



The sorption of inorganic arsenic on modified sepiolite: the effect of hydrated iron(III) oxide

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Abstract: The sorption of inorganic arsenic species, As(III) and As(V), from water by sepiolite modified with hydrated iron(III) oxide was investigated at 25 °C through batch studies. The influence of the initial pH value, the initial As concentration, the contact time and the type of water on the sorption capacity was investigated. Two types of water were used, deionised and groundwater. The maximal sorption capacity for As(III) dissolved in deionised water was observed at an initial and final pH value 7.0, while the bonding of As(V) was observed to be almost pH independent for pH value in the range from 2.0 to 7.0, while a significant decrease in the sorption capacity was observed at pH values above 7.0. The sorption capacity at initial pH 7.0 was about 10 mg g⁻¹ for As(III) and 4.2 mg g⁻¹ for As(V) in deionised water. The capacity in groundwater was decreased by 40 % for As(III) and by 20 % for As(V). The Langmuir model and pseudo-second order kinetic model revealed good agreement with the experimental results. The results showed that Fe(III)-modified sepiolite exhibits significant affinity for arsenic removal and it has the potential for application in water purification processes.

Keywords: arsenic(III); arsenic(V); Fe(III)–sepiolite; sorption; pH influence.

INTRODUCTION

The inorganic arsenic occurs naturally in rocks and soils as a constituent of many minerals (arsenides, sulphides, oxides, arsenates and arsenates).¹ These forms are insoluble, but under certain conditions the minerals might be dissolved, or arsenic could be leached from them, becoming in this way mobile. The mobilization is caused by specific geochemical and hydrogeological triggers.² The two

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main geochemical triggers are: 1) an increase of the pH value to over 8.5, when arsenic releases protons and becomes negatively charged that leads to desorption from minerals; 2) the occurrence of reducing conditions below neutral pH values, when oxides of Fe(III) and Mn(IV) dissolve and become reduced to Fe(II) and Mn(II), which also enables arsenic species from these minerals to dissolve. This is still not sufficient for high levels of arsenic to form in water; the hydrogeological conditions must also be fulfilled: a slow process of ground water exchange in aquifers has to occur or the aquifers have to be very young.² Besides all above mentioned, smaller, but still significant amounts of arsenic are released in water from ore smelting.^{2,3} Due to all this, arsenic is present in many natural waters, primarily in ground water, in the form of arsenic and arsenous acid and in the form of their anions.³ These inorganic species could be methylated by bacteria, fungi and yeasts to organic arsenic compounds.⁴ All these compounds are highly toxic and carcinogenic.

Arsenic may cause acute and chronic poisoning. Symptoms of chronic poisoning are not specific (weakness, gastritis, colitis, tiredness, hair loss); hence, they are not easily recognizable. Longer exposure to water with arsenic leads to severe health problems: skin lesions, disruptions of cardiovascular, respiratory, digestive, renal and nervous systems, and skin, lung, bladder, liver and kidney cancers.^{4,5} There are some indications that inorganic arsenic may also be diabetogenic.⁶ Acute poisoning may cause vomiting, dryness of the mouth and throat, muscle cramps, hallucinations, diarrhoea, or even death due to hepatic and renal failure or heart attack.⁴ Arsenic compounds accumulate in the body, so even small concentrations in drinking water could be very dangerous. As a of this, the WHO (World Health Organization) reduced the recommended maximum concentration in drinking water from 50 to 10 $\mu\text{g dm}^{-3}$ in 1993.⁷⁻⁹ At least 60 million people in the world drink water with high levels of arsenic (over 50 $\mu\text{g dm}^{-3}$) every day.² The most risky areas are in Bangladesh, India, Argentina and Mexico, but many individual wells can also be polluted, which is the case in the Panonian Basin region (parts of Serbia, Romania and Hungary).^{10,11}

Many techniques have been developed for arsenic removal from water: coagulation by salts of Fe(III) and Al, modified limestone softening, oxidation with filtration, ion exchange, membrane techniques and others.^{10,12} This paper investigates sorption, which is still not widely used, although it is a very simple and inexpensive technique. The main issue when sorption is applied is that it is relatively slow process and it lacks in selectivity.

