

# Heavy Metals Removal Using Natural Jordanian Volcanic Tuff

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**Abstract**— The removal performance and the selectivity sequence of separate metal ions ( $\text{Fe}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$  and  $\text{Ni}^{2+}$ ) in aqueous solution were studied by adsorption process on untreated and natural volcanic tuff. A series of experiments were conducted in batch-wise and fixed-bed columns to investigate the removal efficiency of natural Jordanian volcanic tuff as low cost and an effective adsorbent for heavy metal ions and to examine its economical application in water purification and treatment practices.

Water and wastewater samples containing metal ions with concentrations ranging from 1 to 15 mg/L were used. The plexi glass columns were filled with natural occurring volcanic tuff particles ranging between (0.350 – 3.000) mm. Photometric methods were used for laboratory analysis of samples.

The experiments were carried out under changing conditions as a function of different pH-values (2,4,6 and 7), initial solute concentrations (1, 5, 10, 15) mg/L, and different room temperatures (20, 25 and 30 C°), and varying tuff particle sizes (0.35 -3.0) mm. The breakthrough curves were derived by plotting the normalized effluent metal concentrations (C/C0) versus bed volume.

Obtained results showed that natural Jordanian volcanic tuff has an adsorption capacity of 0.417 mg/g for  $\text{Fe}^{2+}$  and 0.151mg/g for  $\text{Cr}^{2+}$ . Factors in the reaction medium such as pH and ionic strength influenced the adsorption process. The quantity of particular ionic species ( $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Cr}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Zn}^{2+}$ ) bound in dependence on the initial concentrations, indicates that the removal efficiency from the liquid phase follows the sequence  $\text{Fe}^{2+} > \text{Cu}^{2+} > \text{Pb}^{2+} > \text{Cr}^{2+} > \text{Zn}^{2+}$  when keeping the pH at 4 and follows the sequence  $\text{Cu}^{2+} > \text{Zn}^{2+} > \text{Fe}^{2+} > \text{Cr}^{2+} > \text{Pb}^{2+}$  when keeping the pH at 6. Equilibrium modeling of the removal showed that the adsorption of the metal cations  $\text{Cr}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Fe}^{2+}$  were fitted to one of the adsorption isotherms.

**Keywords**- Natural volcanic tuff, heavy metals, sorption, ion exchange, water, wastewater, Jordan.

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## Novelty Statement

This research work deals with the use of local, low cost soil material existing in huge amounts in Jordan. A country lacking on water resources, where municipal wastewater is discharged together with polluted industrial wastewater containing industrial pollutants, in particular, heavy metals. Existing wastewater treatment plants are not capable of coping with these kinds of pollutants, unless a separate stage may be included to capture heavy metals. Rain and storm water subjected to be stored in dams may also involve heavy metals added to these waters. It is of great benefit to run these waters over beds of soil materials that are proved to be efficient in removing heavy metals.

## Highlights

Natural volcanic tuff as a low-cost material is efficient material in removing a mixture of heavy metal ions, in particular copper [ $\text{Cu}^{+2}$ ], zinc [ $\text{Zn}^{+2}$ ], chromium [ $\text{Cr}^{+3}$ ], lead [ $\text{Pb}^{+2}$ ] and iron [ $\text{Fe}^{+2}$ ], from aqueous solutions.

The column experiments provide useful information about the transport behavior of the model contaminants.

Acidic metal solutions reduce the performance of natural volcanic tuff in retarding the movement of heavy metals and affect the mineralogical composition of the materials.

## 1.0 Introduction

Wastewater containing heavy metals are generated by a wide variety of industries, such as metal finishing, automobile manufacturing, electronics manufacturing, textile industry and others.  $\text{Pb}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Fe}^{+2}$ ,  $\text{Zn}^{+2}$  and  $\text{Cr}^{+3}$  are especially common metal ions that tend to accumulate in organisms, causing numerous diseases and disorders [1].

Due to their significant toxicity heavy metals are a global concern as a pollutant of drinking water and groundwater. The

problem is the considerable volume of wastewater containing heavy metals throughout the world and the lack of effective and economical treatment and, more particularly, the removal of heavy metals. Heavy metal ions can be removed from contaminated and wastewaters using a wide range of methods such as precipitation, solvent extraction, vacuum evaporation, membrane technologies, adsorption and ionic exchange. On a global scale, different kinds of zeolites and clays also have a large applicability for decontamination, purification of urban and industrial residual waters.

Jordan is a country with limited water resources and this necessitates that much effort is put into water conservation and environmental protection. The expansion of industrial activities, including metal-based industries, and the excessive use of chemicals increase the pollution of waters with heavy metals. All these require the availability of low-cost technology and materials for wastewater treatment. However, the country has huge reserves of volcanic tuffs, especially in the northeast where Phillipsite is the dominant mineral [2]. Volcanic tuff deposits, rich in phillipsite and zeolite, were first discovered in Jordan in 1987 in the eastern part of the country at Jebal Aritain. Since then zeolites have also been found in other locations, mainly in the basaltic areas in the northeastern region of Jordan [3]. The huge reserves and the availability of the volcanic tuff as low cost material encouraged the authors to carry out this research. In addition The development of new and cost effective methods to remove heavy metals from ground water, drinking water and wastewater also becomes one of the research priorities.



