A few particles displayed a diameter as large as 40–45 μm, whereas the diameter of 90% of the particles was less than 26.9 μm. Thereby, the PSD results for our synthesized ZVI particles indicated that the particles were of microscale diameter.

3.2. Effect of surfactant

The decolorization of RB5 in aqueous solution by ZVI particles mostly occurs via the reductive degradation of dye molecules, and the reductive cleavage of the azo bonds of RB5 is accompanied by the oxidation of Fe^0, resulting in the decolorization of RB5 [34].

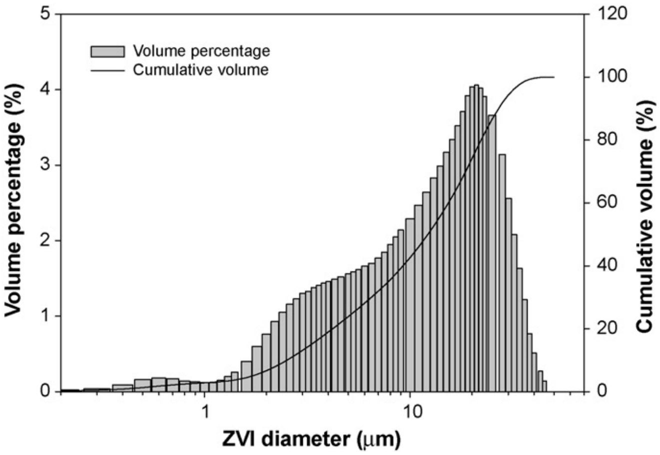

Fig. 2. Particle size distribution (PSD) and cumulative volume for ZVI particles.

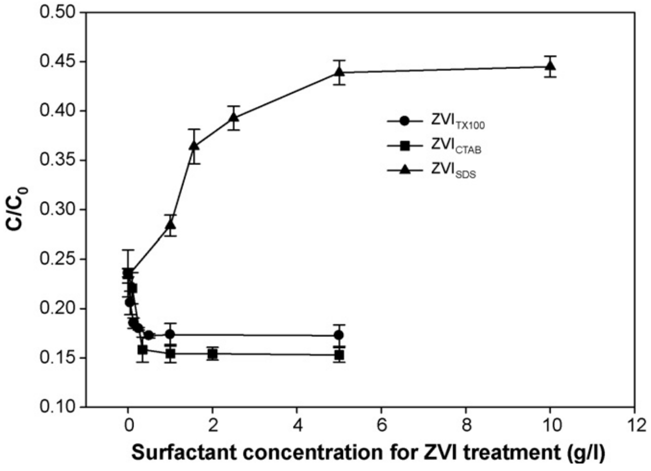

Fig. 3. Effect of surfactant treatment of ZVI particles on decolorization; RB5, 500 mg/l; ZVI, ZVI_{TX100}, ZVI_{CTAB}, and ZVI_{SDS}, dosages, 2.5 g/l; pH 6.

$$Fe^0 + 2H^+ \rightarrow Fe^{2+} + H_2 \tag{3}$$

$$-N{=}N- + 2H^+ + 2e^- \rightarrow -NH{-}NH- \tag{4}$$

$$-NH{-}NH- + 2H^+ + 2e^- \rightarrow -NH_2 + H_2N- \tag{5}$$

The heterogeneous reaction using ZVI involves five steps: (i) mass transfer of RB5 to the ZVI surface from the bulk solution, (ii) sorption of dye on the ZVI surface, (iii) reductive degradation of RB5 at the ZVI surface, (iv) desorption of the reaction product from the ZVI surface, and (v) mass transfer of the product into the bulk solution [35].

Fig. 3 shows the effect of surfactant treatment (TX100, CTAB, and SDS) of ZVI particles on the normalized residual concentration (C/C_0) of RB5 (500 mg/l) by ZVI. The C/C_0 value is the ratio of residual concentration (C) of RB5 and initial dye concentration (C_0). The removal efficiency of RB5 was found to be varied with surfactant dosage. The C/C_0 value of ZVI without TX100 treatment was 0.236. ZVI_{TX100}, after treatment with TX100 at a concentration of 0.5 g/l, exhibited maximum color removal efficiency and the C/C_0 value of RB5 in the solution was 0.172. As shown in Fig. 3, ZVI_{CTAB} after treatment with CTAB at a concentration of 1 g/l, decreased the C/C_0 value of RB5 from 0.236 to 0.154, and a further increase in the CTAB concentration did not produce any significant effects on removal efficiency. Both CTAB and TX100 treatment exhibited positive effects up to a certain concentration on color removal by ZVI particles. Beyond that concentration, they did not have any additional effects. In contrast to those results, SDS treatment significantly reduced the decolorizing power of ZVI, and the C/C_0 value increased steadily with an increase in the SDS concentration during the treatment process.

