



## Lead removal by a reusable gel cation exchange resin containing nano-scale zero valent iron



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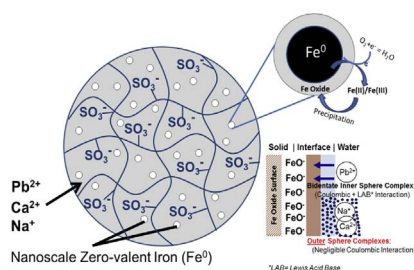
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### GRAPHICAL ABSTRACT



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### ABSTRACT

The practical applications of nano-scale zero valent iron (nZVI) particles in flow-through treatment systems are limited due to unfavorable mechanical and hydraulic properties caused by their tiny size. On the other hand, nZVI, when dispersed within suitable host material, can overcome these limitations to have synergistic improvement in its adsorption capacity. This study aims to synthesize, characterize, validate the performance of hybrid cation gel exchanger dispersed with nZVI particles, named as C100-Fe<sup>0</sup>, for selective trace Pb(II) removal from contaminated water. C100-Fe<sup>0</sup> had Fe content of approximately 22% w/w as determined by double acid digestion method. The characterization studies revealed that nZVI particles of size range 20 nm were well dispersed throughout the gel phase of the polymeric resin beads; XANES spectral analysis confirmed the presence of zero oxidation state of Fe nanoparticles within both the newly synthesized and regenerated C100-Fe<sup>0</sup>. Equilibrium batch studies demonstrated that Pb(II) adsorption capacity was unaffected by the presence of high concentration of competing ions such as Na<sup>+</sup> and Ca<sup>2+</sup> ions whereas, presence of SiO<sub>2</sub> decreased the capacity. The batch adsorption isotherm fitted well with Freundlich model. During column study with C100-Fe<sup>0</sup>, breakthrough of USEPA permissible limit of 15 µg/L was observed after the passage of 4200 bed volumes of challenge water (NSF/ANSI Std.53) having Pb(II) concentration of 150 µg/L. More than 87% of adsorbed Pb(II) could be recovered within 15 bed volumes during regeneration with acid. Wide availability and low price of iron salts as well as of cation exchange resins, combined with the possible reusability make C100-Fe<sup>0</sup> an attractive option for use in the field for removal of Pb(II) from contaminated water.

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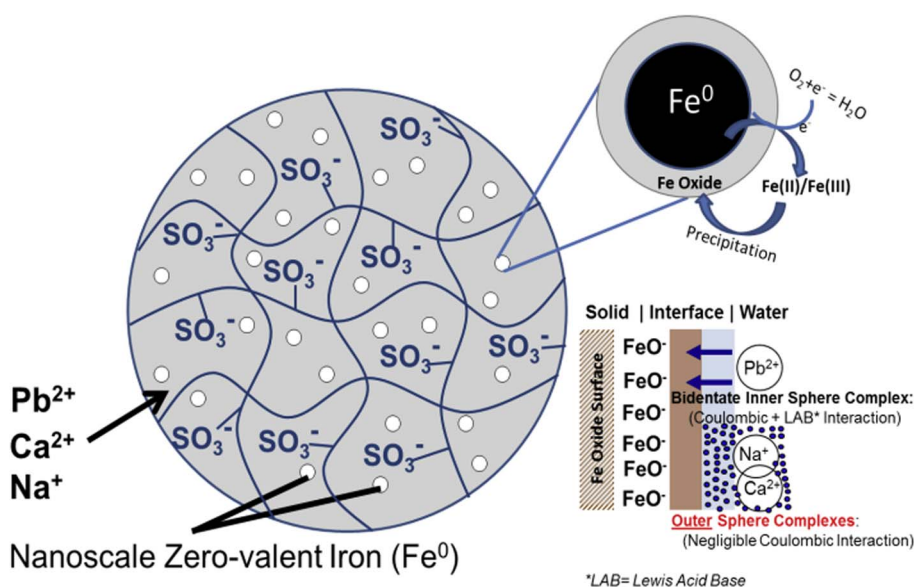


Fig. 1. Diagram of a reusable gel cation exchanger containing nano-scale zero valent iron (C100-Fe<sup>0</sup>).

## 1. Introduction

The problem of heavy metal contamination in the environment is a major concern due to their toxicity and tendency to accumulate and bio-magnify within the food chain, causing wider damage to the ecosystem. Most of these heavy metals can be extremely toxic even at trace concentrations. Lead (Pb<sup>2+</sup>) is one such toxic heavy metal that is released to the environment in large quantities every year. The sources of lead released into the environment can be both natural weathering processes and anthropogenic activities namely, electroplating, battery manufacturing, mining. In human being, lead is known to be accumulated mainly in kidneys, muscles, bones, nervous, and blood circulatory system causing several manifestations such as abdominal pain, fatigue, depression/mood changes, diminished cognitive performance and hand dexterity [1–4]. The USEPA (EPA, 2013) has set a permissible limit of 15 µg/L in drinking water while Thailand Pollution Control Department (PCD) has set the standard of lead in the drinking and industrial effluents as 50 and 200 µg/L, respectively.

