

SORPTION OF ARSENATE BY STANNUM(IV)-EXCHANGED ZEOLITE P

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Abstract

Zeolites are crystalline, hydrated aluminosilicate containing exchangeable alkaline and alkaline earth cations in their structural frameworks. Previous studies have shown that modification of zeolites with an active metal cations yield sorbents with a strong affinity for certain anions. Recently hydrous tin oxide was found effective to remove arsenic anions from aqueous solution. In this study, sodium ion of zeolite P was exchanged with stannum(IV) and used for sorption of arsenate (As(V)) ion. Among parameters investigated were effect of pH, As(V) initial concentrations, contact time, temperature and effect of foreign ions. The results show that As(V) sorption by stannum(IV) exchanged zeolite P (SnZP) was maximum at about pH 2. The sorption capacities increase with increasing initial As(V) concentrations and follows Langmuir model with maximum sorption capacity of 83.33 mg/g at 25°C. The sorption capacity of As(V) by SnZP increased as temperature increases from 25 to 70°C showing that the process is endothermic with ΔH° value of 83.84 kJ mol⁻¹. The free energy change (ΔG°) for the sorption was negative showing that the sorption of As(V) ions is spontaneous. The kinetics study shows that the As(V) sorption follows second order kinetic model. The free energy (E) was 12.91 kJ mol⁻¹ which shows the sorption is an ion-exchange process. The As(V) sorption by SnZP was not affected by the presence sulphate, chloride, nitrate and carbonate but was significantly reduced by phosphate.

Keywords: Stannum-exchanged zeolite P, arsenic removal, kinetic, thermodynamic.

Introduction

Environmental pollution of arsenic from industrial wastewater streams as a consequence of the industrialization process is one of the major problems that have to be solved or controlled. Many industrial activities such as mining, combustion of fossil fuels, and the production of glasses, nonferrous alloys, semiconductor materials, arsenic pesticides, herbicides and crop desiccants discharge arsenic and other heavy metals via their waste effluents [1, 2]. Due to its toxicity and possibly carcinogenic, only 0.5 ppm or less in waste effluent is allowed to be discharged in surface water [3]. Methods for the removal of arsenic ion from waste streams include chemical precipitation, membrane filtration, ion exchange, carbon sorption and coprecipitation/adsorption. However conventional method such as precipitation does not always satisfactory to meet the pollution control limits. Therefore many researches were carried out for effective alternative technologies or sorbents for treatment of arsenic from the waste streams resulted in search for unconventional methods and materials [4]

Recently hydrous tin oxide was found effective for sorption of arsenate ion [5]. However, the compounds dissolved significantly even in mild acidic pH. In order to improve the stability, the active metal ion or its oxide can be incorporated into other stable adsorbent or a polymer matrix [6]. Our previous work showed that Sn(IV)-loaded poly(hydroxamic acid) chelating resin effective in removal of arsenate ions from an aqueous solution [7]. In this paper, sodium ion of zeolite P was exchanged with stannum(IV) and used for sorption of arsenate (As(V)) ion. Among parameters investigated were effect of pH, As(V) initial concentrations, contact time, temperature and effect of foreign ions.

Experimental

Material

A 0.05 M Stannum(IV) solution was prepared by dissolving SnCl₄·5H₂O (BDH) in 200ml 0.5 M HCl solution. Arsenic (V) stock solution was prepared from Na₂HAsO₄·7H₂O (Sigma). Phosphate solution was prepared from Na₂HPO₄ (Unilab). Sulphate, chloride, nitrate and carbonate solutions were prepared by dissolving their sodium salts. Zeolite P prepared from rice husk silica was donated by Ibnu Sinar Laboratory, University Technology Malaysia. Sn(IV) exchange zeolite (SnZP) was prepared by adding 5.0 g of zeolite P in 90.0 ml (4000 ppm) Sn(IV) in 0.01M of acid hydrochloric solutions. The mixture was stirred overnight at room temperature. The exchange zeolite was separated from the solution using vacuum filtration. The resulting exchanged zeolite was rinsed with distilled water and dried in oven (60°C). The zeolite was ground using mortar. The amount of stannum ion in the supernatant and in initial solutions were determined using

Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES). The exchange capacity of Sn(IV) in zeolite was calculated as the mg of Sn(IV) sorbed per gram zeolite from mass balance. The average exchange capacity was found to be 225 mg Sn per gram zeolite.