The presence of surfactant in the solid–water interface could beneficially affect the contaminant interaction with the metal surface by enhancing the surface affinity, or adversely by blocking the reactive sites. Cationic surfactants are the most likely candidates to accumulate at the solid–water interface of ZVI particles [27,36]. CTAB-treated ZVI particles (ZVI_{CTAB}) exhibited more reductive degradation of RB5 than normal ZVI particles and increase in CTAB concentration up to its CMC (0.34 g/l) increased color removal efficiency by ZVI_{CTAB}. This observation could be explained by the fact that increase in CTAB dosage would increase the RB5 concentration near ZVI surfaces by electrostatic interactions between the positively charged head groups of CTAB molecules and the negatively charged RB5 molecules and partly through hydrophobic interactions between a hydrophobic tail of CTAB and hydrophobic

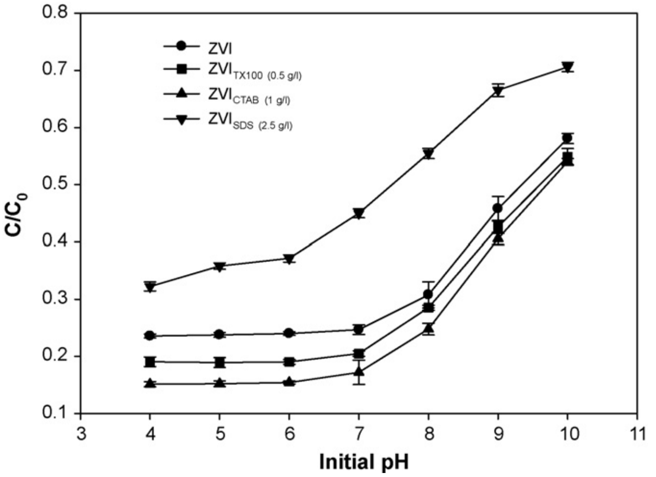

Fig. 4. Effect of initial pH on decolorization; RB5, 500 mg/l; ZVI, $ZVI_{TX100,\ 0.5\,g/l}$, $ZVI_{CTAB,\ 1.0\,g/l}$, and $ZVI_{SDS,\ 2.5\,g/l}$ dosages, 2.5 g/l.

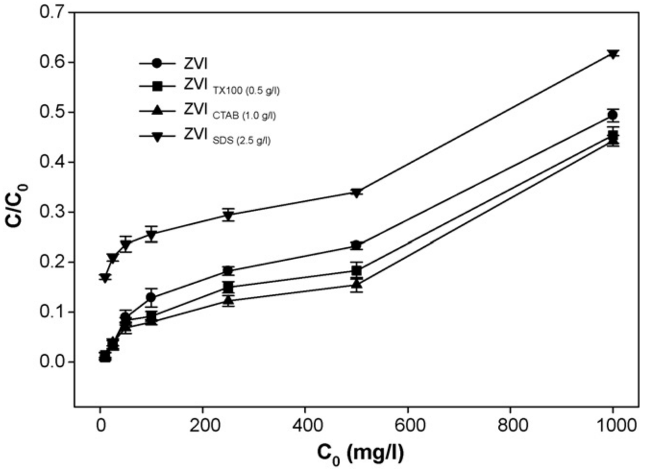

Fig. 5. Effect of the initial dye concentration on decolorization; ZVI, $ZVI_{TX100,\ 0.5\,g/l}$, $ZVI_{CTAB,\ 1.0\,g/l}$, and $ZVI_{SDS,\ 2.5\,g/l}$ dosages, 2.5 g/l; pH 6.

moieties of RB5. The increase in CTAB dosage beyond CMC hardly increased its color removal efficiency, indicating CTAB adsorption on ZVI particles might have reached the saturation limit. ZVI_{TX100} particles would increase the decolorization by increasing the RB5 concentration near metal surfaces through hydrophobic interactions between sorbed TX100 and RB5 molecules. The enhancement of the color removal efficiency by ZVI_{TX100} with increasing TX100 dosage above CMC (0.11 g/l) up to 0.5 g/l might be due to increased hydrophobic interactions between dye and TX100 molecules. TX100 dosage beyond 0.5 g/l did not produce any further increase because of its saturation limit of adsorption on ZVI particles. ZVI_{SDS} exhibited less decolorization than ZVI particles due to electrostatic repulsion between sorbed SDS and RB5 molecules. However, the increase in SDS concentration during the treatment process gradually reduced the removal efficiency by ZVI_{SDS} because of negatively charged groups to its surface. Sometimes, sulfate groups of SDS passivate reactive sites of ZVI by forming secondary minerals, or accelerate non-specific corrosion of ZVI particles [37].

