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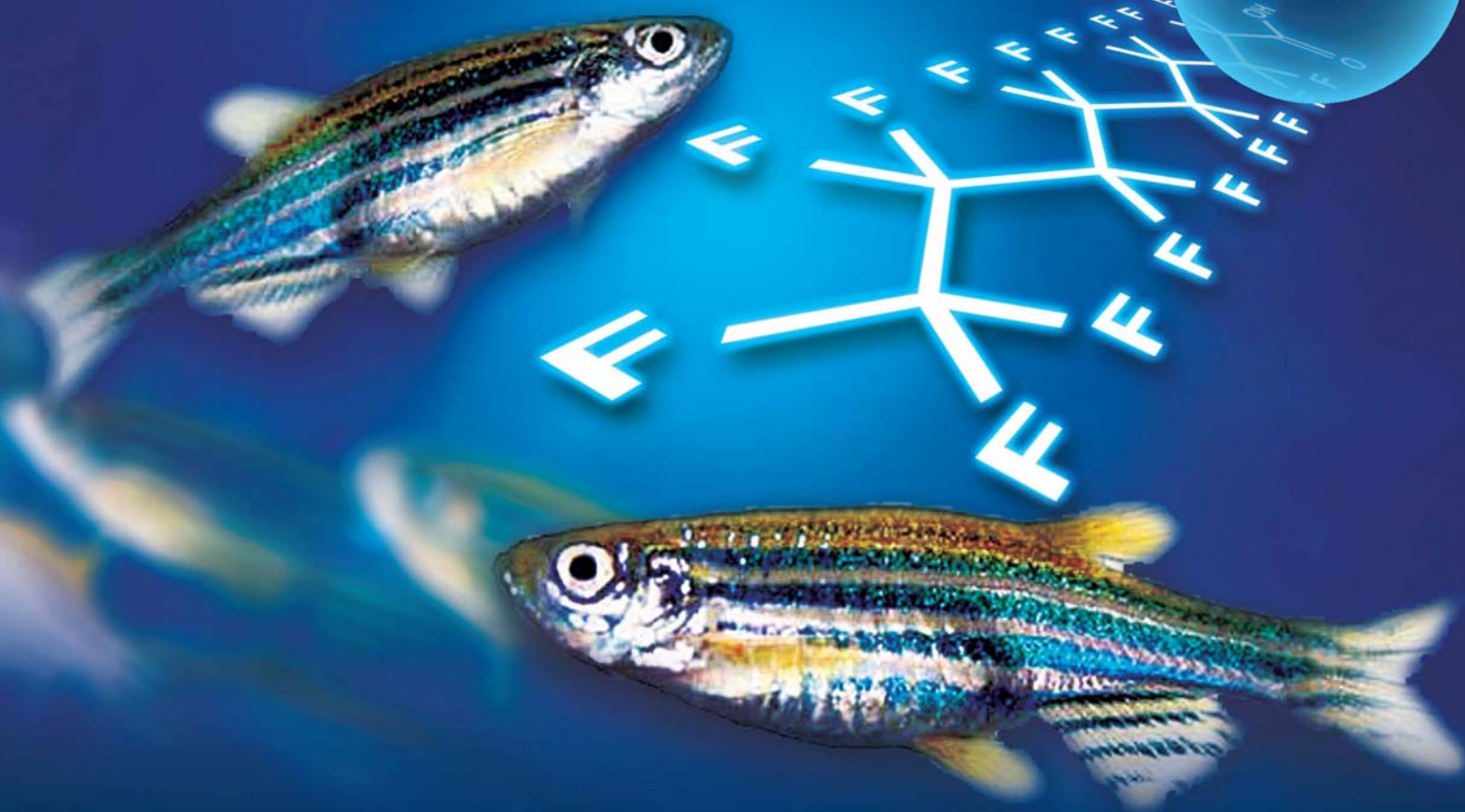
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Interaction between Cu^{2+} and different types of surface-modified nanoscale zero-valent iron during their transport in porous media

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ABSTRACT

This study investigated the interaction between Cu^{2+} and nano zero-valent iron (NZVI) coated with three types of stabilizers (i.e., polyacrylic acid [PAA], Tween-20 and starch) by examining the Cu^{2+} uptake, colloidal stability and mobility of surface-modified NZVI (SM-NZVI) in the presence of Cu^{2+} . The uptake of Cu^{2+} by SM-NZVI and the colloidal stability of the Cu-bearing SM-NZVI were examined in batch tests. The results showed that NZVI coated with different modifiers exhibited different affinities for Cu^{2+} , which resulted in varying colloidal stability of different SM-NZVI in the presence of Cu^{2+} . The presence of Cu^{2+} exerted a slight influence on the aggregation and settling of NZVI modified with PAA or Tween-20. However, the presence of Cu^{2+} caused significant aggregation and sedimentation of starch-modified NZVI, which is due to Cu^{2+} complexation with the starch molecules coated on the surface of the particles. Column experiments were conducted to investigate the co-transport of Cu^{2+} in association with SM-NZVI in water-saturated quartz sand. It was presumed that a physical straining mechanism accounted for the retention of Cu-bearing SM-NZVI in the porous media. Moreover, the enhanced aggregation of SM-NZVI in the presence of Cu^{2+} may be contributing to this straining effect.

