

## Short Communication

# Removal of Lead from Aquatic Solution Using Synthesized Iron Nanoparticles

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(Received: 26 June 2014 and Accepted: 22 August 2016)

### **Abstract**

*Due to its ability in chemical oxidation of contaminants, iron nanoparticle is a material of choice to remove lead ions from aquatic solutions. In this study a reduction method in solution phase was applied to synthesize the nanoparticles. Afterwards, the size of the synthesized particles were confirmed by Scanning Electron Microscopy. It is worth noting that the nanoparticle dose-variations were examined in the range of 0.02-0.5mg while pH and exposure time were respectively investigated in the ranges of 3-11 and 1-40 min. Meanwhile, the removal efficiency of various concentrations of lead ions were evaluated in the range of 1-50 mg/l. The results indicated that the best removal efficiency (92.5%) occurred in the concentration range of 1 to 40 mg/l for a dose of 0.1 mg nanoparticles. By increasing concentration of lead ions to 50 mg/l, the optimum dose was achieved in 0.2 mg. Improved removal was observed with increasing exposure time up to 10 minutes while no improvement was recorded for exposure times of 20 minutes or longer. The results confirmed the effectiveness of synthesized iron nanoparticles in removing lead ions from aquatic solution.*

**Keywords:** Iron Nanoparticle, Lead, Chemical Oxidation, Heavy Metal, Aquatic Environment.

## **1. INTRODUCTION**

Lead and lead compounds are generally toxic pollutants which has bioaccumulation property in tissues of human body [1, 2]. Human body consists of about 120 mg of lead. Intestines absorb approximately 10-20% of lead. Colics, skin pigmentation and paralysis are some of the symptoms of over overexposure to lead. There are generally two types of lead poisoning effects: neurological or teratogenic. Neuron necrosis is a result of organic lead [3] and axonal degeneration and demyelination are due to inorganic lead. In addition, edema and congestion result from organic and inorganic lead. Absorption of organic lead compounds takes place faster and is thus riskier. Organic lead derivatives may be carcinogens. Men are generally

less sensitive to lead poisoning than women [4]. Menstrual disorder, infertility and spontaneous abortion are caused by lead. Lead increases stillbirth risk. Mothers are less sensitive to lead poisoning than fetuses and are in general protected from lead poisoning by fetuses. A larger amount of lead per unit body weight (up to 40%) may be absorbed by children compared with adults. Therefore, children are generally more sensitive to lead poisoning than adults. Lower IQs, behavioral changes and concentration disorder are the symptoms of lead poisoning. Leg tissue is where lead accumulates. Encephalopathy is a result of the most severe type of lead poisoning.

The reaction of lead ions with free

sulfhydryl groups of proteins, such as enzymes induces lead toxicity. Enzymes are deactivated as a result. In addition, lead can interact with other metal ions. Organic lead compounds and lead (II) salts are the most harmful from an eco-toxicological point of view. Lead salts such as lead acetate, oxide, nitrate, and carbonate are associated with water hazard class 2 and are thus harmful. Chlorophyll synthesis in plants is decreased by lead [5]. However, up to 500 ppm of lead can be taken up by plants from the soil. Plant growth is adversely affected by high lead quantities. Lead enters food chains via plant uptake. As a result, the application of lead pesticide is banned in most countries. Lead is collected in organisms, sediments and sludge. Lead present in wastewater mostly originates from streets and roofs. The presence of lead in water may be due to the application of lead and PVC pipes in addition to spill of sewages from such industries as battery making, metal plating, electrical equipment, chemicals, steel, iron and copper [6]. The factors for estimation of lead concentration in drinking water due to pipelines used include consumer behavior and patterns of water use, water characteristics (pH, hardness, and orthophosphate content), piping material, pipe dimensions and piping equipment. Methods to remove lead from water include adsorption process: and the use of adsorbent materials such as activated carbon [7, 8] bio-sorbents such as alga [9] chemical precipitation [10] ion exchange [11] and biofilters [12]. One of the popular methods to eliminate heavy metals is the use of reductants [13, 14]. Chemical oxidation is a fast, highly effective process which uses reductions to remove lead from water [15, 16]. Among available reductions, iron has priority due to its abundance, low cost, non-toxicity, rapid reaction potential and high efficiency in decomposition of contaminants. Particle size and surface-to-volume ratio are of the great importance in reduction and elimination of pollutants by the particles

