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Natural Diatomite (Rudovci, Serbia) as Adsorbent for Removal Cs from Radioactive Waste Liquids

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Abstract:

The removal of Cs (I) ions from aqueous solution was studied using natural diatomite as adsorption materials originated from Rudovci, Serbia. The microstructure of natural diatomite has been characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) while the degree of Cs adsorption was evaluated by atomic emission spectroscopy. The cation exchange capacity (CEC) values for natural diatomite was 50 meq/100g. Depending on whether the Cs adsorption occurred in the acidic and alkaline media at a temperature of 298.15 K in acidic media ΔG^0 values was -12.674 kJ/mol, while in alkaline media ΔG^0 was -13.142 kJ/mol and a change of ΔS^0 to 42.51 J/molK in acidic media and 44.08 J/molK in alkaline medium.

Keywords: Diatomite; Cesium; Adsorption; Radionuclides; AFM

1. Introduction

¹³⁷Cs, with a half-life of 30.17 years, is currently the largest source of environmental radioactivity generated and released from the nuclear weapon tests and Chernobyl accident. Due to its high solubility, it incorporated into aquatic ecosystems and in dissolved form and in contacts with sediment and soil material it is rapidly and strongly adsorbed onto the solid phase. The binding of Cs to clay minerals has been reported to be so strong as to be nearly irreversible [1]. The "preferential" retention of Cs in soils and sediments is believed to take place in a small number of highly selective sites located at the frayed edges of the clay particles [2]. This preference has been attributed to the large ionic radius and uncomplexing nature of Cs, but most importantly to its low hydration energy [3,4]. Research and development of effective and low-cost heavy metals and radionuclide sorbents is very important in the fields of environmental and health protection [5]. Natural materials of both organic and inorganic nature (chitosan, zeolites, clay and clay minerals, etc) and certain waste

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products from industrial operations (fly ash, coal and oxides) were investigated as low-cost adsorbents in previous studies [6-8]. A number of sorbents (mainly with tunneled and layered structures) are currently being developed for removing ^{137}Cs and/or ^{90}Sr from highly alkaline tank wastes, acid wastes and groundwater [9-11].

Between different natural materials, diatomite has a unique combination of physical and chemical properties (high bulk, good absorbency, chemical inertness and large surface area), which makes it applicable as an adsorbent for the pollutants and as flocculants used in waste water treatment [12-16]. Diatomite is fine-grained, low-density biogenic sediment, which consists essentially of amorphous silica ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) derived from opalescent frustules of diatoms. Due to the presence of silanol groups, spread in silica matrix, diatomite can react with many polar functional groups [17]. Also, due to the adsorption properties [18-21] it has been commonly used in water purification, clarifications of liquors and juices and for separation of various oils and chemicals [22] and waste waters filtration [23]. Furthermore, it was found to be a promising agent for immobilization of uranium [24] cesium and cobalt radionuclides [25], The adsorption phenomena of Cu^{2+} ions onto diatomite have been investigated by Sljivić et al. (2009) [26]. Also, diatomite and modified diatomite have been tested as possible adsorbents for removal of Pb^{2+} ions from solutions [27].

To date, the removal of Cs radioisotopes from radioactive waste effluents has been relied largely on ion exchange methods. However, other monovalent cations, especially Na^+ and K^+ can considerably impede Cs^+ adsorption [28]. As an alternative, Osmanliogly [14] treated radioactive liquid by using the natural Turkish diatomite and its radioactivity was reduced to 15% of its original activity. His investigations showed that properly diatomite media can be used for removal of ^{137}Cs , ^{134}Cs and ^{60}Co from radioactive liquids.

The aim of this work was to determine the efficiency of natural raw diatomite originated from Rudovci, Serbia for the removal of cesium from aqueous solutions under kinetic and equilibrium conditions. This area is situated near the mining Kolubara, the largest open pit coal mine in this part of Europe.

2. Materials and methods

2.1. Physic-chemical characterization of diatomite

The elemental composition analysis of natural raw diatomite (Rudovci, Serbia) was done by XRF (type UPA XRF 200) and modified forms expressed as weight percentages of metal oxides.

