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## Lead Removal from Contaminated Water Using Seaweed-Iron Oxide Nanoparticles Composite

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### ABSTRACT

Water scarcity is one of the most global concerns in the last decades, which can be solved by reusing wastewater. Lead, as a potentially toxic element, is one of the most dangerous potentially toxic elements that have to be eliminated from contaminated water resources. Bio-sorbents, like macroalgae (seaweeds) have been recently investigated for removing toxic elements from water. It is hypothesized that mixing seaweeds with nanoparticles (e.g. iron oxide) might improve removal efficiency of  $Pb^{2+}$  ions. In this study seaweeds, iron oxide nanoparticles and their composite were used for lead removal from contaminated water with up to  $50.0 \text{ mg L}^{-1}$ . The results showed that all sorbents under investigation were successful for  $Pb^{2+}$  sorption, and the engineered composite was superior with removal efficiency of 91%. In addition, the engineered composite showed an ease of separation following sorption process using a magnet. Freundlich isotherm model was the best for describing the adsorption of  $Pb^{2+}$  onto the sorbents material under this investigation. This study is a step on the way to improve the separation of bio sorbents from aqueous solutions after the removal of contaminants.

**Keywords:** Lead, nanoparticles, seaweeds.

### INTRODUCTION

Removal of potential toxic elements (PTEs) is a challenging issue, which is highly required to prevent/reduce their toxic effects on human body and disturbance of ecological balance (Jaishankar *et al.*, 2014). Recently, nanotechnology is being exploited with most of the conventional techniques for the removal of PTEs from water system to improve the removal efficiency (Butt, 2020). Nanotechnology represented one of the most interesting fields at emerging and established technologies, that's for production of new substances can improves many industries.

The high functionality of nano substances (the large surface area in particular) can provide promising sorbent materials for large-scale application (Chowdhury and Yanful, 2011). In addition, the heterogeneity of its physicochemical characteristics support its reactivity with wide range of contaminants in wastewater treatment (Gupta *et al.*, 2011 and Parvin *et al.*, 2019). On the other hand, sorbents with nano-sized scale can act as adsorbents, catalysts and polymeric relative to the conventional treatment techniques (i.e., ion exchange, reverse osmosis and membrane technology).

Biomass and biomaterials are major chunk of waste in most countries. They have a great potential to be used for various energy generation, material production, and waste treatment technologies (Jamshaid *et al.*, 2017). Biomaterials have a high potential for eco- friendly wastewater treatment techniques like adsorption. Macroalgae, (or seaweed), are a group of fast-growing aquatic organisms including about 9000 species. They are commonly classified into three groups according to thallus color, which correspond to the Rhodophyta (red algae), Chlorophyta (green algae) and

eterokontophyta phylum, class Phaeophyceae (brown algae) as reported by Ortiz-Calderon *et al.*, (2017). Alginate polymers in cell wall composition of algae are the primary responsible for potential toxic elements sorption in brown algae, and their capacity to bind toxic elements directly depends on the number of binding sites on this polymer and the affinity between sorbent and sorbate. This high affinity for metals can be explained by the functional groups such as carboxyl, hydroxyl, sulfate, phosphate, and amine groups, that play a significant role in the metal binding.

Iron oxides are abundantly available minerals present in the earth's crust. There are about 16 iron oxides and hydroxides, whereas magnetite ( $Fe_3O_4$ ), goethite ( $\alpha FeOOH$ ), maghemite ( $\gamma Fe_2O_3$ ), and hematite ( $\alpha Fe_2O_3$ ) are widely used in remediation processes. The high reactivity of iron oxide nanoparticles including their large surface area, pore size per unit of mass, and crystalline structure support their potential utilization in wastewater remediation. (Macera *et al.*, 2020). Furthermore, iron oxides and their derivatives are distinguished by their surface modification ability, magnetic properties that aid magnetic separation, reusability, and relatively low production cost. In addition, due to its variable oxidation states, it can be bonding with other elements Jian *et al.*, (2018).

Sharma *et al.*, (2018) reported that iron oxide is more effective in nanoparticle size than that in microparticle size for removal of PTEs like Pb, Cd, Cu, Cr, Co, Hg, and As from water. Ghafoor and Ata, (2017) also reported that using iron oxide nanoparticles is effective in potential toxic elements removal, as well as magnetic separation- after adsorption of potentially toxic elements- affected of external magnetic force.

