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Development of Simple and Cost Effective Method for Arsenic(III) Removal

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Abstract: In the present study, eight locally available low cost adsorbents (raw and heat treated) have been screening for its capability to remove As(III). The shale sedimentary rock (SSR) was selected as a good adsorbent based on high As(III) removal. The activation of adsorbent was carried out by heat (250 - 550°C) and acid treatment (0.1 - 1 M H_2SO_4) in order to chose the best treatment method for As(III) removal. Sorption experiments were conducted to examine the effect of initial As(III) concentration, contact time and initial pH on As(III) removal by SSR. Results show that, only by heating the adsorbent at 500°C for 1h, 0.2 g of SSR adsorbent capable to reduce the residual As(III) concentration below than maximum concentration limit (10µg/L) for initial concentration from 100 to 700 µg/L and optimum pH ranges between 3 to 9 after 24 h of contact time. The adsorption data fits well with Langmuir isotherm and yielded Langmuir monolayer capacity of 0.987 mg As(III)/g of SSR at pH 7. The SSR adsorbent has been successfully used for the removal of arsenic from seven real groundwater samples containing arsenic in the range of 20.2 to 697.34 µg/L with 86.73 to 97.86% removal efficiency.

Key words: As (III) removal · Shale sedimentary rock · Heat treatment · Langmuir isotherm · Adsorption

INTRODUCTION

Arsenic exists in natural water predominantly as inorganic arsenite (As(III)) and arsenate (As(V)) [1]. Arsenite is about 60 times more poisonous than arsenate and 70 times more toxic than the methylated species. Manifestation of higher doses of inorganic arsenic compounds in the human body leads to specific disease called arsenocosis. Arsenic contamination is of mostly geogenic origin and is known for its high toxicity and its ability to induce skin cancer on long-term ingestion [2]. Most treatment technologies, such as adsorption and precipitation, are effective in removing As(V) from waters because of positive charge on the surface of the adsorbents. On the other hand, under mildly reducing conditions such as in ground waters, As(III) is the thermodynamically stable form, which is presented as non-ionic form of arsenious acid (H_3AsO_3) at neutral pH [3]. Thus, As(III) is more difficult to be removed from water by means of adsorption and coprecipitation due to the lack of electrostatic attraction [4].

In the present work, 8 low cost adsorbents have been screening for the purpose of removing arsenic from water. The potential adsorbent such as shale sedimentary rock (SSR) was then activated by heat and acid treatment. The adsorption parameters, namely, initial As(III) concentration, shaking time and initial pH have been optimized with respect to arsenic spiked deionized water for As(III). Attempts have been made to ascertain appropriate adsorption isotherms. Finally, the SSR has been used for the removal of arsenic from seven real life arsenic contaminated groundwater samples. The overall objective of the present study was to ascertain the capability of the SSR for removing As(III) from water in a single step operation and without any pretreatment.

MATERIALS AND METHODS

Reagents and Glassware: All the reagents used, including acids, were of Analytical grade. HCl and HNO_3 solutions were prepared from concentrated stock solutions and NaOH solutions were prepared from pellets. Stock arsenic

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solutions (1000 mg/l) were prepared for As (III) from sodium arsenite (NaAsO$_2$), using deionized water with water conductivity of 18.2 MÙ. All glassware and sample bottles used were soaked with 10% (v/v) HNO$_3$ and rinsed with deionized water three times. The procedure guarantees decontamination of glassware for working with As.

Preparation and Activation of Adsorbent Materials: A total of 8 different low cost materials were initially screening for their capability to remove As(III) from water. The materials comprise of natural occurring rocks (shale sedimentary rock (SSR) and river sand (RS)), ceramic factory by-products (porcelain glaze (PG), full body porcelain black (FBPB), ceramic glaze (CG), full body porcelain white (FBPW) and clay (C)) and common commercially available water purifier, activated carbon (AC). Some materials (clay and activated carbon) have been previously used for arsenic removal but others were of unknown potential. Natural occurring rocks were collected from residential college at Universiti Teknologi Malaysia, Johor Bharu, Malaysia and ceramic factory by-products were obtained from one of the ceramic manufacturing premises at Johor Bharu, Malaysia. The adsorbent materials obtained were washed with tap water in order to remove unwanted particle and dry in oven for 24 h at 100 °C. After drying process, the materials were crushed using mortar and pestle before being sieved through a 106 μm stainless steel siever. The material size between 106 to 75 μm was collected for adsorption study. In order to study the effect of heating on As(III) removal, all adsorbent material were heated at 180 °C for 1h. After heat treatment, weight loss was determined. For the screening purposes, the removal capacity of raw (RA) and heat treated (HTA) adsorbent were compared and the adsorbent with highest removal of As(III) was chosen for further study.