The sorption occurs until dynamic equilibrium is established, when the rates of sorption and desorption become equal. This equilibrium could be presented by sorption isotherms in form of sorption capacity *vs.* concentration diagrams. Several models describe this process mathematically, while the Langmuir and the Freundlich models are the most used ones.¹² The Langmuir model assumes

monolayer sorption onto a homogeneous surface with a finite number of identical active sites. Exchange between sorbed species is negligible, which presumes strong bonds between sorbate species and surface active site, *i.e.*, chemical bonds. The Freundlich isotherm model describes non-ideal and reversible sorption, not restricted to the formation of monolayer.¹³ This empirical model can be applied to multilayer sorption, with non-uniform distribution of sorption heat and affinities over a heterogeneous surface. The amount sorbed appears as an overall effect of adsorptions on all sites (each having a particular bond energy), the stronger binding sites are occupied first. The energy of adsorption decreases exponentially upon the completion of the sorption process. That implies that chemisorption (if the active sites are strong enough) is followed by physisorption.

Many materials have been investigated as arsenic sorbents: oxides, salts, activated carbon, biologic materials, organic polymers and waste materials.¹⁰ Moreover, many modifications of these materials are being investigated in order to improve the sorption capacity. It is known that iron oxyhydroxides have good affinities to arsenic, but their flaw is their small specific surface area, which results from agglomeration. A solution could lie in the deposition of Fe(III) oxyhydroxides on porous minerals, such as zeolites, bentonites and sepiolites.^{5,7,8,14–17}

Sepiolite is a fibrous hydrated magnesium silicate^{18,19} with a unit cell formula $Mg_8Si_{12}O_{30}(OH)_4(H_2O)_4 \cdot nH_2O$, $n = 6–8$. Their structures have blocks and channels running parallel to the fibre axis, which provide excellent potential for the retention of micropollutants from water.²⁰ Although natural sepiolite has not shown noticeable capacity for inorganic arsenic,¹⁰ because of its large specific surface area, it is a scientific challenge to attempt modification of a sepiolite surface with Fe(III)-oxyhydroxides, which are considered to be the best sorbents for inorganic arsenic species.

The aim of this study was to evaluate the capacity of Fe(III)-modified sepiolite to remove As(III) and As(V) from aqueous media *via* sorption. The influence of the initial pH value, the initial As concentration, the contact time and type of aqueous media were investigated. Two types of aqueous media were used: deionised water and ground water. The Fe(III)-modified sepiolite used in this study was fully characterized in a previous study.¹⁴ The chemical composition, morphology, phase composition, specific surface area and pore volume, qualitative composition and thermal analysis showed that modified sepiolite retained the structure of natural sepiolite with 31.45 wt. % of amorphous Fe₂O₃. To the best of our knowledge, iron(III)-modified sepiolite has not previously been used for arsenic sorption from aqueous media.

EXPERIMENTAL

Natural sepiolite from Andrići in Serbia, was dried for 2 h at 150 °C and ground in porcelain mortar. Particles under 250 µm were chosen for the modification. Sepiolite (20 g)

was mixed with 200 cm³ of 0.5 M FeCl₃ solution and 360 cm³ of 1.0 M NaOH solution in a polyethylene bottle. The obtained suspension was diluted to 2 dm³ and then heated to 70 °C for 48 h. The precipitate was washed, dried and used as the sorbent, designated as Fe-SEP.¹⁵ XRD analysis, FTIR spectroscopy and DTA/TGA¹⁵ confirmed that Fe-SEP maintained the basic structure of sepiolite. The presence of new crystalline Fe phases was not observed, indicating that only amorphous Fe species were present. The textural properties of Fe-SEP,¹⁹ determined by nitrogen adsorption and desorption at liquid nitrogen temperature, were similar to the properties of natural sepiolite, meaning high porosity and high specific surface area were maintained.

Stock solutions of As(III) (0.5 g dm⁻³) and As(V) (1.0 g dm⁻³) were prepared by dissolving appropriate quantities of As₂O₃ (Merck, 99.5 %) with NaOH (Lach-Ner, 98 %) and Na₂HAsO₄·7H₂O (Carlo Erba, 99 %) in deionised water (DW) with a conductivity under 0.2 μS cm⁻¹ and in ground water (GW). The composition of ground water from a well by the bank of the Sava River was determined by the inductively coupled plasma-optical emission spectrometry (ICP-OES) method (ICP SPECTRO, GENESIS FEE, with the accuracy of 10 μg dm⁻³) and is presented in Table I. As(III) stock solutions were kept in amber bottles.