[17]. Extensive laboratory studies showed that iron nanoparticles can be used for removal of groundwater contaminants such as chlorinated organic compounds [18] nitrates [19] and heavy metals (e.g. copper, cobalt [20, 21], nickel [27, 28] and chromium (VI) etc. [24]. The steps of lead removal by iron nanoparticles are; 1) Mass transfer of the reagents from the solution mass to iron surface, 2) Surface absorption and chemical reaction of the reagents on the surface, 3) Desorption of reduction products from nanoparticle surface, 4) Mass transfer of the products to the solution mass. The characteristics of iron nanoparticles, which make them appropriate for surface absorption, are high surface area to volume ratio and their easy removal from the media using an externally applied magnetic field. Iron nanoparticles have been used in surface absorption of different organic and inorganic pollutants from wastewaters. The most effective parameters in surface absorption are nanoparticle size and media temperature and pH. Iron nanoparticles can be used as effective absorbents due to their good surface absorption and ease of removal [26].

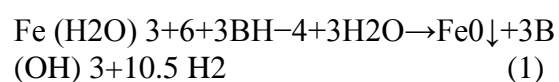
## 2. EXPERIMENTAL

### 2.1. Chemicals

In this research, chemicals used to synthesize iron nanoparticles included ferric chloride hexahydrate ( $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$ ), sodium borohydride ( $\text{NaBH}_4$ ), sodium hydroxide ( $\text{NaOH}$ ), hydrochloric acid ( $\text{HCl}$ ) and ethanol. All the solutions were prepared with de-ionized water and the materials used were of high quality.

### 2.2 Synthesis of Iron Nanoparticles

The applied nanoparticles were prepared using reduction method in solution phase. The nanoparticle extracted is shown by the symbol of  $\text{Fe}^{\text{BH}}$  [25]. The synthesize follows from Eq.1.



For this purpose, two main solutions were initially prepared: Fe III ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) solution 0.1 M (27.019 g in 1000 cc of deionized water) and sodium borohydride ( $\text{NaBH}_4$ ) solution 0.16 M (6.08 g in 1000 cc of 0.1 M sodium hydroxide); the latter is reductant solution. Then reductant solution was added drop-wise (5ml per minute) to 0.1M Fe (III) solution which was simultaneously mixed with the speed of 500 rpm. At the end of the synthesis, the produced iron nanoparticles deposited in the solution. The synthesized nanoparticles were then washed three to four times with 0.0001M HCl and transferred into a dark, glass container with a layer (less than 2cm) of ethanol over them to prevent oxidation. The container was kept at a temperature below 4 °C.

### 2.3. Characterization

In order to measure synthesized nanoparticles, X-ray diffraction analyses were performed on the particles with Co  $K\alpha$  radiation of wavelength 1.78 Å at a power of 40 kW and beam current of 40mA in the step scanning mode. The  $2\theta$  scanning range was from 5 to 90° in steps of 0.0167°. The applied device was of X'Pert brand. SEM (LEO 440i) was used to characterize the structure of the synthesized nanoparticles. Iron nanoparticles agglomerate due to their magnetic properties. Atomic Absorption Spectrophotometer (AAS) (Varian Spectra 200) was used to determine the amount of lead in solution and the effect of nanoparticles on lead elimination.