Figure 1: The natural Jordanian volcanic tuff used in experiments.

Volcanic tuff containing zeolite particles are good carriers of bacteria, which adsorb on the zeolite surface resulting in increased sludge activity. They are considered to be, next to clay iron-oxide-coated sands and activated carbons, low-cost sorbents and offer a potential for a variety of industrial uses.

In literature there are many studies dealing with removing heavy metal by using natural zeolite in batch experiments [ 1, 2, 3, 4, 5, 6, 7, 8, 9], but not enough data about using the column experiment are available.

The present research work relates to water and wastewater treatment and aims to study the adsorption/adsorption behavior of different heavy metal ions  $\text{Cr}^{3+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Cu}^{2+}$

and  $\text{Fe}^{2+}$  on natural untreated volcanic tuff material as low cost soil material.

## 2. Materials and methods

### 2.1 Materials used in the study

Glass columns with different heights (40 -70 cm) were used in the batch and column experiments. As fill and ion exchanger material, Jordanian natural tuff aggregates were used.

The conventional mineral processing techniques of Jordanian zeolitic tuff are starting with crushing the materials followed by autogenously tumbling mill and then low intensity magnetic and gravity separation. Zeolite tuff was ground and then sieved to different fractions of which the fraction of 0.5 - 3.0 mm were selected and used.

The grain size distribution of the used volcanic tuff material is presented in table 2.1:

| Grain size (mm) | Weight (g) | Percentage (%) |
|-----------------|------------|----------------|
| < 3             | 1000       | 100            |
| <2              | 954.3      | 95.43          |
| <1              | 439.15     | 43.92          |
| <0.5            | 46.68      | 4.67           |

Table 1: grain size distribution of the used volcanic tuff material

The tuffs are not exposed to any pre - treatment or modification. Samples were washed and dried at 103.5 °C and kept in desiccators for one day to dry completely out.

The initial aqueous solution of metals (1, 5, 10, 20, 30, and 50) mg/L were prepared using standard solution for each metal.

### 2.2. Design of Experiments

In the present work, a batch-scale system using coarse and fine zeolite packed columns was operated to investigate the continuous removal of heavy metals. In these experiments, the effects of flow rate, hydraulic detention time, particle size of zeolite tuffs, column height, inlet solution concentration and the pH on removal efficiency were investigated.

In one-dimensional experiments with volcanic tuff aggregates filled columns of different sizes (fine, middle and coarse aggregates), were fed with different types of wastewater (natural raw wastewater, synthetic prepared wastewater) from top.

The fixed bed columns, which will allow the most experimental variations, are filled with a layer of fine aggregates < 1mm at the bottom, over which a layer of tuff (different grain sizes) comes. The hydraulic conductivity can be controlled by recording the time required to collect the injected sample outflow (hydraulic detention time). Samples from the wastewater inflow and outflows should be taken for chemical analyses, whereas zeolite tuff samples are to be taken for chemical parameters analysis.

The dry mass of volcanic tuff used in one column is about 79.52 g which forms a height of 10 cm. The volume of wastewater sample poured in the columns is 40 ml each time.

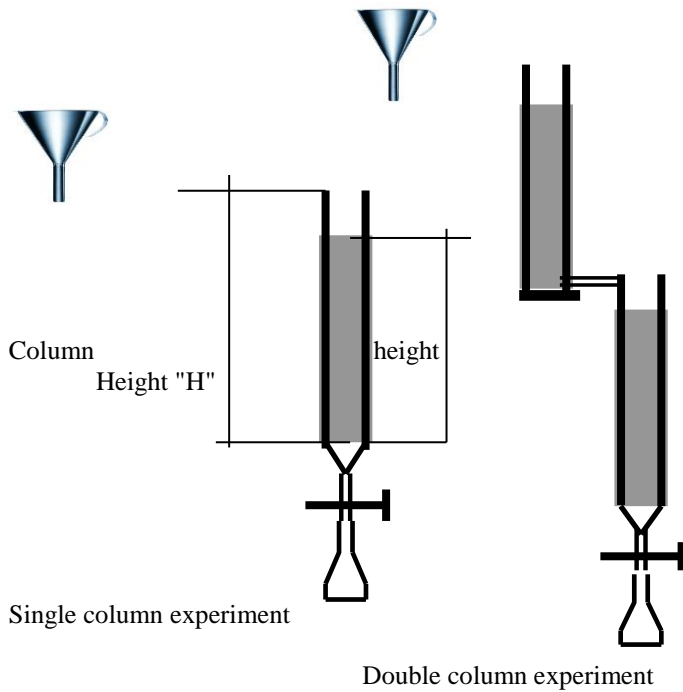


Figure 2: Column Experiment

### 2.3 Methodology

Jordanian zeolite tuff was studied for its potential use as an adsorbent for different metal ions ( $\text{Cu}^{+2}$ ,  $\text{Pb}^{2+}$ ,  $\text{Cr}^{2+}$ ,  $\text{Fe}^{2+}$  and  $\text{Zn}^{2+}$ ) from aqueous solutions. The batch technique of metal ions from solution was carried out at specific conditions, these are:

