

# Preparation, Characterization and Application of Nanoscaled Zero-Valent Iron (NZVI)

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## Abstract

In this research, nanoscaled zero-valent iron (NZVI) was prepared with ethanol and sodium borohydride by reduction method in the atmospheric condition and then NZVI was supported by clay during synthesis. Three samples of NZVI (A, B and C) were prepared by depending on ethanol content. Characterization of NZVI and clay-supported NZVI were performed by using XRD, EDXRF, SEM and TG-DTA spectroscopic methods. The synthesized materials were applied as adsorbents for removal of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions from aqueous solution. The resultant data indicate that clay-supported zero-valent iron nanoparticles are promising reactive materials for environmental applications. Various parameters such as contact time, concentration of metal ion solution and sorbent dosage were carried out to obtain the optimum conditions. According to the results, the maximum removal efficiency of  $\text{Co}^{2+}$  93.04% by NZVI-B, 98.57% by clay-supported NZVI-B and that of  $\text{Cu}^{2+}$  87.54% by NZVI-B, 97.19% by clay-supported NZVI-B were obtained with 250 ppm metal ion solutions for 50 minutes contact time and 0.2 g of adsorbent dosage.

**Keywords :** Nanoscaled zero-valent iron, clay-supported NZVI, reduction method, adsorbents, removal efficiency

## Introduction

### Some Aspects of Nanoparticles

Iron nanoparticle technology represents perhaps one of the first generation nanoscale environmental technologies. Various synthetic methods have been developed to produce iron nanoparticles. Nanoparticle is a very small object and consists of different size nanocrystals. As the nanoparticle works the process by which the removal heavy metals and treatment of wastewater, they can be used as effective and efficient adsorbent. They are also used in biomedical, electronics, consumer products and environmental applications.

In nanotechnology, a particle is defined as a small object that behaves as a whole unit in terms of its transport and properties. It is further classified according to size, in terms of diameter, fine particles cover a range between 100 and 2500 nanometers, while ultrafine particles are sized between 1 and 100 nanometers. Nanoparticles may or may not exhibit size-related properties that differ significantly from those observed in fine particles or bulk materials. Nanoclusters have at least one dimension between 1 and 10 nanometers and a narrow size distribution. (Biswas and Wu)

The structure of a nanoparticle of a material is generally determined by the chemical composition of the material, the number of atoms in the particle, and the character of the chemical interactions between atoms. Nanoparticles can have a regular crystalline structure, can be amorphous, or can form a pseudo close packing indescribable by any of the crystallographic space groups. For each of these structural states of a nanoparticle, there is a certain set of numbers of the atoms involved in the particle that corresponds to optimum stable configurations. These numbers are usually called the magic numbers.

The crystal structures of matter are traditionally described in terms of structural crystallography, which is based on the concept of an infinite perfect lattice with a translational symmetry (Filipovich and Kalinin). A particular structure (an amorphous structure, a regular crystalline structure, or a nanocrystallographic pseudoclose packing) of nanoparticles of a material under given external conditions is determined by the chemical composition of the material, the character of the chemical interaction between atoms, and the size factor.

Nanoparticles are expected to help improve products used in biomedical, electronics, consumer products and environmental applications.

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### **Some Aspects of Clay**

Clay is a substance which we know as a deposit which is extensively distributed over the surface of the earth. It is found almost everywhere but differs greatly in its purity. This deposit had been produced by the decomposition of various rocks. As these decomposed products have been acted upon by water, the various original materials have been separated, leaving deposits of various kinds. Some of these we know, as sand, clay and various mixtures. Clay in its pure condition is comparatively rarely found, the larger portion of the deposit being composed of impure clay. Pure clay is a silicate of aluminum and when found in large quantities it is called kaolin.

The typical clay such as kaolin has the formula  $2\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $2\text{H}_2\text{O}$ . Its percentage of composition would be accordingly  $\text{SiO}_2$ , 47.1;  $\text{Al}_2\text{O}_3$ ; 39.2;  $\text{H}_2\text{O}$ , 13.7. The impure clays contain feldspar such as orthoclase,  $\text{K}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$ ,  $6\text{SiO}_2$  and there will be also present mica having the formula  $\text{K}_2\text{O}$ ,  $2\text{Al}_2\text{O}_3$ ,  $4\text{SiO}_2$  and quartz.