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Introduction

With the advancement of industrialization, agricultural and urban activities, the levels of groundwater pollution have increased many folds in the last few decades (Zhang, 2003; Zeng et al., 2013a, 2013b). The application of nanotechnology for the remediation of contaminants may yield promising results in the future (Zhang, 2003; Gong et al., 2009; Xu et al., 2012; Tang et al.,

2014). Nanoscale zerovalent iron (NZVI) is being increasingly studied and tested for the *in-situ* remediation of contaminated soil and groundwater sites that contain chlorinated solvents and heavy metals (Grieger et al., 2010). Although at appropriate doses, NZVI can eliminate or transform certain pollutants rapidly, its transport to target locations and its reactivity may be limited by its rapid aggregation (Grieger et al., 2010). In order to decrease aggregation and enhance the mobility of

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NZVI for in-situ remediation, surface stabilizers can be coated onto the surface of the particles to provide electrostatic repulsion, and steric or electrosteric stabilization (He and Zhao, 2005; Quinn et al., 2005; Saleh et al., 2005, 2007, 2008; He et al., 2007; Kanel et al., 2007; Hydutsky et al., 2007; Phenrat et al., 2007).

While the use and development of surface-modified NZVI (SM-NZVI) are understandably heralded as one of the most promising environmental nanotechnologies, fundamental questions remain on its fate, transport, and environmental and health risks in the environment (Grieger et al., 2010; Dong et al., 2012). One potential issue to consider when evaluating the environmental and health risks of SM-NZVI is the possibility of mobilizing contaminants. It has been reported that naturally occurring colloids in the soil, which range in size from 1 to 1000 nm, can either facilitate or retard the transport of metallic contaminants (Fang et al., 2011). Mobile colloids may therefore act as contaminant carriers, for example facilitating the transport of strongly adsorbed Pb out of a contaminated zone and through a non-contaminated zone in a noncalcareous soil (Grolimund and Borkovec, 2005). Co-transport of colloids and adsorbed contaminants in subsurface environments has attracted considerable attention in recent years, especially for nanoscale colloids such as buckminsterfullerene and TiO_2 (Fang et al., 2011; Zhang et al., 2011). Naturally occurring nanoscale iron oxide particles (which are a product of NZVI oxidation) have also been found with heavy metals, such as copper, bound to their surface many kilometers downstream from mining sites (Hochella et al., 2005). This indicates that the introduced engineered iron nanoparticles, either in ZVI or iron oxide form, may be capable of transporting adsorbed contaminants. It is thus presumed that the transport of SM-NZVI in porous media could potentially involve the migration of the adsorbed contaminants.

Although the SM-NZVI particles themselves may be proven to not pose a threat to human health or the environment, the particles may be capable of binding with contaminants (or byproducts) and transporting them offsite. This may, in the end, increase the problem at hand rather than add to the solution. To date, however, little attention has been paid to the potential environmental risks of application of SM-NZVI in subsurface environments contaminated by heavy metals and the co-transport behavior of SM-NZVI with metal ions (e.g., copper, which is commonly found in metal-contaminated soil (Wang et al., 2011)). The copper released from industrial effluent into soil and groundwater has led to a growing concern about the fate and transport of copper in the environment under acidic conditions (Sharma et al., 2009). It is expected that SM-NZVI, with its large negatively charged surface area, could possibly exhibit a high uptake capacity for copper. Additionally, NZVI coated with different modifiers (carrying different functional groups) may exhibit different affinities for copper.

Therefore, the objectives of this study were: (1) to investigate the uptake of copper ions by the different types of SM-NZVI, and the colloidal stability and mobility of copper-bearing SM-NZVI in saturated porous media and (2) to investigate the retention behavior of copper-bearing SM-NZVI in the porous media by examining the distribution of both copper and SM-NZVI along the length of porous media. NZVIs modified with three different

classes of surface stabilizers (i.e., PAA (polyelectrolyte), Tween-20 (nonionic surfactant) and starch (hydrophilic biopolymer)) were employed in this study for comparison of their affinities for copper ions.

1. Materials and methods

1.1. Chemical reagents

Chemicals such as NaCl, $\text{Cu}(\text{NO}_3)_2$, polyacrylic acid (PAA) (average MW ~2000), Tween-20 (polyoxyethylene sorbitan monolaurate) and potato starch were all reagent grade (purchased from Sigma-Aldrich), and their stock solutions were prepared by dissolving chemicals in deionized water (DI water). DI water was purged with nitrogen gas for 1 hr prior to usage. HCl and NaOH (1.0 mmol) were applied to adjust the initial pH values of the samples. All samples, unless otherwise specified, were prepared at pH 6.

1.2. Preparation and characterization of SM-NZVI

Three types of SM-NZVI (i.e., PAA-, Tween-20- and starch-modified NZVI) were prepared and used in this study. The methodologies for the preparation and characterization of the SM-NZVI are described in detail in Supporting Information (SI). Briefly, two commercial NZVIs in aqueous dispersion form were supplied by the NANOIRON® Company (Czech Republic, EU): pristine NZVI (Nanofer 25, produced from nanosized ferrihydrite) and NZVI surface-coated by PAA (Nanofer 25S). Nanofer 25 (referred to as CNZVI in this study) was used for further modification by using Tween-20 or starch. PAA-modified CNZVI (i.e., Nanofer 25S), Tween-20-modified CNZVI and starch-modified CNZVI are referred to as P-CNZVI, T-CNZVI and S-CNZVI, respectively, in the text.

1.3. Characterization of porous media

Standard Ottawa sand ($\text{SiO}_2 > 99.7\%$), with an average grain size of 300 μm , was obtained from U.S. Silica Company (ASTM-C778). Prior to use, the sand was cleaned thoroughly by a procedure described elsewhere (Zhou et al., 2011) to remove any metal oxide and absorb clay on the sand surface. The ζ -potential of the quartz colloid was measured using an Electro Kinetic Analyzer (SurPASS, Anton Paar GmbH, Graz, Austria) by the method described by Zhou et al. (2011) and was determined to be -29 mV .