- at pH of 1.0, 3.0, 4.0, 6.0 and 7,
- at temperatures 10, 20.0°C, 30.0°C and 45.0°C,
- at different contact time and with the same ionic strength (0.1M  $\text{NaClO}_4$ ).

Analysis of data will be based on adsorption models such as Langmuir, or Freundlich isotherms. Adsorption kinetics were applied in order to determine adsorption mechanism and adsorption characteristic constants.

Representative samples of Jordanian zeolitic tuff were investigated by using optical microscopy, scanning electron microscopy (SEM), X-Rays diffraction (XRD).

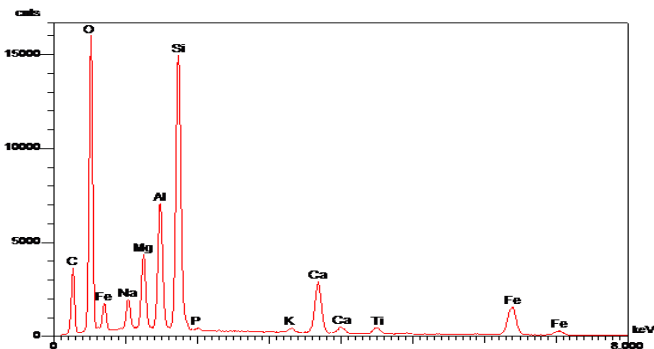


Figure 3: X-Rays diffraction spectrum of Jordanian volcanic tuff

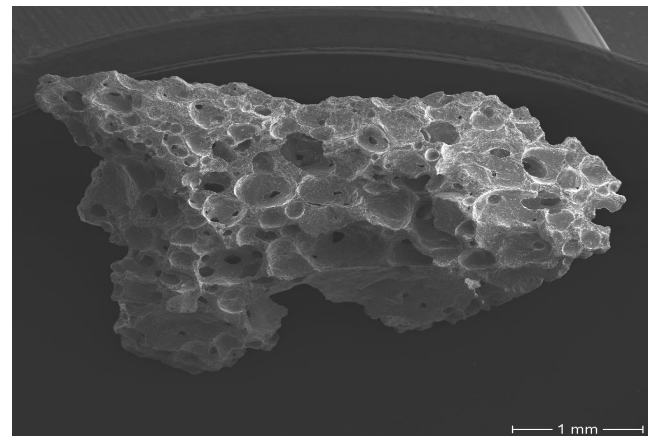
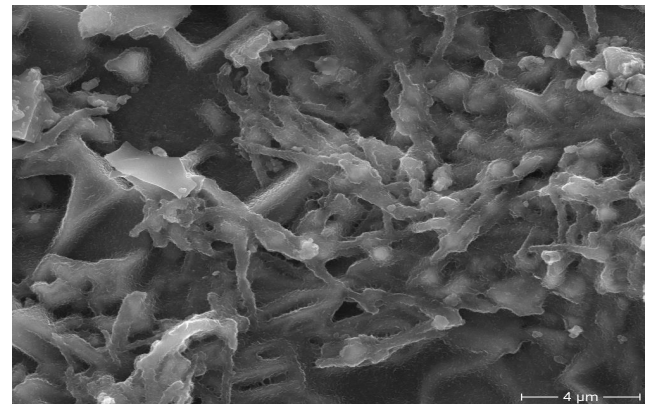


Figure 4: SEM images of Jordanian volcanic tuff shows tabular crystals associated with small fibrous crystals

For a better understanding the removal efficiency of zeolitic tuff, a chemical analysis of the mineral is performed. The chemical composition in (%) of the zeolitic material examined in work is presented in table 3.

|                                |       |
|--------------------------------|-------|
| SiO <sub>2</sub>               | 70.08 |
| MnO                            | 0.04  |
| Al <sub>2</sub> O <sub>3</sub> | 11.72 |
| Na <sub>2</sub> O              | 1.55  |
| CaO                            | 3.18  |
| K <sub>2</sub> O               | 3.78  |
| MgO                            | 0.96  |
| TiO <sub>2</sub>               | 0.16  |
| Fe <sub>2</sub> O <sub>3</sub> | 1.20  |

