Adsorption Characteristics of Different Adsorbents and Iron(III) Salt for Removing As(V) from Water

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Summary

The aim of this study is to determine the adsorption performance of three types of adsorbents for removal of As(V) from water: Bayoxide® E33 (granular iron(III) oxide), Titansorb® (granular titanium oxide) and a suspension of precipitated iron(III) hydroxide. Results of As(V) adsorption stoichiometry of two commercial adsorbents and precipitated iron(III) hydroxide in tap and demineralized water were fitted to Freundlich and Langmuir adsorption isotherm equations, from which adsorption constants and adsorption capacity were calculated. The separation factor R_L for the three adsorbents ranged from 0.04 to 0.61, indicating effective adsorption. Precipitated iron(III) hydroxide had the greatest, while Titansorb had the lowest capacity to adsorb As(V). Comparison of adsorption from tap or demineralized water showed that Bayoxide and precipitated iron(III) hydroxide had higher adsorption capacity in demineralized water, whereas Titansorb showed a slightly higher capacity in tap water. These results provide mechanistic insights into how commonly used adsorbents remove As(V) from water.

Key words: arsenic removal, drinking water, adsorption

Introduction

The prevalence of toxic arsenic in drinking water is a significant worldwide problem. Most developed countries have implemented strict regulations based on World Health Organization guidelines that allow no more than 10 μg/L of arsenic in drinking water (*1*). However, many developing countries do not have the infrastructure or water resources to implement or enforce such a policy, leading to a recommendation that they initially implement a maximum contaminant level of 50 μg/L of As to provide at least some protection against higher As concentrations (*2*). For all countries, affordable and efficient arsenic removal systems are crucial for creating and maintaining a safe drinking water supply.

Arsenic can be present in water in an oxidation state of –3, 0, +3 or +5, depending on the pH and redox potential of the water. It occurs in water most often as trivalent arsenite (H_3AsO_3, $H_2AsO_3^-$, $HAsO_3^{2-}$ and AsO_3^{3-}) and pentavalent arsenate (H_3AsO_4, $H_2AsO_4^-$, $HAsO_4^{2-}$ and AsO_4^{3-}). Arsenite, or As(III), is usually present as an uncharged molecule (H_3AsO_3) in the water with low redox potential and a pH between 6.5 and 9.2, while pentavalent arsenate, or As(V), is usually present as a negatively charged molecule ($H_2AsO_4^-$) in the water of the same pH range but with a more oxidized aerobic environment (*3*).

The oxidation state of arsenic determines whether it can be efficiently removed from drinking water by standard methods, which usually include a multistep process involving oxidation followed by adsorption or separation (*4*). For example, because arsenite can be uncharged, it is harder to remove it from water than negatively charged arsenate (*5*). Oxidation can be used to convert arsenic into an oxidation state favourable for removal, followed usually by either adsorption or separation. Separation usually involves filtering the contaminated water and separat-

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ing arsenite and arsenate from the water by membrane-based processes such as reverse osmosis or nanofiltration (6).

Adsorption, an alternative method to separation, may involve various types of natural or synthetic adsorbents (4), including precipitated iron or aluminium hydroxides, ion exchangers, metal oxides such as iron, manganese and titanium, laterites, zeolites, activated charcoal and natural red earth (7–13). In cases when water is not heavily contaminated with arsenic and no oxidation or pH correction is necessary, commercially available granular iron(III) hydroxide and oxide are widely used because they have high adsorption capacity (11,14) and can be disposed of safely. The adsorbent titanium dioxide (TiO_2) actually plays a dual role of photocatalyst and adsorbent for arsenic removal. In the presence of artificial or natural UV light, TiO_2 can photocatalyze the oxidation of As(III) to As(V), which then adsorbs onto the TiO_2 surface. In the absence of UV radiation, TiO_2 acts only as an adsorbent and shows adsorption capacity and adsorption kinetics superior to those of iron-based adsorption media. Pena *at al.* (15) showed granular TiO_2 to have higher adsorption capacity than similarly sized granular iron(III) oxide.