### 2.4 The Applied Experimental Method

In this study, the iron nanoparticles were freshly prepared and kept in liquid phase. After synthesis by the method described in section 2.2, they were first allowed to deposit in the container as much as possible so that ethanol content was not more than 2 cm above the nanoparticles. The remaining liquid was removed by pipette. The lid was then closed and the container shaken well to mix uniformly. Thereafter, 10 cc of this solution was poured by pipette in a clean petri dish,

dried at desiccator and weighed before using. Subsequently, it was placed in the oven at 75 ° C to dry completely. After drying, the petri dish was again placed in desiccator to cool down without absorption of moisture, and then, re-weighed. Thus the exact weight of the nanoparticles was determined in 10 cc of the solution. The nanoparticles were added to the samples in liquid form. This had three major advantages: Due to the use of nanoparticles in solution phase, 1) they can be uniformly dispersed throughout the sample only by using a shaker. If solid nanoparticles were used, due to their strong electromagnetic properties, it would have been necessary to use devices such as ultrasonic mixer to add the particles to the sample and distribute them in the whole sample. 2) The presence of very small amounts of ethanol prevents oxidation of nanoparticles before adding them into the sample, and 3) drying of nanoparticles, an expensive and time consuming step, was not required. To analyze the effectiveness of the synthesized iron nanoparticles, five parameters including contact time, optimal dose of nanoparticles, lead solution concentration, pH and the interfering ions were examined.

### 2.5 Determination of Optimum Conditions

In order to determine optimum contact time, this was varied from 1 to 40 min at two concentrations (1 and 5 mg/L) of lead solution containing 0.1 mg of nanoparticles. Then the optimum dose of nanoparticles was studied. By applying the optimum contact time from the first step, optimum amount of nanoparticles was derived among seven different doses (0.02, 0.04, 0.06, 0.08, 0.1, 0.2, and 0.5 mg) for three different concentrations of lead solution (1, 5, and 50 mg/L). In the next step, the impact of optimum dose of nanoparticles within the optimum contact time was analyzed on five different concentrations (1, 5, 10, 20, and 50 mg/L) of lead solution. Besides, another test was conducted to determine the best pH for elimination of lead ions from the solution using nanoparticles. For this purpose, five

pHs (3, 5, 7, 9, and 11) were considered to assess the removal efficiency at pre-defined optimal conditions. The last parameter investigated was the effect of interfering ions in the solution. Accordingly, three ions; sulfate (250 and 500 mg/L), nitrate (10 and 50 mg/L) and copper (5 and 60 mg/L) were selected to assess the removal efficiency of lead from solutions with lead ion concentrations of 5 mg/l and 50 mg/l.

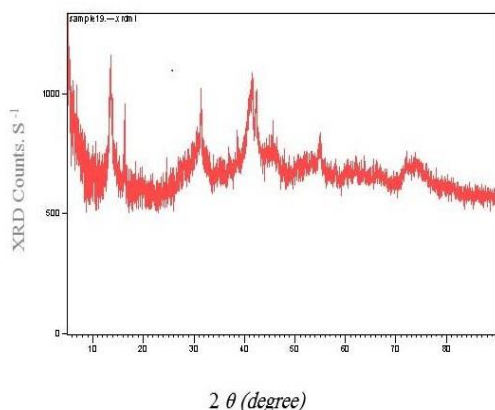
### 3. RESULTS AND DISCUSSIONS

The XRD analysis result indicated that the highest peak corresponds to Fe<sup>0</sup> (figure 1). The size of nanoscale grains can be determined through the results obtained from XRD analysis (Fig.1) and Scherrer formula [Eq. (2)]. In this research, the size of the nanoparticles determined by Scherrer formula was 21 nm.

Eq.2.

$$D = 0.9 \lambda / \beta \cdot \cos \theta$$

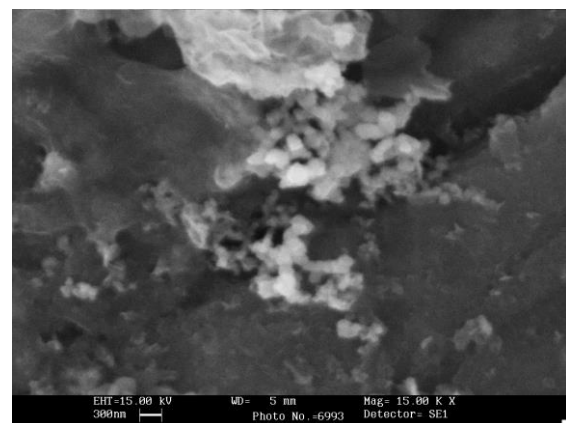
where, D is the size of the crystallite grain,  $\lambda$  is X-ray wavelength in Å and;  $\beta$  is the same as full width at half maximum (FWHM) in the list of analyses. In other words, it is maximum peak width at half its height.



