REMOVAL OF SELENATE AND COBALT BY GOETHITE AND NANO PARTICLES ZERO-VALEANT IRON FROM AQUATIC SYSTEMS.

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ABSTRACT

The objective of this study is to investigate the effect of pH variation from 3 to 12 on adsorption of Se and Co on synthetic goethite and nano particles zero-valeant iron. Therefore, series of well-controlled laboratory experiments were carried out on the previous materials.

Maximum adsorption on goethite and nano particles zero-valeant iron (nZVI) for selenate occurred at the initial concentration of 10µg Se mL^{-1} where nearly 99.8% and 100% of the total applied selenate was adsorbed at a pH range of 3-4 for goethite and pH 3 for (nZVI). With increasing pH to more than 4, selenate adsorption tended to decline sharply to be 3.3% and 3.5% of the total Se added at pH 11 for goethite.

Selenate adsorption decreased almost linearly from almost 3119.7 µg g^{-1} goethite at pH 3.0 to 104.4 µg g^{-1} goethite at pH 11 and corresponding to 24855 µg g^{-1} (nZVI) at pH4.0 to 4160 µg g^{-1} nZVI at pH11.

The lower adsorption of selenate at high pH values is probably attributed to an increase in repulsion between the more negatively selenate species and negatively charged surface sites. Adsorption of Co showed an opposite behavior to selenate as its adsorbed quantities increased with increasing pH. The highest Co adsorption was noticed at pH 8.0 with a constant adsorption values with pH> 9.0. The highest Co adsorption was 2771 µg g^{-1} goethite and 20240 µg g^{-1} (nZVI), which corresponded to 89% and 81% of total added Co for goethite and (nZVI), respectively.

Keywords: Removal – Selenate - Cobalt - Goethite -Nano particles Zero-Valeant Iron - pH – Adsorption.

INTRODUCTION

Selenate and cobalt are known to be human carcinogenic elements. Knowledge of reaction kinetics at the solid liquid interface is extremely important in understanding the adsorption /desorption processes controlling solubility of selenate and cobalt in soil and water systems.

Contamination of soil with heavy metals possess serious risks for biota and human health.Cobalt and other heavy metals reach the soil from the air, phosphatic fertilizers and by the incorporation of sewage sludge. Adsorption and desorption strongly affect the bioavailability of heavy metals and therefore toxicity in soil (Renella et al., 2004).

Selenium is present in the environment in both inorganic and organic forms, and in the solid, liquid, and gas phases. Although it can exist in six oxidation states, yet the II^{+} state is not known to exist in nature (Lenz 2008). Heavy metal contamination exists in the aqueous waste streams of many industries, such as metal plating facilities, mining operations, nuclear power plant and tanneries. The soils surrounding many military bases are also contaminated and possess a risk of ground water and surface water
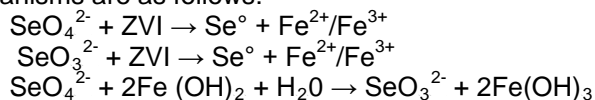
contamination due to the heavy metals. Some metals associated with these activities are cadmium, chromium, cobalt, lead and mercury. Heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders (Susan et al., 1999, Netzer and Hughes1984and Gomez-Lahoz et al., 1993).

Inorganic forms include SeO_3^{2-}, SeO_4^{2-}, Se^0, H_2Se, and metal selenides (MeSe). Organic forms include alkyl selenides such as dimethylselenide (DMSe) and dimethyldiselenide (DMDSe), selenoaminoacids, and seleno-proteins including selenocysteine and selenomethionine. Selenocystein and selenomethionine are analogues of the sulfur-containing proteins methionine and cystein. Both DMSe and DMDSe are volatile, sparingly soluble, and relatively non-toxic, although both are bioavailable (Shrestha et al., 2006).

