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Experimental study of As(V) adsorption onto different adsorbents

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The As(V) adsorption onto natural bentonite (B), Fe enriched bentonite (FB) and synthetic magnetic particles (MP) from the aqueous solutions was investigated by batch as well as column tests. The natural materials did not show such good adsorption properties towards As(V) in the batch experiments as MP, whose Q_{max} was calculated to 23 mg/g. However, in column experiments the adsorption effect of natural materials was comparable with MP. The model solution with the concentration 30 mg As(V)/L was percolated through the columns filled with the B and FB sample. After two cycles the As(V) concentration was lower than 2 mg/L. The second series of columns were filled with FB/MP/QS and B/MP/QS. Two cycles with the solution concentration of 50 and 100 mg As(V)/L were performed. After the second cycle 74 and 100% of As(V) were removed by FB/MP/QS and B/MP/QS, respectively. In the third series two most effective columns were selected for the second percolation of 100 mg As(V)/L solution. While the adsorption effect of the QS/MP column decreased at 36%, for the QS/B/MP column it was about 24%. The first experimental study showed that natural materials should be used effectively as column fillers to fix finer magnetic particles. On the other hand, their selectivity towards Cu(II) and Zn(II) ions could be beneficial for regeneration of media used in the process of soil bioremediation.

Keywords: arsenic, bentonite, Fe-bentonite, magnetic particles

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INTRODUCTION

Subsequent toxic contamination of soils is one of the main environmental problems of urbanisation and industrialisation and has recently become a serious concern in the world. Toxic elements are generally non-biodegradable in the soil matrix. They can be transported and accumulated in sediments and agricultural products and pose a risk to the food safety and human health. Therefore, the remediation of environment with the aim of its decontamination is a global environmental interest.

The treatment of contaminated soils and sediments can be achieved by either physical or chemical methods. Although these techniques have been extensively applied in practice, they show some limitations, such as low efficiency and high cost. Therefore, the bioremediation has received a great deal of attention in recent years [1]. In the past many branches of industry in Slovakia produced a plenty of waste and in recent times a problem with contamination of soils and sediments due to the mobilisation of metals and metalloids from the waste dumping, mining works and heaps occurs. One of them is also a locality Krompachy, loaded by the metallurgical industry. The biological-chemical leaching and usage of resistant bacteria was applied for Cu(II), Zn(II) and As(V) removal from the contaminated samples of soils and sediments [2–4], and the effect of bioleaching in static and dynamic conditions was studied [5]. These first experiments have brought a new ecological way of contaminated soil treatment, but also the question of obtained leachates processing. The reverse immobilisation of toxic elements from the water environment after the bioleaching process could be realised by adsorption and precipitation onto selected mineral materials and synthetic adsorbents. Naturally occurring clays and clay minerals are of paramount importance in the field of environmental and waste management. To the universal usable materials belongs bentonite whose unique properties are determined from its structure. There are few significant deposits of bentonite in the East part of Slovakia (Kuzmice, Fintice, Lastovce) as well as in Central Slovakia (Stará Kremnička – Jelšový potok and Lieskovec). These bentonites are used for industrial purposes, and their geological, geotechnical and sorption properties are still studied by Slovak researchers for their wider utilisation [6–14].

Iron oxides as natural compounds of soils, sediments and environment containing water have a large specific surface area and are able to adsorb a significant amount of toxic ions. They represent dominant adsorbents because they could be also fine dispersed and used as surface coatings of other particles [15]. Magnetic properties allow their easier separation from water. The magnetite, hematite and maghemite particles could be synthetised by different chemical methods, including their modification by functional groups (carboxyl acid) to prevent their agglomeration. The review of the published results about magnetic particles and their nanomaterials as adsorbents of As(III), As(V) and other heavy metal ions was done by Pragnesh and Lakhan [16]. The natural goethite and hematite were studied as adsorbents of Cu(II) and Zn(II) from waters. The synthetic oxides were more efficient, especially for Cu(II) removal, what corresponds with their better surface properties in comparison with natural materials [17].

In this study the As(V) adsorption onto natural bentonites and synthetic magnetic particles from the aqueous solutions was investigated by batch tests as well as in dynamic conditions. The natural and synthetic materials were combined as bedding in column to achieve the effective filter which could be used for regeneration of media utilising in the bioremediation process.

EXPERIMENTAL

Adsorbents

The natural clay samples originated from the Slovak deposit – locality Stará Kremnička – Jelšový potok. Two bentonite samples were studied: bentonite (B) and Fe-enriched bentonite (FB) from the same deposit. The FB sample was visually more ocherous in comparison with the B sample. The clay samples were dried at 40°C for 24 h and sieved to obtain two fractions of particles, below 0.5 mm and between 0.5–1 mm. The chemical composition of the studied clay samples is presented in Table 1.

