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Influence of chitosan on modified nanoscale zero-valent iron for arsenate removal

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ABSTRACT

Chitosan-modified nanoscale zero-valent iron (CNZVI) was synthesized for arsenate removal from water. Batch experiments were carried out to examine the influence of weight percent of chitosan loading in the CNZVI, and the initial solution pH including acidic, neutral, and basic conditions. As a result, the arsenate removal rate increased with higher weight percent of chitosan loading, but decreased with higher initial pH. In addition, the efficiencies of CNZVI for arsenate removal did not change significantly for the three investigated pH conditions, when higher percentage of chitosan loading was applied. The results demonstrate that CNZVI has the potential as an effective agent for field arsenic removal.

Keywords: Adsorption; Arsenic removal; NZVI; Chitosan

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1. Introduction

Arsenic (As), which is a well-known carcinogenic substance, has been detected in groundwater at a significant level in many countries. The predominant oxidation states of inorganic arsenic in water are arsenate (As(V)) and arsenite (As(III)) [1]. In oxic condition and pH 5–12, the dominant arsenic species is As(V) which requires further removal. Several conventional methods have been used to remove arsenic, such as precipitation–coagulation, ion exchange, membrane filtration, and adsorption. Adsorption is a process using solid material to remove the contaminants by physical and chemical forces. It was found that iron adsorbents have been developed for applying in arsenic removal such as granular ferric hydroxide, iron oxide-coated sand, and zero-valent iron (Fe^0 or ZVI) [2–5]. Nanoscale zero-valent iron (NZVI or Fe^0) was found to be an effective adsorbent for removing both As(V) and As(III) in water. According to the laboratory studies, the NZVI of 1–100 nm size has more potential in arsenic removal than microscale ZVI, due to its larger surface area and higher surface reactivity [6,7]. However, the problem of *in situ* field application is that NZVI tends to agglomerate. The agglomeration causes the decrease in reactive surface area. Large

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particle sizes also have the limitation of their mobilities in the subsurface area [8,9].

Chitosan has been used as a modifier for the preparation of metallic nanoparticles, such as silver, gold, platinum, and palladium [10]. These metallic nanoparticles were reported to disperse well in the presence of chitosan [11]. Chitosan consists of N-acetyl glucosamine and glucosamine units, produced by the partial deacetylation of chitin under alkaline condition, as shown in Fig. 1 [12]. Chitosan is a biopolymer which is claimed as a nontoxic substance.

Due to hydroxyl (–OH) and amino (–NH_2) groups in the structure, chitosan can dissolve in acidic solution and bind with metal ions. Guibal [13] reported that the main reactive unit of chitosan for metal ions is its amine sites. Furthermore, the presence of positive charge, because of the protonation of amine groups in acidic solution, facilitates the attraction of metal anions.

In this study, the chitosan-modified nanoscale zero-valent iron (CNZVI) particles were synthesized and characterized for the morphology and point of zero charge. In addition, the arsenate adsorption performance of CNZVI was evaluated and compared with the bare NZVI. Important influent factors, including the amount of chitosan in the modified-NZVI (weight percent), and the initial pH (acidic, neutral, and basic condition), were investigated.

2. Materials and method

2.1. Materials

Arsenate solution was prepared by dissolving $Na_2HAsO_4{\cdot}7H_2O$ of reagent grade (Panreac Quimica SA, Spain) in deionized (DI) water. Sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH) were used for adjusting the solution pH. The NZVI was synthesized by using sodium borohydride ($NaBH_4$) for the chemical reduction of ferric (Fe^{3+}) ions ($FeCl_3{\cdot}6H_2O$). Chitosan with a molecular weight of 100,000–300,000 (Dalton) was purchased from Acros Organics.