Heat and acid treatment methods were used to activate the SSR adsorbent in order to compare and select the best activation method for As (III) removal. For heat treatment method,1g of each SSR adsorbent was placed in porcelain dishes and calcined at various temperatures (250, 300, 400, 500, 550 and 600 °C) for 1 h, respectively. In order to find the optimum time for calcinations, the temperature that give high As(III) were chosen and 1 g of each SSR adsorbents were calcined at various time interval (10, 30, 60, 90, 120 min) respectively. At the end of heat treatment process, weight loss of each samples were determined. In the second method, SSR adsorbent was activated through acid treatment. For this purpose, 1 g of each SSR adsorbent were suspended in 20 mL of 0.2-1.0 M HCl solutions and the suspensions were shaken at 200 rpm for 3 h to allow a good contact between acid and adsorbent. The treated SSR adsorbent was separated from the acid solution by filtration and the adsorbent was washed three times with distilled water to remove the residual acid and soluble compounds. It was then dried at 100 °C for 24 h and used for adsorption study.

Batch Adsorption Study: Batch adsorption experiment were performed to examine the effect of initial As(III) concentration (100 -1000 μg /L), contact time (0 - 48h) and pH (pH 2 - 10) on adsorption performance. Adjustment of pH to 7 was done before addition of adsorbent by adding 0.1 M HCl solution. Unless otherwise stated, all experiments were carried out at room temperature 30°C using 0.2 g of adsorbent dose with particle size fraction between 75 to 106 μm. After introducing 100 mL of 500 μg/L As(III) bearing solution, the fiasks (250 mL) were placed on a orbital shaker and shaken for 24 h at the rate of 200 rpm (SW-20C, JULABO, Germany). After the required time, the suspension was filtered through a filter paper (Whatman No. 1) prior to analysis. All samples were analyzed using inductively coupled plasma-mass spectrometry (ICP-MS) (Perkin Elmer DRC-e). An average and standard deviation of three readings for each sample was reported. Samples were filtered through a filter paper (Whatman No. 1) before diluted to a maximum of 60 μg/L total arsenic and acidified to 1% (v/v) HNO$_3$. All data represents the mean of triplicates. The amount of As(III) adsorbed onto the adsorbents was calculated using the following equation:

$$q = V(C_i - C_t)/M \tag{1}$$

where q is the mass of As(III) adsorbed; V is volume of solution: C_i and C_t are initial As(III) concentration at time t respectively; and M is mass of adsorbent. All standard deviation were calculated using Microsoft Excel 2007.

Application of the Adsorbents to Real Life Groundwater Samples: The adsorbent studied was used for the removal of arsenic in six natural groundwater samples collected from Kandal Province and Kg. Cham, two of the most severe arsenic affected districts of Cambodia. The adsorbent (0.1 g) with particle size of 75 - 106 μm was mixed with 50 mL groundwater sample and shaken at 200 rpm, 30 °C for 24 h.

RESULT AND DISCUSSION

Screening of Low Cost Materials for As(III) Removal:
From the screening tests, it was found that all materials were able to reduce As(III) concentration at different extent (Figure 1). The percentage removal range from as low as 0.4 to 60% for raw adsorbent and a slight increment was observed for the heated adsorbent (1.8 to 70%). The screening tests initially indicated that SSR was the best performing adsorbents (raw and heated) since it could remove 60 to 70% of 500 µg/L As(III) in the solution. From the experiment, it was found that, mild heating introduced could increase the removal efficiency of the adsorbent. For SSR adsorbent, the removal was efficiency was increase from 61.25 to 72.59% upon heating. This thing happen might be due to the contaminants present in natural occurring rocks. The present result (heated adsorbent > raw adsorbent) could well be related to the blockage of the rocks surface by the contaminants, thereby preventing access of As(III) to the adsorption site. Contaminants such as organic compound that burn along the heating process was evidence by the weigh loss of the adsorbent. After heating, the blockage by organic compound was reduced and this site now available for As(III) adsorption [5]. Studies by Altundogan *et al.* 2002 [6], revealed that thermal treatment of hydrous metal oxide (in this case iron oxide) strongly influenced the adsorption of metal ion. Therefore SSR was chosen for further adsorption study.