The Fe_3O_4 magnetic particles were synthetised by chemical precipitation: 15.9 g $FeCl_3$ and 14.0 g $FeSO_4 \cdot 7H_2O$ were dissolved in 100 mL of deionized water and heated under rigorous stirring up to 60°C. During next heating at 90°C 50 ml of 25% NH_4OH was added dropwise. The precipitate was heated and stirred for one hour to complete

Table 1. **Chemical composition of natural clay samples**

Sample	Annealing loss, %	SiO_2, %	Al_2O_3, %	MgO, %	CaO, %	Fe_2O_3, %
FB	15.24	57.2	17.28	3.89	2.72	2.56
B	13.98	57.9	19.66	2.64	2.1	2.15

the reaction. Then it was separated magnetically and washed with 700 mL of hot deionized water to remove free ions. The slurry was then dried at room temperature. The sample was labelled as MP.

Textural study

Textural properties of natural samples were determined from the adsorption and desorption isotherms by the method of physical adsorption of nitrogen at −196°C measured with a NOVA 1200e Surface Area & Pore Size Analyzer (Quantachrome Instruments, USA). First, the studied samples were degassed at 100°C in a vacuum oven under a pressure lower than 2 Pa for 16 h. The measured data were processed by the BET (Brunnauer–Emmet–Teller) isotherm in the range of relative pressure p/p_0 0.05–0.2 to obtain the value of a specific surface area (S_A) [18]. The values of the external surface (S_{ext}) and the volume of micropores (V_{micro}) were calculated from the *t*-plot using the Harkins–Jura standard isotherm [19]. The value of the total pore volume (V_{tot}) was estimated from the maximum adsorption at relative pressure close to saturation pressure. The pore size distribution was obtained from the desorption isotherm using the BJH (Barrett–Joyner–Halenda) method [20].

Zeta potential

The zeta potential (ZP) was measured using a Zetasizer Nano ZS (Malvern, Great Britain). The Zetasizer Nano measures the electrophoretic mobility of the particles, which is converted to the zeta potential using the Helmholtz–Smoluchowski equation built into the Malvern zetasizer software. The zeta potential of the samples (concentration of 2 g/L) was measured within a different pH range, which was adjusted by the addition of NaOH or HNO_3.

Adsorption experiments

Model solutions of As(V) were prepared from KH_2AsO_4 (per analysis quality) and distilled water, in the concentration of 1000 and 100 mg As(V)/L. The model solutions were diluted to the obtained different concentrations in the range 10–50 mg As(V)/L. The pH of the solution was treated by diluted HNO_3 and NaOH to be 4. The adsorption experiments were performed as batch and column tests. The adsorbent dosage for batch experiments was 10 g/L for the clay samples and 1 g/L for a magnetic adsorbent. The suspensions of the model solution and the dosed adsorbents were shaken in a rotary shaker in polyethylene bottles at laboratory temperature (20°C) for 24 h at 30 rpm. The products were separated by filtration and the filtrates were analysed for the residual As content by atomic absorption spectroscopy (AAS). The adsorption experiments were done duplicate. The adsorption data were fitted with the Langmuir equation [21–22], providing the parameters of adsorption (Q_{max}, maximum equilibrium sorption capacity; Q_t, theoretical sorption capacity; R^2, coefficient of determination; K_L, Langmuir adsorption constant), which were used for the comparison of As(V) adsorption affinity to natural clays and synthetic magnetite.

For the dynamic regime, 5 g of the sample (quartz sand or quartz sand with clay/magnetic particles) was filled into the glass column with a fritted end. The synthetic magnetic particles are very fine, therefore another bearer in the column is required. Quartz sand is a cheap, stable material with a good permeability and could be used to fix fine magnetic particles in the column. Natural quartz sand with 0.5 g of the synthetised sample as middle bedding was used in the column. The solution of the As(V) concentration in the range 30–100 mg/L was percolated through the columns. The experiments were done duplicate. The effect of As(V) retention and the possibility to use column fillers more times was studied. Finally, the effect of adsorbents combination in the column beddings on As(V) adsorption was also tested.

RESULTS AND DISCUSSION

Textural analysis

The natural clay samples showed a similar run of adsorption/desorption isotherms with the difference in