3.3. *Effect of initial pH*

The effect of the initial solution pH on the color removal efficiency of RB5 by ZVI particles is shown in Fig. 4, with the initial dye concentration of 500 mg/l. The pH study for decolorization using surfactant-treated ZVI particles ($ZVI_{TX100,\ CTAB\ or\ SDS}$) was also performed. The results showed that the color removal was much higher in acidic pH (pH 4–6), and decolorization occurred in this pH range through the direct reduction on ZVI particles, as shown in Eqs. (3)–(5). The pH change was monitored during decolorization and no acid or alkali was added to maintain pH throughout the process. Preliminary tests showed that pH increased to 6.0, 6.4, and 7.0 from initial pH 4.0, 5.0, and 6.0, respectively, after 3 h using ZVI particles as well as surfactant-treated ZVI particles ($ZVI_{TX100,\ CTAB\ or\ SDS}$). At neutral pH (pH 7.0), no pH change was found at the most and the color removal might be due to sorption of RB5 molecules onto ferrioxyhydroxide (FeOOH) formed on the ZVI particles. It is known that iron reacts with water in aqueous environment to form a layer of oxyhydroxide on the surface.

$$Fe^{0}_{(S)} + 2H_2O \rightarrow FeOOH + 1.5H_{2(g)} \qquad (6)$$

The low decolorization in alkaline pH levels (pH 8–10) could be explained by the fact that the ZVI surface might be covered by the corrosion products formed in alkaline conditions, and this phenomenon causes a lowering of the reducing power of ZVI and mass transport limitations on ZVI surface. The charge of the ZVI surface also changes from positive to negative with an increase in the solution pH [38], which diminishes the sorption of RB5 molecules onto ZVI surfaces. No pH change during decolorization was found when initial pH of RB5 solution was maintained in alkaline range (pH 8–10). Therefore, the optimized standard condition for conducting the reductive degradation of RB5 is in an acidic pH.

As shown in Fig. 4, the C/C_0 values of RB5 by $ZVI_{TX100,\ 0.5\,g/l}$ or $ZVI_{CTAB,\ 1.0\,g/l}$ were lower than those of ZVI particles at all pH levels. $ZVI_{SDS,\ 2.5\,g/l}$ showed higher C/C_0 values than normal ZVI particles at all pH levels tested herein, indicating less color removal by $ZVI_{SDS,\ 2.5\,g/l}$ than ZVI. Thereby, the influence of surfactant on decolorization seems not to depend much on the initial pH of the solution.

3.4. *Effect of initial dye concentration*

The effect of initial RB5 concentration on color removal efficiency by ZVI particles is shown in Fig. 5, with different initial concentrations (10, 25, 50, 100, 250, 500, and 1000 mg/l). The C/C_0 values of RB5 by ZVI particles were found to be varied with the initial dye concentration. The observed pH range (initial pH-final pH) during decolorization by ZVI particles was 6.0–6.4 with 10, 25, and 50 mg/l initial concentrations of RB5, and 6.0–7.0 with other initial concentrations (100, 250, 500, and 1000 mg/l). In general, as the initial dye concentration increased, the normalized residual concentration value (C/C_0) increased from 0.011 to 0.493, indicating that the removal efficiency by ZVI decreased with increase in initial dye concentration. The range of C/C_0 value obtained by $ZVI_{TX100,\ 0.5\,g/l}$ and $ZVI_{CTAB,\ 1.0\,g/l}$ was 0.0144–0.454 and 0.007–0.443, respectively, with initial RB5 concentration range of 10–1000 mg/l, indicating that the removal efficiency by ZVI particles could be enhanced after TX100 as well as CTAB treatment. The C/C_0 values in the range of 0.170–0.620 by $ZVI_{SDS,\ 2.5\,g/l}$ with initial dye concentration range (10–1000 mg/l) indicated its less removal efficiency than ZVI.