Effect of Method of Activation on as (III) Removal:
Activation of SSR adsorbent was carried out to enhance As(III) adsorption. The removal efficiency of heat and acid treatment method was compared in order to select the best activation method for As(III) removal. From the Figure 2 (a) and (b), the removal efficiency of heat treated adsorbent was higher as compared to acid treated adsorbent. The removal efficiency for heat treated adsorbent calcined at 250 to 600 lies between 73 to 99.6%. It was clearly observed that, as the calcinations temperature increase, percentage removal also increased. This might be due to the decrease contaminant level in the adsorbent and provide an active site for As(III) removal. Other than that, calcinations at temperature above 350°C will change the properties of iron oxide present in the adsorbent where the amorphous state of iron oxide will converted to crystalline state. In this sense, the transformation from the amorphous to crystalline state may be advantageous for the long-term stability of the

iron oxides and high affinity for As adsorption. Therefore, upon calcinations over 400°C, the residual As(III) concentration down below the MCL (Figure 2 (c)). However, heating above 550°C caused a slight reduction on As(III) removal. This could attribute to clogging of the pores by melted silica.

From the result of As(III) adsorption tests of SSR activated by acid treatment, the percentage removal vary between 71 to 81% with residual As(III) concentration of 78-145 µg/L. As observed from Figure 2(d), an increase in concentration of acid used in acid treatment up to 1.0 M causes an increase in adsorption efficiency of SSR adsorbent. Low As(III) adsorption using acid treated adsorbent might be due to covering the silicic acid of the active oxidic sites. Since heat treatment shows promising result as compare to acid treatment, therefore, heat treatment was chosen as the method for adsorbent activation. The optimum calcinations temperature used for subsequent study was 500°C. The study on the effect of calcinations time, the adsorbent was calcined at 10 to 120 min in order to determine the optimum time for adsorbent calcinations. As shown in Figure 3, all calcinations time show percentage removal greater than 99% with residue As(III) solution below MCL. Sixty minute calcinations time was chosen as an optimized calcinations time since it can lowest the As(III) concentration below 2 µg/L. Subsequent experiment was carried out using SSR adsorbent calcined at 500°C for 1 h.

Adsorption Study for As(III) Removal:
Effect of Initial As(III) Concentration: The effect of initial arsenic concentration was studied by varying the content of arsenic in the solution between 100 to 1000 µg/L. These concentrations were selected due to the results of groundwater sampling from Cambodia. The adsorption of As (III) with varying the initial As concentration are presented in Figure 4 (a) and (b), which represent the percentage As(III) removal, as well as the residual As concentration.

Interestingly, only by introducing 2g/L of SSR adsorbent, it can efficiently remove more than 99% of all As (III) concentration used. SSR adsorbent was able to remove As(III) from the solution to below the maximum concentration limit (MCL) for As (10 µg/L) as set by United State Environmental Protection Act (USEPA) for all concentration except for initial As(III) concentration of 1000 µg/L. As observed in Figure 4 (b), as the initial As(III) concentration increased, the residual As(III) concentration increased or in other word, increasing the
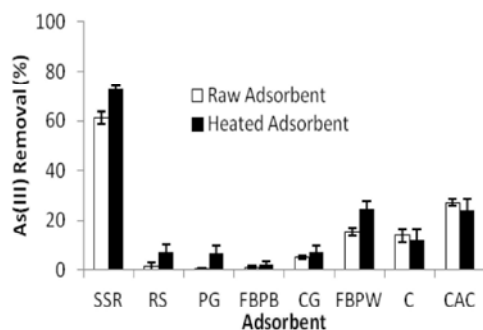
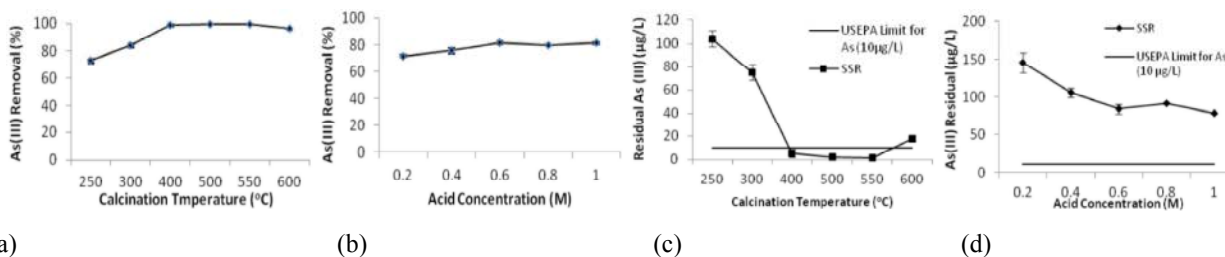Fig. 1: As (III) removal performance of raw and heated low cost adsorbents.