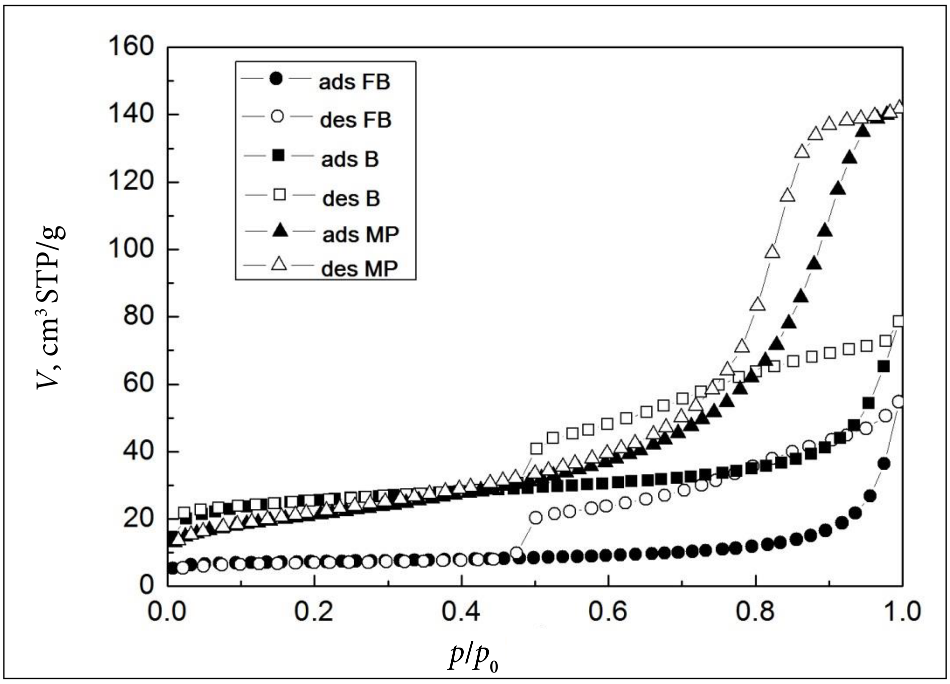

Fig. 1. Adsorption/desorption isotherms of the studied bentonite samples and magnetic particles

the volume of adsorbed/desorbed gas (Fig. 1). Lower values of the adsorbed nitrogen volume showed the Fe-enriched sample. Probably, the present amorphous Fe forms on the clay surface filled the pores and reduced their volume or restrained the gas adsorption into them. The measured isotherms correspond with the Type I of Brunauer–Deming–Deming–Teller (BDDT) classification pointing at the presence of micropores [23]. Type I isotherms are given by microporous solids having relatively small external surfaces, the limiting uptake being governed by the accessible micropore volume rather than by the internal surface area [18]. The adsorbed volume in the range 0.01–0.2 probably corresponded with the quasi-multilayer adsorption in supermicropores (larger micropores with diameter approx. 1–2 nm) and the range p/p_0 = 0.2–0.3 corresponded with a lower limit of mesopores. Over the relative pressure $p/p_0 > 0.3$ the volume of the adsorbed gas increased moderately for both samples, almost linearly up to $p/p_0 = 0.8$. In general, the hysteresis appearing in the multilayer range of physisorption isotherms is usually associated with the capillary condensation in mesopore structures (pores of widths between 2 and 50 nm). Both natural samples also showed a wide hysteresis loop between the adsorption and desorption branch similar to type H4, according to the IUPAC report, which relates with the presence of mesopores of a slit-like shape.

For the magnetic particles the volume of adsorbed gas increased continuously in the whole range of relative pressure. The hysteresis loop is similar to type H1 often associated with porous materials known, from other evidence, to consist of agglomerates or compacts of approximately uniform spheres in a fairly regular array, and hence to have narrow distributions of pore size [18]. Considering that the synthetic magnetite belongs to the class of porous materials formed by loose packing, the obtained loop is related with the gas adsorption into the interparticle space of agglomerates.

The experimental data were processed by the BET isotherm in the range of relative pressure 0.05–0.2 p/p_0 to obtain the value of a specific surface area (Table 2). The BET isotherm showed a convex shape with a negative intercept for both studied clay samples, what indicated again the presence of a higher volume of micropores. From the reason of the negative values of intercept, as well as the C_{BET} constant obtained from the mathematic model the values of a specific surface area are not of real physical meaning. These results are in agreement with the previous statements. The BET method is appropriate for meso- to macroporous materials, that is, materials with a pore size above 2 nm. For microporous materials, other data evaluations than BET should be made [24].

Table 2. **Textural parameters of the studied samples determined from the low temperature nitrogen adsorption method**

Sample	S_{BET}, m²/g	C_{BET}	S_t, m²/g	V_{tot}, cm³/g	V_{micro}, cm³/g
FB	25.2	−143.8	7.2	0.0847	0.0082
B	86.5	−167.7	35.0	0.1216	0.0239
MP	75.3	144.5	67.3	0.2192	0.0033

Therefore, according to the equipment possibilities, the texture of the studied samples was evaluated by the *t*-plot method using the Harkins–Jura isotherm. That allows determination of the micropore volume and value of an external surface area (external surface area + specific surface area of mesopores). The higher micropore volume V_{micro} = 0.0239 cm³/g calculated for the B sample corresponds to a higher position of the adsorption isotherm and a higher value of the specific surface area S_{BET} = 86.5 m²/g. But the real surface area, corresponding to the external surface of materials particles, is only S_{ext} = 35.0 m²/g. That means that about 20% of adsorbed nitrogen was concentrated in micropores, while for the FB sample only 10%.

For the MP sample the S_{BET} value was very close to the S_t value, only a small content of micropores should be expected according to the results obtained from the *t*-plot method. This sample showed the highest value of the total pore volume, what corresponds with the measured isotherm.