### **Some Aspects of Clay Supported Nanoscaled Zero-Valent Iron**

Clay minerals are basically hydrous aluminum silicates having small particle size ( $< 2\mu\text{m}$ ). Mg and Fe may exist instead of aluminum and the clay may contain alkali or rare earth elements as constituent. A pure clay mineral consists of two basic blocks; a sheet of silicon tetrahedrons and aluminum octahedrons. The type as the clay minerals is determined by the stacking of these two sheets into layers, the properties of the bonding between layers and existence of different ions substituted for aluminum and silicon. Clay minerals have plasticity when wet, they are thermally chemically stable in geochemical media in a wide range of pH in addition to their low costs and wide availability. Clays of concern in this study, kaolinite and montmorillonite, are potential heavy metal adsorbents (Liu, et.al). These properties make clay minerals suitable filling and/or support materials for zero-valent iron nanoparticles for *in situ* remediation methods.

### **Characterization of Zero-Valent Iron Nanoparticles**

Zero-valent Iron Nanoparticles can be characterized by X-ray Diffraction (XRD), Energy Dispersive X-ray Fluorescence (EDXRF), Scanning Electron Microscopy (SEM) and Thermogravimetric Differential Thermal Analysis (TG-DTA).

### **Aim and Objectives**

The main aim of this research is to increase the utilization of clay-supported nanoparticles of zero-valent iron as an adsorbent for removal of heavy metals such as  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  from aqueous solution.

The objectives are

- ❖ To collect the clay samples
- ❖ To prepare sample solutions for preparation of nanoscale zero-valent iron
- ❖ To synthesize nanoscaled zero-valent iron (NZVI)
- ❖ To synthesize clay-supported nanoscaled zero-valent iron
- ❖ To characterize the synthesized nanoscaled zero-valent iron and clay-supported nanoscaled zero-valent iron

- ❖ To study the removal efficiencies of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by synthesized nanoparticles

### **Materials and Methods**

The chemicals used in this research were from British Drug House Chemical Ltd. Poole, England. All chemicals were of reagent grade and used as received.

#### **Sampling**

In the experimental work, clay samples were collected from Bokalay village, Gwa Township Rakhine State. Three samples of Clay A, Clay B and Clay C were examined for the experiments.

#### **Preparation of iron (III) chloride solution**

Accurately 0.292 g of iron (III) chloride was weighed in a mettler balance and was put into a beaker. The ferric chloride was dissolved in 100 mL deionized water to make 0.018 M iron (III) chloride solution.

#### **Preparation of sodium borohydride ( $\text{NaBH}_4$ ) solution**

Accurately 2.8 g of sodium borohydride ( $\text{NaBH}_4$ ) was dissolved in 50 mL of deionized water.

#### **Preparation of nanoscaled zero-valent iron**

Nanoparticles were prepared from  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  with ethanol and  $\text{NaBH}_4$  by colloidal method. Prepared 0.018 M iron (III) chloride (100 mL) solution was dissolved into 1L of 70% ethanol aqueous solution in a 2 L beaker to make iron (III) chloride solution for the iron source. Then, the  $\text{NaBH}_4$  aqueous solution was dropped (drop speed of 7-7.5 mL/min) into the prepared iron (III) chloride solution with mechanical stirring for 13 min at 500 rpm. After all the synthesized nanoparticles were settled, the supernatant was washed with ethanol and deionized water to remove the by-product salts and to replace the remaining water with ethanol. After washing, the resulting black particles were dried into oven for 1 hour. The weight of synthesized iron-nanoparticle was weighed by a mettler balance. The yield percent (44.52%) of NZVI-A was obtained.

Similarly, NZVI-B and NZVI-C were also prepared by using 80% ethanol and 90% ethanol solutions. The yield percentages of NZVI-B (54.79%) and NZVI-C (51.36%) were obtained.