TABLE I. Ground water composition, determined by the ICP-OES method

Cation	Al	B	Ba	Ca	Fe	K	Mg	Mn	Na	Sr	As, Be, Cd, Co, Cr, Cu, Hg, Li, Mo, Ni, Pb, Sb, Sn, Zn
<i>c</i> / mg dm ⁻³	0.08	0.03	0.03	50.8	0.09	3.01	13.3	0.02	14.3	0.25	<0.01

The sorption experiments were performed by batch technique in a thermostated water bath with a shaker (MEMMERT), in which the temperature was maintained at 25±0.1 °C. A mass of 0.02 g of Fe-SEP in 100 cm³ solutions was used in all sorption experiments. Arsenic concentrations were determined by the ICP-OES method (ICP SPECTRO, GENESIS FEE).

The effect of pH value on As(III) and As(V) sorption from solutions in DW was investigated with arsenic solution of 0.500 mg dm⁻³ concentration, by varying the initial solution pH (pH_i), from 2.0 to 10.0. After 24 h of equilibration, the suspensions were filtered and the final pH (pH_f) and the As concentration were determined.

The sorption capacity was determined after equilibration of different concentrations (from 0.25 to 7.0 mg dm⁻³) of As(III) or As(V) solutions in DW and GW. The initial pH values were adjusted to 7.0 using 0.01 M KOH or HNO₃ solutions. The equilibrium sorption capacity, *q_e* (mg g⁻¹), was calculated according to the following equation:

$$q_e = (c_0 - c_1)V/m \quad (1)$$

where *c₀* and *c₁* are the arsenic concentrations before and after sorption (mg dm⁻³), *m* is the mass of sorbent (g), and *V* is a volume of solution (dm³).

The kinetic analysis of sorption was realised using As(III) and As(V) solutions in DW of 0.50 and 3.00 mg dm⁻³, at an initial pH value of 7.0±0.1. The suspensions were equilibrated for 1, 2, 4, 8, 16 and 24 h. The amount of As sorbed after time *t*, *q_t*, were calculated according to Eq. (1), where *c₁* is As concentration after time *t*.

RESULTS AND DISCUSSION

Effect of initial pH

The pH value of the solution is an important controlling parameter in the arsenic sorption process because it has an important effect on the chemistry of

arsenic,²¹⁻²³ as demonstrated in Fig. 1, and on the surface properties of the sorbent, and hence on the interactions between arsenic and the sorbent surface.

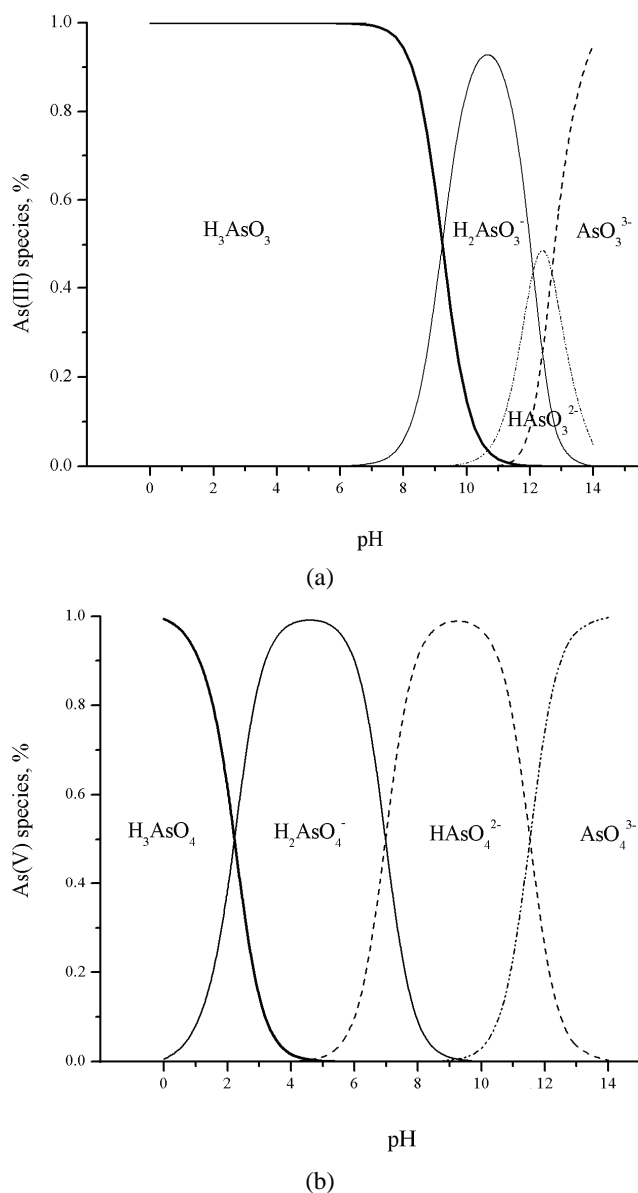


Fig. 1. The distribution of As(V) and As(III) species as a function of the pH value of the water.^{12,21-23}