Oxidized inorganic forms of selenium exist as highly soluble oxyanions in aqueous systems. Neither Se(VI) (selenate) nor Se(IV) (selenite) anions react with common cations such as calcium or magnesium, therefore they tend to remain in solution. Se(IV) species are more reactive than Se(VI) species and are easier to reduce to elemental selenium (Chapman, 2000). Nevertheless, elemental colloidal selenium particles are easily transported through aqueous systems (Haygarth, 1994).

Selenium speciation and therefore remediation is highly dependent on pH and redox potential (Eh). Selenate and selenite are often found outside their predicted thermodynamically stable zones (Microbial Technologies, 2005).

Nano particles zero-valent iron (nZVI) is used to reduce selenium oxyanions to elemental selenium. Ferrous cations can also reduce selenate to selenite and subsequently remove selenite by adsorption to iron hydroxides. In an aqueous environment, ZVI can be oxidized to ferric (Fe^{3+}) and ferrous (Fe^{2+}) ions. These ions react with hydroxyl ions present in water to form ferric and ferrous hydroxides. Selenate is reduced to selenite while ferrous iron is oxidized to ferric iron. Selenite is then adsorbed on the ferric and ferrous hydroxide surfaces and is removed from solution. Iron reduction removal mechanisms are as follows:

$$SeO_4^{2-} + ZVI \rightarrow Se° + Fe^{2+}/Fe^{3+}$$
$$SeO_3^{2-} + ZVI \rightarrow Se° + Fe^{2+}/Fe^{3+}$$
$$SeO_4^{2-} + 2Fe(OH)_2 + H_2O \rightarrow SeO_3^{2-} + 2Fe(OH)_3$$

Available data suggest that the best pH for selenite adsorption ranges between 4 and 6, producing 85-95 % removal of selenite, on the other hand, pHs greater than 7 show drastic decreases in adsorption. Selenate adsorption may be strongly affected by the presence of sulphate, bicarbonate, and aqueous silica species (Lovett 2008). The measured mobility of Co also depends on pH, and the ionic strength of the extracting (Naidu et al,1994).

Selenium also accumulates in soils through the use of fertilizers and irrigation water containing Se (Bisbjerg, 1972). In fertilizers, ammonium sulphate contains up to 36 mg kg-1, phosphate rocks up to 55 mg kg^{-1}, and single superphosphate up to 25 mg kg^{-1} Se (Swaine, 1962 and Bisbjerg, 1972).However, since single superphosphate has generally been replaced by

triple superphosphate, which contains up to 4 mg kg^{-1} Se, fertilizer inputs of Se to soils have fallen.

Cobalt, a natural element present in certain ores of the Earth's crust, is essential to life in trace amounts. It exists in the form of various salts. Pure cobalt is an odorless, steely-gray, shiny, hard metal. Everyone is exposed to low levels of cobalt in air, water and food. An average of 2 gdm^{-3} in drinking water has been estimated. Cobalt has both beneficial and harmful effects on health. Important natural sources of cobalt in the environment are soil, dust and sea water. Cobalt and its salts are used in nuclear medicine, enamels and semiconductors, grinding wheels, painting on glass and porcelain, hygrometers and electroplating; as a foam stabilizer in beer, in vitamin B12 manufacture, as a drier for lacquers, varnishes and paints, and as a catalyst for organic chemical reactions. The permissible limits of cobalt in the irrigation water and livestock watering are 0.05 and 1.0 mgdm^{-3}, respectively (Environmental Bureau of Investigation, Canadian Water Quality Guidelines).

A number of technologies have been developed over the years to remove toxic cobalt from water, including chemical precipitation, electro flotation, ion exchange ,reverse osmosis and adsorption on activated carbon (Poon, 1986).

The removal of heavy metal pollutants at high concentrations from water can be readily accomplished by chemical precipitation or electrochemical method. At low concentrations, removal of such pollutants is more effectively implemented by ion exchange or adsorption on solid sorbent such as activated carbon (Sigworth and Smith., 1972).

The IRN77 and SKN1 are effective for removal of Co (II) over the pH range 2–8. For comparison, revealed that precipitation curve at different pH values in the absence of resins. The precipitation of Co (II) was observed at pH 8.5.