One of the most common methods currently used to remove arsenic from drinking water is adsorption on precipitated hydroxides used for coagulation of colloidal particles with iron or aluminium salts. Coagulation is usually used in water treatment to remove turbidity and suspended solids, but hydroxides that form from coagulant salts can bind As(V) by either incorporating it into the hydroxide matrix or by adsorbing it electrostatically onto the positively charged hydroxide surface (16). This method in combination with sand filtration as a final step in the process sometimes does not remove arsenic to meet the maximum contaminant level of 10 µg/L in the treated water. This is primarily due to the inefficiency of sand filtration, which does not successfully remove particles smaller than 10 µm on which adsorbed As(V) can be located (17).

Researchers continue to search for more efficient methods to adsorb arsenic from water, which has led to a broad range of adsorbents (4). Though the efficiency of these adsorbents can vary depending on physical factors that influence the complex chemistry of arsenic in water, relatively few studies have compared the functional characteristics of several types of adsorbents (7,11,18,19). In particular, little is known about the comparative functional characteristics of iron coagulant and adsorbents based on iron(III) oxide or titanium oxide.

To provide some of these data and thereby create a basis for improving arsenic adsorption methods, we investigated and compared the removal of As(V) from model solutions using commercial adsorptive media Bayoxide® E33 (granular iron(III) oxide), Titansorb® (granular titanium dioxide) and iron(III) chloride as a precipitated suspension of iron(III) hydroxide. Results of As(V) adsorption stoichiometry on the three used adsorbents in tap and demineralized water were fitted to Freundlich and Langmuir isotherm models to gain insights into the adsorption mechanism.

Material and Methods

Water samples

Three types of adsorbents were investigated for the removal of As(V) from tap and demineralized water spiked with As(V). The characteristics of the water used in this study are shown in Table 1. $AsHNa_2O_4 \cdot 7H_2O$ (Fluka Chemie GmbH, Buchs, Switzerland) was used to prepare solutions of tap or demineralized water containing the following concentrations of As(V): 100, 200, 300, 400, 500 and 600 µg/L.

Table 1. Characteristics of tap and demineralized water

Parameter	Tap water	Demineralized water
pH	7.2	7
γ(suspended matter)/(mg/L)	0	0
Electrical conductivity/(µS/cm)	680	<0.1
Total hardness/(mg $CaCO_3$/L)	375	0
Alkalinity/(mg $CaCO_3$/L)	340	0
γ(As)/(µg/L)	<1	0
γ(Fe)/(µg/L)	0.2	0
γ(Mn)/(µg/L)	0.2	0
γ(Cl^-)/(mg/L)	15	0
γ(SO_4^{2-})/(mg/L)	27	0
γ(PO_4^{3-})/(mg//L)	<0.01	0

Adsorbents

The following three types of adsorbents were studied: Bayoxide® E33, Titansorb® and precipitated suspension of iron(III) hydroxide. Bayoxide® E33 is granulated iron(III) oxide medium developed by LANXESS GmbH, Leverkusen, Germany. Manufacturer's specifications are as follows: >70 % content of Fe_2O_3, bulk density of 0.6 kg/L, grain size of 0.5–2.0 mm and specific surface area of at least 250 m^2/g. Titansorb® is granulated titanium dioxide developed by Watch GmbH, Mannheim, Germany. Manufacturer's specifications are as follows: grain size of 0.5–1.5 mm, bulk density of 0.6 kg/L and specific surface area of 350–450 m^2/kg. Precipitated iron(III) hydroxide was prepared by adding $FeCl_3$ (Sigma-Aldrich, Saint Louis, MO, USA) to model solutions to achieve desired concentrations of precipitated hydroxide, which were then expressed as Fe(III) in mg/L.