1.4. Determination of Cu^{2+} uptake by SM-NZVI

Sorption kinetics experiments were conducted using a 100 mg/L SM-NZVI (P-CNZVI, T-CNZVI or S-CNZVI) suspension and 0.1 mmol Cu^{2+} in background solution (10 mmol NaCl) at pH 6. The final suspensions (40 mL) in 41 mL glass vials sealed with Teflon caps were shaken in an end-over-end rotator at 26 r/min, at room temperature (25°C). At pre-determined time intervals, the suspensions were subjected to magnetic separation, followed by filtration using 0.2- μm pore size cellulose nitrate filters. The filtrates were acidified and analyzed using an Atomic Absorption Spectrophotometer (AAS) (Varian 220FS, Varian, Inc., CA, USA) for total Cu concentration.

1.5. Aggregation and sedimentation of SM-NZVI in the absence/presence of Cu^{2+}

For all aggregation and sedimentation experiments, the concentrations of CNZVI/SM-NZVIs were 100 mg/L. The SM-NZVI solutions in the absence and presence of Cu^{2+} were prepared in background solution (10 mmol NaCl) and sonicated for 5 min just prior to measurement. Aggregation of the nanoparticles was monitored by measuring the time-dependent hydrodynamic diameter via dynamic light scattering (DLS) (Zetaplus, LaborScience S.A., Greece), according to the method described by Chen et al. (2007). Sedimentation was measured by UV scanning ($\lambda = 508$ nm, GE Healthcare, Ultrospec 4300 Pro, USA) in a drive-time mode for 1 hr (Phenrat et al., 2007). The measurements were made at room temperature (25°C) and all the experiments were run in duplicate or triplicate. The sedimentation and aggregation data for CNZVI are provided in the Fig. S1 (SI).

1.6. Transport of SM-NZVI

Column transport experiments were conducted to investigate the SM-NZVI transport in water-saturated sand columns. Vertical columns, 3.6 cm in internal diameter and 15 cm in height, were packed in small incremental steps with dry porous media. The porosity was determined to be 0.45. A schematic of the column experiments for the transport of SM-NZVI is shown in Fig. S2 (SI). The packed columns were first flushed with a background solution (10 mmol NaCl), for the purpose of removing background turbidity and to provide a steady flow. Next, one pore volume (PV) of SM-NZVI suspension was injected (phase 1), followed by injection of the background solution until complete breakthrough of the mobile fraction of particles (phase 2). The background solution and the nanoparticle suspensions were drawn into the column using a peristaltic pump, with upward flow maintained to ensure the column remained fully saturated and sustained steady-state flow. Flow velocity of 10^{-3} cm/sec was employed to simulate the typical groundwater velocity (Lai et al., 2006). The feed slurry of SM-NZVI (100 mg/L) was prepared prior to each experiment, and the slurry source reservoir was sonicated with ultrasonic equipment and sparged using N_2 gas to minimize both aggregation and oxidation during the slurry injection.

The effluent samples were collected at time intervals of 0.1 PV for iron concentration measurement by AAS after acid digestion. The SM-NZVI in suspension (7 mL) was dissolved by acidifying each sample with concentrated HCl (50 μL), followed by sonication for 5 min. At the end of each column experiment, the column was dissected into small increments (3 cm long) and the amount of retained SM-NZVI in each layer was analyzed to determine the depth profile of the retained SM-NZVI in the column. The SM-NZVI was extracted from the section material by adding DI water (50 mL) and concentrated HCl (1 mL), followed by sonication for 30 min. The total SM-NZVI recovery rate was calculated from the ratio of the total amount of SM-NZVI eluted from the column and retained in porous media over the total amount of SM-NZVI injected into the column.

1.7. Transport of Cu-bearing SM-NZVI

Cu-bearing SM-NZVI suspensions were prepared by mixing Cu^{2+} solution and SM-NZVI suspensions in 10 mmol NaCl solution. The final concentrations of SM-NZVI and Cu^{2+} in suspension were 100 mg/L and 0.1 mmol, respectively. The Cu-bearing SM-NZVI suspensions were allowed to react for 30 min to reach equilibrium before use (As shown in Fig. 1). The zeta potentials of the Cu-bearing SM-NZVI suspensions were measured using a zeta potential analyzer (Zetaplus, LaborScience S.A., Greece).

The transport of Cu-bearing SM-NZVI suspension was examined in column tests using the same procedure as described above. In brief, the packed columns were first saturated with background solution. After injection of 1 PV of Cu-bearing SM-NZVI suspension, elution and retention of SM-NZVI and Cu in the column were examined. The dissolved Cu^{2+} in the aqueous phase and the adsorbed Cu on the surface of SM-NZVI in the eluted sample were differentiated by magnetic separation, followed by filtration using 0.2- μm pore size cellulose nitrate filters. The collected solids were dissolved into 7 mL solution for the measurement of Fe and Cu concentrations by AAS. At the end of each column experiment, the column was dissected into 3 cm increments and the amount of retained SM-NZVI and Cu in each layer was analyzed to determine the depth profile of the retained SM-NZVI and Cu in the column.