At any pH, the removal of metal cation is much greater by adsorption than by hydroxide precipitation. The adsorption of the metal cation on adsorbent depends on the nature of the adsorbent surface and species distribution of the metal cation. For the IRN77 and SKN1 resins, the adsorption at pH above 8 shows a decreasing trend because of the formation of hydroxyl complexes of cobalt, Co(OH)$_2$ (Periasamy et al. 1991).

Precipitation may also occur, especially in the high pH range. Precipitation of hydroxides and carbonates that are formed above pH 8 may be misinterpreted as large distribution coefficients, especially if the batch-equilibration method is used. The pH in this investigation was limited to less than 7 so that no precipitation would occur.

The objective of this study is to investigate the effect of pH variation on adsorption of Se and Co on synthetic goethite and zerovalent iron.

MATERIALS AND METHODS

This experiment was carried out to evaluate the adsorption of selenate or cobalt under different pH values by synthetic goethite and zerovalent-iron with a back ground of 0.1M NaCl.

Synthesis of goethite:

Goethite was synthesized through a laboratory experiment according to the procedure of Schwartzman and Cornell (1991). Iron was measured in the last solution using the Atomic Absorption Spectroscopy of Perkin Elmer 3300. The concentration of goethite in the final suspension was 32 gL^{-1}. Small portion of goethite suspension was taken, freeze-dried and the identity of goethite was confirmed by powder X–ray diffraction analysis as presented in Fig. (1).
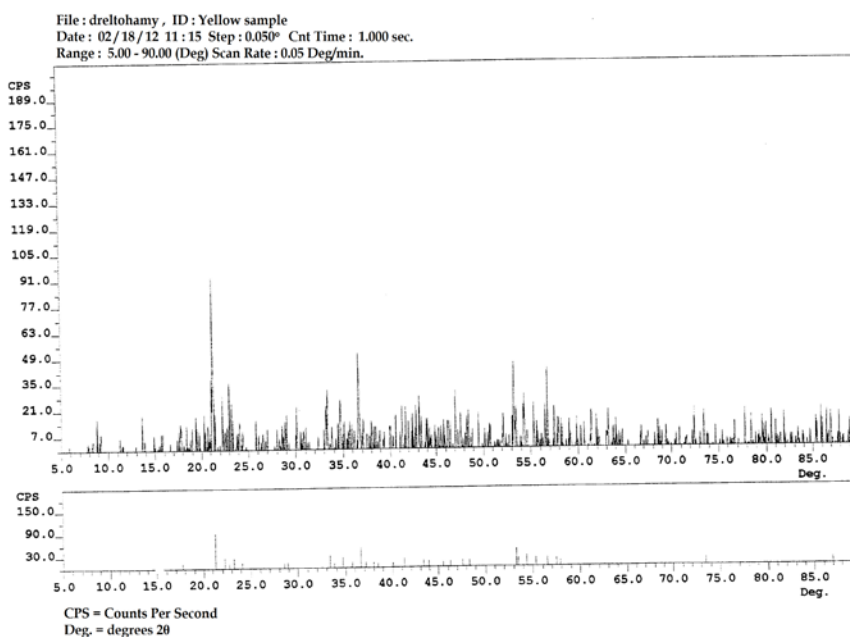

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CPS = Counts Per Second
Deg. = degrees 2θ

Fig (1): X–ray diffraction analysis of goethite

Selenate and cobalt adsorption envelopes (adsorption versus pH):
Goethite and Zero-Valent iron

To study the effect of pH on selenate and cobalt adsorption on goethite and zero-valent iron (iron metal, fine powder); an experiment was conducted as follows: to a 50-mL reaction vessel 2.5 mL goethite suspension or 0.01 g zero-valent iron, 2.5 mL of electrolyte back ground (0.1 M NaCl) and 250 µL of selenium in the form of selenate or cobalt (stock solution 1000µg mL^{-1}) were added and the volume was completed to be 25 mL with deionized water.