Adsorption tests

All adsorption experiments were conducted at a constant temperature of (20±1) °C and pH=8±0.1 in a Jar Tester ZR 4-6 (Zhongrun Water Co. High-Tech Industrial Park, Shenzhen, Guangdong, PR China) equipped with six 1.5-litre square acrylic beakers, each with its own mixing paddle centrally controlled. Model solutions for adsorption experiments were prepared in volumes of 1 L. At the beginning of each experiment, pH was corrected by the addition of 1 mol/L of hydrochloric acid (Sigma-Aldrich) and 1 mol/L of sodium hydroxide (Sigma-Aldrich) to achieve desired pH after the addition of adsorbents.

For testing arsenic adsorption using a precipitated suspension of iron(III) hydroxide, different amounts of $FeCl_3$ were added to 1 L of model solutions of tap or demineralized water containing 100 µg/L of As(V) to give final Fe(III) concentrations of 0.68, 1.37, 2.06, 2.75, 3.44 and 4.12 mg/L. Then, a corresponding volume of $FeCl_3$ stock solution was added to achieve 3.44 mg of Fe(III) in the model solutions containing As(V) concentrations of 100, 200, 300, 400, 500 or 600 µg/L. The iron(III) hydroxide suspension was mixed with paddles rotating at 200 rpm for 1 min and then at 20 rpm for 24 h.

For testing arsenic adsorption by Bayoxide E33 and Titansorb, these adsorbents (0.25 g) were added to 1 L of model solutions of tap or demineralized water containing As(V) concentrations of 100, 200, 300, 400, 500 or 600 µg/L. Solutions were mixed with paddles rotating at 150 rpm for 24 h.

After adsorption experiments, model solutions were filtered through a 0.45-µm filter (Whatman, GE Healthcare, Dassel, Germany), and 10 mL of concentrated HNO_3 (Fluka Chemie GmbH) were added to 1 L of each sample in order to preserve them until later analysis by inductively coupled plasma optical emission spectrometry (ICP-OES).

Adsorption column tests

Bayoxide E33 and Titansorb were examined in column tests. Bayoxide E33 (V=95 mL), bulk density of 0.6 kg/L and grain size of 0.5–2.0 mm or Titansorb (V=95 mL), bulk density of 0.6 kg/L and grain size of 0.5–1.5 mm were placed in a glass cylinder with an 11-mm diameter, and tap water spiked with 100 µg of As(V) per L was pumped through the column at 5 m/h using a peristaltic pump (ProMinent, Heidelberg, Germany), where empty bed contact time (EBCT) was 12 min. From the mass of the used adsorbent and from the volume of spiked water that passed through the column, adsorption capacity of adsorbent (q) was calculated. Spiked water was run through the column until the concentration of As(V) in effluent exceeded γ=50 µg/L. Each day, a 100-mL sample of effluent was taken and preserved by adding 1 mL of concentrated HNO_3 for later analysis by ICP-OES.

Calculation of Freundlich and Langmuir constants

Freundlich and Langmuir constants were calculated using MS Excel (Microsoft, Redmond, WA, USA) from the initial mass concentrations of As(V) (γ_0/(mg/L)), measured equilibrium As(V) concentration (γ_e/(mg/L)) and calculated mass of adsorbate on the adsorbent at equilibrium for Freundlich (q_{eF}/(mg/g)) and for Langmuir (q_{eL}/(mg/g)) isotherms [20].

Freundlich constants were calculated from the linear region of a log-log diagram (Eq. 1), and then plotted as log q_{eF} *vs.* log γ_e. This plot was used to calculate the Freundlich isotherm constant (K_F/(mg/g)·(L/g)^n), adsorption intensity (n) and coefficient of determination (R^2).

$$\log q_{eF} = \log K_F + 1/n \cdot \log \gamma_e \qquad /1/$$

The linear Langmuir equation (Eq. 2) was plotted as $1/q_{eL}$ *vs.* $1/\gamma_e$. The slope ($1/Q_0$) and y-intercept ($1/(K_L \cdot Q_0)$)

were used to calculate the Langmuir isotherm constant (K_L/(L/mg)), maximum monolayer coverage capacity (Q_0/(mg/g)) and coefficient of determination (R^2). To determine linearity, favourability or unfavourability of Langmuir isotherms, the dimensionless constant separation factor, commonly used as separation factor (R_L), was calculated according to Eq. 3 [20,21].