#### **Purification of clay samples**

For precipitation of impurities, 5 g of natural clay were dispersed in 500 mL water. After 24 hours of decantation process, impurities were separated from the clay water mixture by filtration. The suspension was centrifuged at 5000 rpm for 15 min and then filtrated in order to separate the precipitated clay minerals. Pure clay was obtained by removing the liquid part of the suspension in a vacuum oven for 24 hours.

#### **Preparation of clay-A supported nanoscaled zero-valent iron (NZVI-B)**

Prepared 100 mL of 0.018 M iron (III) chloride solution was put in a 2 L beaker. 80% ethanol aqueous solution (100 mL) was added into the beaker. Then 1.5 g of clay-A was added to this solution and the mixture was held on a magnetic stirrer to be mixed. Then, the prepared  $\text{NaBH}_4$  aqueous solution was dropped (drop speed 7-7.5 mL/min) into the prepared iron (III) chloride and clay mixture solution, with mechanical stirring for 15 min at 5000 rpm. And then the beaker was removed from the stirrer. After all the synthesized nanoparticles was settled, the supernatant was removed and the precipitated nanoparticles was washed twice with anhydrous

ethanol and deionized water to remove the by-product salts and to replace the remaining water with ethanol. After washing, the resulting black particles were dried into oven for 1 hour. The yield percent of clay-A supported NZVI-B (85.37) was obtained. The dried samples were kept in air-tight container to prevent moisture changes and contamination.

### **Characterization of Nanoscale Zero-Valent Iron and Clay-A Supported of NZVI-B**

The characterization by XRD, EDXRF, SEM and TGDTA were made to examine the chemical, morphological and thermal properties of NZVI and clay-supported NZVI.

### **Preparation of Co<sup>2+</sup> Solution**

The sample solution (1000 ppm) of Co<sup>2+</sup> solution was prepared. Accurately 1.009 g of CoCl<sub>2</sub>.6H<sub>2</sub>O was weighed by using mettler balance and was placed in a beaker. 2 mL of HCl (1 M) solution was added into the beaker. The solution was stirred thoroughly by a glass rod. Then the solution was dissolved in 250 mL of deionized water.

Similarly, 500 ppm, 250 ppm and 125 ppm of CoCl<sub>2</sub>.6H<sub>2</sub>O solutions were prepared respectively.

### **Preparation of Cu<sup>2+</sup> Solution**

The sample of (1000 ppm) Cu<sup>2+</sup> solution was prepared. Accurately 0.982 g of CuSO<sub>4</sub>. 5H<sub>2</sub>O was weighed and was placed in a beaker. 2 mL of HCl (1 M) solution was added into the beaker. The solution was stirred thoroughly by a glass rod. And then the solution was dissolved in 250 mL of deionized water.

Similarly, 500ppm, 250 ppm and 125 ppm of CuSO<sub>4</sub>.5H<sub>2</sub>O solutions were prepared respectively.

### **Sorption Studies of Selected Metal Solutions on Prepared Sorbent NZVI-B and Clay-A Supported of NZVI-B**

#### **Contact time experiment**

Accurate weigh 0.1 g of NZVI-B was placed in each 10 mL of each 1000 ppm sample solution. The flask was stirred in the shaker at room temperature for 10 minutes. After 10 minutes, the sample was taken and removed by the filter paper. The residual content of this solution in the filtrate was determined by UV spectrophotometer.

The above procedure was also made with the solutions of Co<sup>2+</sup> and Cu<sup>2+</sup> at 500 ppm, 250 ppm and 125 ppm.

The remaining concentration of Co<sup>2+</sup> and Cu<sup>2+</sup> ions are determined by UV spectrophotometer. The resulting data are shown in Table 3.2 and 3.3.

#### **Effect of concentration of metal solutions**

The percent removal of Co<sup>2+</sup> and Cu<sup>2+</sup> were also determined within 50 minutes from 125 ppm, 250 ppm, 500 ppm, 1000 ppm solutions by 0.1 g of adsorbent dosage under same conditions. The results are shown in Table 3.4 and 3.5.