The pore size distribution curves of the studied samples were calculated using the BJH method from the desorption isotherms (Fig. 2). The sharp maximum at the distribution curves of clay samples (at about 1.9 nm) relates with the skip on the desorption isotherm associated with the so-called forced closure of the hysteresis loop and does not respond to real pores [25]. The B and FB samples showed a wider distribution in the range from 4.6 to19 nm, with a maximum at 6 and 9 nm, respectively. A quite narrow distribution of MP with a Gaussian character and a maximum at 11.4 nm pointed at the presence of interparticle spaces with a diameter from 3.2 to 24 nm.

As(V) adsorption

The study of pH on the sorption capacity of adsorbents for individual ions is important from

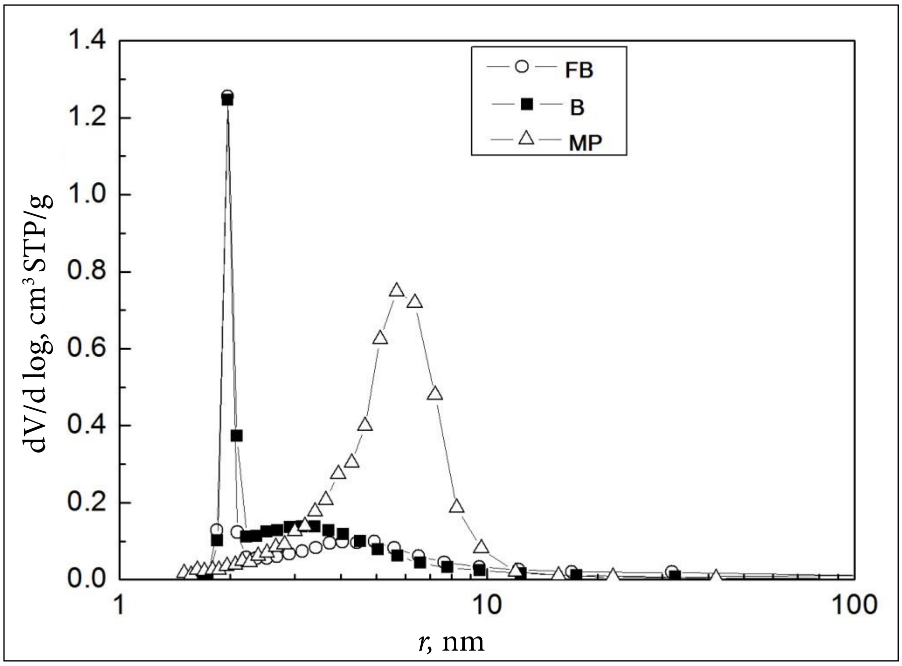

Fig. 2. Pore size distribution curves of the studied bentonite samples and magnetic particles (STP – standard temperature and pressure – 0 °C and 101325 Pa)

the point of sorption processes. As(V) ions are present in the water solutions in different forms depending on pH of the solution. Additionally, the value of pH affected the ionization degree of functional groups, the surface charge of the adsorbent and, finally, the interaction between the metal ions and the adsorbent. In our previous study, the natural bentonite showed the highest equilibrium adsorption capacity at pH 4. As it can be seen from the Zeta potential measurement, the zero point charge of the B and FB sample is at 4 and 3.5, respectively (Fig. 3). In the solution of lower pH, the dissolution of Fe ions occurs as well as for clay samples as for the magnetic particles. From this reason the batch experiments were performed at pH 4 with the exception of MP higher adsorption capacity.

The clay samples did not show an expressive As(V) uptake in comparison to MP (Fig. 4), what corresponds with the result from the zeta potential measurement. Comparing the natural adsorbents, Fe-enriched bentonite showed higher equilibrium adsorption capacity in the solutions with a lower concentration (up to 30 mg As(V)/L). By increasing of initial ions concentration an expressive ions uptake was observed for the B sample, while for FB the adsorption equilibrium was reached.

Adsorption isotherms are mathematical models which are used for the study of the adsorption