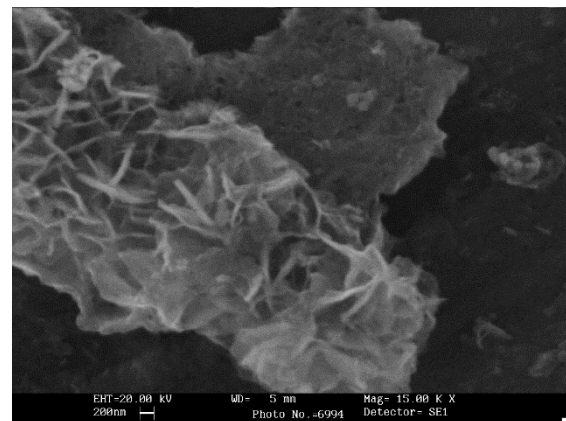
**Figure 1.** Synthesized nanoparticles XRD patterns

As can Fig. 2, be seen in Fig. 2, the size of nanoparticles were determined equal to 50 nm. Based on SEM images, nanoparticles are predominantly composed of iron. In this study, based on SEM results, the size

of the synthesized nanoparticles were determined about 50 nm. The optimum dose of the nanoparticles for solutions containing less than 50 mg/L lead was 0.1 g, and for those containing 50 mg/L lead was 0.2 g. Besides, the proper contact time was achieved at 10 minutes. Considering that in all concentrations of lead solution, more than 99% removal efficiency was achieved, the optimum pH was equal to 11 . It is noteworthy that the presence of interfering ions has no particular effect on lead removal.



**Figure 2.** The image of the nanoparticles in the scales 300 nm

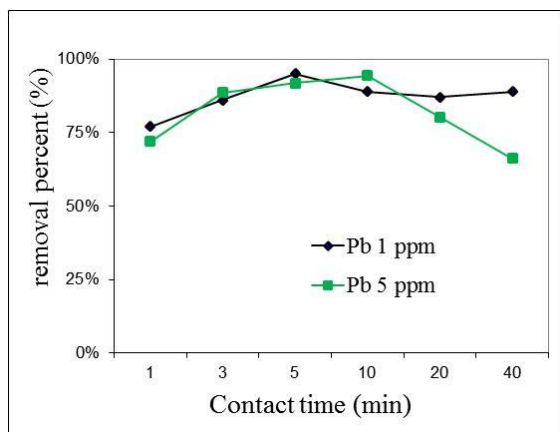


**Figure 3.** The image of the nanoparticles in the scales 200 nm

#### 3.1 Equilibrium Time

As shown in figure 4, maximum elimination of lead ions occurred at a contact time of 10 minutes for both 1 and 5m g/L of lead concentrations. By increasing the contact time up to 10

minutes, lead concentration showed a drop; but for contact times of 20 and 40 minutes in solution with 5 mg/L of lead concentration, sudden increase in concentration was observed. Therefore, in this research, contact time of 10 minutes was considered as optimum contact time.