#### **Effect of adsorbent dosage**

The percent removal of Co<sup>2+</sup>, Cu<sup>2+</sup> within 50 minutes from 125 ppm, 250 ppm, 500 ppm, 1000 ppm solution were also determined by varying the adsorbent dosage from 0.1 to 0.3 g under same conditions. The percent sorption with respect adsorbent dosage is shown in Table 3.6 and 3.7.

## **Results and Discussion**

Nanoparticles usually ranging in dimension from 1-100 nanometers (nm) have properties unique from their bulk equivalent. With the decrease in the dimensions of the materials to the atomic

level, their properties change. The nanoparticles possess unique physicochemical, optical and biological properties which can be manipulated suitably for desired applications.

### **Synthesis of Nanoscaled Zero-valent Iron (NZVI)**

Nanoscaled zero-valent iron can be synthesized by several methods (Sun, *et al.*, 2006). The most widely used method for environmental purposes is the borohydride reduction of Fe(III) ion in aqueous media. The method consists of dropwise addition of sodium borohydride ( $\text{NaBH}_4$ ) to a solution of Fe (III) (Wang and Zhang, 1997). Synthesis of NZVI and even the experiments for the adsorption of several species were performed in inert conditions to keep iron in its zero-valent form. In this present work, NZVI is synthesized in open air in the presence of ethanol to prevent massive oxidation (Liu, *et al.*, 2005).

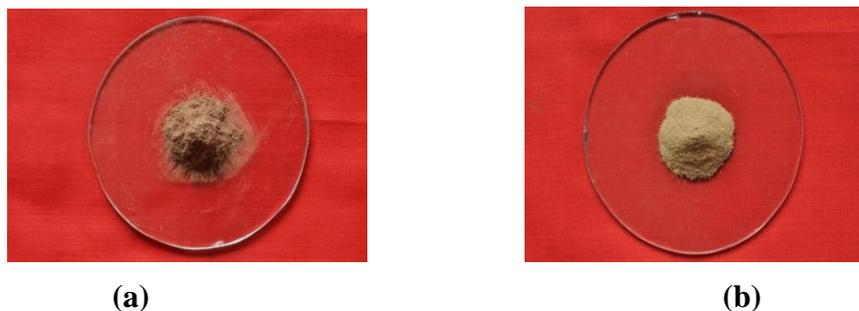


Figure 1. Photographs of (a) NZVI-B (b) clay supported NZVI-B

### **Characterization of Nanoparticles**

The characterization by XRD, EDXRF, SEM, FT-IR and TGDTA were made to examine the morphological, thermal and chemical properties of NZVI and clay-supported NZVI. To characterize the crystallographic structure, X-ray Powder Diffraction (Powder XRD) is used. X-ray diffraction is one of the most important characterization tools used in industry and science. This method can be used to gather qualitative and quantitative information about the compounds in the sample. Images in nanoscales are recorded by Scanning Electron Microscopy (SEM). The principle of SEM is the scanning of the surface of a solid material in a raster pattern with a beam of electrons as source. To determine the elemental analysis of samples, EDXRF is used. Thermal properties of prepared nanoscaled particles are detected by TG-DTA. Ultraviolet Spectrometry (UV) analysis is performed to determine the aqueous concentrations of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions.

#### **XRD analysis**

Figures 2 (a), (b) and (c) show XRD patterns of clay, NZVI-B and clay supported NZVI-B. Clay sample showed characteristic of crystalline structures but NZVI-B and clay supported NZVI-B showed characteristic of amorphous structures.

#### **EDXRF analysis**

Figures 3 (a), (b) and (c) show EDXRF spectra of clay, NZVI-B and clay supported NZVI-B. The relative abundance of elements for each sample was determined by the resultant data.

#### **SEM analysis**

Figure 4 (a), (b) and (c) show SEM images of the cross-sectional surface of clay, NZVI-B and clay supported NZVI-B. Nanoporous materials consist of a regular organic or inorganic framework supporting a porous structure.

#### **TG-DTA analysis**

Figure 5 (a), (b) and (c) show simultaneous thermogravimetric and differential thermal analysis (TG-DTA) of clay, NZVI-B and clay supported NZVI-B. According to the TG-DTA, there is no significant weight loss that has found in clay and clay supported NZVI-B but significant weight loss that has found in NZVI-B.