$$1/q_{eL} = 1/Q_0 + 1/(K_L \cdot Q_0) \cdot 1/\gamma_e \qquad /2/$$

$$R_L = 1/(1 + K_L \cdot \gamma_0) \qquad /3/$$

ICP-OES and other analyses

As(V) concentration in tap and demineralized water was measured using inductively coupled plasma optical emission spectrometer (Iris Intrepid II XSP; Thermo Electron, Thermo Fischer Scientific Inc., Waltham, MA, USA). Other analyses used to determine pH, temperature, suspended matter, electrical conductivity, total hardness, alkalinity, iron, manganese, chlorides and sulphates in all samples were performed according to standard methods [22].

Results and Discussion

Adsorption experiments

The removal of As(V) from spiked tap and demineralized water by precipitated iron(III) hydroxide, Bayoxide E33 and Titansorb was compared. $FeCl_3$ served as the source of iron(III) hydroxide, since it hydrolyzes when added to water and later precipitates as insoluble hydroxide. Based on the criteria of Cornell and Schwertmann [23], it was assumed that all $FeCl_3$ added to the model solutions hydrolyzed and precipitated as iron(III) hydroxide because the pH was above 2. Since the other two adsorbents were granular, they were added directly to the spiked solutions.

Freundlich constants in tap water were 5.85 for iron(III) hydroxide, 0.13 for Bayoxide E33 and 0.07 (mg/g)·(L/g)^n for Titansorb (Table 2), suggesting that iron(III) hydroxide had the highest adsorption capacity. The value of $1/n$ of all three adsorbents did not differ significantly, which suggested that they all had similar adsorption intensity (Fig. 1). This is consistent with the adsorption mechanism of Edwards [16], who proposed a three-step process in which arsenate precipitation is followed by its incorporation into the growing floccules of precipitated hydroxide, and finally by bonding between negatively charged arsenate and positively charged hydroxide. Titansorb showed the highest adsorption intensity ($1/n$) of 0.4731 (Table 2), indicating the formation of a stable complex between the adsorbent and As(V).

According to the calculated constants for Freundlich isotherms, iron(III) hydroxide and Bayoxide E33 achieved better results in demineralized compared to tap water (Table 2). Iron(III) hydroxide had a K_F of 10.30 (mg/g)·(L/g)^n, which was 1.7-fold higher than the value in tap water. Nevertheless, when we used the calculated Freundlich constants and a γ_e(As(V))=5 µg/L in Eq. 1, we found q_{eF} to be 20.80 and 24.70 mg of As(V) per g of Fe(III) in tap and demineralized water, respectively. This suggests a similar amount of adsorbed As(V) in the two types of wa-

Table 2. Freundlich and Langmuir isotherm constants for As(V) removal from tap and demineralized water by three adsorbents

	Adsorbent	Freundlich isotherm constants				Langmuir isotherm constants				
		K_F $\dfrac{}{(mg/g)\cdot(L/g)^n}$	$1/n$	q_{eF}* $\dfrac{}{mg/g}$	R^2	Q_0 $\dfrac{}{mg/g}$	K_L $\dfrac{}{L/mg}$	R_L	q_{eL}* $\dfrac{}{mg/g}$	R^2
Tap water	Iron(III) hydroxide	5.85	0.7880	20.80	0.9571	376.2	10.4	0.49	18.60	0.9685
	Bayoxide E33	0.13	0.7535	0.42	0.9510	8.0	9.5	0.51	0.36	0.9954
	Titansorb	0.07	0.4731	0.14	0.9458	1.2	11.6	0.46	0.06	0.9380
Demineralized water	Iron(III) hydroxide	10.30	0.5454	24.70	0.9012	181.9	23.9	0.29	24.30	0.9766
	Bayoxide E33	0.47	0.3639	0.85	0.9277	1.9	229.5	0.04	1.04	0.9848
	Titansorb	0.05	0.5195	0.11	0.9114	1.4	6.5	0.61	0.04	0.9971