Sorption of As(V) by SnZP at various pH

Sorption capacity of As(V) by SnZP at various pHs was carried out as follow. A series 25 of mL of 160 ppm As(V) solution whose pH was adjusted to predetermined values by either hydrochloric acid or sodium hydroxide. Exactly 5.0 mL of the solution was taken for the an amount determination of As(V) as initial concentration. Exactly 0.1g of SnZP was added to the other of 20 ml portion As(V) solutions. The mixture was shaken for 16 h at room temperature. The SnZP was filtered and the final pH for each sample was determined. The concentration data verification of the ICP-AES of As(V) and Sn(IV) was analyzed by ICP-AES and sorption capacity was calculated. The sorption capacity, q (mg As/g SnZP), was calculated from the change in the metal concentration before and after equilibrium and the weight of the dry SnZP as follows:

$$q = (C_o - C_e)V/W \quad (1)$$

where C_o and C_e are the initial and equilibrium concentrations of the metal ion in aqueous phase (mg dm⁻³), V is the volume of the aqueous phase (dm³), and W is the dry weight of the SnZP (g).

Sorption kinetic of Sn(IV)-zeolite P for As(V)

The sorption rate was measured by shaking 0.1g of the SnZP in 20 mL of a 160 ppm As(V) solution at optimum pH 2 for the different time periods (15, 30, 60, 120, 240, 480, 960 and 1440 min) at 25, 50 and 70°C. The initial and equilibrium As(V) concentrations was measured by using ICP-AES and sorption capacity was calculated as above.

Sorption isotherm of SnZP for As(V)

The sorption isotherm was studied by shaking 0.1g of the SnZP in 20ml of As(V) solutions with different concentrations (80, 160, 320, 640, 960, 1600 and 3200 ppm) for 16°C at optimum pH 2 and at 25°C, 50°C and 70°C. The initial and the equilibrium As(V) concentration was measured by ICP-AES and the sorption capacity was calculated as above.

Effect of foreign ions on As(V) sorption by SnZP

Effect of foreign ions on As(V) sorption was carried out by shaking 0.1g of SnZP in 20 ml of 160 ppm As(V) solution containing either sulphate, nitrate, phosphate, carbonate, and chromate with different concentrations (0, 80, 160, 320, 640, 1600 and 3200 ppm). The solutions were then analyzed for As(V) by ICP-AES and the sorption capacity was calculated as above.

Results and Discussion

Effect of pH on As(V) sorption by SnZP

The pH of a solution is an important parameter in sorption process because of the pH dependencies of the electrostatic interactions in the sorption processes at the sorption surface. Since tin could form an amphoteric compound the pH dependencies of arsenate sorption has to be determined. Variation of the metal uptake with pH is shown in Fig. 1. The sorption capacity was found to be low at higher pH values and increased with decrease in pH. This can be explained with a coulombic force between negative arsenic and the positively charged charge of the Sn on the resin. As the pH decreased, the sorbent surface becomes more positive and therefore electrostatic attraction between the arsenic anion and SnZP surface is likely to be increased. The optimum pH values at which the maximum metal uptake could be achieved, was obtained as 2. A similar trend of pH dependent was reported by other workers for the sorption of As(V) by hydrous tin oxide [5]. Fig. 1 also shows that the Sn on zeolite is stable at this optimum pH. For all subsequent experiments, this optimum pH value was used.

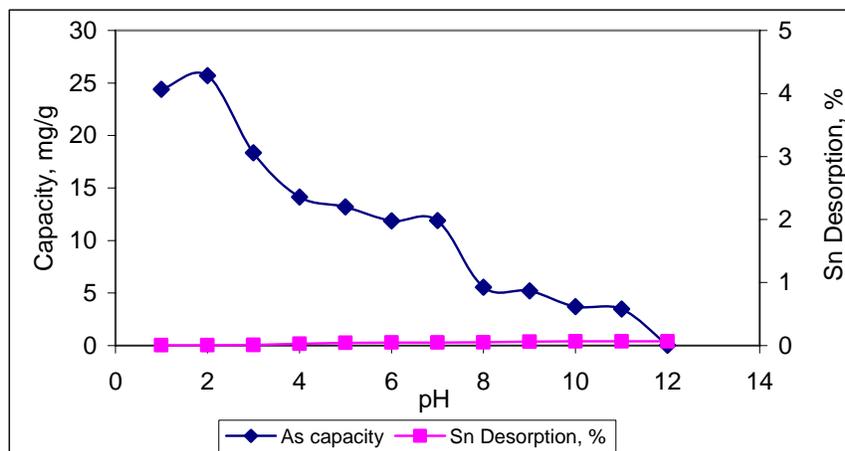


Fig. 1. The effect of pH on the sorption of As(V) by SnZP resin. Initial As(V) 160 mg dm⁻³; volume 20 cm³; sorbent dosage 0.1 g; t: 16; temperature 25°C.