(a)   (b)   (c)   (d)

Fig. 2: Effect of activation method on As(III) removal, (a)effect of heat treatment on As(III) removal, (b) effect of acid treatment on As(III) removal, (c) effect of heat treatment on residual As(III) concentration and (d) effect of acid treatment on residual As(III) concentration
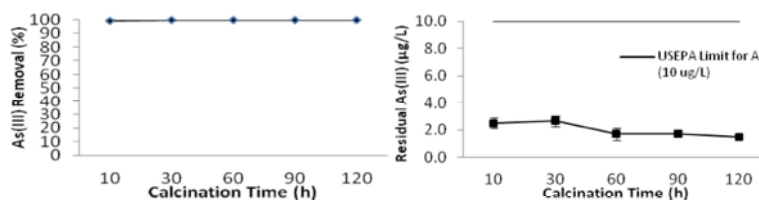

Fig. 3: Effect of calcinations time on As(III) removal and residual As(III) concentration
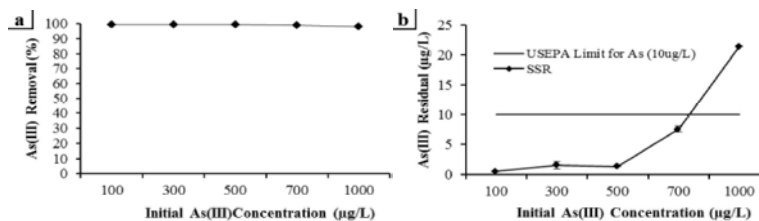

Fig. 4: Effect of initial As(III) concentration on SSR removal based on (a) percentage removal (b) As residual.

initial As concentration was decreased the removal efficiency of the adsorbent. This is because, high As(III) concentration will lead to more ion competing for the available binding sites and more ions are left unadsorbed in solution due to saturation of adsorption site. Contradict, at lower As(III) concentration, all As(III) ions present in the solution could interact freely with available binding site present in SSR adsorbent.

Effect of Initial pH on As(III) Removal: The solution pH is an important factor for all water and wastewater treatment processes because it affects, among others, the speciation of the metals in water. Therefore, experiments were performed in order to investigate the effect of initial pH of solution to be treated regarding and As(III) removal. From the result, the percentage removal of all pH solution vary from 97 to 99.6% (Figure 5 (a)) and the residual As present in the solution for all pH range were above MCL limit
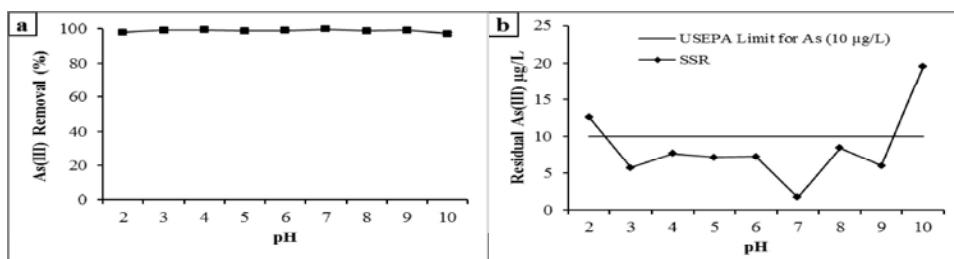
Fig. 5: Effect of initial pH on (a) percentage removal and (b) residual As concentration
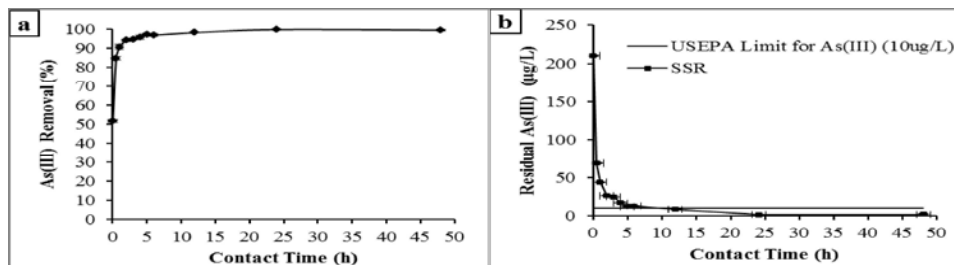