*Calculated amount of adsorbate in the adsorbent at an equilibrium concentration of γ_e=5 µg/L
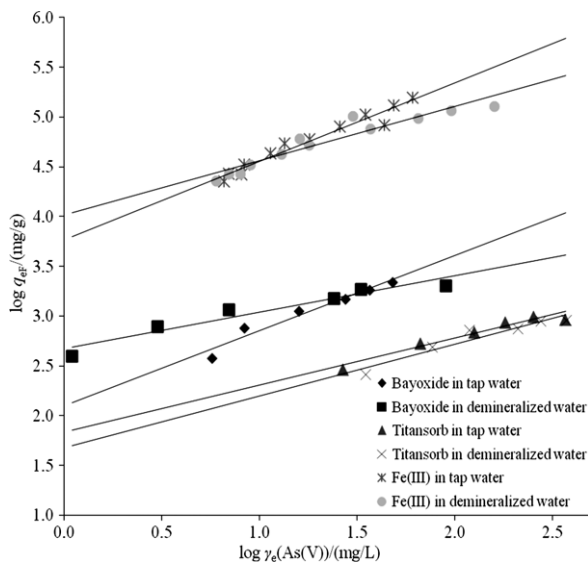

Fig. 1. Dependence of As(V) adsorbed at equilibrium (q_{eF}) on As(V) concentration in a solution, used to calculate Freundlich isotherm constants

ter. Mitrakas *et al.* (*24*) developed a mathematical model for prediction of Fe(III) dose for removal of As(V) at different pH values. Calculated q_{eF} at pH=8 was 15.7, and at pH=7.5 the q_{eF} was 24.7 mg of As(V) per g of Fe(III), which was in accordance with our results.

Similar to the precipitated iron(III) hydroxide, Bayoxide had a K_F of 0.47 (mg/g)·(L/g)^n in tap water, nearly 3.5-fold higher than in demineralized water. When we used the calculated Freundlich constants and an γ_e(As(V))=5 µg/L in Eq. 1, q_{eF} was 0.42 mg of As(V) per g of Bayoxide in tap water and 0.85 mg of As(V) per g of Bayoxide in demineralized water. This large difference probably reflects the high adsorption intensity ($1/n$), which was 0.7535 in demineralised and 0.3639 in tap water. Our measurement of the adsorption capacity of Bayoxide is comparable to that of Westerhoff *et al.* (*11*), who reported a value of 0.5 mg of As(V) per g of adsorbent. Lin *et al.* (*7*) observed a K_F of 16.7 (mg/g)·(L/g)^n for Bayoxide, but they used much higher As(V) concentrations than we did (5.1–20.8 mg of As(V) per L).

In contrast to the other two adsorbents, Titansorb adsorbed more As(V) in tap than in demineralized water, where the Freundlich constant K_F was 0.05 (mg/g)·(L/g)^n. Based on Eq. 1, q_{eF} was 0.14 and 0.11 mg of As(V) per g of Titansorb in tap and demineralized water, respectively. Titansorb had the lowest q_{eF} of all three absorbents, which may be due to the pH values of model solutions ((8±0.1)). For example, Dutta *et al.* (*19*) reported that lower pH enhances the removal of As(V) from water by adsorption onto TiO_2. Furthermore, Bang *et al.* (*25*), who worked with higher concentrations of competitive ions than we did, reported no obvious effect of silica and phosphorus on the adsorption of As(V) onto granular TiO_2.