2. Results and discussion

2.1. Uptake of Cu^{2+} by SM-NZVI

Uptake of Cu^{2+} by CNZVI and each of the three types of SM-NZVIs (i.e., P-CNZVI, T-CNZVI and S-CNZVI) as a function of time is shown in Fig. 1. The uptake of Cu^{2+} by CNZVI was insignificant. The uptake of Cu^{2+} by P-CNZVI was the fastest among the three types of SM-NZVI in the initial stage of

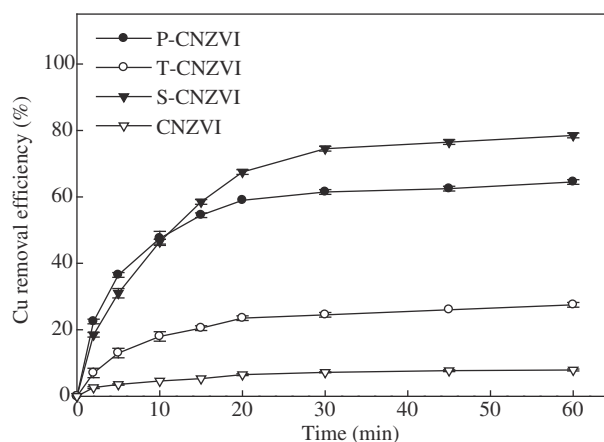


Fig. 1 – Kinetics of Cu^{2+} removal by CNZVI/SM-CNZVI at $\text{Cu}^{2+} = 0.1$ mmol/L and; CNZVI/SM-CNZVI = 100 mg/L. NZVI: nano zero-valent iron; CNZVI: nanofer 25 refers to as CNZVI; P-, T-, and S-CNZVI refer to polyacrylic acid, tween-20, and starch-modified CNZVI.

experiments, while T-CNZVI exhibited the slowest sorption of Cu^{2+} . This should be associated with the surface properties of CNZVI/SM-CNZVI. As demonstrated in Table 1, the surface charges of CNZVI/SM-CNZVI before and after reaction with Cu^{2+} were measured. The CNZVI surface exhibited a positive charge, thus demonstrating a low affinity for Cu^{2+} . The P-CNZVI surface exhibited a more negative charge than S-CNZVI and T-CNZVI, and sorption of Cu^{2+} was thus significantly enhanced by columbic attractions. However, the Cu^{2+} uptake capacity of S-CNZVI was higher than P-CNZVI after approaching equilibrium in 30 min. The equilibrium concentrations of Cu^{2+} in solution after reaction with SM-NZVI are summarized in Table 1. The higher uptake of Cu^{2+} by S-CNZVI than P-CNZVI indicates that there should be other interactions involved between Cu^{2+} and SM-NZVI besides the columbic attractions. The varying Cu^{2+} uptake capacity by the three types of SM-NZVI could also be attributable to the different surface modifiers. The molecule structures of the three surface stabilizers are displayed in Table S1. As seen in the molecule structure of starch, there are 6 hydroxyl groups in each repeat unit of starch, which should be favorable for complexation with Cu^{2+} . In the case of P-CNZVI, it was reported that the PAA was not only adsorbed on the NZVI surface in the structure of bidentate bridging, but also formed a gel network through hydrogen bonding, PAA entanglement and PAA cross-linking (Lin et al., 2010). Consequently, there are few free carboxylic groups on the surface of P-CNZVI for the formation of the Cu^{2+} -carboxylate complexes. Moreover, the interaction between the cross-linked polymer chains and Cu^{2+} could be sterically hindered since the large Cu^{2+} ion requires sufficient space to penetrate the cross-linked polymer chains and react with the carboxylate groups (Kriwet and Kissel, 1996). Tween-20, as a non-ionic surfactant (Kanel et al., 2007), cannot form complexes with Cu^{2+} . Additionally, it should be noted that recent research suggests that Cu^{2+} can also be reduced by the NZVI particles (Ayob et al., 2012). Their results demonstrate that a substantial portion of Cu^{2+} is reduced and transformed to insoluble forms of Cu and Cu_2O . However, no extra attempts were made to identify the degree of Cu^{2+} reduction by the different types of SM-NZVI in this study, which will be one of the central objectives of future research.

2.2. Effect of Cu^{2+} on the colloidal stability of SM-NZVI

The colloidal stability of P-CNZVI, T-CNZVI and S-CNZVI in the absence and presence of Cu^{2+} was examined by sedimen-

tation tests (Fig. 2a–c). The presence of Cu^{2+} exerted no obvious influence on the settling of P-CNZVI and T-CNZVI (Fig. 2a, b). However, in the case of S-CNZVI (Fig. 2c), the particles settled significantly over time in the presence of Cu^{2+} . The sedimentation of SM-NZVI could possibly be initiated by aggregation of the nanoparticles. Thus, the aggregation of SM-NZVI in the absence and presence of Cu^{2+} was examined and the results are shown in Fig. 2d–f. As demonstrated in Fig. 2d, e, the presence of Cu^{2+} did not have an obvious effect on the aggregation of P-CNZVI and T-CNZVI. However, as shown in Fig. 2f, the hydrodynamic radii of S-CNZVI remained almost constant over a period of 1 h in the absence of Cu^{2+} , while it increased significantly from 360 nm to 430 nm after 10 min and gradually increased to 510 nm after 1 hr in the presence of Cu^{2+} . From Fig. 2a–c and Fig. 2d–f, it is clear that the degree of aggregation is positively associated with the degree of settling.