3.5. *Kinetic studies*

The kinetic results of decolorization of RB5 (500 mg/l) with ZVI and surfactant-treated ZVI particles ($ZVI_{TX100,\ CTAB\ or\ SDS}$) are summarized in Fig. 6. The decolorization occurred very fast initially, and the maximum color was removed after 2 h. No further changes in the C/C_0 values were found after 3 h of decolorization. As shown in Fig. 6, the removal rate of RB5 by $ZVI_{TX100,\ 0.5\,g/l}$ and $ZVI_{CTAB,\ 1.0\,g/l}$ were faster than those of ZVI and $ZVI_{SDS,\ 2.5\,g/l}$. Moreover, the normalized residual concentrations after 3 h were 0.182 and 0.154 for

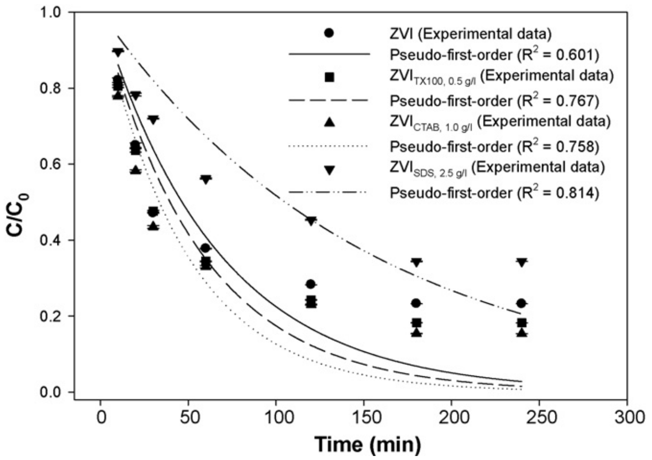

Fig. 6. Kinetics study of decolorization by pseudo-first-order rate equation; RB5, 500 mg/l; ZVI, $ZVI_{TX100,\ 0.5\,g/l}$, $ZVI_{CTAB,\ 1.0\,g/l}$, and $ZVI_{SDS,\ 2.5\,g/l}$ dosages, 2.5 g/l; pH 6.

Table 2
Rate parameters of RB5 decolorization by ZVI with the initial RB5 concentration of 500 mg/l and ZVI dosage of 2.5 g/l.

ZVI type	Pseudo-first-order k_1 (1/min)	R^2
ZVI	0.015	0.601
$ZVI_{TX100,\ 0.5\,g/l}$	0.018	0.767
$ZVI_{CTAB,\ 1.0\,g/l}$	0.021	0.758
$ZVI_{SDS,\ 2.5\,g/l}$	0.007	0.814

$ZVI_{TX100,\ 0.5\,g/l}$ and $ZVI_{CTAB,\ 1.0\,g/l}$, respectively, and those for ZVI and $ZVI_{SDS,\ 2.5\,g/l}$ were 0.232 and 0.344, respectively. The pH variation was detected during decolorization and only sharp rise from pH 6.0 to 7.0 during first 10 min was found. The pH range (initial pH-final pH) of all the systems after 4 h decolorization was 6.0–7.0.

For the ZVI-dye systems, a pseudo-first-order reaction is considered with respect to the dye concentration and the normalized residual concentration (C/C_0) is used to describe the removal kinetic rate [39]

$$\frac{C}{C_0} = e^{-k_1 t} \tag{7}$$

where k_1 (1/min) denotes the pseudo-first-order rate constant and t is the reaction time. C_0 and C denote the initial and residual concentration of the dye after time t, respectively. The values of k_1 obtained from non-linear regression analysis of all the kinetic data in Fig. 6 are provided in Table 2.

As shown in Table 2, k_1 of the systems with $ZVI_{TX100,\ 0.5\,g/l}$ (0.018/min) and $ZVI_{CTAB,\ 1.0\,g/l}$ (0.021/min) was higher than that with ZVI (0.015/min). The k_1 of the system with $ZVI_{SDS,\ 2.5\,g/l}$ (0.007/min) indicated the removal kinetic rate of RB5 by $ZVI_{SDS,\ 2.5\,g/l}$ was lower than $ZVI_{TX100,\ 0.5\,g/l}$ and $ZVI_{CTAB,\ 1.0\,g/l}$. It is known that the reaction involved in the ZVI-contaminant system is surface mediated. Thus, the reaction rate is not only related to the contaminant concentration, but also is related to the reactive sites on ZVI particles. The reaction rate was deviated from a first-order reaction particularly in a later part due to exhaustion of the most active sites on ZVI particles.

4. Conclusions

The synthesized ZVI particles of microscale size exhibited effective decolorization for RB5 dye. The treatment of ZVI with TX100 and CTAB was found to be effective in enhancing the removal efficiency of RB5 by ZVI, while its effectiveness was decreased by SDS treatment. The diminished color removal with SDS compared to TX100 or CTAB is due to an electrostatic repulsion between SDS and RB5 molecules. The normalized residual concentration (C/C_0) was found to be increased with increase in initial dye concentration and decreased with a decrease in the initial pH of the solution. Decolorization kinetics by pseudo-first-order rate equation exhibited that ZVI after treatment with TX100 or CTAB displayed faster decolorization rate than normal ZVI particles.

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