The mineral phases compositions of the materials were characterized by X-ray powder diffraction technique by a Siemens D-500 XRD powder diffractometer with CuK_α radiation (in conjunction with CuK_β nickel filter).

The morphology and microstructure of the materials were studied by using scanning electron microscopy (SEM) technique. SEM micrographs were performed by a JEOL JSM 6460LV and Oxford Instrument INCA-X-sight at 25kV scanning electron microscope. The materials were introduced onto a conductive carbon tape and coated with gold to prevent charging. The powdered samples were dispersed in ethyl alcohol and then left to settle on a disc. After drying, each was coated with thin film of carbon using a vacuum evaporator.

The particle size distribution was determined by the particle size analyzer (PSA) Mastersizer 2000 (Malvern Instruments Ltd., UK). For the particle size measurements, the powder was dispersed in water in an ultrasonic bath (frequency of 40 kHz and power of 50 W), for 5 min.

2.1.1. Naturally occurring radionuclides in the diatomite

Since the diatomite was meant to be used for sorption of cations-activity carriers, complete radiological analysis was performed. The content of radionuclides in diatomite samples were determined gamma spectrometrically by means of semiconductor HPGe spectrometer (GEM30, Ortec with relative efficiency of 37 % and resolution of 1.7 keV at 1332.5 keV). The sample was prepared in accordance with IAEA recommendations IAEA, 1998 [29] and sealed in order to reach radioactive equilibrium. After six weeks sample was measured for 370 ks. A recorded spectrum was analyzed by Genie2k, Canberra's software. Net areas of the peaks were corrected for the background, dead time and coincidence summing effects, applying the calculation method of Debertin and Schötzing [30].

2.2. Cation exchange capacity of diatomite

The cation exchange capacity (CEC) of the diatomite was determined by using a standard procedure that is applicable to both calcareous and noncalcareous soils (EPA, 1986) [31]. The sample was mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations with the matrix cations. Subsequently, the sample was washed with isopropyl alcohol. An ammonium acetate solution was then added, and the adsorbed sodium was replaced with ammonium.

The concentration of displaced sodium was determined by Dionex ion chromatograph instrument. Those cationic species were separated with cationic column Dionex IonPac CS 12 (10-32) and determined using suppressed conductivity detector. The eluent was 0.02M methansulfonic acid, and eluent flow was 0.7 mL/min. The concentration of potassium was determined by using AAS (Aanalyst 700/Perkin-Elmer).

2.3. Adsorption of Cs

2.3.1. Chemical analysis of Cs

The content of Cs in solutions was determined by using the flame atomic emission spectroscopy (AAS Aanalyst 700/Perkin-Elmer). Standard solution (Merck, Darmstadt, Germany) and 18.2 MΩ water were used for preparing calibration standards.

2.3.2. Immobilizations of cesium on diatomite

Raw diatomite was contaminated at 25°C with a Cs (NO₃) (99.99% Alfa Aesar) solution and concentration of 21.47 mg/l. Each sample was dissolved in 10 ml of that solution in the flasks. Equilibrium state of solution was established after a sealing and shaking for 24 h. The concentrations of Cs in the diatomite phase at equilibrium were determined using ICP method (Montinaro et al, 2008). Concentrations in the diatomite phase after 24 h of contact $q_{pb(II)}^{24h}$ (mgkg⁻¹), immobilization efficiency $\eta(t)$ in %, after each treatment interval and the distribution coefficient, K_d , were calculated through the mass balance which is presented in our earlier researching (Nenadovic et al, 2009).

2.4. Thermodynamic study

The thermodynamic (ΔH^0 , ΔS^0 and ΔG^0) for Cs sorption onto raw diatomite can be determined from the temperature dependence of Cs ions concentration. Free energy changes (ΔG^0) were calculated from the relationship:

$$\Delta G^0 = -RT \ln K^0 \quad (1)$$

where, R is the universal gas constant (8.314 J/mol/K), T is the temperature in Kelvin and K^0 is the sorption equilibrium constant. Values of $\ln K^0$ were obtained by extrapolating the $\ln K_d$ vs. C_{eq} graph to zero [32].