Water content ~ 4 %

Table 2: Chemical analysis of Jordanian volcanic tuff used in experiments

### Sampling procedure

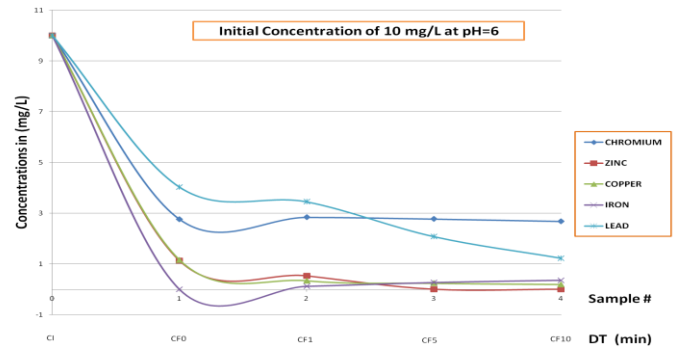
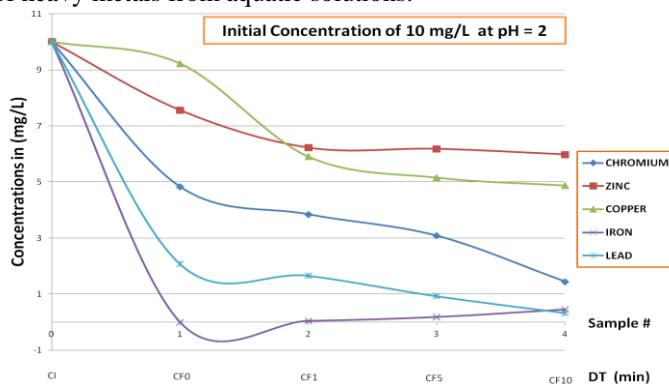
The effect of solution pH on uptake of the metal ions by zeolitic tuff was studied at room temperature to examine and understand the maximum removal efficiency. For these

investigations, a series of 50 mL glass test tubes were employed. Each test tube was filled with 40 mL of a metal ion solution (each batch has different initial concentration) and adjusted to the desired pH from 2.0 to 7.0 by using the pH meter. The pH of the solution was adjusted using dilute solution of hydrochloric acid HCl or sodium hydroxide NaOH. A known amount of zeolitic tuff (10 mg) was added into each test tube. A continuous shake of tubes were performed to maintain an equilibrium and complete mixing. The effluent suspension was diluted (if necessary) to an appropriate concentration range for the elemental analysis by atomic absorption spectroscopy. The detention time denoted as DT in minutes, reflected the real detention time of the aqueous solution within the fixed bed. Each batch consists of 4 effluent samples with detention times varying from zero to 10 minutes. The first sample (sample # 1) is poured over the zeolitic tuff bed and collected immediately at the bottom with detention time zero (denoted by CF0). The second sample (sample # 2) is collected after lasting in the bed for 1 minute (CF2), the third sample (sample # 3) lasts 5 minutes (CF5), where as the fourth sample (sample# 4) is allowed to last 10 minutes (CF10).

### 3. Results and Discussions

#### 3.1 The sorption capacity of volcanic tuff material

In experimental performance by the batch method, due to the small ratio of volumes of zeolite/solution, the kinetics of the removal is determined by diffusion through the natural zeolitic material particles. The experiments are carried out at different pH values (2, 4, 6 and 7) and with different initial concentrations (1, 5 and 10 mg/L). The results show that the ionic exchange reactions took place for all samples considered in the experiments. The most obvious result obtained for all metals considered and at different initial concentrations, is the decrease in the initial concentration. Therefore it is to conclude that volcanic tuffs are active materials in the ionic exchange process and can be strongly employed for removal of heavy metals from aquatic solutions.



Figures (5.1, 5.2): show the decrease of initial concentration (in this case 10 mg/L) of heavy metal ions applied within the fixed volcanic tuff bed by different detention times. Similar behavior of heavy metal ions is also observed when applying aqueous solutions with initial concentrations 1, and 5 mg/l.

It is to pinpoint that all experiments are repeated for every aqueous solutions to a new volcanic bed each time with the aim to avoid the interference of heavy metals among each other.

#### 3.2 Metal uptake as a function of pH

The solutions containing heavy metals ions have been adjusted to different pH values (2, 4, 6 and 7), this is made in order to examine the elimination of these ions at different acidic conditions. The experiments and obtained results show clearly the decrease of initial concentrations of heavy metal ions as a function of pH.