The effect of the initial pH value (pH_i) on the equilibrium sorption capacity for 0.5 mg dm^{-3} solutions of As(III) and As(V) dissolved in deionised water is

presented in Fig. 2, along with the final pH values (pH_f). According to the position of the plateau on the pH_f vs. pH_i dependence, a change in surface charge from positive to negative or *vice versa* occurred at a pH value of around 7.0. For $\text{pH}_i < 7$, the final pH values were higher than the initial ones, implying protonization of the surface functional groups and thus a sorbent with a positive surface charge. For $\text{pH}_i > 7$, the final pH values were lower than the initial ones, indicating deprotonization of the surface functional groups. Accordingly, the adsorbent surface was negatively charged at $\text{pH}_i > 7$.

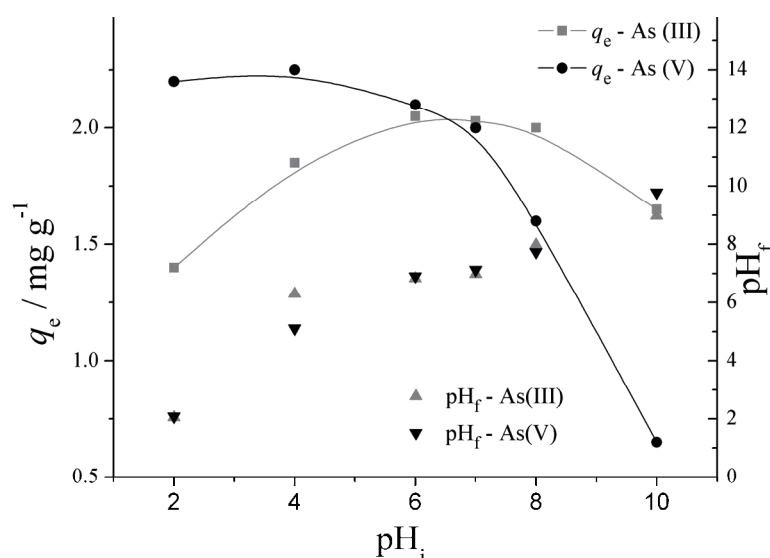


Fig. 2. The influence of the initial pH value on the sorption capacity of Fe-SEP for inorganic arsenic and the dependence of the final pH value of the As(III) and As(V) solutions after the sorption process on the initial pH value of the solutions.

The obtained dependences of the sorption capacity on the pH_i value could be explained by the surface charge of the sorbent and the distribution of arsenic species as a function of pH value. Sorption capacity of Fe-SEP for As(V) was almost constant at initial pH values below 7.0 because the positive charge of the sorbent surface decreased with increasing pH and concomitant increase in the content of anionic species (Fig. 1). The sudden reduction of the sorption capacity with increasing pH_i value above 7.0 could be explained by the change in the surface charge of the sorbent from positive to negative. In the case of As(III), maximal sorption capacity of the sorbent was reached at pH_i values from 6.0 to 7.0, because the sorbent surface was uncharged and arsenic was present mostly in a neutral form of species, H_3AsO_3 (Fig. 1). With decreasing pH_i value, the sorption capacity decreased because the positive charge of the sorbent surface was increased. When the pH_i value increased above 7.0, the sorption capacity for As(III)

decreased because the arsenic was present as anionic specie that is weakly sorbed at the negatively charged surface of the sorbent.

Considering the fact that the sorption capacities were the highest at pH_i values around 7.0 for both As(III) and As(V), that the pH did not change during the sorption for pH_i 7, and that this pH is close to the pH values of natural waters, pH_i 7.0 was selected for all subsequent experiments.