It was reported that multivalent cations can cause the aggregation of nanoparticles, via EDL (Electrical Double Layer) compression, charge neutralization, or complexation (Zhang et al., 2009). Zeta potential measurements were performed on the three types of particles in the presence and absence of Cu^{2+} to gain insights into the correlation between the surface charges of the particles and the sedimentation behavior (Table 1). All three types of particles exhibited a decrease in surface charge in the presence of Cu^{2+} , which suggests that Cu^{2+} was adsorbed onto the particles. However, only the settling of S-CNZVI was significantly influenced by the addition of Cu^{2+} (as demonstrated in Fig. 2c). The surface charge of S-CNZVI became less negative in the presence of Cu^{2+} , which might have resulted in the settling of S-CNZVI. Nevertheless, other studies reported that PAA, Tween-20, and starch stabilize the nanoparticles mainly by steric effects (He and Zhao, 2005; Kanel et al., 2007; Hydutsky et al., 2007). Accordingly, the stability of the three types of nanoparticles should not be significantly influenced by EDL compression or charge neutralization. It can be deduced that the differing aggregation behavior of the three types of SM-NZVI probably resulted from the different interactions of Cu^{2+} with the three stabilizers (i.e., Starch, PAA, Tween-20). As discussed in the previous section, Cu^{2+} can form a network structure with starch via complexation with the hydroxyl groups of the starch molecules, while its complexation with the non-ionic surfactant (Tween-20) and PAA on the surface of nanoparticle is limited. Therefore, it is presumed that S-CNZVI particles undergo attachment with each other via Cu^{2+} complexation with the starch molecules coated on the surface of the particles, thus enhancing the aggregation and subsequent sedimentation of S-CNZVI.

2.3. Transport of SM-NZVI in the absence or presence of Cu^{2+}

As shown in Fig. 3, the transport behavior of three types of SM-NZVI (P-CNZVI, S-CNZVI and T-CNZVI) in the sand-packed columns was examined in the absence and presence of Cu^{2+} . Breakthrough curves of SM-NZVI are plotted as the relative effluent concentration (C/C_0 , where C and C_0 are the effluent and influent concentration of SM-NZVI or Cu-bearing SM-NZVI, respectively) as a function of PVs. Retention profiles of SM-NZVI and Cu are plotted in Fig. 4 as normalized concentration (quantity of the SM-NZVI/Cu recovered in the

Table 1 – Properties of Cu-bearing NZVI suspensions.

	Zeta-potential (mV)		Total Cu ^a (mg/L)	Dissolved Cu ^b (mg/L)
	NZVI	Cu-bearing NZVI		
CNZVI	8.7	9.4	6.4	5.9
P-CNZVI	−38.7	−22.3	6.4	2.4
T-CNZVI	−3.3	−1.6	6.4	4.8
S-CNZVI	−11.4	−3.5	6.4	1.6

^a Total Cu concentration of the Cu-bearing NZVI suspension.

^b Dissolved Cu concentration in the bulk solution. CNZVI, P-, T-, and S-CNZVI refer to Fig. 1 or Section 1.2.