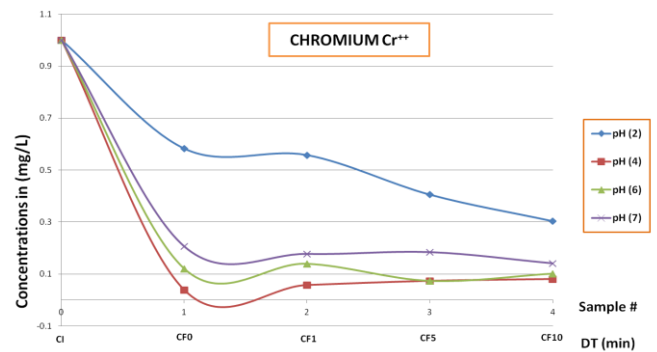


Figure 6: Chromium uptake behavior by different pH

For example: chromium ions show little ability to be absorbed by bed material at pH = 2 when compared to their ability to be absorbed at pH = 4 or pH = 6. Similarly, lead ions show a higher ability to be absorbed by fixed bed material at pH = 4 than at pH = 6 as shown in figure 7.



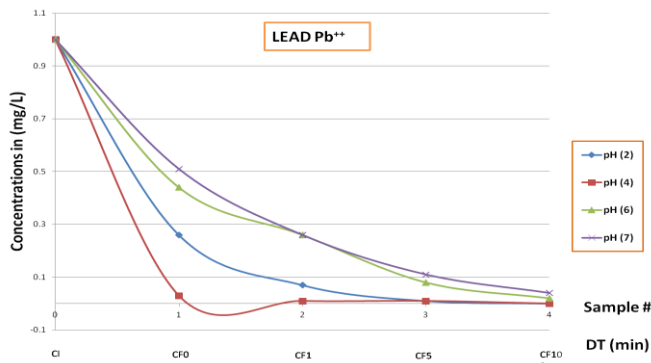


Figure 7: Lead ions uptake behavior by different pH

### 3.3 The uptake capacity of volcanic tuff on metal ions.

The experimental results of the binding of the heavy metals ( $\text{Cu}^{+2}$ ,  $\text{Pb}^{2+}$ ,  $\text{Cr}^{2+}$ ,  $\text{Fe}^{2+}$  and  $\text{Zn}^{2+}$ ) ions on natural volcanic tuff indicate a higher degree of ion removal at (lower/higher) initial concentration area of metal ions. The plots in figure 8.1, 8.2 and figure 8.3 show the uptake degree of each heavy metal ion as a function of the initial concentration. The uptake degree is defined as:  $\alpha = (C_i - C_e/g \text{ fixed bed})$  measured in mg/L/g.

$C_i$  being the initial concentration, and  $C_e$  final concentration of the particular ion. It is to emphasis that the volume of all solutions considered was 40 ml for each application.

The experiments show that applying solutions containing heavy metal ions is an efficient method of removal of heavy metal ions from wastewater containing lower metal concentrations. On the other hand, the metal uptake is shown to be selective. The quantity of particular ionic species ( $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Cr}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Zi}^{2+}$ ) bound in dependence on the initial concentrations, indicates that the removal efficiency from the liquid phase follows the sequence  $\text{Fe}^{2+} > \text{Cu}^{2+} > \text{Pb}^{2+} > \text{Cr}^{2+} > \text{Zi}^{2+}$  when keeping the pH at 4 and follows the sequence  $\text{Cu}^{2+} > \text{Zi}^{2+} > \text{Fe}^{2+} > \text{Cr}^{2+} > \text{Pb}^{2+}$  when keeping the pH at 6.

At pH= 7 the removal efficiency sequence is  $\text{Cu} > \text{Fe} > \text{Pb} > \text{Cr} > \text{Zi}$ . For instance, when flowing through a fixed bed with solutions of different metals, the material prefers copper and iron ions to zinc or chromium at pH =4 . At pH = 6 the material prefers zinc, copper and iron ions to lead or chromium.

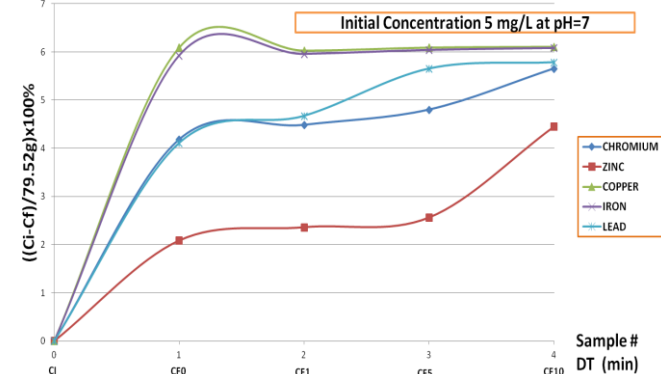
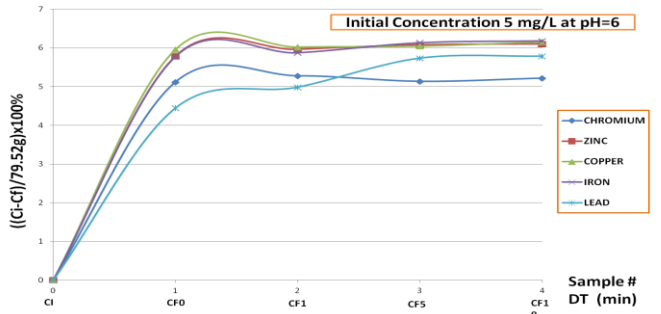
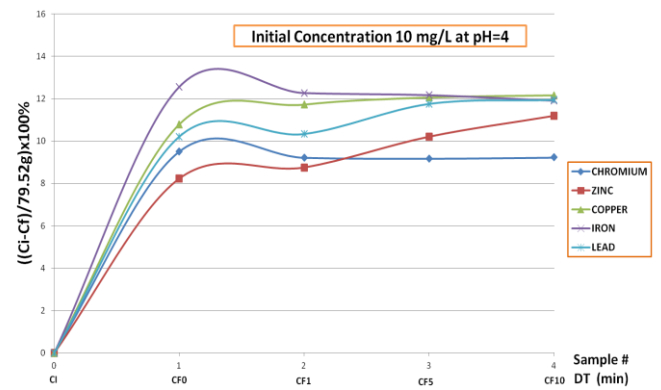
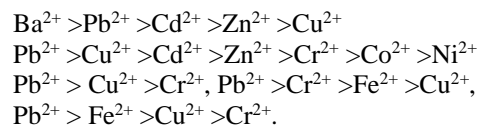