Sorption isotherms studies and modelling

The composition of the aqueous media can have a significant impact on the performance of a sorbent due to the presence of competing ions, complexing anions, dissolved organic matter, *etc.* Therefore, it is important to evaluate potential sorbent materials in aqueous media that represent the conditions under which the adsorbent would most likely be employed. For applications in real water treatment, Fe-SEP was evaluated as a sorbent for As(V) and As(III) in deionised water (DW) and ground water (GW).

The experimental isotherms for the sorption of As(V) and As(III) onto Fe-SEP in DW and GW, as the dependence of q_e on the equilibrium As concentration c_e , are presented in Figs. 3 and 4. All isotherm datasets were fitted to the Langmuir and Freundlich isotherm and the model fits are presented with the experimental data in Figs. 3 and 4. The sorption isotherms constants were determined by non-linear regression analysis using OriginPro 8.5 and the results are given in Table II.

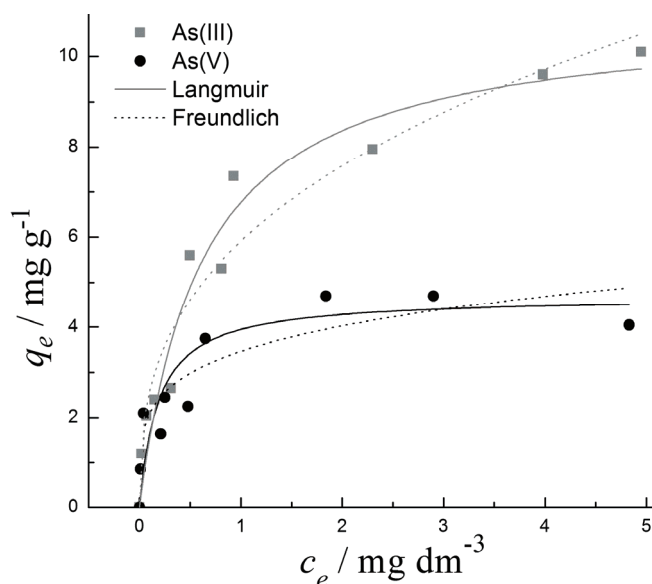


Fig. 3. The sorption isotherms for As(III) and As(V) dissolved in deionised water.

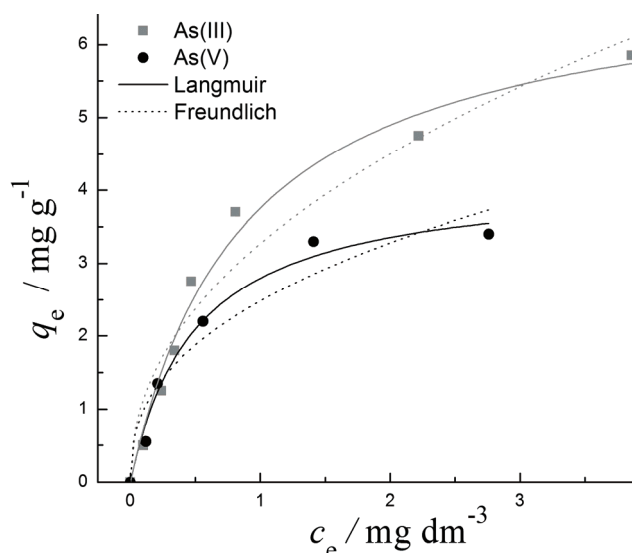


Fig. 4. The sorption isotherms for As(III) and As(V) dissolved in ground water.

Based on the experimental isotherms, Fe-SEP showed capacity of about 10 for As(III) and 4.2 mg g⁻¹ for As(V) in DW, and 5.8 mg g⁻¹ for As(III) and 3.4 mg g⁻¹ for As(V) in GW. As it was expected, the sorption capacities were lower in GW by approximately 20 % for As(V) and 40% for As(III), mainly because the dissolved species present in GW compete with arsenic species for the sorption sites at the surface of the sorbent.