Standard entropy changes were calculated by using the equation:

$$\left(\frac{\partial \Delta G^0}{\partial T}\right)_P = -\Delta S^0 \quad (2)$$

The average standard enthalpy change (ΔH^0) was then calculated from the relationship:

$$\Delta H^0 = \Delta G^0 + T\Delta S^0 \quad (3)$$

3. Results and discussion

3.1. Characterizations of diatomite

The diatomite sample used in this study originates from Rudovci, Serbia which is situated near mining basin "Kolubara". Its chemical composition, obtained by XRF spectroscopy, was: 79.72 % SiO_2 , 10.20 % Al_2O_3 , 1.31 % K_2O , 2.51 % Fe_2O_3 , 0.025 % ZrO_2 , 0.05 % CaO and 0.03 % TiO_2 , while the main constituent minerals, determined by X-ray diffraction (XRD), were quartz, muscovite, kaolin and enstatite.

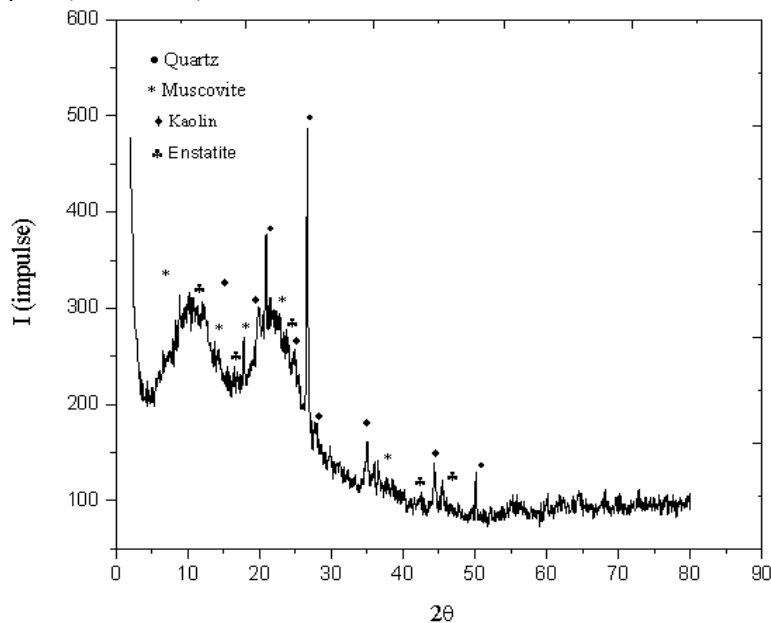


Fig. 1. XRD pattern of diatomite.

Fig. 1 shows the X-ray powder diffraction (XRD) pattern of natural diatomite. The diffraction pattern indicated that the diatomite consists mainly of silica (SiO_2) with small amounts of Al_2O_3 , Fe_2O_3 , CaO and Na_2O . The most abundant phase in the sample were the mineral quartz, muscovite mica group minerals and clay minerals group (mostly kaolin). Minerals of pyroxene-enstatite group were present in small quantities. The presence of amorphous phase was significant in the area of 4° to 20° 2θ and it is probably due to the glass

formation of SiO_2 . The characteristic peaks of SiO_2 appeared at 19° , 21° , 26° and 35° . The XRD showed that the diatomite is poorly crystallized. The structure of the sample was quite similar to that reported by Koseoglu et al [33].

The morphology and microstructure of the natural diatomite are presented in Fig. 2. Diatomite frustules are mainly divided into two categories: centric (discoid) and pinnate (elongated to filiform). The plate indicates both centric and pinnate types of diatom. The centric diatom has a radius of approximately $10\mu\text{m}$, while the length of the pinnate shape is greater than $20\mu\text{m}$. It can be inferred from the scanning micrograph that diatomite has a large void volume, in addition to its highly porous structure. The high porosity of this material was one of the main reasons for choosing it as a potential sorbent for cesium.