Table 2 reports the Langmuir constant Q_0, representing maximum monolayer coverage capacity and the Langmuir isotherm constant K_L, representing the sorption capacity, of the three adsorbents (Fig. 2). Among the three,
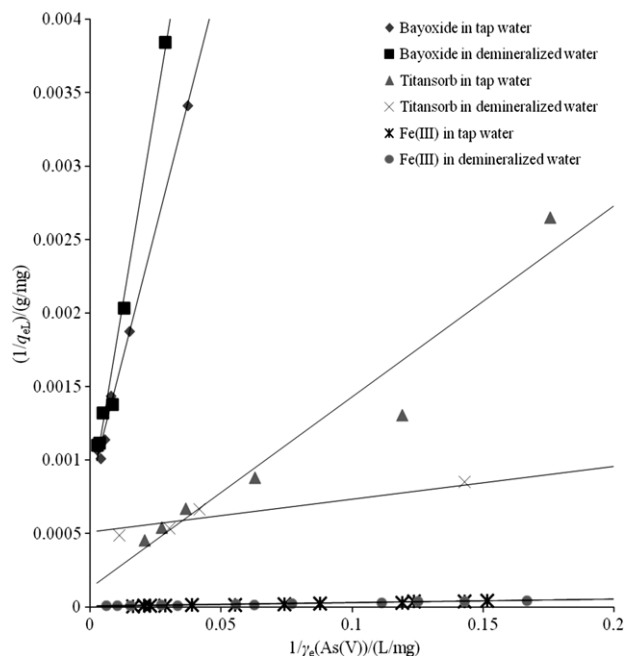

Fig. 2. Dependence of As(V) adsorbed at equilibrium (q_{eL}) on As(V) concentration (γ_e) in a solution, used to calculate Langmuir isotherm constants

iron(III) hydroxide had the highest value of Q_0 (372.6 mg/g) and Titansorb the lowest (1.2 mg/g) in tap water. The same trend was observed in demineralized water, with Q_0 of iron(III) hydroxide equal to 181.9 mg/g, of Bayoxide to 8.0 mg/g and of Titansorb to 1.4 mg/g. Iron(III) hydroxide had a Q_0 almost 2-fold higher in demineralized than in tap water, and Bayoxide had a Q_0 almost 5-fold higher in demineralized water. Titansorb, in contrast, had similar Q_0 in both types of water. However, our study focuses on the removal of low As(V) concentrations, considering drinking water treatment and consequently Q_0 values may not be representative of the adsorbent maximum adsorption capacity due to low initial arsenic concentrations employed.

Even though iron(III) hydroxide and Bayoxide E33 had substantially higher Q_0 values in demineralized water than in tap water, the fact that their calculated K_F values were much higher in demineralized water meant that the calculated capacity of adsorption (q_{eL}) was greater in tap water, as can be seen from the values of q_{eL} calculated from Eq. 2 at γ_e(As(V))=5 µg/L. Titansorb had the smallest value of q_{eL} of all three adsorbents, indicating no significant influence of other ions on As(V) adsorption. Our Langmuir isotherm constants for As(V) removal were lower than those reported by Bang *et al.* (*25*) for granular TiO$_2$, perhaps reflecting the fact that those authors used much higher As(V) concentrations (10–60 mg/L) in their adsorption tests. The same authors reported K_L=0.000172 L/µg, Q_0=40 mg/g and q_{eL}=1.7 mg/g. Like us, they did not observe appreciable interference from other ions. During their column test experiments in which γ_0=32 µg/L, they observed q_{eL} of 0.11 mg/g, which is similar to our results.

The separation factor (R_L) suggests the possibility of using Langmuir isotherm for adsorption of As(V), and it can indicate the adsorption as: unfavourable ($R_L>1$), linear ($R_L=1$), favourable ($0<R_L<1$) and irreversible ($R_L=0$) (*20,21*). Eq. 3 was used to calculate R_L for all three adsorbents: using K_L and γ_0=100 µg/L gave values from 0.04 to 0.61 (Table 2). These values indicate favourable adsorption ($0<R_L<1$) by all three adsorbents.

Although the three adsorbents differed in adsorption capacity, K_F and Q_0 in tap and demineralized water (Table 2), they had similar values of q_{eF} and q_{eL} calculated from Eqs. 1 and 2. In addition, both isotherms gave high coefficients of determination (R^2). Higher coefficients were obtained using the Langmuir model, indicating that adsorption on all three adsorbents may be more accurately described by formation of an As(V) monolayer on the adsorbent surface.