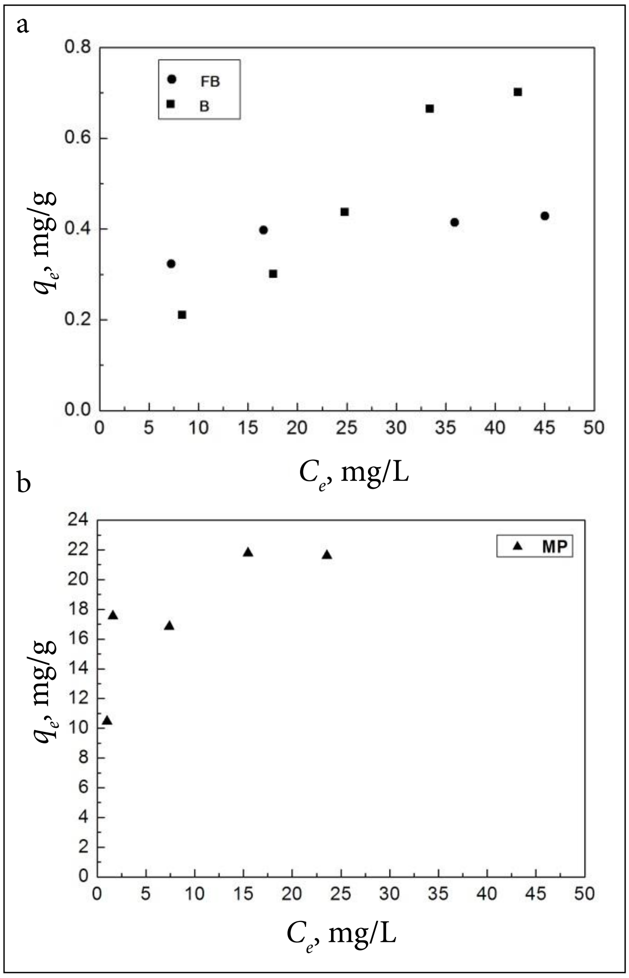

Fig. 4. Adsorption isotherms of the As(V) adsorption onto the bentonite samples (a) and magnetic particles (b)

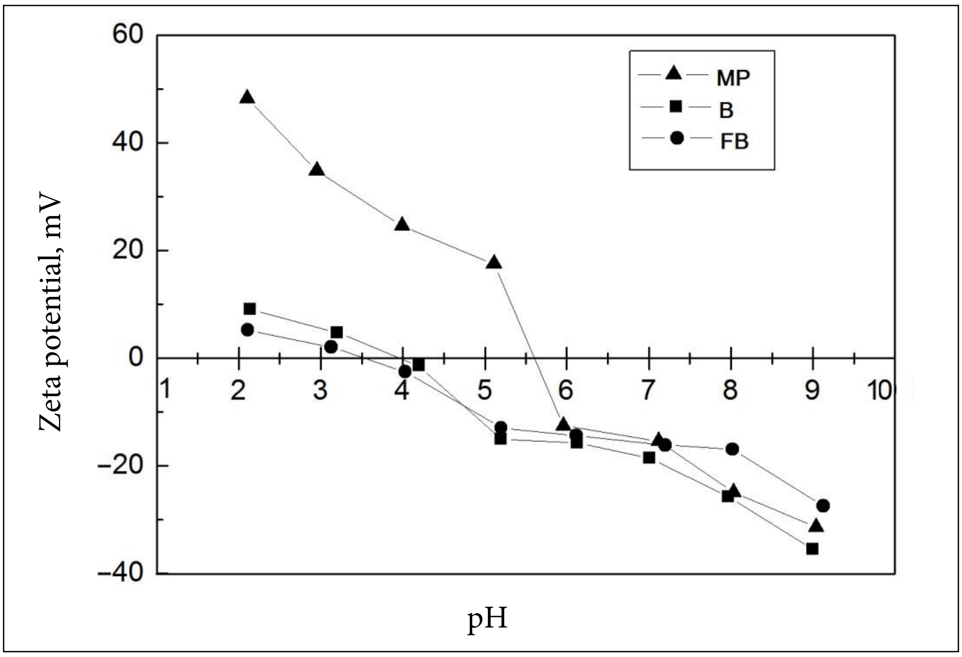

Fig. 3. Zeta potential of the studied bentonite samples and magnetic particles in dependence on pH

mechanism and affinity between the adsorbent (with defined surface properties) and adsorption molecules. Two basic models (Langmuir and Freundlich) were applied for the simulation of adsorption isotherms for the studied adsorbents (Fig. 5). The obtained data of the adsorption equilibrium for As(V) calculated from the linearised Langmuir and Freundlich models are listed in Table 3. From the applied models, the Langmuir better described the adsorption onto the MP and FB sample. The theoretical value of the maximum sorption capacity equal to 23 mg As(V)/g was calculated for the MP. The natural materials did not show so good adsorption properties towards As(V) in the batch experiments, they should be used effectively as column fillers to fix finer magnetic particles. On the other hand, whereas the leachates after the soil bioremediation also contain other toxic elements, their selectivity towards Cu(II) and Zn(II) ions could be beneficial for the leachates regeneration.