Figure 8.1, 8.2 and 8.3: Binding of metals on fixed bed material at different pH values as a function of initial concentration applied.

Several selectivity sequences have been reported in the literature (mainly conducted in a single solution) for natural zeolites [10, 11, 12, 13]:



The observed differences in selectivity sequences were considered to be due to the specifics of the adsorbents and to the differences in the experimental techniques used.

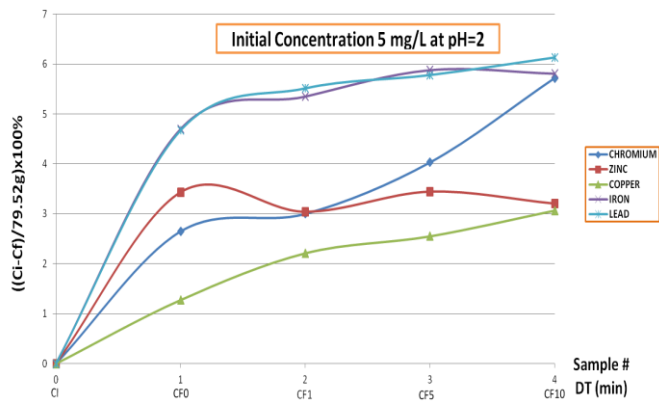


Figure 9: metal uptake at pH = 2 and initial concentration of 5 mg/L

### 3.4 Breakthrough curves

The breakthrough curve experiment of heavy metals on natural zeolite was carried out using the batch method. Batch experiments were conducted using 10 g of adsorbent with 40 ml of solutions containing heavy metal ions of desired concentrations at constant temperatures (25° C).

Two metals iron and copper are applied to a fixed bed column filled with 10 g of volcanic tuff in order to examine the time of saturation of material with these two metal ions.

The batch adsorption experiment were conducted (started first with iron solution) with an initial concentration of 10 mg/l. 40 ml of this solution is applied to the column containing 10 g of bed material every 10 minutes. The discharge is taken and analyzed. After exactly 335 minutes the analysis of the sample taken shows that the final concentration of iron in samples is still low, which means that the adsorption capacity of material is high. In order to fasten the saturation of the bed material, the experiment is then continued with an initial concentration of iron = 20 mg/l also here with 10 minutes tact. After exactly 580 minutes the final concentration stabilizes and starts to increase, this gives a belief that the material is saturated with iron and reaches its ultimate absorbing capacity.

It is to emphasize that iron is selected with the belief that because iron is a major component of the natural material and the absorbing capacity of the material for iron can be limited when compared to the absorbing capacity of tuff on other metal ions that are less present. It is conclude that the material possesses a high absorbing capacity on heavy metals ions similar or even higher that its capacity to absorb iron ions. This gives the evidence that the material is very suitable to remove heavy metal ions from aqueous solutions when coming to contact with the material.

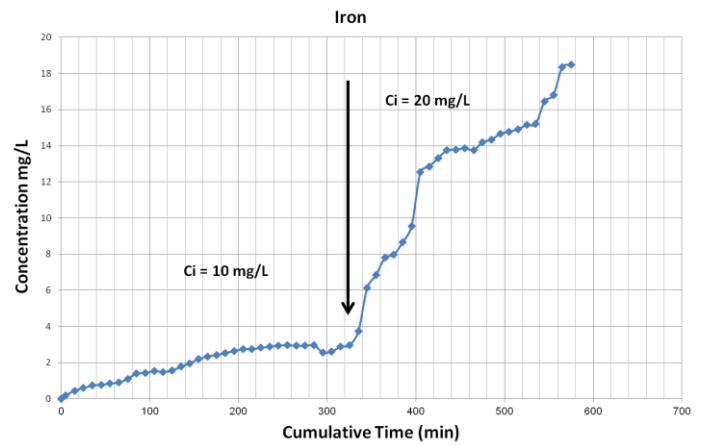


Figure 10.1: Iron breakthrough curve

Figure 10: shows the breakthrough curve for copper. The experiment is repeated for copper solution with an initial concentration of 10 mg/l using the same bed that is previously brought to saturation with iron ions. It is to observe that after exactly 192 minutes the material is completely saturated with copper ions.