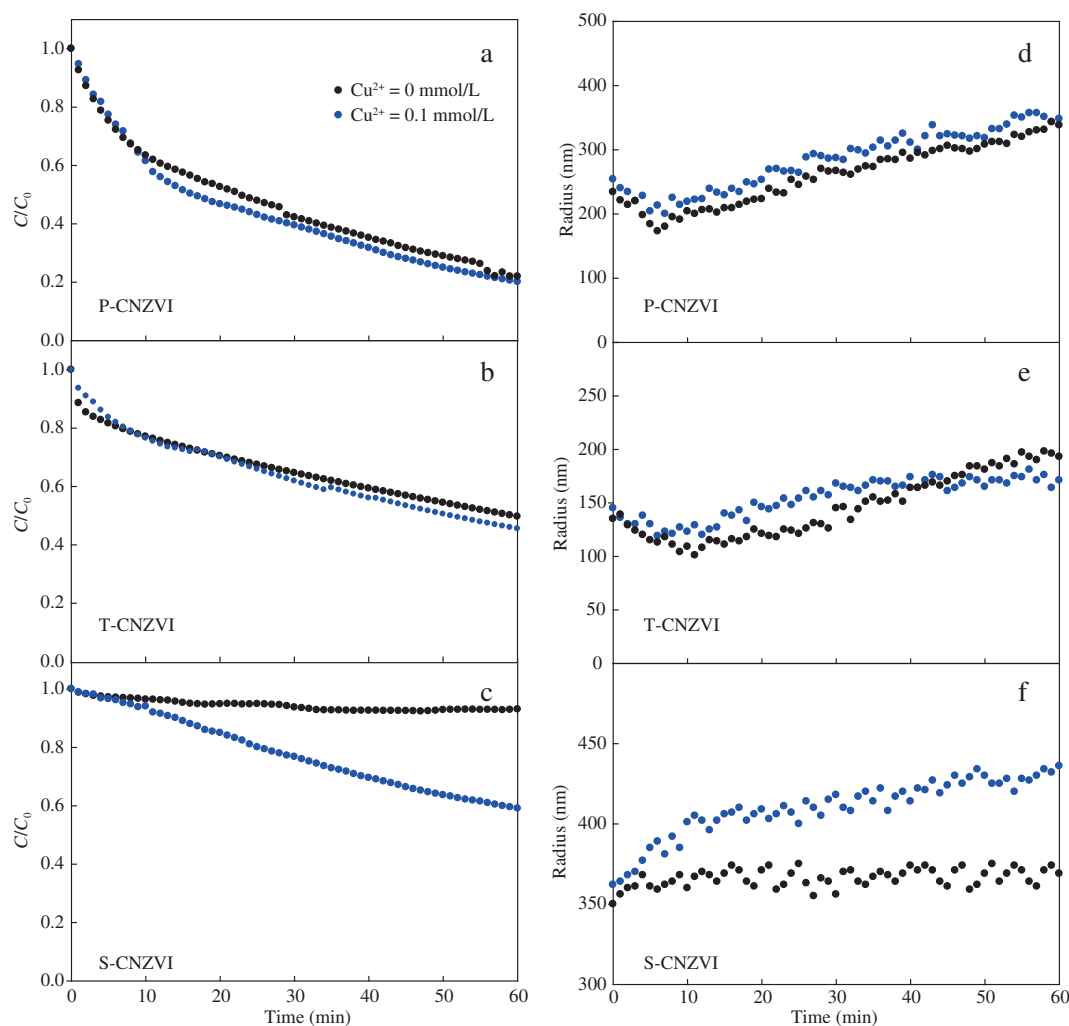


Fig. 2 – Sedimentation and aggregation of (a, d) P-CNZVI, (b, e) T-CNZVI and (c, f) S-CNZVI in the absence and presence of Cu^{2+} as a function of time. The results presented are the average of the duplicate or triplicate runs. CNZVI, P-, T-, and S-CNZVI refer to Fig. 1 or Section 1.2.

column section divided by the total quantity of the input SM-NZVI or Cu-bearing SM-NZVI suspension) as a function of distance from the column inlet. The corresponding mass recovery of SM-NZVI (in the absence or presence of Cu) and Cu in the effluent and sand is shown in Table 2. The results showed that 95%–98% and 93%–96% of SM-NZVI and Cu were recovered from the sand columns, respectively, verifying the accuracy of the method for the iron and copper measurement.

As shown in Table 2, the fraction of T-CNZVI eluted from the column was more than that of P-CNZVI and S-CNZVI. Given that the P-CNZVI and S-CNZVI particles are negatively charged (as demonstrated in Table 1), the negatively charged sand grains resulted in an electrostatically unfavorable condition (electrostatic repulsion) for nanoparticle deposition. However, significant retention of P-CNZVI and S-CNZVI in the sand column occurred despite the unfavorable electrostatic conditions for attachment onto the surface of sand grains. As shown in Fig. 4, the retained T-CNZVI particles in porous media were almost evenly distributed along the length of column (Fig. 4b), while the retention profiles of P-CNZVI or S-CNZVI typically exhibited a hyperexponential shape, with greater retention in

the section adjacent to the column inlet (0–3 cm) and rapidly decreasing retention with depth (Fig. 4a, c). The shape of the retention profiles was not consistent with classical filtration theory, which predicts an exponential shape with depth (Yao et al., 1971). A number of potential explanations for the hyperexponential retention profiles have been proposed in the literature including: secondary minimum and surface charge heterogeneity on the sand grains (Redman et al., 2004; Tufenkji and Elimelech, 2005; Johnson et al., 2010), straining of colloids (Bradford et al., 2002, 2003) and colloid aggregation (Chen et al., 2006; Chen and Elimelech, 2007). Additional experiments were therefore conducted to better understand and quantify the mechanisms of SM-NZVI retention. Under chemically unfavorable attachment conditions (electrostatic repulsion), SM-NZVI may interact with the solid phase by virtue of the presence of the reversible secondary minimum at high ionic strength. In order to further test this hypothesis, we ran three phase transport experiments (shown in Fig. 3). Phase 3 consisted of flushing the column with three pore volumes of ultrapure water (Ionic strength = 0) to eliminate the secondary minimum (Franchi and O'Melia, 2003). The release of