TABLE II. The sorption parameters and correlation coefficients according to the Langmuir and Freundlich model

Mechanism	Langmuir			Freundlich		
Equation	$q_e = \frac{q_m K_L c_e}{1 + K_L c_e}$			$q_e = K_f c_e^{1/n}$		
Type of solution	$K_L / \text{dm}^3 \text{mg}^{-1}$	$q_m / \text{mg g}^{-1}$	R^2	$K_f / \text{mg}^{1-1/n} \text{dm}^{3/n} \text{g}^{-1}$	$1/n$	R^2
As(III), DW	1.62	10.9	0.952	5.91	0.359	0.948
As(V), DW	3.10	4.95	0.863	3.33	0.247	0.807
As(III), GW	1.14	7.04	0.983	3.27	0.461	0.945
As(V), GW	1.99	4.20	0.982	2.48	0.402	0.921

The values of the correlation coefficients, R^2 (Table II), show that the data fit better to the Langmuir than to the Freundlich model, indicating monolayer adsorption on the homogeneous surface of the adsorbent. The Langmuir sorption model serves to estimate the maximum uptake value or the total capacity of the adsorbent, q_m , which could not be reached in the experiments. The values of q_m obtained by the Langmuir model (Table II) were slightly higher than the values

estimated from the experimental isotherms (Fig. 2). The Langmuir constant K_L represents the strength of adsorption:¹³ a higher value of K_L implies a stronger sorbate–sorbent interaction. As can be seen from Table II, the K_L values were higher for As(V) than for As(III), indicating that As(V) formed stronger bonds with Fe–SEP than did As(III). As explained in the previous section, at pH_i 7, As(III) is bonded to the uncharged Fe–SEP surface in the molecular form H_3AsO_3 and those bonds were weaker than those of the anionic species of As(V) with the positively charged Fe–SEP surface.

Kinetics

In order to design appropriate sorption units, it is important to predict the rate at which As is removed from contaminated water. The kinetic data for As(III) and As(V) sorption from DW onto Fe–SEP are shown in Fig. 5 ($c_0 = 0.50 \text{ mg dm}^{-3}$) and Fig. 6 ($c_0 = 3.00 \text{ mg dm}^{-3}$). Two stages are noticed for all the curves. The first stage is intensive, and 80 % of the sorption capacity is attained there. The second phase is considerably slower and q_t value is asymptotically approaching to the equilibrium capacity, q_e . The first stage was faster for higher concentrations because the driving force for sorption was higher.

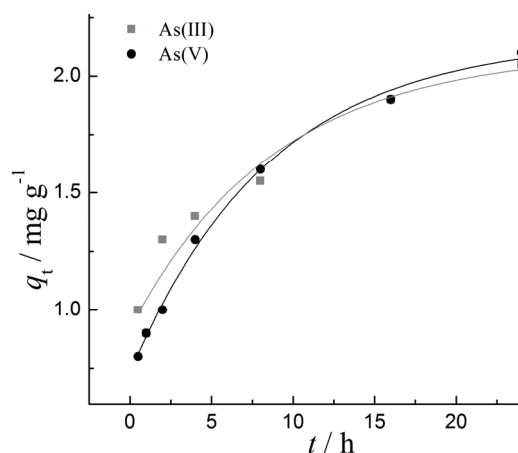


Fig. 5. The sorption kinetics curves for 0.50 mg dm^{-3} As(III) and As(V) solutions.

For a lower concentration (0.50 mg dm^{-3}), sorption curves for As(III) and As(V) almost overlap, while the curves for concentrations of 3.00 mg dm^{-3} have a different shape and plateau position. In this case, Fe–SEP reached considerably higher sorption capacity for As(III) than for As(V), because Fe–SEP has more active sites for As(III) than for As(V) sorption.

The kinetics data were fitted using different models, pseudo-first and pseudo-second order models. Linear forms of these models are presented by Eqs. (2) and (3), respectively:^{24–26}

$$\log (q_e - q_t) = \log q_e - k_1 t / 2.303 \quad (2)$$

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad (3)$$

where k_1 is the rate constant of the pseudo-first order model and k_2 is the rate constant of the pseudo-second order model.

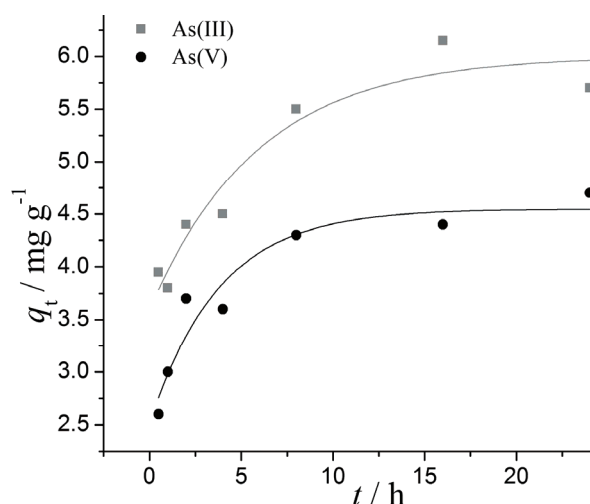


Fig. 6. The sorption kinetics curves for 3.00 mg dm⁻³ As(III) and As(V) solutions.