The concentration of Se or Co in the reaction vessels was 10µg mL^{-1}. The pH in the reaction vessels was adjusted to be between 3 to 12. Then the reaction vessels were shaken for 4 h, centrifuged at 3000 rpm for 30 min,

filtrated and the pH in the filtrates was measured. The filtrates were sealed in vials and kept for Se or Co determination. The adsorbed selenate or cobalt on goethite and zero valent iron was calculated as the difference between the initial and equilibrium Se or Co amounts in the solution, and plotted against the pH values.

RESULTS AND DISCUSSION

Adsorption envelopes (adsorption versus pH) Goethite:
Selenate

Adsorption of selenate on goethite was studied throughout pH range of 3-12.Goethite, an iron oxide commonly found in soil surface consists of a layer of Fe $^{3+}$ ions octahedrally coordinated with OH$^-$ ions and H_2O molecules. Specific adsorption of oxyanions varied with pH. Breaks in slopes of the curves represent anion adsorption over wide range of pH corresponded with pka values for the acid forms of each anion.

The adsorption envelopes for selenate at initial concentration of 10 µg Se mL-1 indicated adsorption of nearly 100% of total selenate added throughout the pH of 3-4 (Table,1). With increasing pH to more than 4, selenate adsorption declined sharply to be 3.3% of the total Se at pH11.

The best pH for selenite adsorption ranges between 4 and 6, producing 85-95 % removal of selenate; pHs greater than 7 show drastic decreases in adsorption (Lovett, 2008).

The adsorption envelopes exhibited broad adsorption maxima for selenate in the pH range of 3-4 which was followed by a gradual decline with increasing pH > 4.

Selenate adsorption decreased almost linearly from almost 3119 µg Se g^{-1} goethite, at pH 3 to 104.4 µg Se g^{-1} goethite at pH 11. The lower adsorption of selenate at high pH values is attributable to an increased repulsion between the more negatively selenate species and negatively charged surface sites (Raven et al., 1998). The species which introduces the least negative charge in the outer plane will be preferred. Loading also affects the formation of protonated bidentate species. At low pH the positive particle charge decreases upon adsorption of negatively charged ions like selenate.

At the highest initial concentration of selenate solution, a distinct adsorption maximum was observed at approximately pH3. The adsorption envelopes for selenate obtained in this study were almost similar to those observed by other workers e.g. (Manning et al., 1998, Raven et al., 1998, Sun and Doner, 1998 and Lovett, 2008).

Cobalt

In case of Co adsorption on goethite, results in Table (2) indicate that, adsorption of Co showed an opposite behavior to selenate as its adsorbed quantities increased with increasing pH. The highest Co adsorption was noticed at pH 8.0 with a constant adsorption values with pH >7.0. The adsorption process may be responsible for decreasing Co concentration in the equilibrium solution, particularly at pH < 7.0. However, at pH values > 7.0 both adsorption and precipitation may be hold the responsibility for