2.2. Preparation of NZVI

The NZVI was synthesized by borohydride reduction method [14]. The solution of 40 mL of 0.25 M $NaBH_4$ was added into 40 mL of 0.045 M $FeCl_3{\cdot}6H_2O$ aqueous solution. By dropwise addition, the mixture was agitated by a revolving propeller. After 10 min of mixing, the ferric ion (Fe^{3+}) was reduced to zero-valent iron (Fe^0) by borohydride, as shown in Eq. (1):

$$\begin{aligned} &Fe(H_2O)_6^{3+} + 3BH_4^- + 3H_2O \\ &\quad \rightarrow Fe^0 + 3B(OH)_3 + 10.5H_2 \end{aligned} \tag{1}$$

2.3. Preparation of CNZVI

CNZVI was prepared with a single-step synthesis procedure, which involves the use of a modifier to react with nanoparticles formed in the mixture. The method of CNZVI preparation was similar to that of NZVI synthesis, except that the solution of chitosan (0.5%, w/v) in 1% acetic acid was mixed with $FeCl_3{\cdot}6H_2O$ solution before the $NaBH_4$ was added. The resulted CNZVI particles were separated from the liquid by a magnet. The amount of modified chitosan was expressed as percent by weight of chitosan based on 0.1 g NZVI denoted as % CNZVI.

2.4. Batch experiments

The capacity of CNZVI for removing arsenate was evaluated by comparing with the bare NZVI. The studied parameters include initial pH (4, 7, and 9), and loadings of chitosan (2.5, 7.5, 15, and 30% wt). Right after the synthesis, each type of nanoparticles was added into 3,000 μg/L of As(V) solution, with a

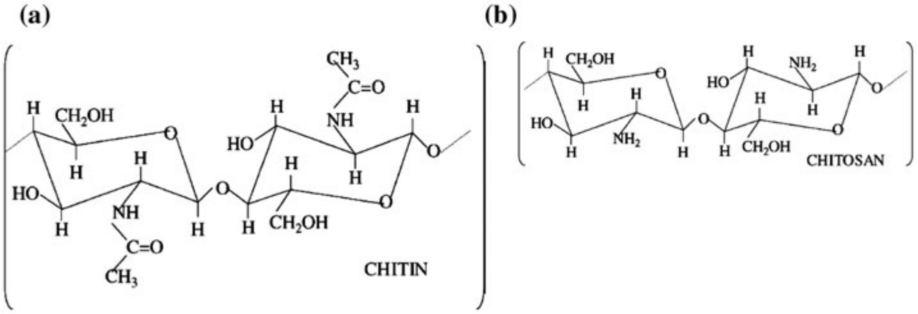

Fig. 1. Structure of (a) chitin (poly (N-acetylglucosamine)) and (b) chitosan.

liquid volume of 1.5 L. The mixture was stirred at 300 rpm. The NZVI dosage used was 0.067 g/L. During the mixing, 5 mL of water samples were taken at different time intervals. The samples were filtered through 0.45 μm membrane and acidified by nitric acid before the analysis of arsenic concentration.

2.5. Analytical methods

Analyses of total dissolved arsenic were carried out using inductively coupled argon plasma (ICP) (Thermo Scientific Model iCAP 6000 series). For the DO measurement, the DO meter (Oxi 330i) was used, while the pH and ORP meters (Suntex TS-1) were used for pH and ORP measurements, respectively. For particles characterization, the synthesized NZVI and CNZVI were vacuum-dried in a freeze drier for 24 h, and then kept in a desiccator. Surface morphology and elements composition of dried particles were analyzed by scanning electron microscope (SEM/EDS: JSM-5800LV). Nanoparticles image and size were examined by transmission electron microscopy (TEM: TECNAI G^2 20 (FEI)). Surface charge was measured by salt addition method [15].

3. Results and discussion

3.1. Characterizations of nanoparticles

Fig. 2 depicts the morphology of pristine NZVI and CNZVI particles that were separated from the solution right after synthesis. The lump of CNZVI particles appeared to be more glossy, and it looked like that the NZVI particles were covered by viscous gel, as shown in Fig. 2(b). In addition, the CNZVI was more difficult to separate from solution by magnet. The coating of chitosan resulted in the reduction in electrostatic interaction force between NZVI particles. Consequently, the particle aggregation tendency would then be mitigated, implying that particle mobility within the porous media will be enhanced. This can be beneficial for *in situ* application [16].