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This study aims to investigate lead removal using brown seaweed and effect of mixing seaweed with iron oxide nanoparticle for improve removal efficiency and the ability of using this composite for separation of sorbent material after equilibrium.

**MATERIALS AND METHODS**

**Materials**

Seaweeds were imported. Lead nitrate Pb(NO<sub>3</sub>)<sub>2</sub> (AR) (assay 99.5%, MW: 331.21), where the reagents that used as a source for Pb<sup>2+</sup> ions in patch sorption experiment. Also, FeSO<sub>4</sub>.6H<sub>2</sub>O and FeCl<sub>3</sub>.6H<sub>2</sub>O were used for synthesis the engineered sorbents experiments were AR grade. NaOH used as technical grade.

**Preparation of Iron oxide nano particles (IONP)**

Preparation of IONP was conducted according to Khandanlou *et al.*, (2013) with a slight modification. Ferric chloride (1M) was added to 0.5M of Ferrous sulfate and mixed well. Then 5M of NaOH was added dropwise with stirring. From first drop of NaOH, a black precipitate was formed and dissolve in the mixture, continuous adding of NaOH till pH 11 resulting a suspended black fine powder. After 20 minutes, black precipitates were separated with filtering from solution, and then washed 3 times with distilled water and one time with ethanol. Then dried at 50°C for 12 hours.

**Preparation of Seaweed-Iron oxide nanoparticles composition (S-I):**

Seaweed (0.5 g) was mixed with 100 mL contained 0.1 g of IONP in 50% ethanol, and then stirred for 3 hours, separated and dried at 50°C overnight. This composite named as S-I

**Characterization of sorbent materials**

The abovementioned synthesized materials (i.e: MCC, SPS, IONP) was characterized by FT-IR spectroscopy using an FTIR - 8201 PC, Shimadzu.

**Effect of the initial metal ion concentration on sorption / removal of lead**

A 0.05 g portion of each of the studied sorbent materials (SW, IONP and S-I) equilibrated with 50 mL of each abovementioned PTEs concentrations. Then agitated under; a constant equilibrium time (120 min), at constant temperature (298 K) and at constant pH (7.0); centrifuged at 7000 rpm, And then, the supernatant were used for determining the considered stuided PTEs ions. The centrifuge step were conducted for SW and IONP sorbent. On the other hand, separation of S-I composite was conducted using magnet. This step was for testing if that separation of seaweed with iron oxide nanoparticles can be available without centrifuge.

**a.Measurements:**

The concentrations of lead ions were measured using a buck science, Type VGP 210 atomic absorption spectrophotometer (AAS). The considered pH values of the solutions were adjusted using a dilute solution of either NaOH or HCl of 0.01M.

**Calculation**

In all experimental runs, the adsorbed amounts calculated as follows:

**Removal percentage**

$$(R\%) = \frac{(C_0 - C_e)}{C_e} * 100 \dots \dots \dots (1)$$

$$q_e = (C_0 - C_e) \frac{V}{W} \dots \dots \dots (2)$$

**Where** q<sub>e</sub> is the amount of element sorbed by a unit mass of an adsorbent (mg g<sup>-1</sup>), W is the weight (g) of the adsorbent, V is the volume of the equilibrium solution, and C<sub>0</sub> and C<sub>e</sub> are the concentrations (mg L<sup>-1</sup>) of each ion before and after adsorption, respectively.

**• Theoretical foundation**

The adsorption equilibrium is usually described by an isotherm equation whose parameters express the surface properties and affinity of the adsorbent, at a preset temperature and pH. The following two widely used isotherms i.e. Langmuir and Freundlich, as summarized by Al-Ghouthi and Da'ana (2020) were applied:

**Langmuir equation:**

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \text{ (the non-linear form)} \quad \frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \text{ (the linear form)}$$

**Where** q<sub>e</sub> is the amount of metal ion sorbed onto a unit mass of a clay minerals (mg g<sup>-1</sup>), K<sub>L</sub> and q<sub>m</sub> are Langmuir constants representing the equilibrium constant for the adsorbate-adsorbent equilibrium and the monolayer sorption pattern, respectively.