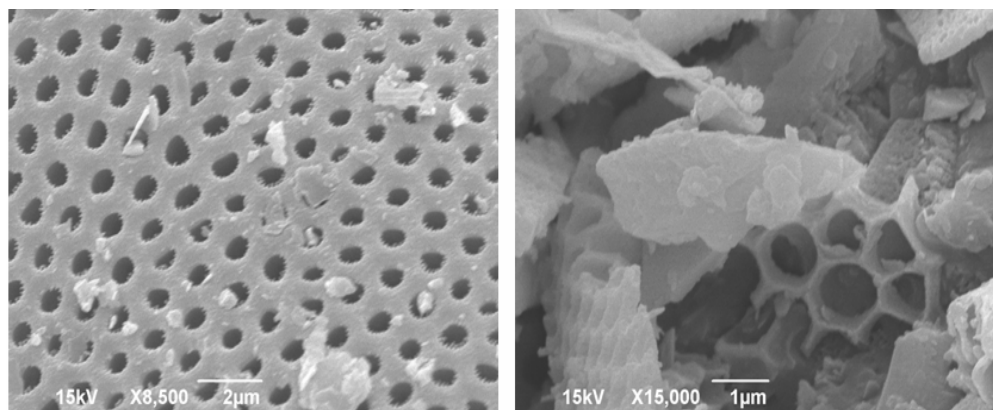


Fig. 2. SEM microphotography of natural diatomite.

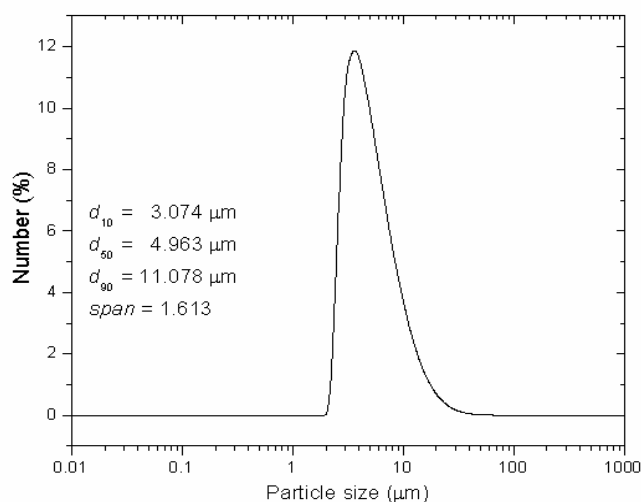


Fig. 3. Particle size distribution of diatomite.

Results of the particle size distribution, based on a number of the analyzed diatomite samples, are presented in Fig. 3. It should be mentioned that the correctness of the measurement depends on the degree of the powder dispersion. So, 5 min after the powder dispersion in water, with the aid of low-intensity ultrasound, the following results were obtained: in K-type (raw diatomite) the particle size distribution was relatively narrow (span = 1.613), 10 % of particles ($d_{0.1}$) have diameter smaller than $3.074\mu\text{m}$, 50 % of particles ($d_{0.5}$) have a diameter of $4.963\mu\text{m}$, while 90 % of particles ($d_{0.9}$) are smaller than $11.078\mu\text{m}$.

One of the important physical properties of the diatomite is its natural radioactivity. The specific activities of U-series, Th-series, ^{235}U , ^{137}Cs and ^{40}K obtained by gamma spectrometry, are given in Tab. I. Quoted uncertainties (the confidence level of 1σ) were calculated by error propagation calculation. The combined standard uncertainties included the efficiency calibration uncertainty and the statistical uncertainties of the recorded peaks. Results show that secular equilibrium in uranium series is preserved in the sample, that activity ratio of uranium isotopes $^{235}\text{U}/^{238}\text{U}$ is natural and that artificial radionuclides were not detected in the sample. Minimal detectable activity concentration of ^{137}Cs , for performed measurement was 0.04 Bqkg^{-1} . The radiological analysis of diatomite from Rudovci region (Serbia) confirmed that it does not contain ^{137}Cs , which is the main recommendation for its usage as a potential material for the removal of ^{137}Cs isotopes from radioactive effluents.

Tab. I Specific activities of diatomite

Sample	Activity concentrations (Bqkg^{-1})				
	U-series	^{235}U	^{40}K	^{137}Cs	Th-series
D	37 ± 4	1.6 ± 0.3	240 ± 20	< 0.04	56 ± 4

3.2. Adsorption process of diatomite

The main adsorption mechanism, involved in cation removal by diatomite is exchange with exchangeable cations such as Ca^{2+} , Mg^{2+} , K^+ and Na^+ [34]. The cation exchange capacity (CEC) of these materials is a measure of the number of negatively charged sites that attract exchangeable cations. CEC values for diatoms are $50 \text{ meq}/100\text{g}$. The negative charge caused by substitutions within the lattice structure is permanent and pH independent, whereas the charges on the edges and the exposed hydroxyls surface depend upon pH. At the same initial pH, increasing Cs concentration diminished the relative amounts adsorbed.