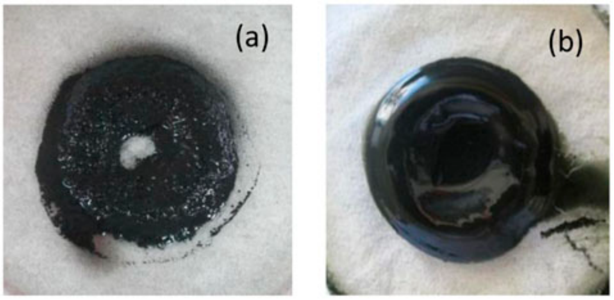

Fig. 2. Morphology of pristine particles of (a) NZVI and (b) CNZVI (30% CNZVI).

3.1.1. Particles morphology and element composition

Surface images of the dried NZVI and CNZVI were obtained by using SEM and TEM, as presented in Figs. 3 and 4, respectively. In Fig. 3(a) and (b), more aggregation was observed in a chain structure for the bare NZVI, whereas dispersion of iron particles was found higher for CNZVI. They are spherical in shape, especially for the CNZVI particles. TEM images of dried unmodified and modified samples are shown in Fig. 4. The thin shell of iron oxides covering NZVI particles is clearly seen in Fig. 4(a). Due to the magnetic properties, there was the chain-like aggregation among the NZVI particles, the diameter of which was in the range of 30–90 nm. In Fig. 4(b), it can be observed that the smaller size nanoparticles are covered by chitosan. The diameter of CNZVI particle was found in the range of 20–60 nm. This could be explained by the chemical properties of chitosan, which tends to bind with metal ions [17]. The chemical reaction of chitosan and iron may reduce magnetic force among the NZVI particles, due to the outer surface of NZVI being coated by chitosan.

As-synthesized nanoparticles were kept in a dessicator for two days before the elemental analysis by EDS. The result is shown in Table 1. The oxygen content in NZVI was 13.75%, whereas only 4.61% was found in 30% CNZVI. The highest percentage of Fe/O ratio was found in 30% CNZVI; meanwhile NZVI appeared as the lowest Fe/O ratio. The detected oxygen content was probably due to iron oxides from oxidation reaction which may occur during the storage. Therefore, this could be assumed that chitosan retarded the oxidation of NZVI particles, resulting in less iron oxides formation.

3.1.2. Point of zero charge (pH_{PZC}) of the nanoparticles

Table 2 shows pH_{PZC} for NZVI and CNZVI with different chitosan loadings. In the higher chitosan loading, the higher value of pH_{PZC} was observed. As is known, the surface charge of particles will be predominantly positive when the solution pH is lower than pH_{PZC} [18,19], whereas, a negative charge will be observed at the pH value higher than pH_{PZC}. This implies that the charge of 30% CNZVI is still remaining positive in the solution until the pH value is higher than 10.1, while the NZVI turns to be negatively charged when the pH is higher than 7.75. With the extension of pH_{PZC} to higher value, as mentioned,

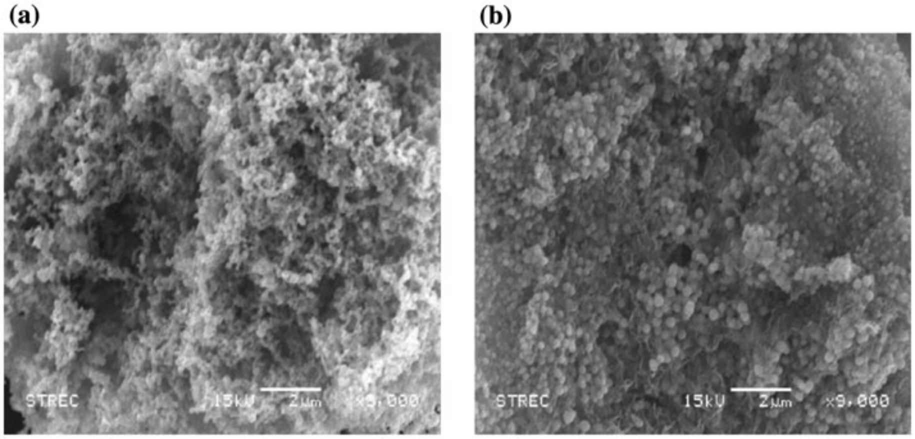

Fig. 3. SEM images of (a) NZVI and (b) 30% CNZVI.