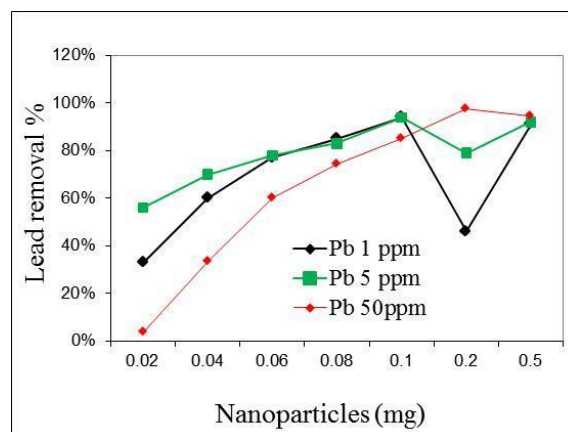
**Figure 4.** Equilibrium time determination

### 3.2 Optimum Dose of Nanoparticles

Figure 5 shows the effect of amount of nanoparticles on lead removal. For 1 and 5 mg/L of lead concentration, the best elimination (91 and 94%, respectively) occurred with 0.1 mg of nanoparticles. For 50 mg/L of lead concentration in the solution, removal efficiency was equal to 85% with 0.1 mg of nanoparticles while the highest removal rate of 98% was achieved with 0.2 mg of nanoparticles. However, with the increase of the nanoparticle to 0.5 mg, the removal rate dropped again to a value of 95%. Therefore, optimum dose of nanoparticles was achieved at 0.1 mg for lead concentrations less than 50 mg/L.

### 3.3 Changes in Lead Ions Concentration

Removal percentages for five concentrations of lead solution containing 0.1 mg nanoparticles are summarized in table 1. It is obvious that maximum removal percentage has occurred at 10 mg/L of lead concentration. However, at



**Figure 5.** Optimum dose of nanoparticles for three different lead concentrations

other concentrations except 1 mg/L, removal percentage was above 80% which is acceptable (Table 1).

**Table 1.** The results of the highest removal percentage at different concentrations of lead ions solution.

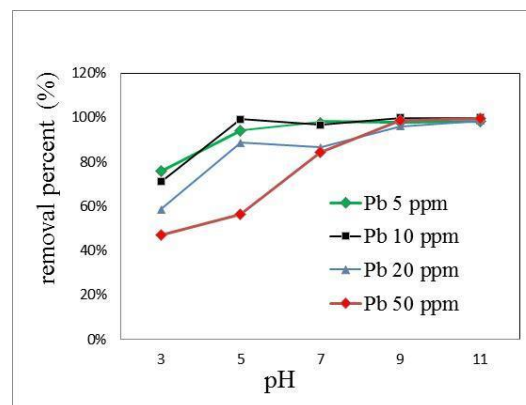
No	Dilution (mg/L)	Removal percent
1	1	50%
2	5	84%
3	10	96%
4	20	90.50%
5	50	85.50%

To prepare samples for lead measurement using atomic absorption, the samples were treated as follows: The samples were centrifuged for 5 minutes at a rate of 2500 rpm to completely remove the nanoparticles. The main reason for complete removal of nanoparticles is that the lead on the nanoparticles may release and get into the sample during the digestion process if nanoparticles are not removed. Since the pollutant to be removed by the nanoparticles is a heavy metal, the samples must be digested after contact with nanoparticles because the metals are capable of being absorbed on other particles. In other words, digestion is performed in order to reduce the interference of organic compounds and the

changes in metal structures for the presence of particles, which interfere with the measurement and analysis of the metals in the atomic absorption equipment. Nitric acid gives a good yield in the digestion and is sufficient for digestion of most samples. However, hydrochloric acid is also recommended for samples containing organic materials, which are difficult to oxidize or silicate containing inorganic compounds [27]. Following centrifugation and complete removal of nanoparticles, 20 mL of 65% nitric acid is added to the solution and the solution is heated to reduce the volume to about 10 mL. 5 mL of 65% nitric acid is added to the solution again and the heating is continued to reduce the volume to about 10 mL. If the solution is still cloudy, 5 mL of 65% nitric acid is added again. This is repeated until a clear solution is obtained. The solution is then cooled and the volume is increased to 50 mL using distilled water and atomic absorption is carried out.