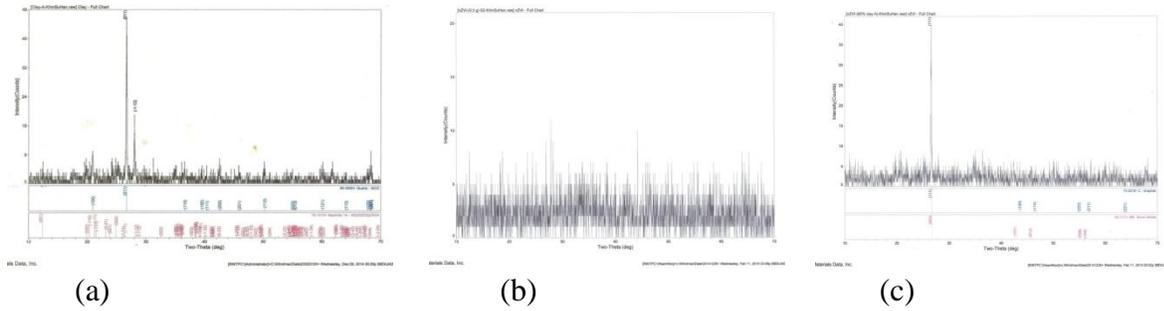


Figure 2. XRD spectra of (a) clay-A (b) NZVI-B (c) clay supported NZVI-B

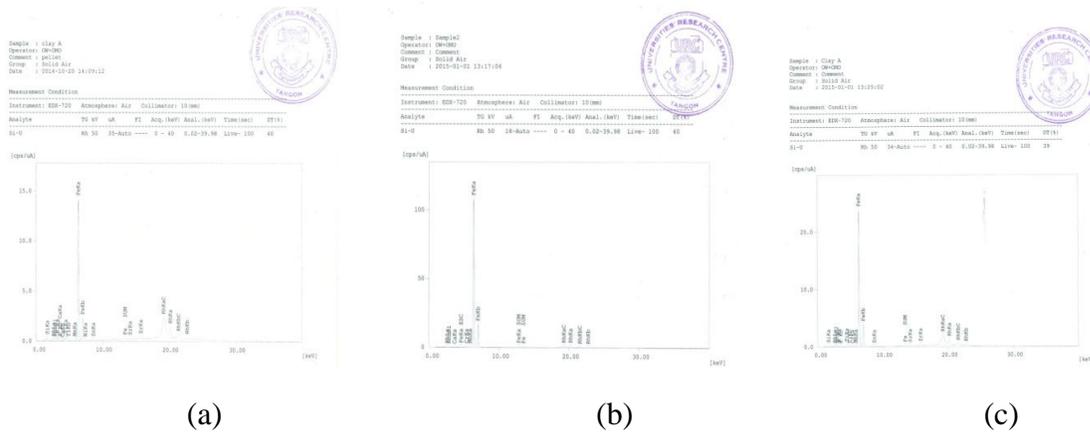


Figure 3. EDXRF spectra of (a) clay-A (b) NZVI-B (c) clay supported NZVI-B

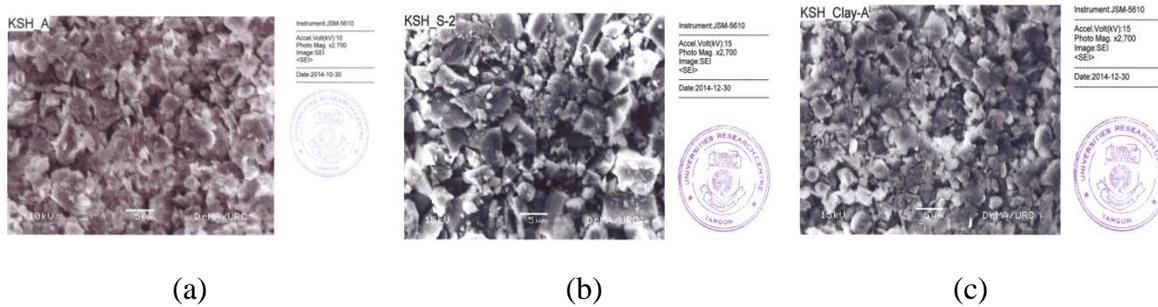


Figure 4. SEM photographs of (a) clay-A (b) NZVI-B (c) clay supported NZVI-B

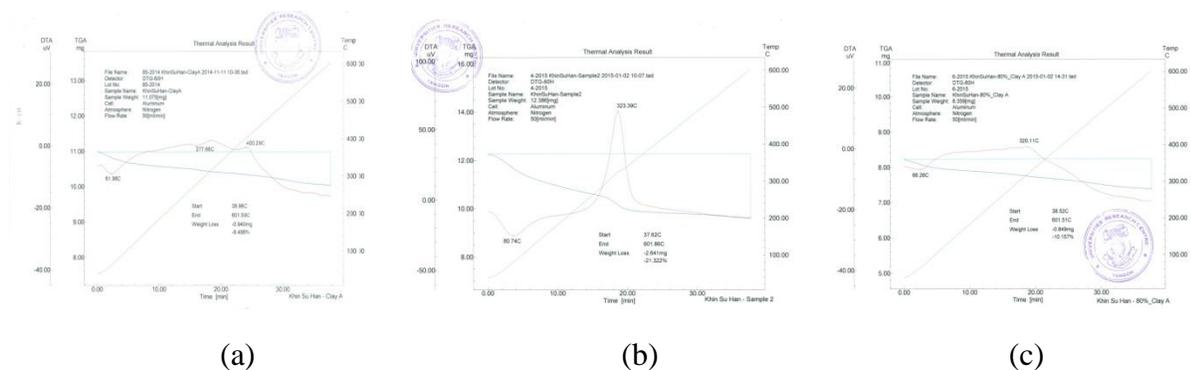


Figure 5. TG-DTA spectra of (a) clay-A (b) NZVI-B (c) clay supported NZVI-B

Table 1. Relative Abundance of Elements in Clay-A, NZVI-B and Clay Supported NZVI-B by EDXRF Analysis

No	Elements	Relative Abundance (%)		
		Clay-A	NZVI-B	Clay Supported NZVI-B
1	Si	65.262	-	43.874
2	Fe	24.596	99.195	49.510
3	K	7.021	-	4.304
4	Ti	1.918	-	1.607
5	Ca	0.660	0.165	0.235
6	Mn	0.245	0.425	0.319
7	Zr	0.108	-	0.092
8	Ni	0.071	-	-
9	Zn	0.065	-	0.051
10	Sr	0.053	-	0.040
11	Cr	-	0.215	0.115

### Metal Uptake by NZVI-B and Clay Supported NZVI-B

Prior to testing the synthesized NZVI-B and clay supported NZVI-B as adsorbents for removal  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions, the