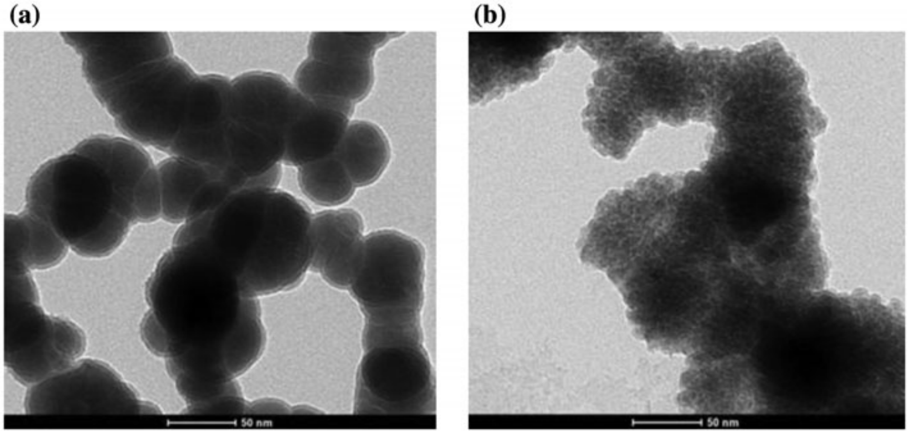

Fig. 4. TEM images of (a) NZVI and (b) 30% CNZVI.

Table 1
EDS analysis of NZVI and CNZVI[a]

Type of particles	Fe (%)	O (%)
NZVI	86.25	13.75
2.5% CNZVI	87.05	12.95
7.5% CNZVI	89.54	10.46
15% CNZVI	91.69	8.31
30% CNZVI	95.39	4.61

[a] Analysis carried out after the synthesis for 2 d.

Table 2
pH_{PZC} of the synthesized nanoparticles

Type of particles	pH_{PZC}
NZVI	7.75
2.5% CNZVI	7.83
7.5% CNZVI	8.3
15% CNZVI	9.2
30% CNZVI	10.1

it could reduce the drawback of poor resistance property of chitosan in acidic condition [20].

3.2. *Effect of chitosan loading*

With an initial concentration of 3,000 μg/L, an influence of chitosan loading on As(V) removal was studied under different pH, such as 4, 7, and 9. As shown in Fig. 5, a higher removal rate on CNZVI is found when compared to that on NZVI, given all initial studied pH. However, the removal rate seems faster, especially in the case of initial pH 7 and 9. At all initial pH, the As(V) was removed faster when the higher loading of chitosan was applied. This could be