### 3.4 Optimization of pH

The solubility of the solute can be affected by the pH of aqueous solution. pH and ionic strength affect the steady state kinetic parameters for reduction of electron transfer. The best pH for all lead concentrations was 11. This is due to Iso-Electric Points (IEP) of iron nanoparticles which occurs between pH 8.1-8.2. When the average pH is higher than the IEP(s) [13, 20] then; nanoparticle surface will attract a negative charge and thus better absorbs  $Pb^+$  ions (Figure 6). In a research conducted at University of South Australia by Xi and Mallavarapu (2009) [13], the best removal rate occurred at acidic pH range that is inconsistent with these research findings. In a research done in 2008 in Turkey by Üzümlü and Shahwan [14] in alkaline pH, close to 100% removal rate was obtained. This is similar to the findings of the current study.



**Figure 6.** Finding optimum pH for solutions with four different lead ions concentrations

### 3.5 Effect of Interfering Ions

The results showed that the presence of interfering ions has no significant effect on lead ions removal from aqueous solution. Furthermore, the amounts of these ions also drop. When there are only lead ions present in the solution, lead removal by 0.1 mg of nanoparticles at a contact time of 10 minutes for 5 and 50 mg/L solutions was 99.2 and 99.8%, respectively. Tables 2 and 3 shows percentage of lead elimination in the presence of three interfering ions (sulfate ions; 250 and 500 mg/L, nitrate ions; 10 and 50 mg/L, and copper ions; 5 and 50 mg/L) for two lead concentrations.

**Table 2.** Percentage of lead ions elimination in presence interfering ions (Lead 5ppm)

lead elimination %	interfering ions elimination %	interfering ions	Lead
98.60%	29.20%	250	5 ppm
99%	31.20%	500	
99%	30%	10	
98.60%	66%	50	
98.20%	98.18%	5	
98.20%	99%	50	
99.20%	-	only lead ions	

**Table 3.** Percentage of lead ions elimination in presence interfering ions (Lead 50 ppm)

lead elimination %	interfering ions elimination %	interfering ions	Lead
99.80%	22.80%	Sulfate 250 ppm	50 ppm
100%	34.00%	Sulfate 500 ppm	
100%	10%	Nitrate 10 ppm	
99.70%	68%	Nitrate 50 ppm	
99.80%	98.80%	Copper 5 ppm	
99.80%	99%	Copper 50 ppm	
99.80%	-	only lead ions	

#### 4. CONCLUSION

In this study, based on SEM results, the size of the synthesized nanoparticles was determined about 50 nm. The optimum dose of the nanoparticles for solutions containing less than 50 mg/L lead was 0.1 g, and for those containing 50 mg/L lead was 0.2 g. Besides, the proper contact time was achieved at 10 minutes. Considering that in all concentrations of lead solution, more than 99% removal efficiency was achieved, the optimum pH was is believed to be 11. It is noteworthy that the presence of interfering ions did not seem to have any particular effect on lead removal.

The removal of lead from aqueous solutions by iron nanoparticles takes place via several mechanisms, which are indirectly related to the core structure of nanoparticles. The mechanisms generally involved in lead removal are electrostatic absorption, complex formation, oxidation,

reduction and precipitation. The contribution of each mechanism depends on the standard electrical potential of each metal ion and the experimental conditions; especially average pH. Given that the reduction potential of  $Pb^{2+}$  (-0.13 v) is greater than that of  $Fe^{2+}$  (-0.44 v), the most effective parameter in lead removal by nanoparticles is oxidation reduction reaction, in which Pb is converted to water insoluble PbO. Another reason for determination of oxidation reduction as the major mechanism for lead removal is the interference of other ions, whereby the same quantity of nanoparticle removed over 90% of copper ions along with lead. If absorption was the major mechanism in removal, the second metal would not be removed since each absorbent has a limited capacity beyond which it will not be able to absorb. One of the benefits of the procedure used in this work is the application of nanoparticles in slurry form which has two advantages. Nanoparticles can be used in ten contaminated site, for example ground waters and the expenses associated with drying and preparation of nanoparticles in powder form will be eliminated.

#### ACKNOWLEDGEMENT

The authors would like to express their gratitude to that management of Iran Mineral Processing Research Center (IMPRC) - for their support.

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