sorption capacity of NZVI-B and clay supported NZVI-B, towards aqueous  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions were investigated. These experiments formed a reference in the assessment of the adsorption capacity of the synthesized materials. For this purpose, batch tests were run with various initial metal concentrations and adsorbent dosage as described in experimental section.

Generally the removal percentages decrease as the initial metal concentrations are increased for  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions uptake. It is clear that clay-supported NZVI-B shows greater removal capacities for  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions in comparison to NZVI-B. The adsorption capacities for removal of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions, within the studied concentration ranges of these ions, are

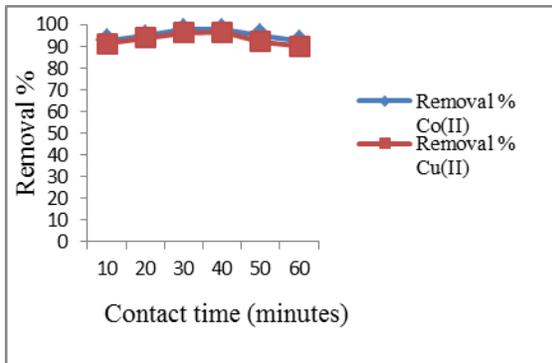
presented in Table 3.2 to 3.7 and Figure 3.6 to 3.8 by NZVI-B and clay-supported NZVI-B, respectively.