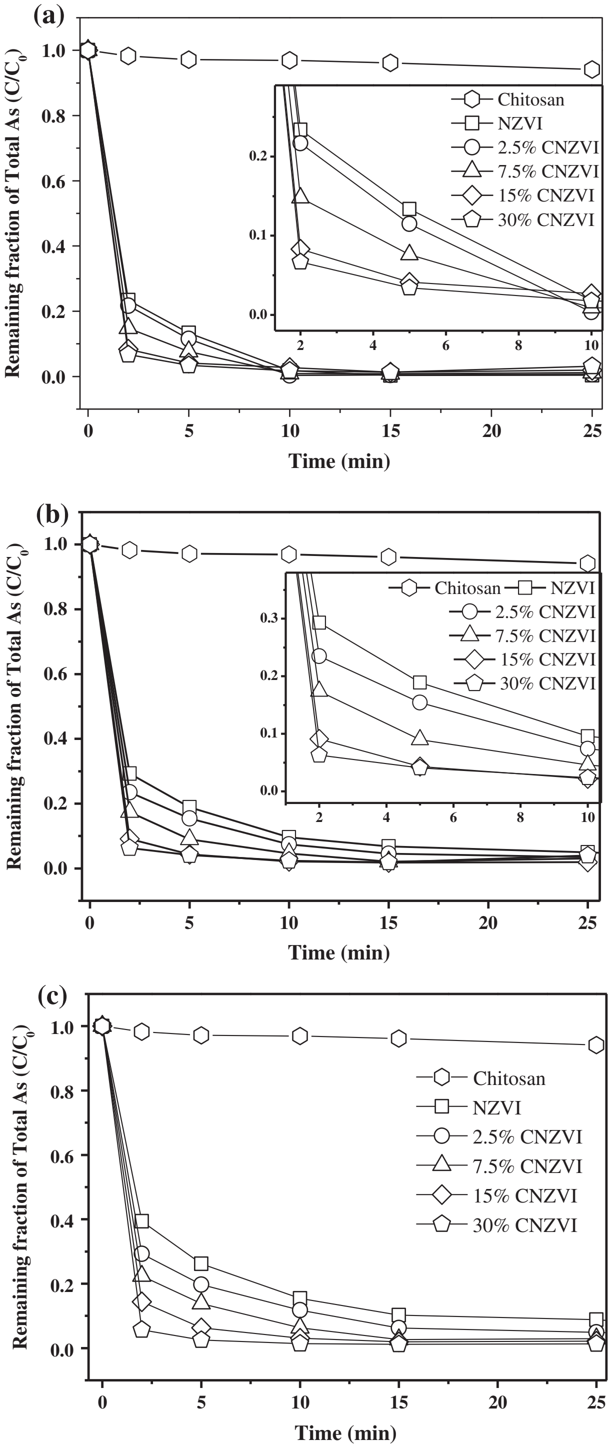

Fig. 5. Effect of chitosan loading in the CNZVI on As(V) removal at initial pH (a) 4, (b) 7, and (c) 9, Conditions: Initial As(V) concentration = 3,000 μg/L, NZVI dosage = 0.067 mg/L.

explained that less aggregation of modified NZVI particles leads to an increase in its reactive surface area. Moreover, chitosan is a cationic biopolymer that can facilitate the attraction of metal anions because of the protonation of amine groups, according to Guibal [13]. It appears that more acidic solution leads to the accumulation of protons on chitosan surface. Tanboonchuy et al. [21] reported that the predominant forms of As(V) in the solution of pH 2–7 and pH 7–12 are $H_2AsO_4^-$ and $HAsO_4^{2-}$, respectively. Therefore, anionic forms of arsenic will be more easily attracted onto CNZVI of positive charge, in view of pH_{pzc}, resulting in a higher removal performance.

The adsorption capacities by related chitosan modifiers were obtained from the adsorption isotherm, which was carried out in a wide range of concentration (up to 10^6 μg/L). In our study, the arsenate adsorption capacity of chitosan-composited NZVI was investigated with a lower arsenate concentration of 3,000 μg/L, and it is noticed here that its capacity was calculated at the end of reaction time. As shown in Table 3, the as-obtained chitosan seems unfavorable to arsenate capture when compared to other reports.

To understand the removal kinetics on NZVI and CNZVI, two kinetic models including pseudo-first-order, and pseudo-second-order models were selected. The integration of pseudo-first-order kinetic is given by Eq. (2), where C_0 is the initial concentration of arsenate solution, C is the concentration of arsenate as a function of time, and k_1 (min^{-1}) is the pseudo-first-order rate constant. Value of k_1 can be calculated from the slope and intercept of the linear plotting of $\ln(C_0/C)$ vs. t.

$$\ln\frac{C}{C_0} = -kt \tag{2}$$

The integration of pseudo-second-order model is expressed in Eq. (3), where k_2 ($\text{L}\,(\mu\text{g min})^{-1}$) is the pseudo-second-order rate constant. k_2 can be calculated from the slope and intercept of $1/C$ vs. t, respectively.

$$\frac{1}{C} - \frac{1}{C_0} = kt \tag{3}$$

The data on the removals of arsenate by NZVI and CNZVI under different loadings of chitosan were further used to analyze their possible kinetic adsorption models, including pseudo-first-order and second-order models. The kinetic parameters of each model are evaluated by linearization of model, as plotted in Fig. 6. The results are tabulated in Table 3. According to the correlation coefficient (R^2), its small values ($R^2 = 0.21–0.34$) indicate poor fit on pseudo-first-order kinetic model. On the other hand, the