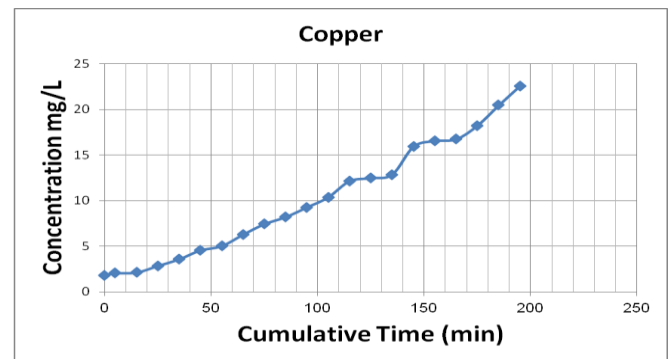


Figure 10.2: Copper breakthrough curve

### 3.5 Uptake capacity factor for Iron and copper

The Uptake capacity UP, to absorb contaminants in this case metal ions, can be determined by using following mass balance equation:

$UP = (V/m) \times (Ci - Cf)$ , where, UP = contaminant loading at a given time period until saturation point [mg/g].

V = volume of the solution that contains the contaminant [mL]. m = mass of the adsorbent material [g]. Ci = solute initial concentration in solution [mg/L]. Cf = solute final concentration in solution [mg/L].

The iron breakthrough curve experiment involves the application of:

35 applications each 40 mL of liquid with an initial concentration of iron = 10 mg/L in addition there are 24 applications of 40 mL of solution with an initial concentration of 20 mg/L. This iron loading is completely absorbed by the 79.52 g bed material before reaching the saturation point. An uptake capacity factor can be calculated using these data as follows:

$$\text{Total iron loading: } 35 \times 0.040 \text{ L} \times 10 \text{ mg/L} + 24 \times 0.040 \text{ L} \times 20 \text{ mg/L} = 33.2 \text{ mg}$$

$$\text{Uptake capacity factor, } UC_{Fe} = 33.2/79.52 \text{ g} = 0.417 \text{ mg/g.}$$

In order to reach the saturation point with copper, there are 30 applications needed with initial concentration = 10 mg/L.

$$\text{Copper loading: } 30 \times 0.04 \text{ L} \times 10 \text{ mg/L} = 12 \text{ mg. Uptake capacity factor, } UC_{Cu} = 12.0 \text{ mg}/79.52\text{g} = 0.151 \text{ mg/g. } UP_{Fe}/UP_{Cu} = 2.76.$$

### 3.6 Adsorption Efficiency of volcanic tuff:

The efficiency of volcanic tuff on absorbing heavy metal ions can be determined using following equation:  $(C_i - C_f / C_i) \times 100\%$ .

The percent adsorption (%) and distribution ratio (Kd) were calculated using the equations:

% adsorption =  $(C_i - C_f / C_i) \times 100$ , where  $C_i$  and  $C_f$  are the concentrations of the metal ion in initial and final solutions, and respectively, and  $K_d = \text{amount of metal in adsorbent} / \text{amount of metal in solution} \times V / m \text{ ml/g}$ , where  $V$  is the volume of the solution (ml) and  $m$  is the weight of the adsorbent (g) [14].

The amount of contaminant fixed on the volcanic tuff is a function of the initial concentration of contaminant in solution  $p = f(C_i)$ . This relationship can be easily seen by examining the breakthrough curves.

Because all experiments are conducted using the procedure and the same weight and volume of fixed bed material and the same volume of aqueous solution, the results of concentration decrease within the fixed bed can be taken to represent uptake capacity of the fixed bed material. The concentration decrease in dependence of the initial concentration of the aqueous solution is presented in following figures.