Fig. 6: Effect of contact time on (a) percentage removal and (b) residual As concentration.

except for solution at pH 2 and pH 10. The significant increase in percentage removal with the increase in pH between 2 and 3 was observed (Figure 5 (b)). At pH > 3, the adsorption capacity generally keeps constant. The results evidently show that the SSR adsorbent can efficiently adsorbed As(III) in a wide pH range.

The optimum pH for As(III) adsorption is at pH 7 and above this pH, arsenic removal was found to be decreased. This observation could be well correlated with the point of zero charge (PZC) of iron oxides. Pure iron oxides, whether they can be identified as having a particular crystal structure or not, typically have PZCs in the pH range 7-9 [7]. Over these PZC values, iron oxides are present in the monomeric anionic form [Fe(OH)$_4^-$], hence inappropriate for adsorbing anionic components. High As(III) residual in solution at pH 2 and 10 might be due to the electrostatic repulsion forces, which thrive between iron oxides and arsenic species over the PZC of hydrous ferric oxide [8]. H$_3$AsO$_3$ (uncharged form) dominates at pH 2.0-9.2 and the adsorption of this species to SSR adsorbents may occur as inner-sphere bidentate surface complexes with iron oxide surfaces [9]. H$_2$AsO^{3-} (monovalent anion) is dominant at pH 9.2-12.7. The adsorption of As(III) in this pH range is unfavorable. The negatively-charged monovalent anion becomes predominant and the oxide surface becomes negatively charged, resulting in electrostatic repulsion between As(III) and oxide surface [10].

Effect of Contact Time on As(III) Removal: It is clearly seen from Figure 6 that adsorption of As(III) on SSR adsorbent increases significantly with an increase in contact time. The adsorption rate is quite high in the first 30 min and gradually increases as the adsorption process continues (up to 6 h). The high adsorption rate attributes to the great amount of adsorption sites and the large gradient of As concentration between solution and solid at the beginning of the kinetic test. About 85% of As(III) is removed within 30 min, while only 5-10% in the following 48 h. The slow adsorption rate is likely due to the decrease in the adsorption sites on the surface of the adsorbents [11].

Actually, the adsorption achieves equilibrium in 120 min, when 94 % of As (III) is removed. However, the residue As concentration was lowered to below MCL after 24 h. Therefore a reaction time of 24 h is chosen for further studies. Three general stages normally occur for As adsorption onto solid particles. Firstly, As is transported to the external surface of the adsorbent from bulk solution across the boundary layer (external mass transfer); then As is adsorbed onto particle surfaces; and finally the adsorbed As is exchanged with the structural elements of adsorbent particles or diffused in the internal surfaces of porous materials (intra-particle diffusion) [12]. At the early stage of kinetic tests, first two steps would be predominated, which always happens very fast. However, major processes would be the intra-particle diffusion at the late stage, being considered to be slow [13].