Table 2. Effect of Contact Time for Removal Efficiency of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by NZVI-B  
initial concentration = 250 ppm  
adsorbent dosage = 0.2 g

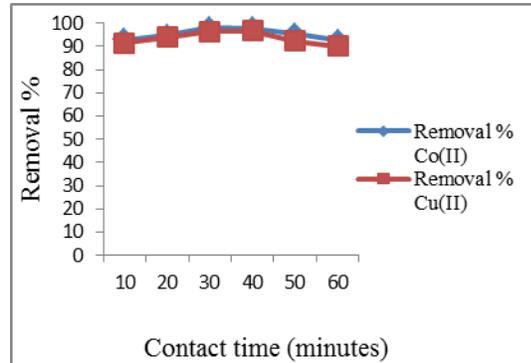
Contact time (minutes)	Removal % by NZVI-B	
	$\text{Co}^{2+}$	$\text{Cu}^{2+}$
10	70.64	66.35
20	79.92	78.19
30	85.36	85.19
40	87.36	86.5
50	91.55	90.04
60	91.49	90.02

Table 3. Effect of Contact Time for Removal Efficiency of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by Clay-A Supported NZVI-B

Contact time (minutes)	Removal % by Clay-A Supported NZVI-B	
	$\text{Co}^{2+}$	$\text{Cu}^{2+}$
10	92.78	91.42
20	94.82	93.87
30	97.91	96.43
40	97.84	96.60
50	97.87	96.19
60	92.48	90.06



(a)



(b)

Figure 6. Effect of contact time for removal efficiency of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by (a) NZVI-B

(b) Clay-A supported NZVI-B

Effect of contact time for removal efficiency of  $\text{Co}^{2+}$

Table 4. Effect of Adsorbent Dosage on the (%) Removal of  $\text{Co}^{2+}$ ,  $\text{Cu}^{2+}$  of NZVI-B

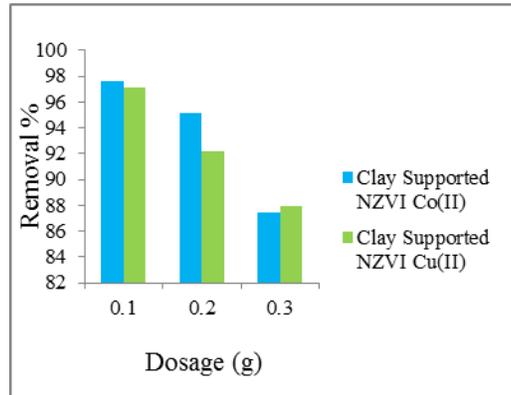
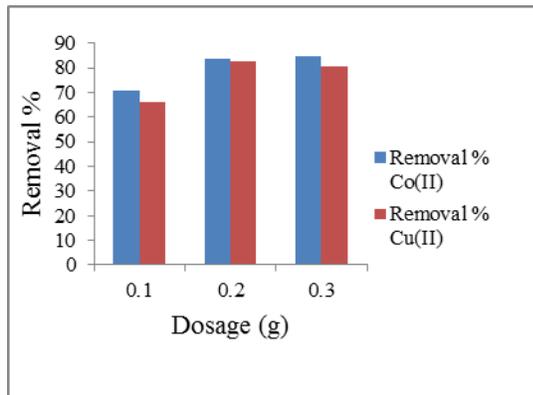
contact time = 50 min  
Initial concentration = 250 ppm

Dosage (g)	Removal % by NZVI-B	
	$\text{Co}^{2+}$	$\text{Cu}^{2+}$
0.1	70.96	66.28
0.2	83.43	82.54
0.3	84.68	80.38

Table 5. Effect of Adsorbent Dosage on the (%) Removal of  $\text{Co}^{2+}$ ,  $\text{Cu}^{2+}$  of Clay-A supported of NZVI-B

contact time = 50 min  
Initial concentration = 500 ppm

Dosage (g)	Removal % by Clay-A Supported NZVI-B	
	$\text{Co}^{2+}$	$\text{Cu}^{2+}$
0.1	95.44	94.14
0.2	98.52	96.75
0.3	94.22	92.68