**Freundlich model:**

The Freundlich model is an empirical equation and can be applied for non-ideal sorption on heterogeneous surfaces and multilayer sorption . It indicates that the ratio of the amount of solute adsorbed onto a given mass of a sorbent to the concentration of the solute in the solution is not constant at different concentrations. The Freundlich equation may be written as:

$$q_e = K_f C_e^{1/n} \text{ (the non - linear form)} \quad \log q_e = \log k_f + 1/n \log C_e \text{ (the linear form)}$$

**Where** q<sub>e</sub> is the amount of solute adsorbed per unit mass of the adsorbent (mg g<sup>-1</sup>) C<sub>e</sub> is the equilibrium concentration of the solute in the bulk solution (mg L<sup>-1</sup>), k<sub>f</sub> constant indicating the relative adsorption capacity of the adsorbent, n is a constant indicating the intensity of the adsorption capacity of the adsorbent (mg g<sup>-1</sup>) and 1/n a constant indicating the intensity of the adsorption.

**Statistical analysis**

Data of the current study were statistically analyzed using Statistical Software Program (PC-Mstat). Means of treatments were compared with the Least Significant Deference (L.S.D) at the 0.05 level.

**RESULTS AND DISCUSSION**

**Characterization of sorbent materials**

The FT-IR spectrums of the SW, IONP and S-I are given in Figure (1). FT-IR spectrum of SW has identified the presence of several surface functionalities such as the peak at wavenumber 467 cm<sup>-1</sup> for hydroxyl group and 540 and 618 cm<sup>-1</sup> for organic chlorides. While the peak of 1584 cm<sup>-1</sup> indicated nitro groups presence. The peak of 3419 cm<sup>-1</sup> related to the vibration of phenolic groups.

The FT-IR spectrum of IONP materials has identified the presence of some surface functionalities such as the peak at wavenumber 565 cm<sup>-1</sup> for metal chloride (may due to the preparation method). In addition, aromatic methyl groups observed at 792 cm<sup>-1</sup>. While the peak of 3144 and 3384 cm<sup>-1</sup> related to the vibration of phenols and carboxylic groups. Similar results were reported by Zaman *et al.*, (2019) and Paulose *et al.*, (2016).

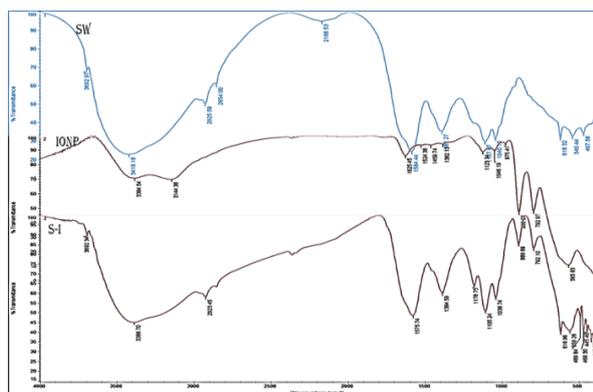
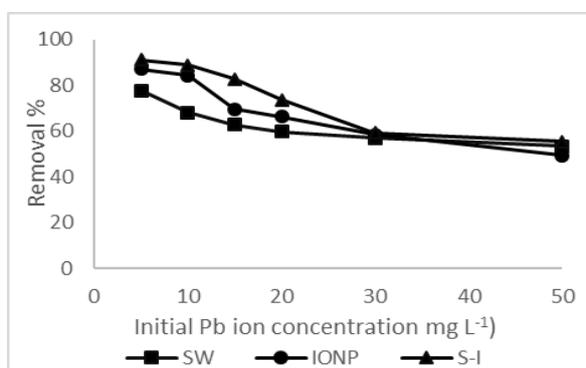


Figure 1. FT-IR spectrums for Seaweed (SW), Iron oxide nanoparticles (IONP) and seaweed-iron oxide nanoparticles composite (S-I)

The FT-IR spectrum of the S-I has identified that the surface functional groups are interfered. The main surface functional groups from two materials appeared in the



spectrum of S-I, and the spectrum showed that some functional groups be more concentrated, especially 1038 and 1105  $\text{cm}^{-1}$  which related to esters and alcohols. Similar of this result appeared for 1575  $\text{cm}^{-1}$ , which referred to amino acids. These results can have indicated that the S-I will have a mixture of properties of its source (SW and IONP). V