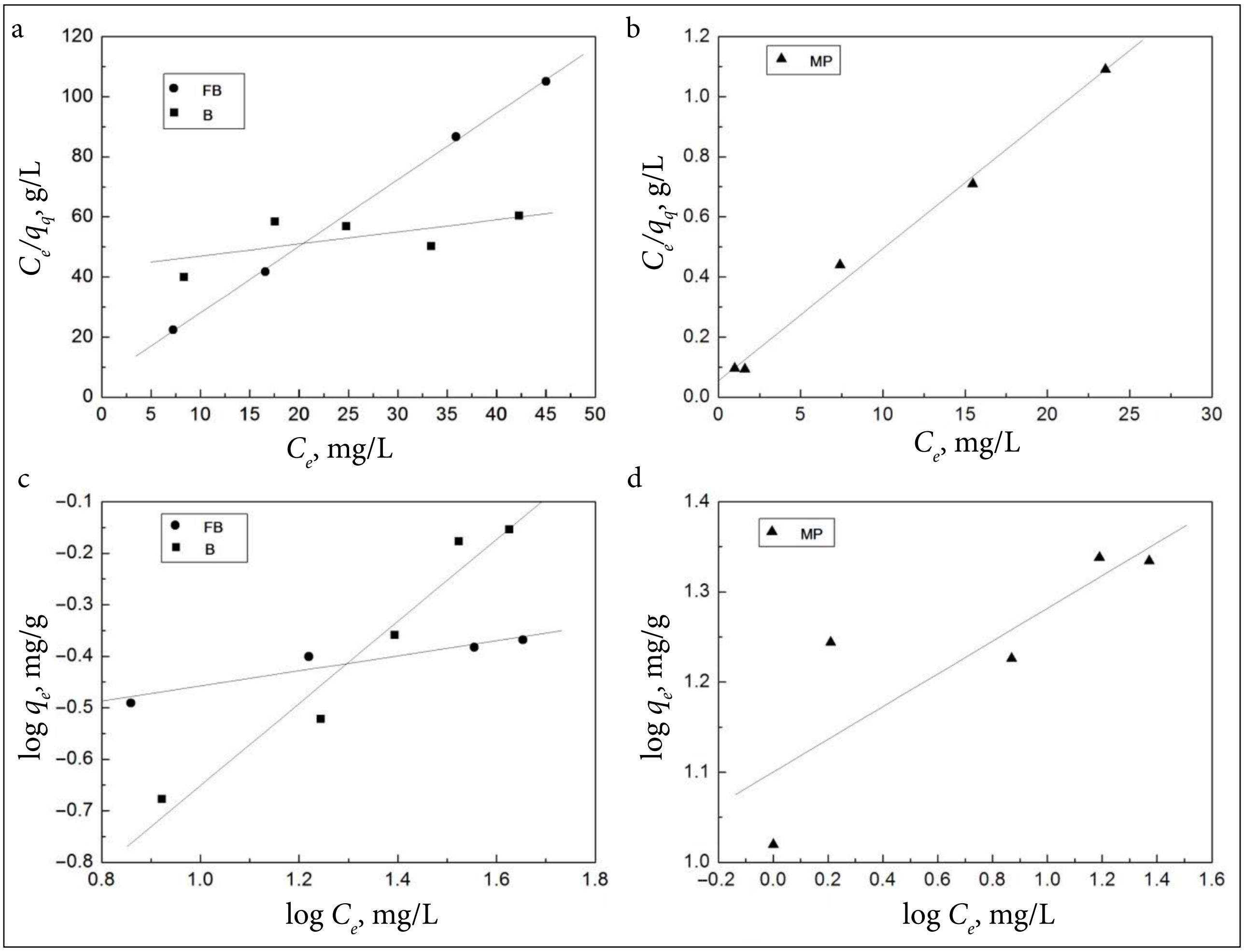

Fig. 5. Linearized Langmuir isotherms of the As(V) adsorption onto (a) bentonite samples and (b) magnetic particles and linearized Fruendlich isotherms of the As(V) adsorption onto (c) bentonite samples and (d) magnetic particles

Table 3. **Langmuir and Freundlich parameters**

Sample	**Langmuir parameters**			**Freundlich parameters**		
	Q_0, mg/g	***b*, L/mg**	**R^2**	**K_f, L/g**	***n***	**R^2**
FB	0.45	0.374	0.9994	0.249	0.714	0.9073
B	2.49	0.009	0.4061	0.0356	0.160	0.9538
MP	22.73	0.818	0.9927	12.595	0.658	0.7111

Column study

Column experiments were performed to study the effect of certain parameters like bedding of samples and their behaviour in columns consisting of different adsorbents as well as of different fractions of natural clay for removal of As(V) from the aqueous solutions. First, the natural bentonite and Fe-enriched bentonite samples were compared. The model solution with the concentration 30 mg As(V)/L was percolated through the columns twice. After the second cycle, the As(V) concentration was lower than 2 mg/L for both columns. However, due to the fine particles of the natural samples the percolation was very slow, retarded, and the flow rate was not continual. In the second series, quartz sand and synthetised iron oxide were added into the columns. Quartz sand is a common and suitable material for column study due to its permeability and it is also able to fix the fine clay or magnetic particles in the column. As was shown above, the magnetic particles showed good adsorption properties in batch experiments. They were used as a slim bedding to enhance the As(V) removal. The columns were filled with 2 g of B/or FB with a layer of QS and MP (together 5 g). Two percolation cycles with solutions of 50 and 100 mg As(V)/L were performed. From this study, more effective was the column filled with B where the As(V) uptake reached 100%, while the FB sample only 74% after the second cycle (Fig. 6), against the expectation that the FB sample will be more effective by the reason of a higher Fe content. Comparing the results obtained from the batch and column experiments it can be concluded that a lower adsorption capacity of the FB sample is caused by the presence of amorphous Fe forms on the clay surface which filled the pores and reduced the potential adsorption sites on the surface. These results correspond with the above-discussed textural analysis, where the B sample also contained a higher volume of micropores and its value of the external surface was higher in comparison with the FB sample.