(a)

(b)

Figure 7. Effect of adsorbent dosage on the (%) removal of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by (a) NZVI-B (b) Clay-A supported of NZVI-B

Table 6. Effect of Concentration for Removal of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by NZVI-B on Various Concentrations

Contact time = 50 min  
Adsorbent dosage = 0.2 g

Initial Concentration (ppm)	Removal % by NZVI-B	
	Co(II)	$\text{Cu}^{2+}$
125	90.55	83.43
250	93.04	87.54
500	89.22	80.21

1000

86.47

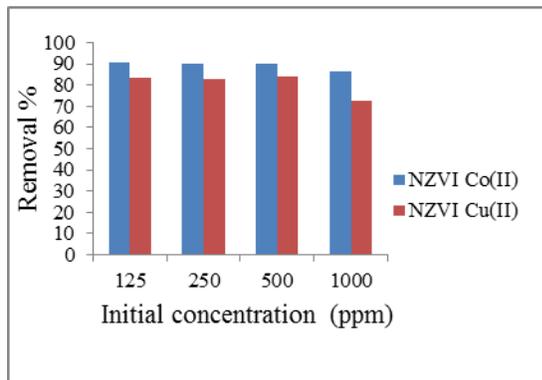
72.61

Table 7. Effect of Concentration for Removal of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by Clay-A supported NZVI-B on Various Concentrations

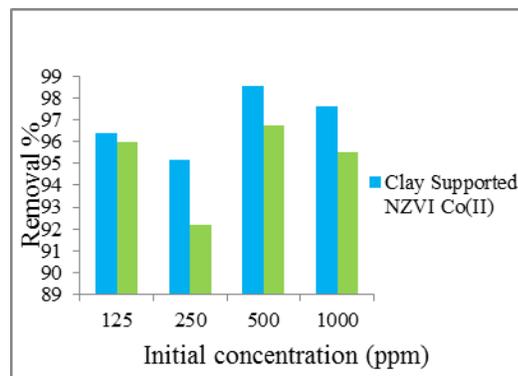
Contact time = 50 min

Adsorbent dosage = 0.2 g

Initial Concentration (ppm)	Removal % by clay-A supported NZVI-B	
	$\text{Co}^{2+}$	$\text{Cu}^{2+}$
125	96.37	95.00
250	98.57	97.19
500	97.52	96.75
1000	94.62	95.50



(a)



(b)

Figure 8. Effect of concentration for removal of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  by (a) NZVI-B (b) clay supported NZVI-B on various concentrations

### Conclusion

In this study, nanoscaled zero-valent iron (NZVI) and clay-supported nanoscaled zero-valent iron were synthesized in ethanol by borohydride reduction method under atmospheric conditions. It was observed that iron nanoparticles are mainly in zero-valent oxidation state and that no significant oxidation took place for weeks of storage under atmospheric conditions.

The yield percent of NZVI-A (44.52%), NZVI-B (54.99%), NZVI-C (51.36%) and clay A-supported NZVI-B (85.37%) were obtained from the synthesized materials.

The characterization of the synthesized materials was performed in terms of XRD, SEM, EDXRF, FTIR and TGDTA. From the XRD spectra, it was found that clay-A is nanoscaled crystalline patterns with 99.58 nm.

The synthesized materials are used for the removal of  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions in aqueous media under the various experimental conditions. The effects of contact time, initial metal concentration and adsorbent dosage were investigated to clarify the adsorption properties of NZVI-B and clay A-supported NZVI-B against  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$ . According to results, unsupported and clay-supported NZVI have a great capacity to immobilize  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$  ions.

For both types of adsorbents and for both ions,  $\text{Co}^{2+}$  and  $\text{Cu}^{2+}$ , under investigation, it was observed that the removal percentages commonly increase with initial concentrations 250 ppm and adsorbent dosage 0.2 g at a contact time 50 minutes.

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