Table 3
Literature data on arsenate adsorption capacity under different conditions

Arsenate adsorption (mg/g)	Conditions	Refs.
93.46_{isotherm}	Chitosan-coated Al_2O_3, pH 4 and C_0 = 104–106 μg/L	[22]
0.56_{isotherm}	Chitosan/PVA	[23]
200_{isotherm}		
	Chitosan/PVA/NZVI pH 4 and C_0 = 100–10,000 μg/L	
1.33_{isotherm}	Chitosan, pH 5.5	[24]
14.16_{isotherm}	Chitosan, pH 3.5 and C_0 = 100–10,000 μg/L	
2.92_{eq}[a]	Chitosan, pH 4 and C_0 = 3,000 μg/L	This study

[a] Calculated at the end of the test.

pseudo-second-order model is satisfactorily fitted by the experimental data (R^2 = 0.94–0.99). The rate constant (k) was found to increase when the percentage of chitosan loading increased.

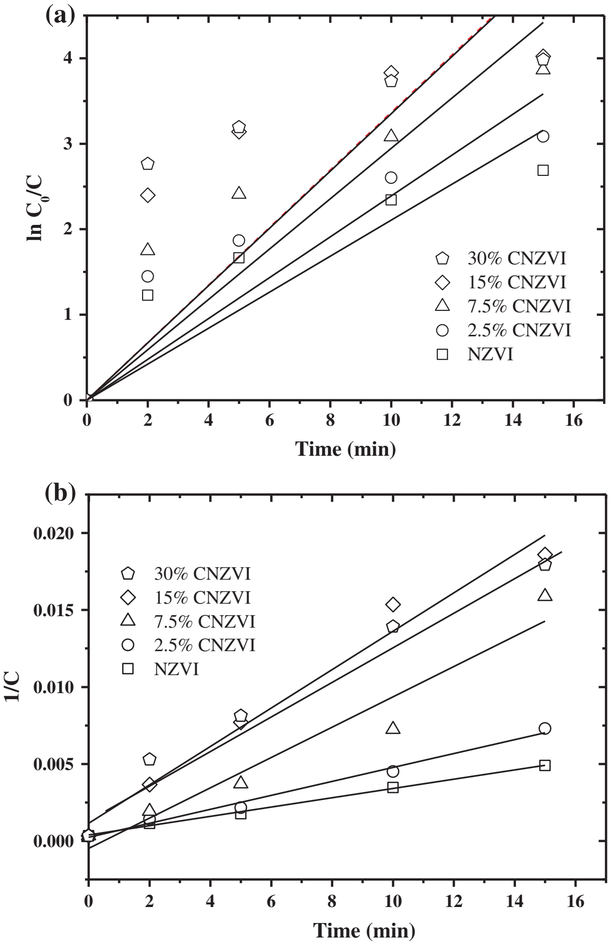

Fig. 6. Linearization of kinetic model under different percentage of chitosan loadings: (a) pseudo-first-order kinetic model, and (b) pseudo-second-order kinetic model.

3.3. Effect of initial pH

The removal of As(V) using pristine NZVI and CNZVI were carried out under the initial pH of 4, 7, and 9 (Fig. 7). It was found that the acidic condition favored the removal of As(V) when the NZVI and 2.5% CNZVI were applied, as shown in Fig. 7. This may be due to electrostatic forces. As presented in Table 2, the solution pH_{PZC}'s for the synthesized NZVI and 2.5% CNZVI are 7.75 and 7.83, respectively. Therefore, the surface of particles would be dominated by positive charges in the acidic and neutral solution. The surface will become negatively charged as the solution pH is higher than its pH_{PZC} value. In addition, the As(V) will exhibit a negative charge under alkaline condition. As a result, the removal efficiency will decrease due to electrostatic repulsion between As(V) and NZVI or 2.5% CNZVI at a higher solution pH.