3.2.1. Effects of pH

The adsorption of Cs was investigated at different values of pH ranging from 2.07 to 10.71. In acidic solutions (pH 2-4) the less significant Cs adsorption was due to competitive adsorption of H^+ ions. The sharp increase of the adsorbed amount of Cs at $\text{pH} > 6$ was due to precipitation of cesium hydroxide. The general trend shows that the uptake percentage of Cs does not change with the change in solution pH. This means that the adsorption of Cs is highly independent on the surface characteristics of the diatomite materials at various pH values. The independence of the distribution coefficient (K_d) on the solution-pH is probably due to the considerable weak hydrolysis of monovalent Cs ion, which might originate from the lack of affinity of Cs (I) towards the hard oxygen atom present in the aluminosilicate diatomite structure.

3.2.2. Adsorption kinetic study

Dependence of Cs (I) adsorption on the natural diatomite of the contact time is shown in Fig. 6. Adsorption isotherm is mainly used to describe adsorption equilibrium, which reflects the degree of interaction between the quantities of adsorbate on the adsorbent. Hence, the maximum affinity of the sorbent at constant temperature and pH could be evaluated. In this respect, the sorption isotherm for the removal of Cs ion from contaminated solutions on the different samples of natural diatomite. Various concentrations of non-radioactive CsNO_3 ranging from 2.07 mg/l to 21.47 mg/l were used as carrier for Cs. Fig. 4 shows that the isotherms are regular concave curves to the concentration axis. The amount of adsorbed Cs

increases rapidly at the low concentration range, and then increases gradually at high Cs ion concentrations. These results reflect the efficiency of the diatomite materials towards Cs nuclide.

Adsorption kinetics of cesium from the aqueous solution onto the diatomite reaches its equilibrium in time, as it is presented in Fig. 4.

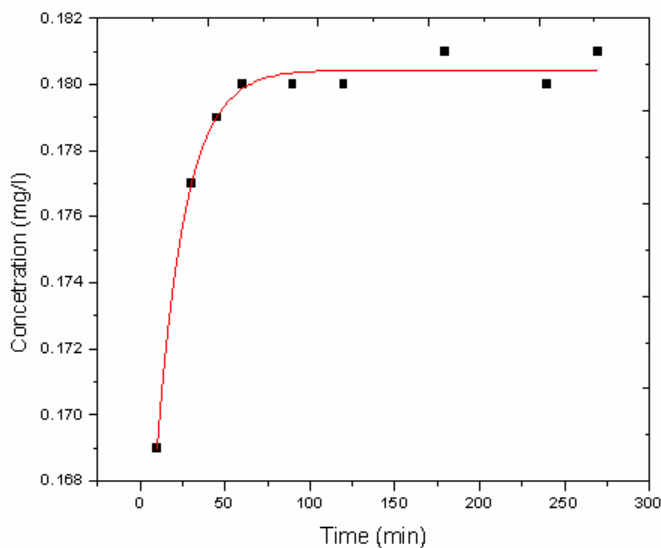


Fig. 4. Cs adsorption as function of time in a) acidic medium and b) alkaline medium.

As one can see in Fig. 6a, adsorption of Cs (I) increased very quickly at the initial contact time. After 60 minutes from beginning of the adsorption process the maximum amount of cesium adsorbed was achieved. In the next stage of the process (for 100 to 120 minutes after start), we could observe desorption (about 25%) of cesium previously adsorbed by the diatomite. This inversion period of adsorption may have been caused by competition between the exchangeable cations and hydrogen ions from the diatomite surface with cesium ions from the solution. The cesium adsorption increased later, but the amount of the adsorbed cesium was significantly lower (about 20%) compared with the first period of the fast adsorption.