Table 1: Arsenic concentration in real groundwater sample before and after adsorption

| Sample	Sampling Location	[As](μg/L) Before	[As](μg/L) After	As Removal (%)
G1	Ecole Primaire Chong Koh, Kandal Province	697.34	15.1	97.36
G2	Villager at Kors Chamroeun, Kandal Province	99.71	6.44	93.54
G3	Villager at Kg. Siem District, Kg. Cham	20.2	2.68	86.73
G4	Villager at Koh Roka Village, Kg. Siem District, Kg. Cham	68.04	8.78	87.1
G5	Primary School, Kg. Siem District, Kg Cham	85.7	2.25	97.37
G6	Villager at Kg. Siem District, Kg. Cham	44.73	3.19	92.86
G7	As patient, Kg. Siem District, Kg Cham	200.59	4.29	97.86
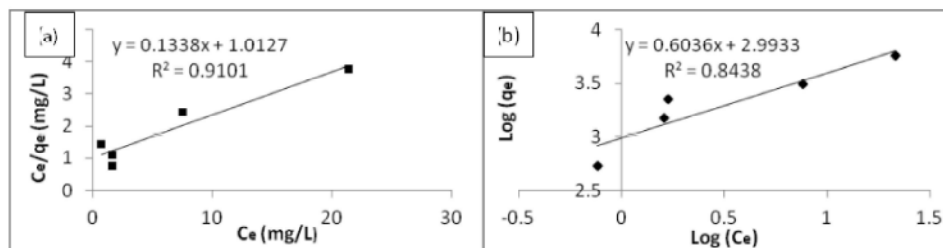
Fig. 7: Langmuir isotherm (a) and Freundlich isotherm (b) of As(III) adsorption on SSR adsorbent.

Application of the SSR Adsorbents to Real Life Groundwater Samples: Seven real groundwater samples obtained from Kandal Province and Kg. Cham were used in order to access the applicability of SSR adsorbent to remove As bearing groundwater. From the result, it can be seen that the As contents of real groundwater sample varied from 20.2 to 697.34μg/L (Table 1). In all water samples, 0.2 g of SSR adsorbent could bring down the As level well below the MCL level except for G1. SSR exhibit higher percentage removals of As in seven groundwater samples, achieving 86.73 to 97.86% efficiency. A slight reduction in As removal efficiency often occurred in ground water could due to the presence of other water contaminant such as Fe_2O_3, phosphate, nitrate and silica. Silica can interfere with the adsorption of As onto iron oxide by forming ferro-silicate.

Adsorption Isotherm: Adsorption isotherm correlate the equilibrium adsorption data with different mathematical models in order to describes a relationship between the amount of chemical adsorbed on adsorbent and the concentration of adsorbate in solution and it is critical in optimizing the use of adsorbents [14,15,16]. The fitness of the equilibrium data obtained from the experiment was evaluated using Langmuir and Freundlich isotherm model. These isotherm equations were used to quantitatively described the effect of increased aqueous As(III) concentrations on the surface loadings of As(III) on SSR adsorbent. The linear form of Langmuir equation is expressed as:

$$C_e/q_e = (C_e/q_m) + (1/K_L q_m) \qquad (2)$$

Where q_e is the amount of As(III) uptake at equilibrium (mg/g), K_L is Langmuir constant related to energy of adsorption, C_e is the equilibrium concentration of As(III) in liquid phase (mg/L) and q_m is the monolayer maximum adsorption capacity (mg/g). The linear form of Freundlich model is given as:

$$\log q_e = \log K_f + (1/n) \log C_e \qquad (3)$$

where K_f and n are Freundlich constants related to adsorption capacity and adsorption intensity respectively. Langmuir and Freundlich isotherms are plotted in Figure 7 (a) and (b) respectively.

The values of Langmuir parameters b and V_m are evaluated from the linear plot of C_e/q_e vs. C_e, while those of the Freundlich constants from the linear plot of log q_e vs. log C_e. Results show that correlation coefficients (r^2) for both the isotherms range between 0.8438 and 0.9101, representing a good fit of observed data. In Freundlich model, the distribution coefficient (K) was 984.69 L/g while the Freundlich constant ($1/n$) was 0.6036. In Langmuir model, the adsorption constant b was 7.57 L/mol while the adsorption capacity (q_m) was 0.987 mg/g. As presented in Figure 7, the correlation coefficients (r^2) of Langmuir model were greater than those of Freundlich model, indicating that Langmuir isotherm was better than Freundlich isotherm at describing the experimental results. The dimensionless constant separation factor (R_L) which indicates whether

