



## Removal of Pb, Cd, Cu and Ni from aqueous solution using nano scale zero valent iron particles



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### ABSTRACT

Nanoparticles offer the potential to improve environmental treatment technologies due to their unique properties. Adsorption of metal ions Pb(II), Cd(II), Cu(II), Ni(II) to nano zero valent iron (nZVI) particles of surface iron oxide was examined. nZVI particles were synthesized by reduction of ferric chloride with sodium borohydride. In this work, transmission electronic microscopy (TEM), scanning electronic microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD) and Fourier transform infrared spectroscopy are applied to study the compositional and structural of nZVI. Batch equilibrium experiments were performed to study the removal efficiency of heavy metal ions. The effect of different parameters such as contact time, initial concentration, pH and nZVI dose on the adsorption process were investigated. The adsorption kinetics well fitted using a pseudo-second order kinetic model. SEM and TEM images showed that of particles were mostly spherical shape in chains with an average size  $43.1 \pm 11.9$  nm. The initial sorption rate for all the metals is in order  $Pb^{2+} > Cu^{2+} > Cd^{2+} > Ni^{2+}$ . Short adsorption equilibrium contact time favors the application of the adsorption process. The formation of FeOOH on the surface of nZVI could be the main factor of cations removal due to its high adsorption affinity for aqueous solutes. Therefore, with the strong adsorption at  $pH < 6.5$  which is representative of several natural groundwater and the ability to adsorb multiple metal ions simultaneously, nZVI may offer a potential remediation method for the removal of metals from water and environment.

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## 1. Introduction

Heavy metals are naturally occurring elements that have a high atomic weight and a density at least 5 times greater than that of water. Their multiple industrial, domestic, agricultural, medical and technological applications have led to their wide distribution in the environment; raising concerns over their potential effects on human health and the environment. The natural and anthropogenic activities are the main sources of heavy metals in aquatic environment [1,2]. Heavy metal ions are non-biodegradable and can accumulate in living organisms [3]. Metal ions such as lead and cadmium classified as human carcinogens according to the U.S. Environmental Protection Agency, and the International Agency for Research on Cancer [4].

Therefore, reliable methods are necessary for the removal of heavy metal ions from aquatic environment. Several methods used to remove metal ions from aqueous solution such as precipitation, ion exchange [5], reverse osmosis, and membrane process [6,7]. In recent years, adsorption has become a popular process for metal ion removal from water due to its physical and chemical properties and low-cost [8]. Many studies have focused on pollutant removal using adsorption techniques with cheap adsorbents such as fly ash, waste carbon slurry, red mud, and many others [9,10].

Iron nanoparticles technology is consider among the first generation of nanoscale environmental technologies proposed for heavy metals and organic substances removal [11]. Nano scale zero valent iron (nZVI) studied as an effective remediate for removing various organic and inorganic pollutants from contaminated water sources because of its large active surface area, in-situ application, low solid waste product (easy to separated and disposed) [12], and stable removal performance regardless of the change of volume of water treated [13–15]. Nano scale zero valent iron is capable to exchange the toxic substances to non-toxic materials by reduction and precipitation methods [16].

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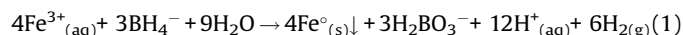
The efficiency of nZVI to remove metal ions is strongly affected by solution properties including pH, initial metal concentration and nZVI dosage. pH influences metal sorption on nZVI by both controlling formation of an outer iron oxide layer as well as changing the surface charge [17].

The objectives of this research are to evaluate the synthesis of nano scale zero valent iron (nZVI) particles, as well as to investigate the effect of contact time, initial metals concentration, pH and adsorbent dosage on the removal of lead, copper, cadmium and nickel metals from aqueous solution.

## 2. Materials and methods

### 2.1. Synthesis of nano scale zero valent iron particles

Nano scale zero valent iron (nZVI) particles prepared by reduction method using two main chemicals which were  $\text{FeCl}_3$  and  $\text{NaBH}_4$  followed the method proposed by Sun et al. [11]. The  $\text{NaBH}_4$  functions as a reducing agent for the ferric chloride  $\text{FeCl}_3$  in form of solution to produce nZVI by mixing equal volumes of 0.94 M sodium borohydride ( $\text{NaBH}_4$ ) and 0.18 M  $\text{FeCl}_3$ . nZVI particles were synthesized in the laboratory via the following reaction:



The borohydried solution was slowly added into the iron chloride with vigorous stirring ( $\sim 400$  rpm). Immediately after the first drop of reducing agent into iron solution, black particle appeared. Then further mixing for 15–20 min produce the maximum yield of black iron particles. Particles were then separated from the solution by vacuum filtration using Whatmann cellulose nitrate membrane filter ( $0.2 \mu\text{m}$ ). The solid particles were washed three times with absolute ethanol to remove all water. This washing process is probably the key step of synthesis since it prevents the rapid oxidation of nZVI. For storage, a thin layer of ethanol added to preserve the nano iron particles from oxidation.