**Removal of lead using different sorbents**

Removal experiments were performed with variable initial concentrations of metal ions from 0 to 50  $\text{mg L}^{-1}$  using 2  $\text{g L}^{-1}$  of SW, IONP or S-I. Results illustrated in Fig. 2 and detailed in Table 1 showed that removal of  $\text{Pb}^{2+}$  decreased with the increase of the initial concentration. Highest removal percentages were 77.87, 87.07 and 91.13% for SW, IONP and S-I, respectively, that for the initial concentration of 5.0  $\text{mg L}^{-1}$ . The lowest removal percentages were 53.47, 49.22 and 55.35% for SW, IONP and S-I, respectively, that for 50  $\text{mg Pb L}^{-1}$  initial concentration.

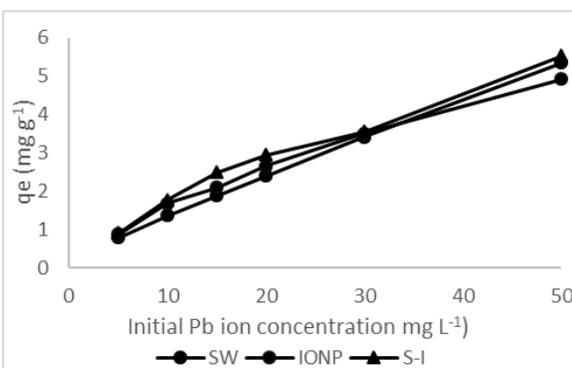


Figure 2. Effect of initial lead ion concentration on removal percentage(left) and sorption (right).

Table 1. Effect of the initial metal ion concentration ( $\text{mg L}^{-1}$ ) on its removal (%) sorption ( $\text{mg g}^{-1}$ ) by the different studied sorbents.

| PTEs ions                       | Sorbent (S) | Initial metal ion concentration ( $\text{mg L}^{-1}$ ) (C) |       |          |       |           |       | Mean  |
|---------------------------------|-------------|--|-------|----------|-------|-----------|-------|-------|
|                                 |             | 5  | 10    | 15       | 20    | 30        | 50    |       |
| Removal (%)                     | SW          | 77.87  | 68.13 | 62.69    | 59.80 | 56.97     | 53.47 | 63.15 |
|                                 | IONP        | 87.07  | 84.30 | 69.62    | 66.33 | 58.36     | 49.22 | 69.15 |
|                                 | S-I         | 91.13  | 88.80 | 82.80    | 73.70 | 59.16     | 55.35 | 75.16 |
|                                 | Mean        | 85.36  | 80.41 | 71.70    | 66.61 | 58.16     | 52.68 | 69.15 |
|                                 | LSD         | S: 0.657   |       | D: 0.929 |       | SD: 1.609 |       |       |
| Sorption ( $\text{mg g}^{-1}$ ) | SW          | 0.78   | 1.36  | 1.88     | 2.39  | 3.42      | 5.35  | 2.53  |
|                                 | IONP        | 0.87   | 1.69  | 2.09     | 2.65  | 3.50      | 4.92  | 2.62  |
|                                 | S-I         | 0.91   | 1.78  | 2.48     | 2.95  | 3.55      | 5.53  | 2.87  |
|                                 | Mean        | 0.85   | 1.61  | 2.15     | 2.66  | 3.49      | 5.27  | 2.67  |
|                                 | LSD         | S: 1.548   |       | D: 2.189 |       | SD: 3.791 |       |       |

SW: Sea Weed, IONP: Iron Oxide Nanoparticles and S-I: Seaweed-iron oxide nanoparticles composite

These results agreed with that found for PTEs removal by different bio-sorbent materials and related to the nature of each studied sorbent especially the surface nature. Functional groups identified by FT-IR analysis for S-I were sharper than both of that obtained for SW and IONP. Sorption results showed that sorbed amounts of  $\text{Pb}^{2+}$  using investigated sorbents increased with increasing initial lead ion concentration. The sorbed amounts ranged from 0.78 to 5.35, 0.87 to 4.92 and 0.91 to 5.53  $\text{mg g}^{-1}$  for SW, IONP and S-I, respectively. These results confirmed that obtained for removal of  $\text{Pb}^{2+}$  using studied materials. Near findings reported by Lee and Park, (2012) and Cardoso *et al.*, (2017) for seaweeds sorption of lead. Moreover, Das and Rebecca (2018) reported similar data for removal of lead using IONP.