Furthermore, interesting results occur in the higher chitosan loading. The As(V) removal seems to be close to each other in the two cases of 15 and 30% CNZVI (Table 4), with their pH_{PZC} values being 9.2 and 10.1, respectively. This implies that both cases still show the positive charges under alkaline condition. Therefore, the enhancement of the As(V) removal can be

Table 4
Kinetic parameters for arsenate removal by NZVI and CNZVI

	Model parameters			
	Pseudo-first-order		Pseudo-second-order	
Samples	k_1 (min^{-1})	R^2	k_2 (L (μg min)$^{-1}$)	R^2
NZVI	0.706	0.21	0.0003	0.99
2.5% CNZVI	0.702	0.24	0.0005	0.99
7.5% CNZVI	0.705	0.29	0.001	0.94
15% CNZVI	0.399	0.34	0.0012	0.98
30% CNZVI	0.227	0.34	0.0012	0.98

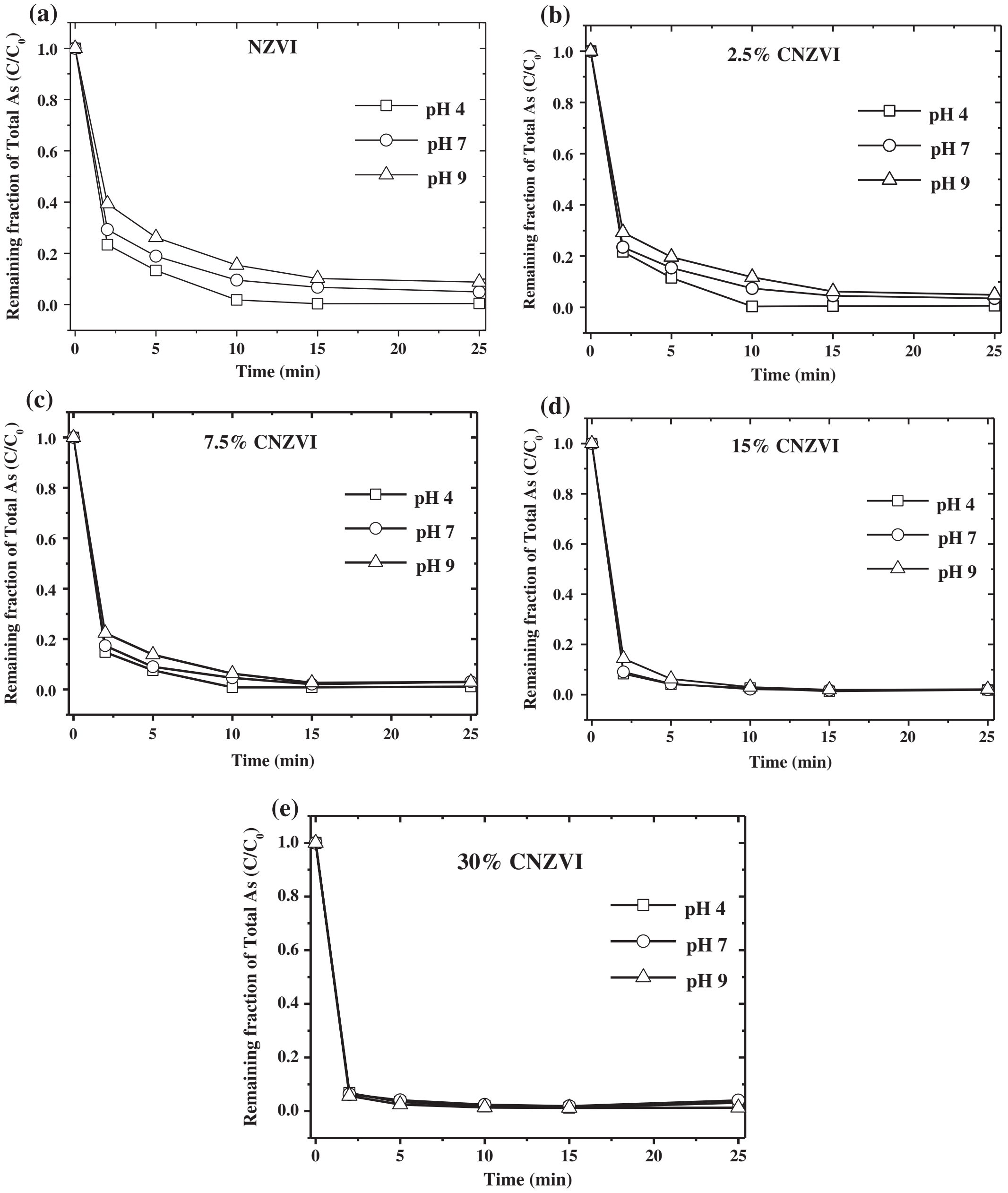

Fig. 7. Effect of initial pH on As(V) removal with the weight percent of chitosan/NZVI of (a) 0, (b) 2.5%, (c) 7.5%, (d) 15%, and (e) 30%. Conditions: Initial As(V) concentration = 3,000 μg/L, NZVI dosage = 0.067 mg/L.

explained by electrostatic attraction between the negative charge of As(V) and the positive charge of CNZVI. The higher amount of chitosan loading can reduce the sensitivity of initial pH effect on the As(V) removal.

3.4. Shelf-life study

In general, the NZVI is a very reactive material. When it is applied to an *in situ* groundwater treatment, it is injected to generate an iron plume. It can be readily oxidized to produce both ferrous (Fe^{2+}) and ferric (Fe^{3+}). These two species can further form a complex with As, resulting in an active sorbent for some other remaining arsenate [17]. In other words, the overall property of NZVI or CNZVI, after its utilization, will be changed and no longer suitable for the reuse purpose, as reported by Nowack [25]. The synthesized NZVI and CNZVI were tested for their shelf-lives. Dried samples