Several studies have shown that higher water pH diminishes adsorption capacity, mostly due to changes in the charge of arsenate species, which then need two active sites on the adsorbent. Also, increase of pH value above the isoelectric point (IEP) of the adsorbent results in more negative charge of adsorbent surface (*26,27*), which can diminish adsorption capacity. Bayoxide and freshly precipitated iron(III) hydroxide usually have IEP ranging between 7 and 7.5 (*23*) and that of TiO$_2$ is lower, which could explain lower adsorption capacity of Titansorb in our study since we conducted experiment at higher pH values. Furthermore, As(V) adsorption is also influenced by the competitive ions present in the water, mostly by OH$^-$,

PO$_4^{3-}$, SO$_4^{2-}$ and NO$_3^-$ (*4,7,28*). Tap water used in our experiments did not have high concentrations of the mentioned ions, but it can be seen from the obtained results that some influence on the adsorption capacity was present, mostly on iron(III) hydroxide and Bayoxide, and less on Titansorb.

Adsorption column tests

Column tests were conducted to measure the adsorption of As(V) by Bayoxide E33 and Titansorb from spiked tap water containing 100 µg of As(V) per L (Fig. 3). Titansorb had a breakthrough point of 10 µg of As(V) per L in the effluent after 1077 bed volumes, while Bayoxide could be used to treat 1436 bed volumes before the As(V) concentration in the effluent reached 10 µg/L. Also, Bayoxide had adsorption capacity (*q*) of 0.24 µg of As(V) per g, and Titansorb of 0.17 µg of As(V) per g, before achieving mass concentration of As(V) of 10 µg/L in treated water. Comparing *q* values of the column test with calculated q_{eF} and q_{eL} values from Table 2, q_e of Bayoxide was higher in the batch tests, indicating that Bayoxide should adsorb more As(V) from water in column test. This difference may be influenced by an EBCT in a column test, which was 12 min. Similar influence of EBCT on the adsorption capacity of adsorbents was observed in the work of Tresintsi *et al.* (*18*). On the other hand, Titansorb in our experiment showed similar *q*, q_{eL} and q_{eF} values, which suggests that the employed EBCT did not influence its adsorption capacity.
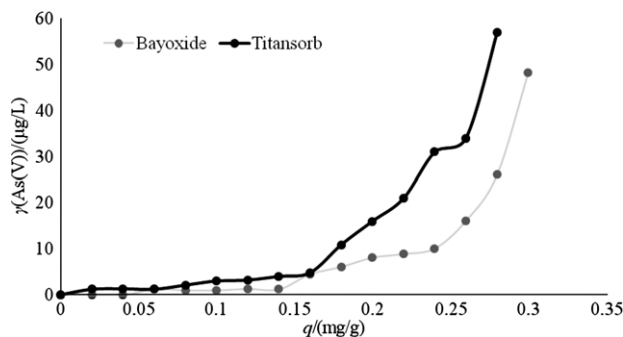

Fig. 3. Adsorption capacity (*q*, in mg of As(V) per g of adsorbent) test used for As(V) removal from tap water

Conclusions

Among the three adsorbents in this study, precipitated iron(III) hydroxide had the highest capacity for As(V) adsorption from both tap and demineralized water. Bayoxide had higher adsorption capacity than Titansorb in both the batch and column tests.

Other ions did not appear to appreciably influence As(V) adsorption by any of the three adsorbents tested here. Nevertheless, iron(III) hydroxide and Bayoxide demonstrated higher adsorption capacities in demineralized water than in tap water, with Bayoxide showing the greatest capacity difference between the types of water. Titansorb exhibited slightly better calculated adsorption capacity q_{eL} and q_{eF} in tap water than in demineralized wa-

ter. All three adsorbents showed good adsorption, as values of separation factor R_L from Langmuir isotherms ranged between 0.04 and 0.61.

Both Freundlich and Langmuir isotherms fit the experimental data with high coefficients of determination (>0.9012) for all adsorbents, indicating that both models may be useful for representing As(V) adsorption. Nevertheless, the Langmuir model showed somewhat better results, which may mean that it represents more accurately As(V) adsorption on these three adsorbents.

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