This data, in general, showed that seaweed sorption capacity was the lowest and improved using IONP, which represented as a harvesting tool to separate the composite from the solution using magnet. Iron oxide nanoparticles, depend on these results, played two rules: improved the sorption (removal) and separation tool.

**Modelling the sorption of Pb, Cd, and Cu as related to their concentrations in the equilibrium solutions:**

Illustration of linear form for both of Langmuir and Freundlich isotherm models are showed in figure 3 and Table 2. The results showed that the most fitting Freundlich isotherm model is sorption of lead on all investigated sorbents. The results showed also high regression for Langmuir isotherm model, but not higher than that for

Freundlich isotherm model. Calculated constants of linear Langmuir isotherm model  $q_m$  which expressed the maximum adsorption capacity ( $\text{mg g}^{-1}$ ), which was highest for adsorption of lead ions on SW, ( $8.53 \text{ mg g}^{-1}$ ), and other values for this constant was ranged from 5.73 to  $5.88 \text{ mg g}^{-1}$  as reported by Wang *et al.*, (2019). Another Langmuir linear fitting isotherm model was ( $K_L$ ), which related to adsorption capacity ( $\text{mg g}^{-1}$ ) that can be correlated with the

variation of the suitable area and porosity of the adsorbent which implies that large surface area and pore volume will result in higher adsorption capacity (Ayawei *et al.*, 2017).

These results can be pointed to, as reported by Al-Ghouti and Da'na (2020), that constants of fitting linear equation for Freundlich isotherm model ( $1/n$ ) which indicated by the slope of linear fitting is generally between 0 to 1, which indicated that adsorption isotherm is favorable.

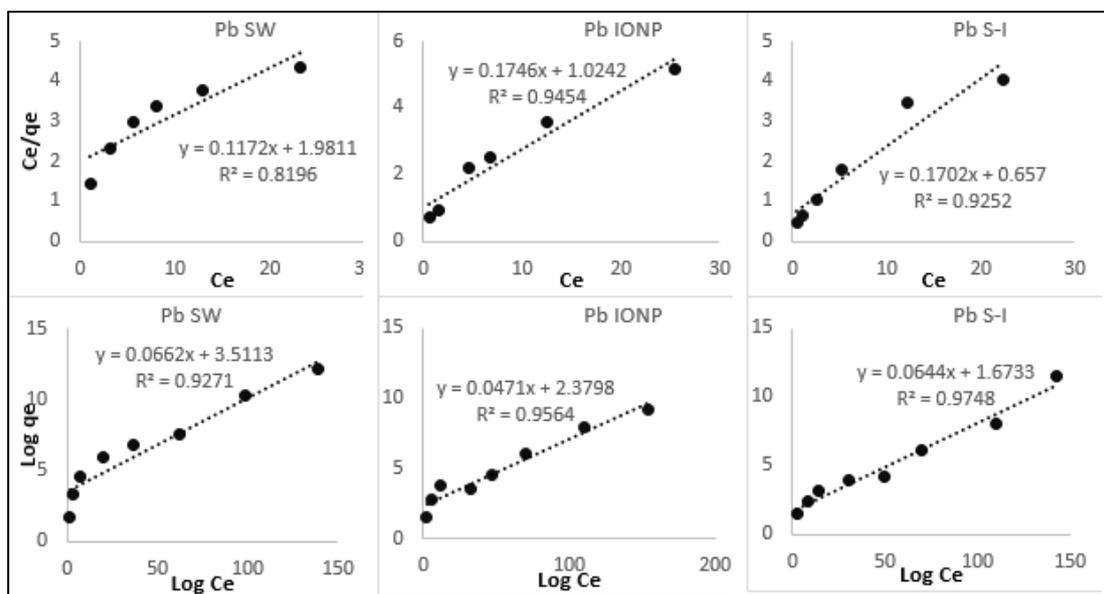


Figure 3. Plotting of linear form for both of Langmuir and Freundlich isotherm models.