decreasing Co concentration in equilibrium solution and account for the retention of Co by goethite. These results confirmed the results of Al-Sewailem et al., (2000) who explained the effect of increasing pH on cadmium adsorption by two complimentary trends: an increasing proportion of $CdOH^+$ ions and a decreasing electrostatic potential of the goethite surface.
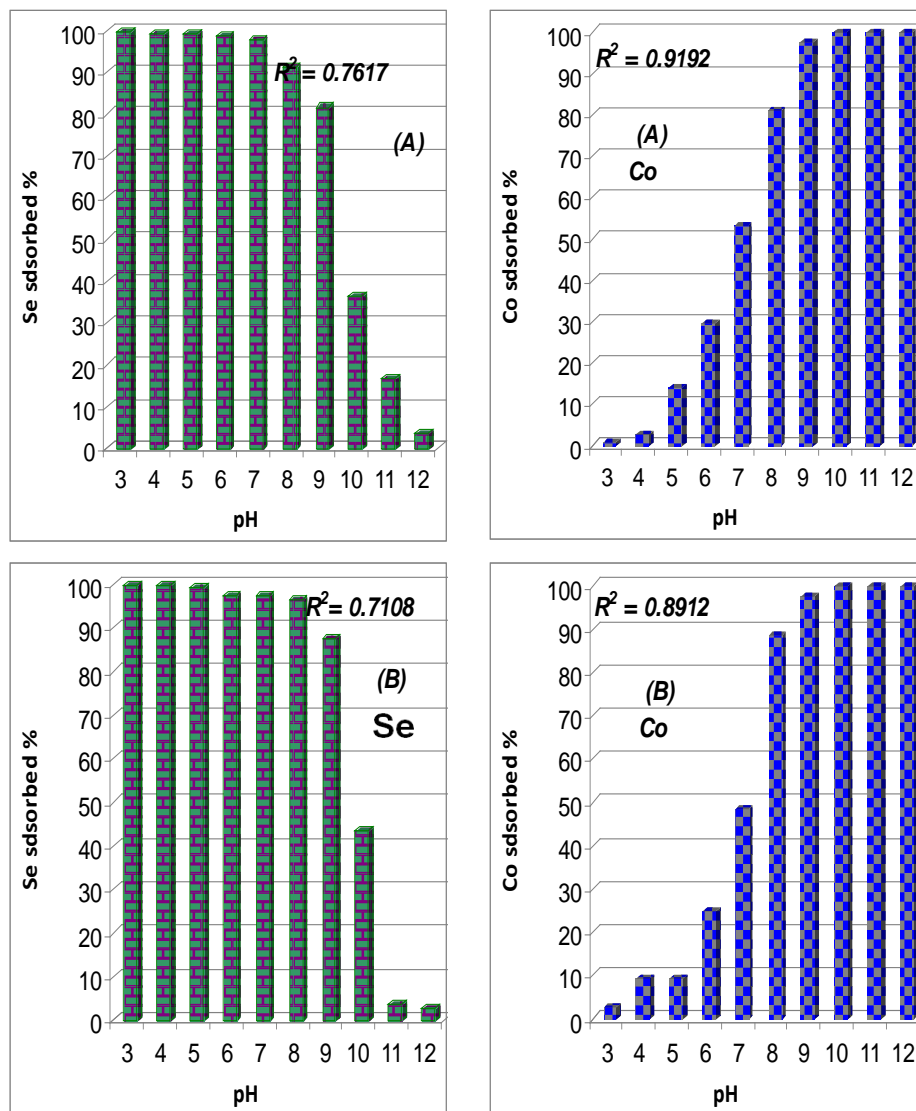

Fig.(2): Selenate and cobalt adsorption on (A) goethite and (B) nZVI as a function of pH (Se and Co 10 µg mL^{-1} , ionic strength, 0.1M NaCl.

As shown in Fig. (2), the statistical relationship between each of the removed selenium and cobalt by on one hand and the pH vales on the other one reveal that there were highly significant relations between each of the adsorbed Se and Co on of goethite and nZVI and the pH values where the values of (R) were 0.84 and 0.94 respectively, in case of goethite while in case of nZVI the corresponding the values of (R) were 0.87 and 0.96, respectively.

Table (1): Selenate adsorption on goethite as a function of pH (Se 10µg mL^{-1} , ionic strength, 0.1M NaCl 32g goethite L^{-1}).

Initial conc. of Se µg mL^{-1}	pH initial	C µg mL^{-1}	q µg g^{-1}	pH Final
10.00	3.00	0.017	3119.7	3.50
10.00	4.00	0.041	3112.2	3.90
10.00	5.00	0.076	3101.3	3.90
10.00	6.00	0.243	3049.1	4.00
10.00	7.00	0.277	3038.4	3.70
10.00	8.00	0.366	3011	8.70
10.00	9.00	1.261	2731	9.30
10.00	10.0	5.663	1355.3	9.60
10.00	11.0	9.666	104.4	11.0
10.00	12.0	9.758	75.6	11.5

C: Concentration of Se in equilibrium solution   q:Adsorbed Se

Table (2): Cobalt adsorption on goethite as a function of pH (Co 10µg mL^{-1} , ionic strength, 0.1M NaCl 32g goethite L^{-1}).