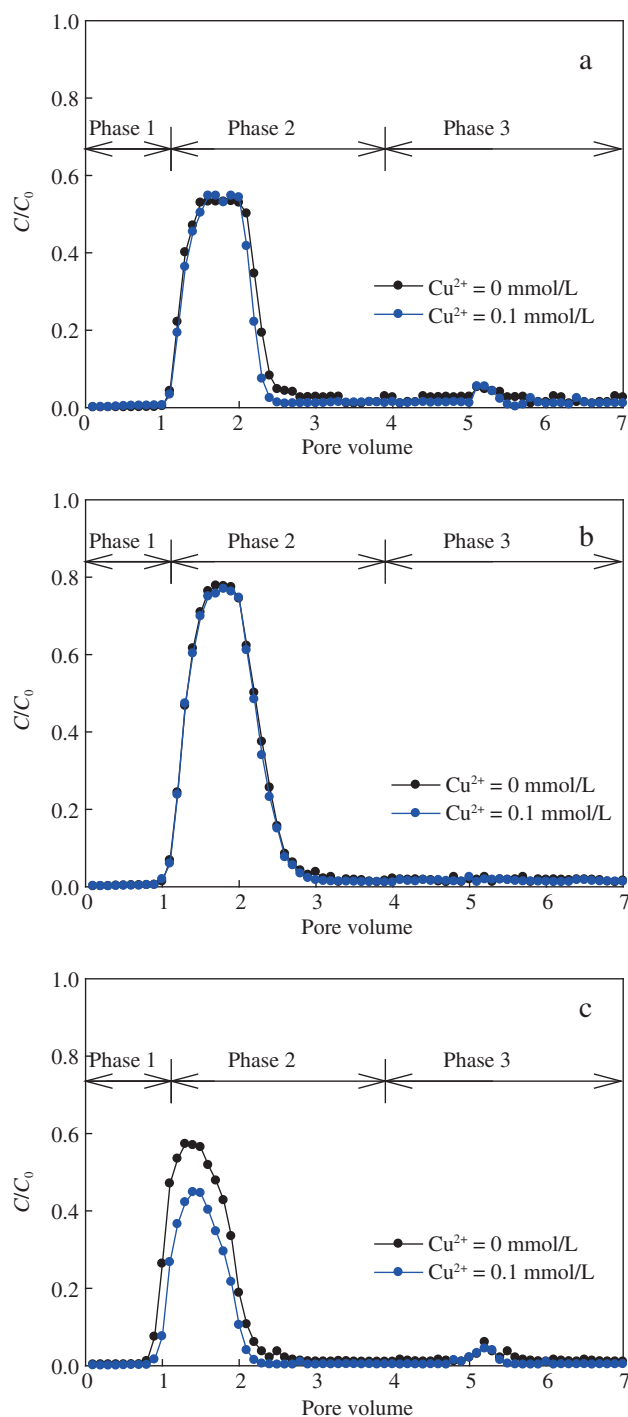


Fig. 3 – Breakthrough curves of P-CNZVI (a); S-CNZVI (b) and T-CNZVI (c) in the absence and presence of Cu^{2+} . Phase 1: Surface-modified NZVI (SM-NZVI)/Cu-bearing SM-NZVI suspension, phase 2: eluted with background electrolyte solution, and phase 3: further eluted with ultrapure water CNZVI, P-, T-, and S-CNZVI refer to Fig. 1 or Section 1.2.

previously deposited colloids through lowering the bulk solution ionic strength has been used as supporting evidence for particle deposition in the secondary minimum (Redman et al., 2004; Tufenkji and Elimelech, 2005; Franchi and O'Melia, 2003). As shown in Fig. 3, rinsing the column with

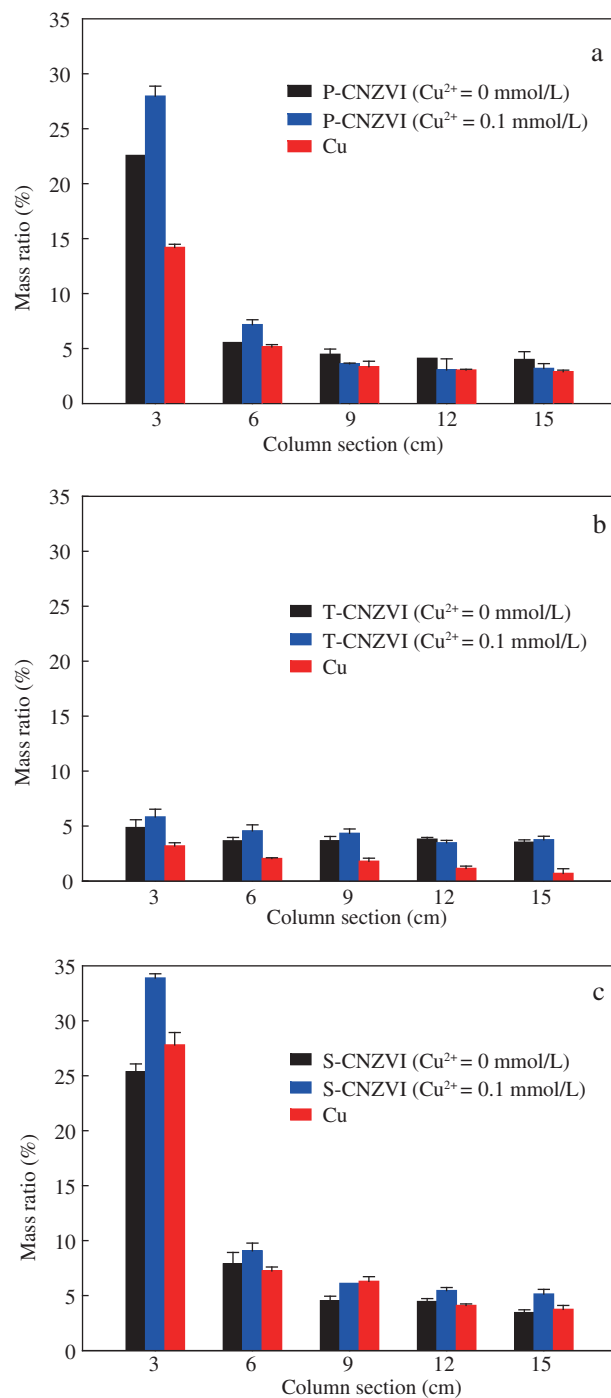


Fig. 4 – Distribution of surface-modified NZVI (SM-NZVI)/Cu-bearing SM-NZVI, and Cu along the length of column at the end of co-transport experiments (phase 1 and phase 2). Column sectioning is from the inlet to the outlet. In X-axis, “3” represents the column section of “0–3 cm” and “6” represents the column section of “3–6 cm”, etc. CNZVI, P-, T-, and S-CNZVI refer to Fig. 1 or Section 1.2.

ultrapure water during phase 3 only resulted in a minor peak ($C/C_0 < 0.05$) and release of retained SM-NZVI in the effluent. This observation suggests that the secondary minimum insignificantly accounted for the SM-NZVI retention for both SM-NZVI and Cu-bearing SM-NZVI systems.

Table 2–Mass balance percentages for surface-modified NZVI (SM-NZVI) and Cu in the saturated packed column experiments (phase 1 and phase 2). CNZVI, P-, T-, and S-CNZVI refer to Fig. 1 or Section 1.2.

SM-NZVI	SM-NZVI (%)			Cu (%)		
	Effluent ^a	Retained ^b	Total ^c			
P-CNZVI	61	35	96	NA		
T-CNZVI	81	14	95	NA		
S-CNZVI	55	43	98	NA		

Cu-bearing SM-NZVI	SM-NZVI (%)			Cu (%)		
	Effluent ^a	Retained ^b	Total ^c			
				Effluent	Retained ^f	Total ^g
				Adsorbed ^d	Dissolved ^e	
P-CNZVI	54	43	97	31	34	28
T-CNZVI	79	19	98	19	70	7
S-CNZVI	37	58	95	24	22	48

NA: not applicable.