In order to comply with the regulatory limits, various technologies relying on conventional methods such as chemical precipitation, ion-exchange, and reverse osmosis have been adopted [5–7]. However, each of these methods suffers from one or more of the following drawbacks: high cost, generation of voluminous sludge, and poor removal efficiency due to high solubility product values of precipitating salts. In contrast, removal using adsorption shows higher efficiency and better economics as a result of potential for selective removal, regeneration and reuse of the adsorbent, and recovery of metals from wastewater.

Nanoscale zero-valent iron (nZVI) particles have shown good potential for removal of various metals such as Co<sup>2+</sup>, Cu<sup>2+</sup>, Pb<sup>2+</sup>, Ni<sup>2+</sup> from contaminated water and wastewater [8]. However, bare nZVI particles also suffer from disadvantages such as formation of aggregates, reduction in specific surface area, production of a less negative redox potential, high pressure drop in fixed-bed column operations, low-to no- reusability, lack of durability and mechanical strength. In order to solve this problem, several attempts have been made to immobilize the n-ZVI into porous host materials. For the last 5 years, nZVI supported various materials have been widely investigated. They can be classified as (1) natural minerals such as pillared clay [9], pumice granular [10,11], acid activated sepiolites [12,13], montmorillonite [14–16], kaolin [17], bentonite [18,19], zeolite [20,21], biochar [22,23], and char coal [24]; (2) biomaterials namely, aquatic plant *Azolla filiculoides* [25], walnut shell [26], macroporous alginates

[27,28]; and (3) synthetic materials such as anion exchange resin [29], chelating resin [30], mesoporous silica carbon [26], activated carbon [31,32], cationic resin [33], graphene oxide [34,35], magnesium (hydr) oxide [36,37], layered double hydroxide [38,39], titanate nanotube [40], carbon nanotube [41], and porous carbon sheet [42]. However, most of the studies focused onto the basic equilibrium batch study only. They lacked the use of column experiments as well as in depth study about the phase change and adsorption mechanism to elucidate interaction between Pb<sup>2+</sup> and nZVI.

From the previous studies, it was understood that toxic heavy metal cations can be concentrated inside a cation exchanger having negatively charged (SO<sub>3</sub><sup>-</sup>) fixed functional groups due to the Donnan membrane effect [43]. Therefore, the dispersion of nZVI inside a cation exchanger host should have improved potential to remove the target heavy metals. Note that, the cation exchangers alone can be used for the removal of heavy metal ions such as Pb<sup>2+</sup>, Cu<sup>2+</sup>, Ni<sup>2+</sup>, etc. However, most of contaminated water has, apart from these heavy metal ions, other competing cations such as Ca<sup>2+</sup>, Na<sup>+</sup>, etc. at a much higher concentrations compared to these heavy metal ions. Therefore, unless there is presence of any specific interaction other than electrostatic interactions for these trace heavy metal ions, it is not possible to effectively remove the target heavy metal cations from the background of the other commonly occurring cations. Chelating resins with specific functional groups such as iminodiacetic acid exhibit high selectivity toward heavy metals compared to these competing cations. However, they have limited applications, primarily due to the high cost of these chelating resins.

This research was taken up to develop a reusable gel cation exchange resin containing nano-scale zerovalent iron (C100-Fe<sup>0</sup>) for trace lead removal from the natural water sources. To the best of our knowledge, this is the first instance to show that impregnating nZVI into a gel type cation exchange resin as a host material improves the target Pb<sup>2+</sup> removal capacity from contaminated water. The mechanism behind the superior Pb<sup>2+</sup> removal by a hybrid cation exchanger impregnated with nanoscale zero valent iron (C100-Fe<sup>0</sup>) is illustrated in the Fig. 1. In the presence of dissolved oxygen or other oxidants, nZVI gets oxidized to Fe<sup>2+</sup>/Fe<sup>3+</sup> and subsequently, there is a formation of Fe<sup>2+</sup>/Fe<sup>3+</sup> oxide layer as shell on the top of the core, which is still Fe<sup>0</sup> or zero valent iron. Because of high density of negatively charge fixed functional groups (SO<sub>3</sub><sup>-</sup>), Pb<sup>2+</sup> ions are attracted inside the hybrid material due to the Donnan membrane effect and then get adsorbed onto the surface of iron oxide on the dispersed nZVI, through the formation of inner sphere complex. The objectives of this study were to:

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