To differentiate the contribution of particular adsorbents in the column to the As(V) removal, the third series of columns were set. The columns filled with QS, QS/B, QS/MP and QS/B/MP were tested and compared. To restrain the column clogging by fine clay particles, a higher fraction of the B sample was used: 0.5–1 mm. Only one cycle with a solution of 100 mg As(V)/L was performed. The column filled with pure QS and QS/B removed only 2.4% and 5.14% of As(V), respectively, the effect of QS/MP was 34.9%. The combination of all studied adsorbents QS/B/MP was most effective in the As(V) removal and reached 77.8% of removed As(V)(Fig. 7). This is a smaller

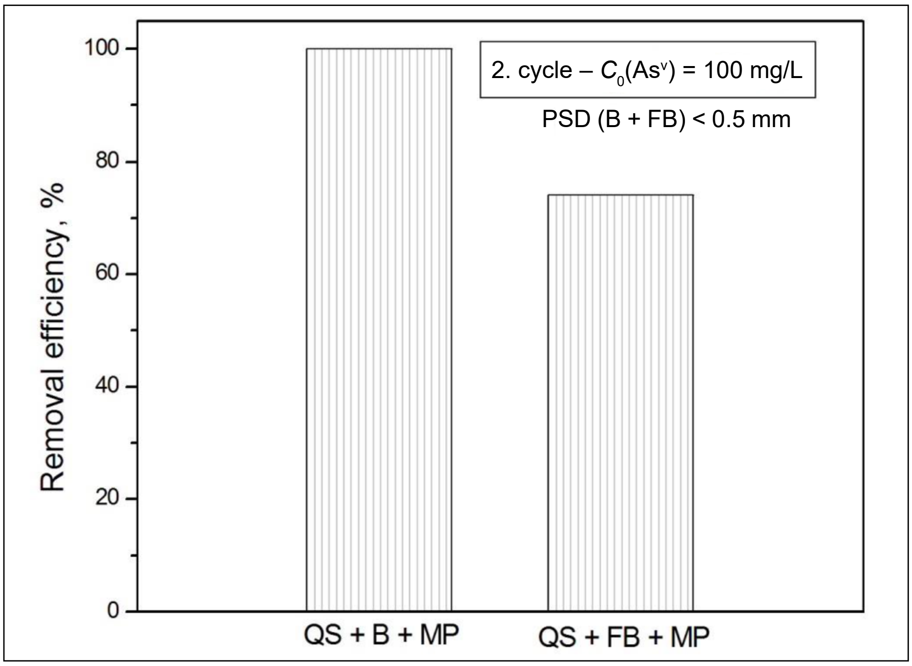

Fig. 6. Effect of the As(V) adsorption onto the quartz sand/clay samples with the clay fraction <0.5 mm and magnetic particles

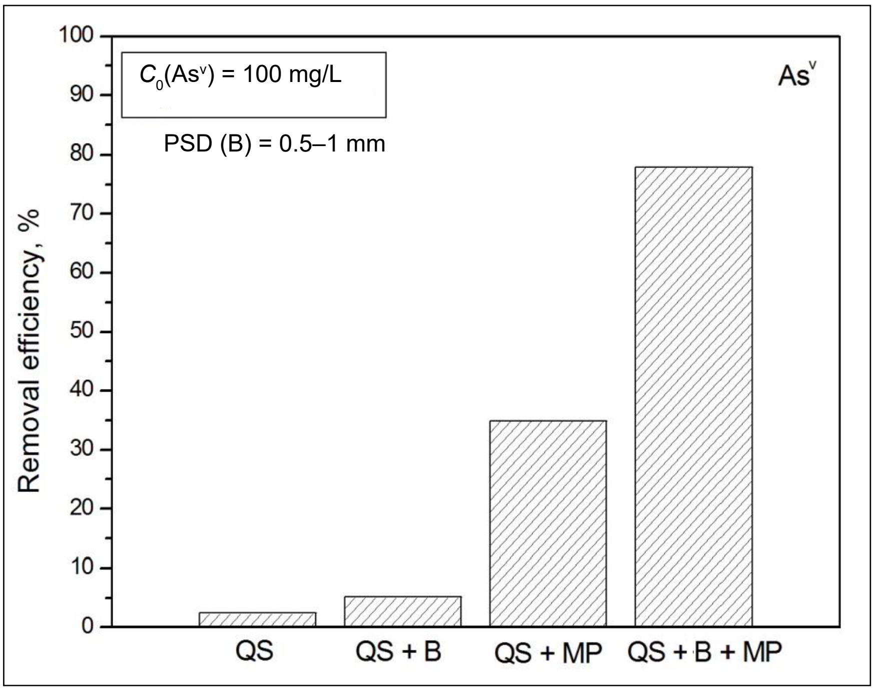

Fig. 7. Effect of the As(V) adsorption onto particular adsorbents and their combination with the clay particle size 0.5–1 mm

uptake comparing the experiment with a finer B fraction (Fig. 6). On the other hand, the percolation was continual and the column was not clogged. These experiments showed that the particle size is also an important parameter that influences the adsorption properties of studied materials.