Initial conc. of Co µg mL-1	pH initial	C µg mL-1	q µg g-1	pH Final
10.00	3.00	9.735	82.8	3.50
10.00	4.00	9.047	297.8	3.90
10.00	5.00	8.090	297	3.90
10.00	6.00	7.507	779	4.00
10.00	7.00	5.125	1523.4	3.70
10.00	8.00	1.134	2771	8.70
10.00	9.00	0.216	3058	9.30
10.00	10.00	0.015	3120	9.60
10.00	11.00	0.007	3123	11.00
10.00	12.00	0.003	3124	11.50

C: Concentration of Co in equilibrium solution   q:Adsorbed Co

Nano particles Zero-Valeant iron (nZVI):
Selenate

The adsorption envelopes for selenate at the initial solution concentration equivalent to 10 µg Se mL^{-1} indicated adsorption of nearly 100% of total selenate added throughout the pH value of 3-4 (Table, 3). With increasing pH value more than 4, selenate adsorption declined sharply to reach at pH 12 be 16% of the maximum adsorption of Se. Similar results were reported by Abd El- Haleem (2001) in which adsorption envelopes of AsO_4^{3-} reveal that the highest AsO_4^{3-} adsorption occurred at low pH (<4.0).

The adsorption envelopes exhibited broad adsorption maxima for selenate in the pH range of 3-4 which was followed by a gradual decline with increasing pH to more the 4. Inflections or maxima in the adsorption envelopes of anions at pH values close to their pKa are well documented phenomenon (Stumm, 1992 and Abd El- Haleem 2001).

Selenate adsorption decreased almost linearly from almost 24960 µg Se g^{-1} nZVI, at pH 3 to 4160 µg Se g^{-1} nZVI at pH 11. The lower adsorption of selenate at high pH values is attributable to an increased repulsion between the more negatively selenate species and negatively charged surface sites (Raven et al., 1998). These results are confirmed with those of Griffin and Shimp (1978) in which maximum adsorption of selenate by kaolinite and montmorillonite, occurred at pH 5. Adsorption of selenate by aluminum and iron oxides has shown an adsorption maximum at pH 3-4 followed by a gradual decrease in adsorption with increasing pH (Hingston et al., 1971; Anderson et al., 1976)

Cobalt

In case of Co adsorption by nZVI, results in Table (4) indicate that adsorption of Co showed an opposite trend to arsenate as its quantities increased with increasing pH. The highest Co adsorption was noticed at pH 8.0 with a constant adsorption values with pH >7.0. The highest Co adsorption was 24973 µg Co g^{-1} nZVI which corresponded to 99 % of total added Co. The adsorption process may be responsible for decreasing Co concentration in equilibrium solution, particularly at pH < 7.0. However, at pH values > 7.0 both adsorption and precipitation may be responsible for decreasing Co concentration in equilibrium solution and account for the retention of Co by nZVI.

Table (3): Selenate adsorption on nano particles zero-valeant iron (nZVI) as a function of pH (Se 10 µg mL^{-1}, ionic strength, 0.1 M NaCl)

Initial conc. of Se µg mL^{-1}	pH initial	C µg mL^{-1}	q µg g^{-1}	pH Final
10.00	3.00	0.016	24960	3.50
10.00	4.00	0.058	24855	3.90
10.00	5.00	0.073	24818	3.90
10.00	6.00	0.103	24743	4.00
10.00	7.00	0.197	24508	3.70
10.00	8.00	0.586	22860	8.70
10.00	9.00	1.83	20415	9.30
10.00	10.00	6.38	9058	9.60
10.00	11.00	8.34	4160	11.00
10.00	12.00	9.65	875	11.50

C: Concentration of Co in equilibrium solution   q:Adsorbed Co

Table (4): Cobalt adsorption on nano particles zero-valeant iron (nZVI) as a function of pH (Co 10 µg mL^{-1} , ionic strength, 0.1 M NaCl)