The kinetic curve of cesium adsorption onto the diatomite in alkaline medium is shown in Fig. 6b. It can be observed that about 95% of cesium was removed for the first 60 minutes of the adsorption process. The inversion phenomenon with desorption prevalence (near 10%) in this case was apparent from 90 to 120 minutes of the adsorption. Cesium adsorption slowly increased with time reaching a plateau after 250-300 min of the process duration.

3.2.3. Effects of immobilization

Fig. 5 represent the dependence of the immobilization efficiency (η) of initial Cs concentration in acidic and alkaline medium.

The minimum value of the Cs immobilization effect (62%) was for 21.47 mg/l of $\text{Cs}(\text{NO}_3)$ solution in acidic media while the greatest immobilization effect (96.9 %) was in alkaline media (with 2.07 mg/l $\text{Cs}(\text{NO}_3)$ concentration). The effect of immobilization increases with decreasing concentration, therefore the concentrations ratio is very important for the efficiency of immobilization. Compared with acidic media, the effect of immobilization in alkaline medium is higher and less dependent of initial concentration of Cs.

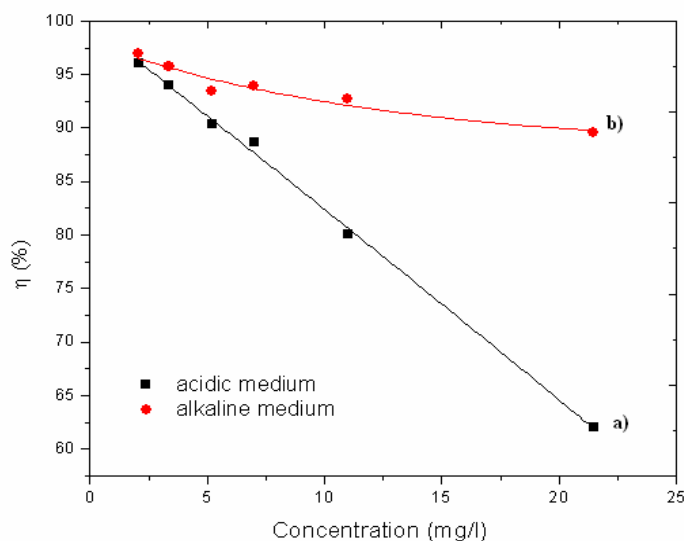


Fig. 5. Immobilization effect dependence of the initial Cs concentration in different media a) acidic and b) alkaline.

3.3. Thermodynamic parameters

The distribution coefficient K_d is a function of many factors such as the mineralogical and chemical composition of the solid media, and the chemical composition of the liquid transport media. Many studies in which the distribution coefficient K_d value was derived placed emphasis on clay minerals for their negative electricity and high cation exchange capacity (CEC) [35].

Based on the Equation (1), and linear fit of $\ln K_d = f(C_{pb(II)}^{24h})$ [32] value of $\ln K^0$ (see Fig. 8) and other thermodynamic parameters (ΔG^0 and ΔS^0) for Cs(I) sorption onto diatomite have been obtained.

Depending on whether the Cs adsorption occurred in the acidic and alkaline media different values of thermodynamic parameters were obtained. At a temperature of 298.15 K in acidic media ΔG^0 values was -12.674 kJ/mol, while in alkaline media ΔG^0 was -13.142 kJ/mol. There was a change of ΔS^0 to 42.51 J/molK in acidic media and 44.08 J/molK in alkaline medium.

The evaluation of thermodynamic parameters provides an insight into the mechanism of Cs sorption on diatomite. The free energy of Cs sorption on diatomite is more negative at 298.15 K, which suggests that the spontaneity of the process increases with the rise in temperature. A positive value of the standard enthalpy change (ΔH^0) indicates that the sorption is endothermic. The low value of ΔH^0 also suggests that the endothermic process of Cs sorption on diatomite is weak. This conclusion is supported by the weakly increasing sorption with the increase in temperature. Since the free energy changes are negative and accompanied by positive entropy changes, the sorption reactions are spontaneous with a high affinity.