Higher correlation coefficients were obtained for the pseudo-second order model than for the pseudo-first order equation (Fig. 7), and the obtained parameters for this model are given in Table III. Since pseudo-second order implies chemisorption,^{25,26} it could be observed that the results of the kinetic analysis are consistent with those of the isothermal studies, *i.e.*, with the applicability of Langmuir model to the isotherm studies. Sorption of As(V) is in better agreement with the pseudo-second order model than As(III) sorption, which is consistent with higher values of Langmuir constant K_L for As(V) than for As(III) sorption.

Comparison of Fe-SEP with other Fe(III)-based sorbents

The sorption capacity of sepiolite toward arsenic was significantly enhanced by the modification, similarly to literature reports for some other natural minerals modified by iron(III) oxyhydroxides. Recently, several Fe(III)-based binary oxide sorbents were synthesized that had surprisingly high sorption capacity for both arsenic(III) and arsenic(V).^{33,34} The list of materials and their sorption capacities reported in the literature are summarized in Table IV. The analysis of these values clearly show that sorption capacity of Fe-SEP, being about 10 mg g⁻¹ for As(III) and 4.2 mg g⁻¹ for As(V), was comparable with the capacities of some other Fe(III)-based sorbents.

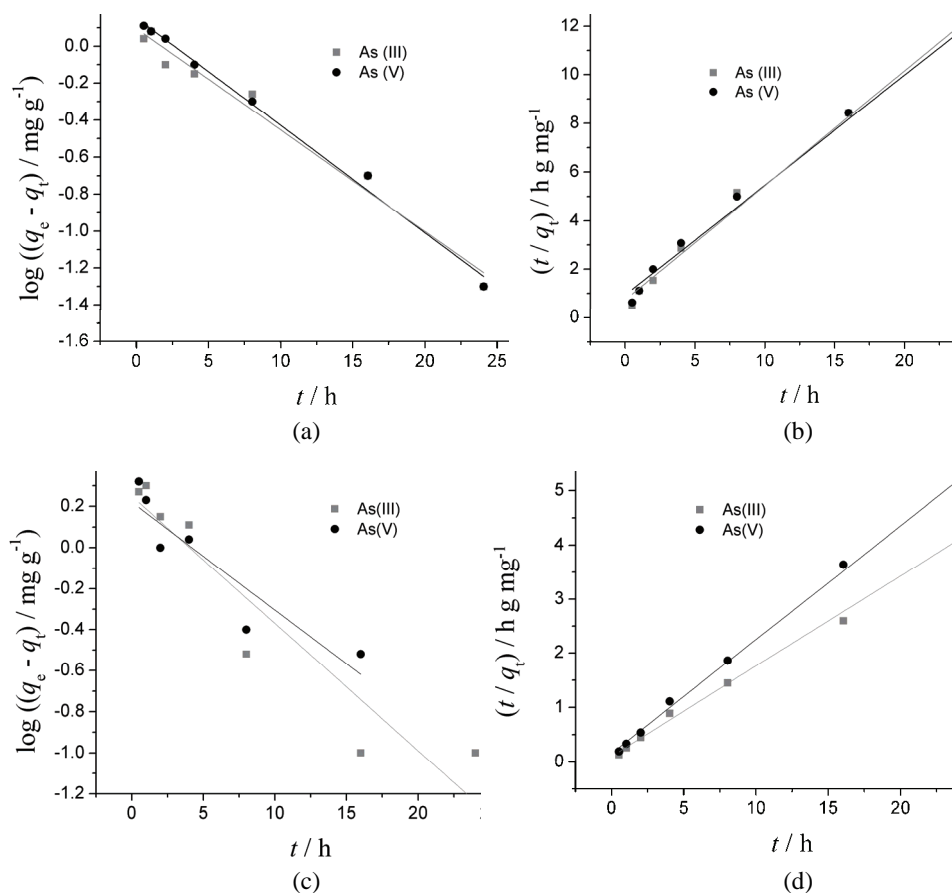


Fig. 7. Experimental data fitting for As(III) and As(V) sorption onto Fe-SEP for 0.50 mg dm^{-3} solutions using: a) the pseudo-first and b) the pseudo-second order model, and for 3.00 mg dm^{-3} solutions using: c) the pseudo-first and d) the pseudo-second order model.