^a Refers to the effluent percentage of SM-NZVI recovered from column experiments.

^b Refers to the retained percentage of SM-NZVI recovered from column experiments.

^c Refers to the total percentage of SM-NZVI recovered from column experiments.

^d Refers to the percentage of Cu adsorbed on SM-NZVI in the effluent.

^e Refers to the percentage of dissolved Cu recovered in the effluent.

^f Refers to the retained percentage of Cu recovered from column experiments.

^g Refers to the total percentage of Cu recovered from column experiments.

This retention of nanoparticles is presumably due to a physical mechanism such as straining. Straining refers to both wedging (retention of particles at two bounding surfaces) and bridging (when multiple particles collide and are retained in a pore constriction) (Bradford and Torkzaban, 2008; Zhang et al., 2012). Straining occurs when a nanoparticle is physically removed from flow by the size of the channel, which is prevalent in cases with larger nanoparticles or smaller porous media size. Bradford et al. (2002, 2003) observed systematic trends of lower effluent concentrations and increasing colloid retention in sand near the column inlet with decreasing sand grain size or increasing colloid size. Data from Li et al. (2004) indicates that straining may occur when the particle size to the median grain size ratio (d_p/d_{50}) > 0.002. In this study, SM-NZVI particles tended to aggregate with time (as demonstrated in Fig. 2), and the diameter ratio of SM-NZVI aggregates to sand was more than 0.002, especially for P-CNZVI and S-CNZVI. Physical straining of SM-NZVI aggregates at grain–grain contacts was therefore considered to be important.

The fraction of SM-NZVI eluted from the column in the presence of Cu^{2+} was less than that in the absence of Cu^{2+} (Table 2). It is expected that the increased ionic strength compresses double layer thickness and reduces double layer repulsion between nanoparticles and grain surfaces, thus inducing the deposition of more nanoparticles (Kretzschmar et al., 1999). However, the decrease in the fraction of S-CNZVI eluted in the presence of Cu^{2+} was much more significant compared with P-CNZVI and T-CNZVI. This may be associated with the effect of Cu^{2+} on the deposition and aggregation of SM-NZVI. The differing effects of Cu^{2+} on the colloidal stability of S-CNZVI, P-CNZVI and T-CNZVI were observed in the previous section, which are related to the different interactions of Cu^{2+} with the three stabilizers (i.e., Starch, PAA, Tween-20). Significant aggregation and sedimentation of S-CNZVI particles

was induced by the presence of Cu^{2+} . As shown in Fig. 4c, the retention profile of SM-NZVI in the presence of Cu^{2+} exhibited an even more pronounced hyperexponential shape compared with that in the absence of Cu^{2+} . For example, approximately 25% of the total injected S-CNZVI was retained near the column inlet (0–3 cm) in the absence of Cu^{2+} , whereas the value was as high as 34% in the presence of Cu^{2+} . The retention profile of Cu^{2+} was consistent with that of SM-NZVI. Accordingly, the Cu-induced aggregation of SM-NZVI may be contributing to the straining effect as mentioned above. Moreover, the high deposition of S-CNZVI particles could also possibly be attributed to bridging complexation in which the adsorbed Cu^{2+} serves as a bridge between the surface of sand grains and the hydroxyl groups of the starch molecules on the S-CNZVI particles.

3. Conclusions and environmental implications

This research provides a context for understanding the interaction between Cu^{2+} and SM-NZVI during their transport in saturated porous media. The use of NZVI with varying surface stabilizers and their interaction with Cu^{2+} provide insight into the potential environmental risks of application of different types of SM-NZVI in subsurface environments contaminated by heavy metals and the co-transport behavior of SM-NZVI and the metal ions. The results from this research show that NZVI coated with different modifiers (carrying different functional groups) exhibited different affinities for Cu^{2+} . It is expected that particles binding with heavy metals may pose a threat to human health or the environment. However, the uptake of Cu^{2+} decreased the transport of SM-NZVI in porous media, resulting from the enhanced nanoparticle aggregation. It appears that the complexation between surface stabilizers and Cu^{2+} has profound

implications for SM-NZVI stability and mobility. For instance, S-CNZVI particles undergo attachment with each other via Cu^{2+} complexation with the starch molecules coated on the surface of the particles, thus enhancing the aggregation and subsequent retention of S-CNZVI in porous media. However, due to the weak complexation stability between the PAA or Tween-20 coatings and Cu^{2+} , only a slight influence on the stability and mobility of P-CNZVI and T-CNZVI was observed in the presence of Cu^{2+} . This suggests that, even though the SM-NZVI has a strong affinity for Cu^{2+} , the Cu-bearing SM-NZVI will finally settle out in porous media, which would not pose any threat to human health or the environment. On the contrary, NZVI modified with surface stabilizers (e.g., starch) that can form complexes with cations provides an option for the removal of cationic pollutants in the subsurface environment.

The results from this study are based on a single water system (NaCl solution). Such studies are a necessary first step toward understanding the behavior of SM-NZVI in natural systems. However, these studies should be extended to a wider and more representative range of conditions, such as divalent cations and other constituents in groundwater. For instance, divalent Ca^{2+} may compete with Cu^{2+} for ion-exchange sites of SM-NZVI, leading to enhanced dissociation of Cu^{2+} from SM-NZVI, and thus altering the co-transport behavior of Cu^{2+} with SM-NZVI.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.jes.2014.09.043>.

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