### 2.2. Characterization of nZVI particles

The chemical composition of the synthesized nZVI was identified by an energy dispersive X-ray spectroscopy system (EDX) (Module Oxford 6587 INCA x-sigh) coupled to the SEM at 17 kV after gold coating using SPI-Module sputter coater. Also, X-ray fluorescence (XRF) analysis of nZVI done with JSX-3222 analyzer to analyzed main elemental compositions of the minerals that are present in nZVI particles before and after Pb adsorption. XRD patterns obtained on a Bruker D8 (Bruker-AXS, Karlsruhe, Germany) diffractometer using filtered  $\text{Cu K}\alpha$  radiation with an accelerating voltage of 40 kV and current of 30 mA.

The surface morphology of nZVI characterized by a scanning electron microscopy (SEM) (JEOL JSM-5600 LV). Transmission Electron Microscopy (TEM) images of the particles size distribution of nZVI particles recorded with a EM (208S Philips, Netherlands) at 80 kV, and then photos were taken by digital camera (Canon, Japan). Samples prepared by depositing a few droplets of dilute nZVI solution on carbon-coated copper grids.

Fourier Transformed IR spectra (FT-IR) of nZVI particles recorded in the transmission mode at room temperature using KBr pellet technique. Shimadzu (Japan) infrared spectrophotometer used to determine the spectra of the sample mixed with spectrally pure KBr and pressed to form thin plates (radius 1 cm, thickness 0.1 cm), then subjected to IR spectroscopic analysis (FT-IRNICOLET iS10 Thermo Scientific) in the spectral range 500 and  $4000 \text{ cm}^{-1}$ .

### 2.3. Batch experiments

Batch experiments were carried out to evaluate the efficiency of nZVI particles for adsorb toxic metals ( $\text{Pb}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Ni}^{2+}$ ). Batch adsorption experiments were conducted using a known amount of the prepared adsorbent and metal ions solution at the working pH at various time intervals. Samples were collected after shaking in a rotary shaker at 200 rpm then filtered using  $0.2 \mu\text{m}$  and the concentrations of the studied metal ions were determined. The effects of contact time (5–120 min) with initial metals

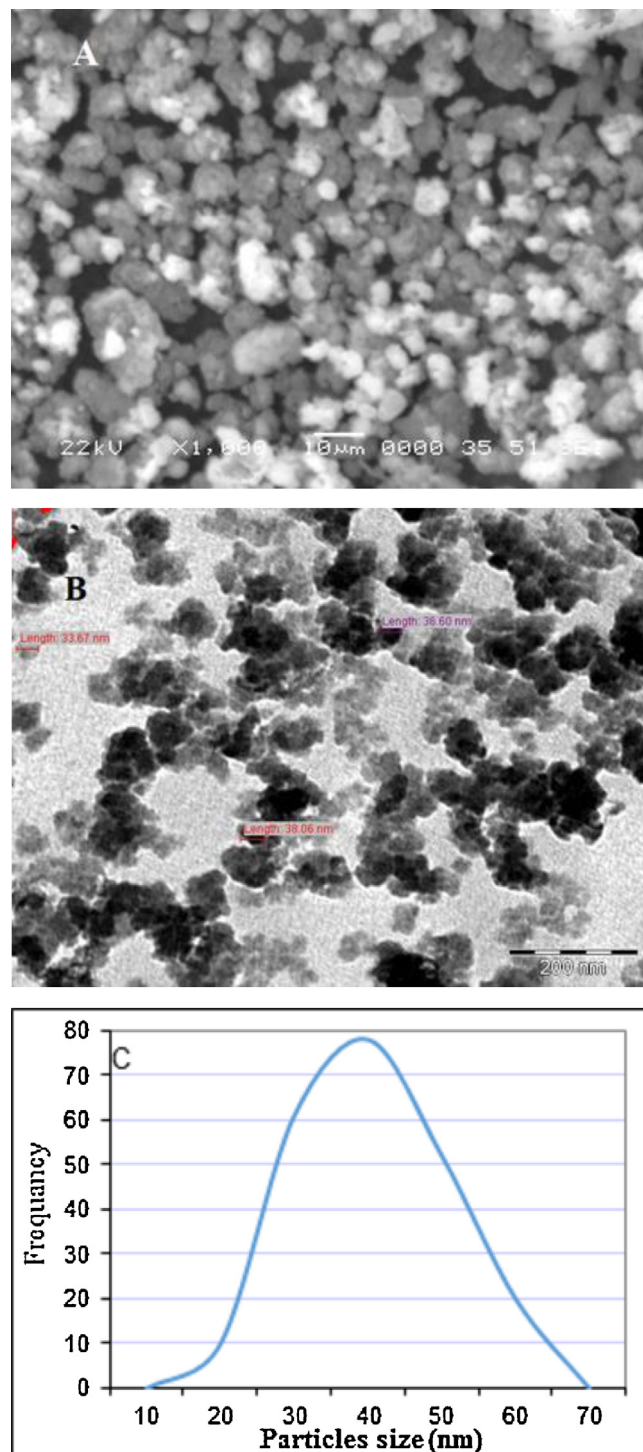


Fig. 1. a) Images of nZVI particles SEM b) TEM of shell iron oxide with core nZVI c) Particles size distribution.

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