TABLE III. Kinetic parameters for the pseudo-second order model obtained from Fig. 7

Initial concentration, mg dm^{-3}	Dissolved species	$q_e / \text{mg g}^{-1}$	$k_2 / \text{g mg}^{-1} \text{h}^{-1}$	R^2
0.50	As(III)	2.12	0.302	0.990
	As(V)	2.21	0.218	0.989
3.00	As(III)	6.02	0.271	0.995
	As(V)	4.76	0.279	0.998

TABLE IV. Sorption capacities, $q_e / \text{mg g}^{-1}$, of some Fe(III)-based sorbents for As(III) and As(V)

Material	Species		Reference
	As(III)	As(V)	
Zeolite modified with Fe(III)	–	0.68	27
Kaolinite modified with Fe(III)		1.74	28
Graphite oxide modified with Fe_3O_4 and MnO_2	14.04	12.22	9

TABLE IV. Continued

Fe ₂ O ₃ nanoparticles	20.0	4.90	29
Fe ₃ O ₄ nanoparticles	5.68	4.78	29
Hematite	–	0.20	30
Mn(II) and Fe(II) in a polymeric matrix	13.5	14.5	31
Fe(III)–Ti(IV) mixed oxide	85.0	14.3	32
Crystalline hydrous ferric oxide	33.3	25.0	32
Fe–Zr mixed oxide	120.0	46.1	33
Fe(III)–Cu(II) binary oxide	122.3	82.7	34
Goethite	22	4	10
Fe–SEP	9.5	4.2	This study

CONCLUSIONS

Sepiolite modified with hydrated iron(III) oxide was demonstrated to be a very efficient sorbent for the removal of As(III) and As(V) from aqueous solutions. Sorption capacity in deionised water was approximately 10 mg g⁻¹ for As(III), and 4.2 mg g⁻¹ for As(V) solutions, at an initial pH value of 7.0. In the case of the sorption from groundwater, the capacity amounts were 5.4 mg g⁻¹ for As(III), and 3.4 mg g⁻¹ for As(V).

Sorption isotherm data in all cases showed better fitting to the Langmuir than to the Freundlich model, which indicates single-layer coverage of the sorbent, with a homogeneous distribution of the active sites on the surface. The values of the Langmuir constant showed that As(V) formed stronger bonds with Fe–SEP than As(III). Investigations of kinetics showed that the sorption of inorganic arsenic followed the pseudo-second order model, which is consistent with the applicability of the Langmuir model to the isotherm studies.

The sorption of both As(III) and As(V) species was the best at pH values from 6.0 to 8.0. These are typical pH values of natural waters, so in the case of the application of sepiolite modified with hydrated iron(III) oxide as the sorbent, the water preparation would be easier and less costly.

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ИЗВОД

ПРОУЧАВАЊЕ СОРПЦИЈЕ НЕОРГАНСКОГ АРСЕНА ИЗ ВОДЕНИХ РАСТВОРА НА МОДИФИКОВАНОМ СЕПИОЛИТУ: УТИЦАЈ ХИДРАТИСАНОГ ГВОЖЂЕ(III)-ОКСИДА

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Објект испитивања у овом раду је сепиолит модификован хидратисаним гвожђе(III)-оксидом и неорганске врсте арсена As(III) и As(V), које представљају најчешће облике арсена у природним водама. Сорпција је изведена у шаржним условима, на 25 °C.

Испитани су утицаји почетне рН вредности раствора, почетних концентрација арсена, времена уравнотежавања и врсте медијума на сорпциони капацитет сорбента. Коришћени су раствори арсена у деминерализованој води и реалној бунарској води. Утврђено је да се максимални сорпциони капацитет за As(III) постиже при константној вредности, рН 7, а за As(V) у ширем интервалу рН вредности од 2 до 7. Сорпциони капацитет при рН 7 је око 10 mg g⁻¹ за As(III) и 4,2 mg g⁻¹ за As(V) растворених у деминерализованој води. У бунарској води капацитет је нижи за приближно 40 % у случају As(III) и 20 % за As(V). Експериментални резултати су показали добро слагање са Ленгмировим моделом и кинетичким моделом псеудо-другог реда. Добијени резултати указују на добар потенцијал сепиолита модификованог гвожђе(III)-оксидом за уклањање арсена из природних вода.

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