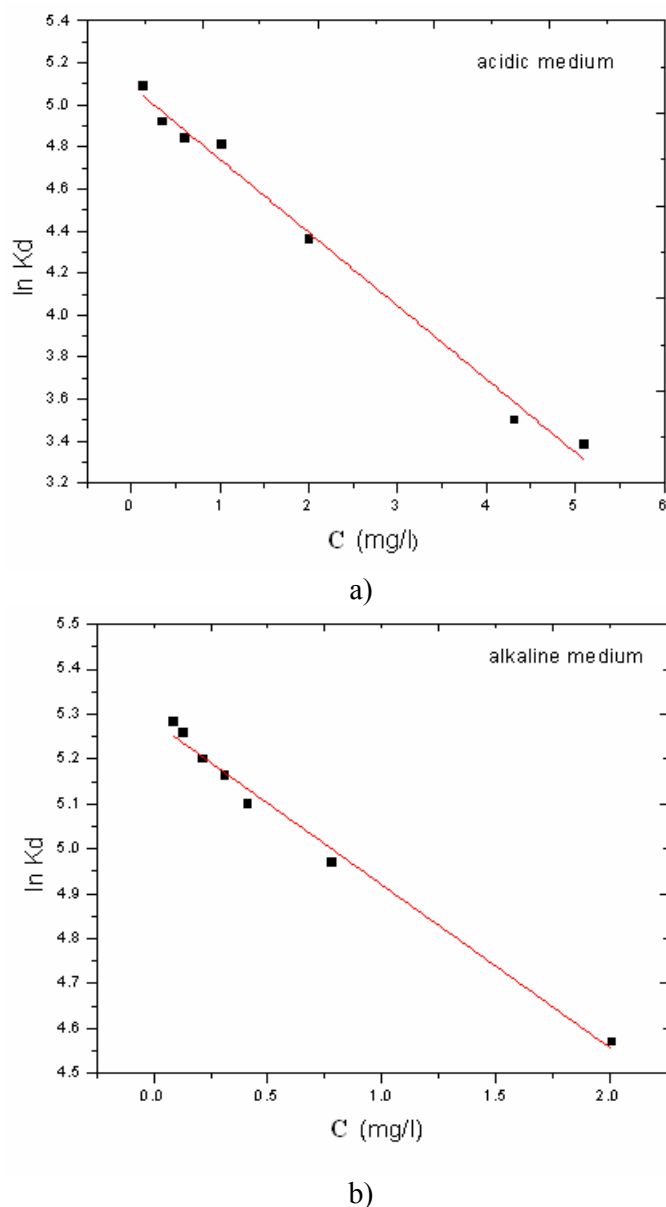


Fig. 6. The distribution coefficient, K_d , vs. C_s concentration in a) acidic medium and b) alkaline medium.

4. Conclusions

Comprehensive investigation of diatomite properties, regarding the Cs immobilization from liquids, was conducted and presented in this paper. The adsorption characteristics of cesium metal ion are strongly affected by initial metal ion concentration. Kinetic experiments clearly indicate that adsorption of cesium metal ion on diatomite samples is more or less a two step process: a rapid adsorption of cesium metal ion to the external surface followed by inter-particle diffusion into the interior of adsorbent.

Sorption for diatomite samples are lower than 50 kJ/mol indicating that the sorption processes were controlled by physical mechanism rather than chemical and process is endothermic and spontaneously. The heat of physical adsorption involves relatively weak intermolecular forces such as van der Waals and mainly electrostatic interaction. Obtained data can be used for optimization of the routine purification processes of liquids that involves adsorption of Cs on diatomite.

5. Acknowledgement

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6. References

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Садржај: Природни дијатомеј који води порекло из Рудоваца, Србија је коришћен као адсорпциони материјал за уклањање Cs (I) јона из водених раствора. Микроструктура природног дијатомеја је карактерисана рендгенском дифракцијом (XRD) и скенирајућом електронском микроскопијом (SEM), док је степен адсорпције Cs испитиван атомском емисионом спектроскопијом. Вредност капацитета катјонске измене (CEC) за природни дијатомеј је 50 meq /100 g. У зависности да ли се адсорпција Cs дешава у киселој или базној средини на температури од 298.15 K, вредност ΔG^0 у киселој средини је била -12.674 kJ/mol, у базној ΔG^0 је -13.142 kJ/mol, док је у киселој средини ΔS^0 42.51 J/molK, а у алкалној средини 44.08 J/molK.

Кључне речи: Дијатомеј; Цезијум; Адсорпција; Радионуклиди; АФМ