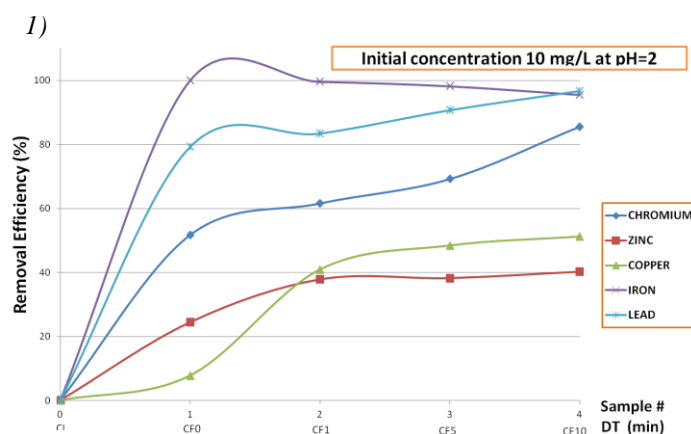
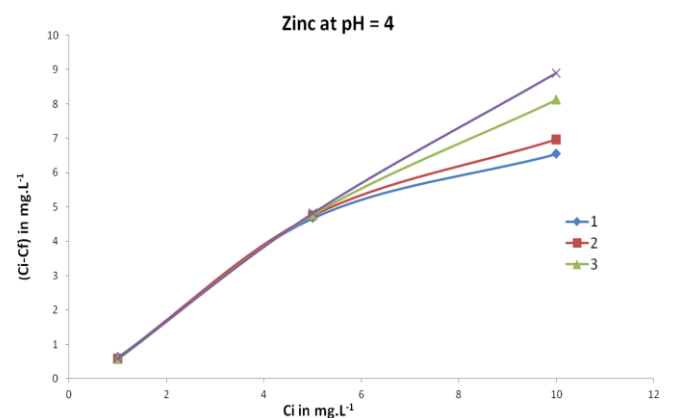
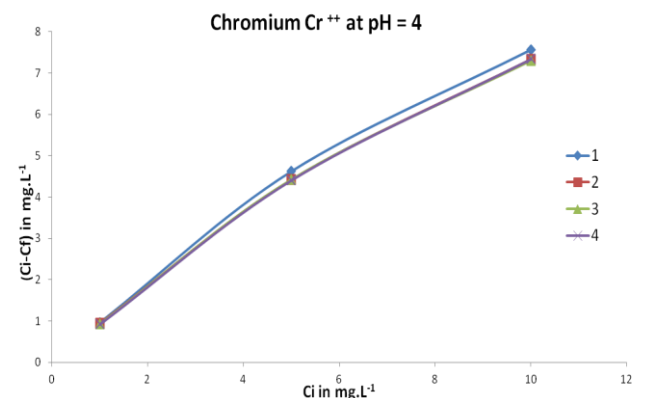
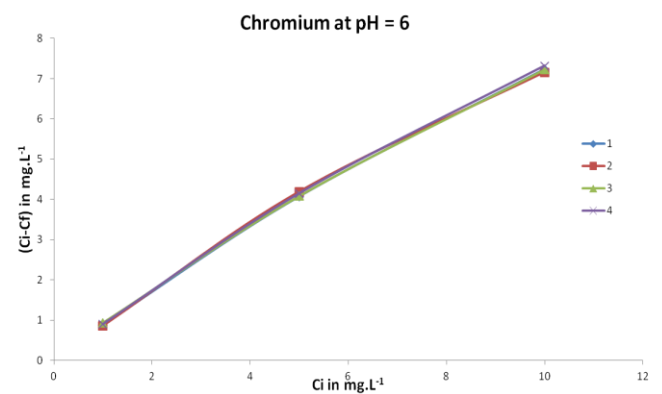


Figure 11: The results show different performance efficiency of used material in absorbing heavy metal ions.



Figures 12.1 , 12.2 and 12.3 represent the uptake of ions by the fixed bed material in relationship with the increase initial concentration of ions.

In fitting these obtained data to one of the adsorption isotherm models it is to conclude that the results clearly show that the uptake capacity of the material is behaving following the linear adsorption model.

#### 4.0 Conclusions

The present study showed the ability of the natural volcanic tuff as a low-cost material in effectively removing a mixture of heavy metal ions , in particular copper [Cu<sup>+2</sup>], zinc [Zn<sup>+2</sup>], chromium [Cr<sup>+3</sup>], lead [Pb<sup>+2</sup>] and iron [Fe<sup>+2</sup>], from aqueous solutions. The column experiments provide useful information about the transport behavior of the model contaminants. Relevant parameters for effective removal are the time to first breakthrough, the retardation factors, the volume of water treated and the amount of contaminant bound on the Ion exchangers.

The Jordanian natural volcanic tuff possess, on the one hand good retention capacity for cationic metals. On the other hand, the metal uptake is selective. For instance, when flowing a solution with two metals through the fixed bed, the material prefers some ions to others. It showed that the selectivity sequence of metal ions by the adsorbents was dependent on the system employed, and was mainly dependent on the initial concentrations of the metal ions and the initial pH of the solution.

Acidic metal solutions reduce the performance of natural volcanic tuff in retarding the movement of heavy metals and affect the mineralogical composition of the materials. Results obtained were modeled using one of the adsorption isotherm models. The results clearly show that the uptake capacity of the used tuff is behaving following the linear adsorption model.

#### Acknowledgements

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