Based on the presented results two most effective columns were selected for the second percolation. The solution concentration was 100 mg As(V)/L again. While the adsorption effect of the QS/MP column decreased at 36%, for the QS/B/MP column it was about 24% (Fig. 8).

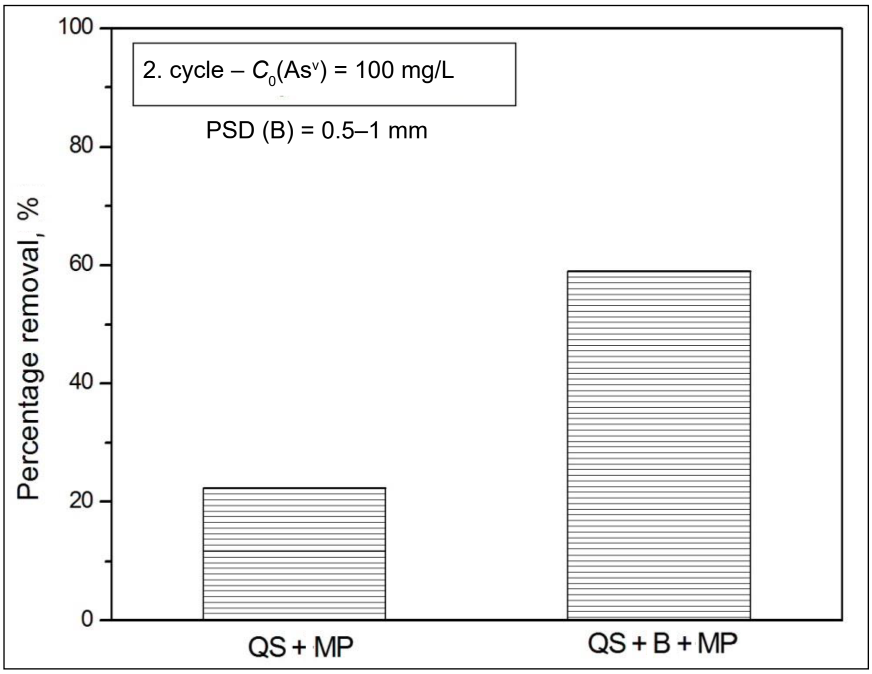

Fig. 8. Effect of the As(V) adsorption onto the quartz sand/magnetic particles and quartz sand with clay/magnetic particles after the 2nd cycle of percolation

CONCLUSIONS

The adsorption properties of natural bentonite, Fe-enriched bentonite and synthetised iron oxide towards As(V) was studied. The batch experiments pointed at the low adsorption capacity of natural materials. The natural bentonite sample was more effective in the As(V) removal as compared to Fe rich bentonite, showed a higher volume of micropores and a higher value of the external surface area. The best adsorption properties towards As(V) showed synthetised magnetic particles. The column studies performed by different type of beddings indicated that the particle size is an important factor and influences the adsorption capacity of bentonite. The column filled with QS/B/MP with a particle size of bentonite below 0.5 mm removed 100% of As(V), while the column with a particle size of bentonite between 0.5–1 mm reached 77.8% of As(V) uptake in the first cycle. On the other hand, a fine fraction of bentonite affected the clogging of the column and flow continuity. These introductory experiments proved that it is suitable to combine the clay adsorbents with natural materials such as quartz sand as fillers in columns because of their permeability as well as to fix finer magnetic particles to enhance the column effect in As(V) removal. Additionally, natural clays were proved as good adsorbents of other toxic elements such as Cu(II) and Zn(II) [26]. The combination of quartz sand, clays and magnetic particles in the column beddings was most effective also after the 2nd percolation of the media of higher As(V) concentration. A clay filler could be an advantage in the treatment of leachates containing also Cu(II) and Zn(II) from the contaminated soil after the bioremediation process.

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EKSPERIMENTINIS As(V) ADSORBCIJOS ANT SKIRTINGŲ NEŠĖJŲ TYRIMAS

Santrauka

Tirta As(V) adsorbcija iš vandeninių tirpalų ant gamtinio bentonito (B), geležimi praturtinto gamtinio bentonito (FB) ir sintetinių magnetinių dalelių (MP). Nustatyta, kad stacionariomis adsorbcijos sąlygomis MP yra našesnis adsorbentas už abu gamtinius adsorbentus. Didžiausia jo adsorbcijos geba siekia 23 mg/g. Kita vertus, adsorbcijos kolonėlėje sąlygomis visi tirtieji adsorbentai pasižymėjo panašaus dydžio adsorbcine geba.