Table 2. Langmuir and Freundlich isotherm linear regression equation and parameters of sorption of PTEs on sorbents

| Sorbents (S)      | Linear equation                     | Regression R <sup>2</sup> | Parameters                                 |                                 |
|-------------------|-------------------------------------|---------------------------|--|---------------------------------|
| <b>Langmuir</b>   |                                     |                           |  |                                 |
|                   |                                     |                           | $q_m$<br>( $\text{mg } 100\text{g}^{-1}$ ) | $K_L$<br>( $\text{L mg}^{-1}$ ) |
| SW                | $C_e/q_e = 0.117 C_e + 1.981$       | 0.820                     | 8.53                                       | 0.06                            |
| IONP              | $C_e/q_e = 0.175 C_e + 1.024$       | 0.945                     | 5.73                                       | 0.17                            |
| S-I               | $C_e/q_e = 0.170 C_e + 0.567$       | 0.925                     | 5.88                                       | 0.26                            |
| <b>Freundlich</b> |                                     |                           |  |                                 |
|                   |                                     |                           | $K_f$<br>( $\text{mg } 100\text{g}^{-1}$ ) | $N$<br>( $\text{g/L}$ )         |
| SW                | $\log q_e = 0.630 \log C_e - 1.677$ | 0.991                     | 0.68                                       | 1.59                            |
| IONP              | $\log q_e = 0.442 \log C_e + 0.063$ | 0.974                     | 1.16                                       | 2.26                            |
| S-I               | $\log q_e = 0.129 \log C_e - 0.181$ | 0.951                     | 0.66                                       | 7.71                            |

SW: Sea Weed, IONP: Iron Oxide Nanoparticles and S-I: Seaweed-iron oxide nanoparticles composite

### CONCLUSION

Usage of seaweed for bio sorption of potential toxic elements (like lead) had a good concern for years. This way of treatment face a problem of separation after sorption. This study try to introduce iron oxide nanoparticle as a solution for this problem. Nano iron is paramagnetic beside its capacity for metal ion sorption. Seaweed and IONP, individually, were effective to remove (adsorb) lead ions from its aqueous solution. Separation of Seaweed-iron oxide nanoparticles composite was effective for remove (adsorb) and separate of removed lead from solution using magnet. Adsorption of lead was agreed with Freundlich isotherm model for all sorbent materials. This study –in future- may

be supported by more work on binding bio sorbents with paramagnetic nano materials

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## إمكانية إزالة الرصاص من المياه الملوثة باستخدام مركب من جزيئات أكسيد الحديد النانوية والطحالب البحرية

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الندرة المائية هي واحدة من أكثر الاهتمامات العالمية في العقود الماضية والتي يمكن حلها عن طريق إعادة استخدام مياه الصرف الصحي. الرصاص كعنصر سام محتمل هو أحد المشاكل التي يجب إزالتها من المياه المعالجة. المواد الماصة الحيوية، مثل الطحالب الكبيرة (الطحالب البحرية) استخدمت مؤخرًا لإزالة العناصر السامة من الماء. علاوة على ذلك، يمكن أن يؤدي خلط الطحالب البحرية مع جزيئات أكسيد الحديد النانوية إلى تحسين القدرة على الإزالة. في هذه الدراسة، تم استخدام الطحالب البحرية وجسيمات أكسيد الحديد النانوية ومركب مكون منهما معًا لإزالة الرصاص من المياه الملوثة حتى ٥٠ ملليجرام/ لتر، ويعتمد الفصل بعد التوازن باستخدام المغناطيس على خصائص الحديد. أظهرت النتائج أن جميع المواد الماصة قيد الدراسة كانت ناجحة لإبصار الرصاص، وتحسنت الإزالة باستخدام الجمع بين المادتين معًا في مركب واحد بنسبة تصل إلى ٩١٪. ويمكن استخدام الفصل باستخدام المغناطيس لحصاد المواد الماصة المركبة بعد التوازن. كان نموذج Freundlich هو الأفضل لوصف إدمصاص الرصاص على المواد المدمصة قيد البحث. هذه الدراسة هي خطوة على طريق تحسين فصل المواد الماصة الحيوية عن المحاليل المائية بعد إزالة الملوثات.