the adsorption process is unfavorable ($R_L>1$), linear ($R_L=1$), favorable ($0< R_L<1$) or irreversible ($R_L=0$) can be calculated as:

$$R_L = \frac{1}{1+bC_o} \qquad (4)$$

The RL value was found to be less than 1 for adsorption of As(III) onto SSR adsorbent indication that the adsorption process was favorable [17,18]. The *n* value of Freundlich isotherm is related to the strength of adsorption. The calculated *n* is 1.657, denoting favorable adsorption of As(III) onto the SSR adsorbent. The greater the value of n, the stronger the adsorption bond is. The value of *n* usually lies between 1.4 and 5 for similar adsorption on soil [1]. Interestingly, the Langmuir isotherm for As(III) was characterized by a very strong adsorption bond energy as indicated by the constant 'b', typical of monolayer adsorption which clearly indicated that the present SSR was an adsorbent for As(III). This was further supported by a low q_m value which indicated a monolayer adsorption. A good fit of the data with Langmuir isotherm obtained suggest that the adsorption of As(III) is monolayer sorption. In this study, As(III) adsorption capacity of the SSR at 30 °C, estimated from Langmuir isotherm, is 0.987 mg/g. It has been reported that the values of q_m, the Langmuir constant related to saturated monolayer adsorption capacity for natural hematite [19], hematite [20], iron oxide coated sand [21], iron oxide coated cement [22] are 0.104, 0.18, 0.028 and 0.69 mg/g, respectively (synthetic siderite).

CONCLUSION

From the screening of potential low cost adsorbent for As(III) adsorbent, the adsorbent which gave high As(III) removal was chosen for further study namely SSR. Heat treatment method was found the best method for activation of SSR adsorbents based on its removal efficiency. The optimum condition for SSR adsorbent activation is at 500 °C for 1 h. From the adsorption study, 0.2 g of SSR adsorbent could reduce the residual As(III) concentration below than MCL for initial concentration from 100 to 700 µg/L and pH 3 to pH 9 with 24 h contact time. SSR adsorbent also manages to reduce As in real groundwater sample with 86.73 to 97.86% efficiency with the initial As concentration between 20.2 to 697.34 µg/L. A good fit of the data with Langmuir isotherm obtained suggest that the adsorption of As(III) is monolayer sorption.

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Persian Abstract

چکیده

در مطالعه حاضر، هشت جاذب سطحی کم هزینه و محلی قابل دسترس (بصورت خام و تیمار شده با حرارت) به منظور تعیین قابلیت آن ها برای حذف آرسنیک(III) غربالگری شده اند. پوسته سنگ های رسوبی (SSR) به عنوان یک جاذب خوب بر اساس حذف بالای آرسنیک(III) انتخاب شده اند. فعال سازی جاذب به وسیله گرما (C° ۵۵۰-۲۵۰) و تیمار اسیدی (۰/۱-۱ M H₂SO₄) به منظور انتخاب بهترین روش تیمار انجام شد. آزمایش های جذب برای بررسی اثر غلظت اولیه آرسنیک (III)، زمان تماس و pH ورودی بر روی حذف آرسنیک(III) بوسیله SSR صورت گرفت. نتایج نشان می دهد که، تنها با حرارت دادن جاذب در دمای C° ۵۰۰ به مدت ۱ ساعت، ۰/۲ گرم از SSR توانایی کاهش غلظت آرسنیک(III) باقیمانده به پایین تر از حداکثر غلظت محدود شده (۱۰ μg/L) برای غلظت ورودی ۷۰۰-۱۰۰ μg/L و محدوده pH بهینه ۳ تا ۹ بعد از ۲۴ ساعت از زمان واکنش را دارد. داده های جذب سطحی با معادله Langmuir isotherm به خوبی منطبق شده و تک لایه Langmuir ظرفیتی از ۰/۹۸۷ mg As(III) / g SSR در pH هفت را به همراه داشت. جاذب سطحی SSR به طور موفقیت آمیزی برای حذف آرسنیک از هفت نمونه آب زیرزمینی واقعی که شامل μg/L ۶۹۷/۳۴-۲۰/۲ آرسنیک بودند با بازدهی حذف ۸۶/۷۳ تا ۹۷/۸۶ درصد استفاده شد.