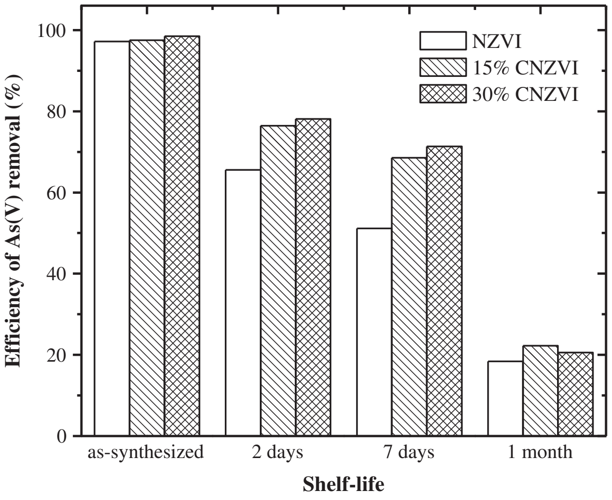

Fig. 8. Effect of shelf-life on As(V) removal efficiency by NZVI, 15% CNZVI, and 30% CNZVI. Conditions: Initial As(V) concentration = 1,000 μg/L, NZVI dosage = 0.067 mg/L, initial pH of 7.

were kept in desiccator at ambient temperature for 2 d to 1 month. As shown in Fig. 8, the arsenate removal efficiencies for all kinds of stored nanoparticles dropped along with the storage period. It was also found that NZVI lost their activity faster than CNZVI. Considering NZVI, the removal efficiency decreases by ca. 20 and 30% were observed after 2 and 7 d storage, respectively. For 15% CNZVI, a removal efficiency decrease by ca. 20 and 30% were observed after 2 and 7 d storage, respectively. As a result, the oxidation over active iron might be retarded by a layer of chitosan [11]. However, the tremendous loss was found for all adsorbents after one month storage.

4. Conclusion

This study describes the effects of weight percent loading of NZVI-modified chitosan and the initial pH of solution on arsenate removal. Batch experiments show that higher amount of chitosan accelerates the removal of As(V) from water. The acidic condition favors the removal of As(V) when the NZVI was applied. However, the effect of initial pH on As(V) removal may be overlooked when higher amount of chitosan was loaded onto NZVI. This outcome is beneficial to *in situ* arsenic removal from groundwater, where higher degree of pH variation is observed. The experimental data of arsenate removal fit quite well with the pseudo-second-order kinetic model. The rate constant (*k*) was found to increase with the increasing percentage of chitosan loading.

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