Initial conc. of Co µg mL^{-1}	pH initial	C µg mL^{-1}	Q µg g^{-1}	pH Final
10.00	3.00	9.90	253	3.70
10.00	4.00	9.72	705	3.80
10.00	5.00	8.57	3575	3.50
10.00	6.00	7.01	7473	3.80
10.00	7.00	4.66	13345	4.00
10.00	8.00	1.90	20240	3.20
10.00	9.00	0.243	24393	4.20
10.00	10.00	0.011	24973	8.70
10.00	11.00	0.001	24998	9.12
10.00	12.00	0.001	24998	11.0

C: Concentration of Co in equilibrium solution   q:Adsorbed Co

CONCLUSIONS

Goethite and nano particles zero-valent iron were used as adsorbents for both Se and Co. The results proved that the sorption capacities of Goethite and nano particles zero-valent iron were pH dependent. These materials can be used for removing contaminants from waste water plants before dumping it into the canals and the work of this material filters.

The pH is an important factor affecting the sorption of selenium and Co by Goethite and nano particles zero-Valeant iron, and it can change the affinity of the sorption sites for heavy metals. Selenium and Cobalt adsorption can be achieved using an initial solution pH. There for adsorption capacity of materials using was different with change of pH selenium is more adsorped in acid medium while cobalt is more adsorbed in alkaline side.With a careful selection of the other conditions, especially the metal ion and slurry concentrations, to avoid masking of sorption by chemical precipitation.

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إزالة السلينات والكوبلت بواسطة الجيوثايت وبرادة الحديد المتناهية الصغر من الأنظمة المائية

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الهدف من هذه الدراسة هو دراسة تأثير التغير فى درجة الحموضة والقلوية الـpH من 3 إلى 12 على إدمصاص السلينيوم والكوبلت على الجيوثايت وبرادة الحديد المتناهية الصغر ولذلك تم إجراء العديد من التجارب المعملية المتحكم فيها على مواد الإدمصاص السابق ذكرها.

وجد أن أعلى إدمصاص للسلينيوم على الجيوثايت وبرادة الحديد المتناهية الصغر ، عند تركيز المحلول الذى يعادل 10 ميكروجرام سلينات لكل ملل ووجد أن نسبة الإدمصاص تقترب من 99.8 إلى 100% من الكمية المضافة من السلينات فى مدى الـ pH من 3.0 إلى 4.0 وذلك للجيوثايت وعند الـ pH 3.0 لبرادة الحديد المتناهية الصغر ، ومع زيادة الـ pH عن 4 فإن إدمصاص السلينات يقل بشدة حتى يصل إلى 3.3 % و 3.5 % من الكمية الكلية المضافة من السلينات عند الـ pH 11.0 وذلك للجيوثايت. وعند قيم الـ pH المنخفضة يقل معظم إدمصاص السلينات خطياً من 3119.7 ميكروجرام لكل جرام جيوثايت عند الـ pH 3.0 إلى 104.4 ميكروجرام لكل جرام عند الـ pH 11.0 و 24855.0 ميكروجرام لكل جرام من برادة الحديد المتناهية الصغر عند الـ pH 4.0 إلى 4160.0 ميكروجرام لكل جرام من برادة الحديد المتناهية الصغر عند pH 11.0.

انخفاض إدمصاص السيلينات يرجع إلى زيادة الشحنة السالبة على الأسطح. وأخذ الكوبلت الاتجاه المعاكس فى سلوكه بالنسبة للسلينات حيث تزايدت الكمية المدمصة بزيادة رقم الـ pH ولوحظت أن أعلى قيمة للإدمصاص عند الـ pH 8.0 وتكون قيم الإدمصاص ثابتة عند الـ pH أعلى من 9 وكانت أعلى قيمة لإدمصاص الكوبلت هى 2771.0 ميكروجرام لكل جرام جيوثايت و 20240.0ميكروجرام لكل جرام من برادة الحديد المتناهية الصغر والتى تعادل 89% و 81 % من الكمية الكلية المضافة من الكوبلت على كلاً من الجيوثايت وبرادة الحديد المتناهية الصغر على التوالى.

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