Sorption kinetics

Metal ion uptake capacities were determined as a function of time to determine an optimum contact time for the sorption As(V) on SnZP. Fig. 2 shows time course of the sorption equilibrium of As(V) ion onto SnZP resin. As it can be seen from the figure, the sorption process is rapid in the initial stages of contact time but gradually decreases with time until equilibrium is attained at 16 h. Therefore 16 h of contact time was chosen as the equilibration time in further experiments unless otherwise stated, to ensure that equilibrium condition were achieved. Kinetic characteristic in a sorbent depends not only on the presence of active metal site on it but also depends on the accessibility of the metal site without sterical hindrance which is greatly determined by the matrices of the sorbent. The equilibrium sorption capacity of As(V) onto SnZP was also affected by temperature and increased with increasing temperature from 25 to 70°C. This indicates that the sorption of As(V) onto SnZP surface was favored at higher temperatures and it is controlled by an endothermic process. Sorption process is known to be controlled by different kinds of mechanisms such as mass transfer, chemical reactions and particle diffusion. Hence, several sorption models were applied to evaluate the experimental data. For this purpose Lagergren's pseudo-first-order kinetic model, pseudo-second-order kinetic model and intra particle diffusion model were applied.

First-order kinetic: The linearized form of the first-order rate equation by Lagergren [8] is given as

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

where q_e and q_t are the amount of the arsenic ions sorbed (mg g⁻¹) at equilibrium and at time t (min), respectively and k_1 is the sorption rate constant (min⁻¹). Fig. 3 shows the plots of $\log(q_e - q_t)$ versus t gives a straight line. The value of k_1 can be calculated from the slope while the theoretical capacity $q_{e(\text{theor})}$ can be calculated from the intercept at y axis. In order this model is applicable a part from high correlation coefficient value of the plot, the calculated equilibrium sorption capacity value, $q_{e(\text{theor})}$ should be in agreement with the experimental $q_{e(\text{exp})}$ value [9]. Experimental and theoretically calculated q_e values and the correlation coefficient of the plot (r^2) are given in Table 1. As it can be seen the linear correlation coefficient of the plots using first order kinetic model is not good and experimental and calculated q_e values are not in agreement with each other. So, the result suggests that the sorption of As(V) by SnZP resin is not a first-order reaction.

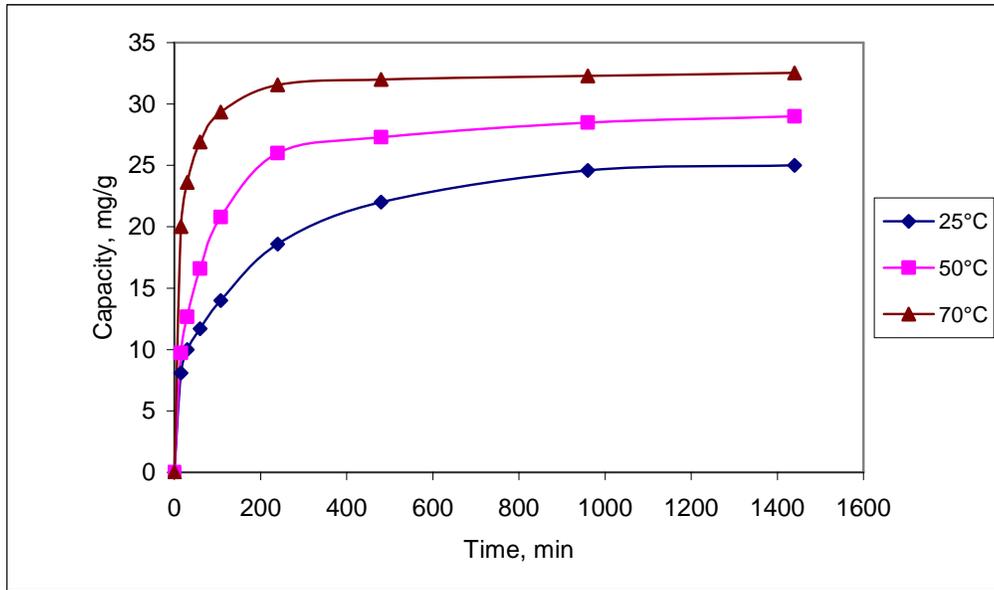


Fig. 2. Sorption rates of As(V) ions onto SnZP resin at various temperatures. Initial As(V) 160 mg dm^{-3} ; volume 20 cm^3 ; pH 2; sorbent dosage 0.1 g.

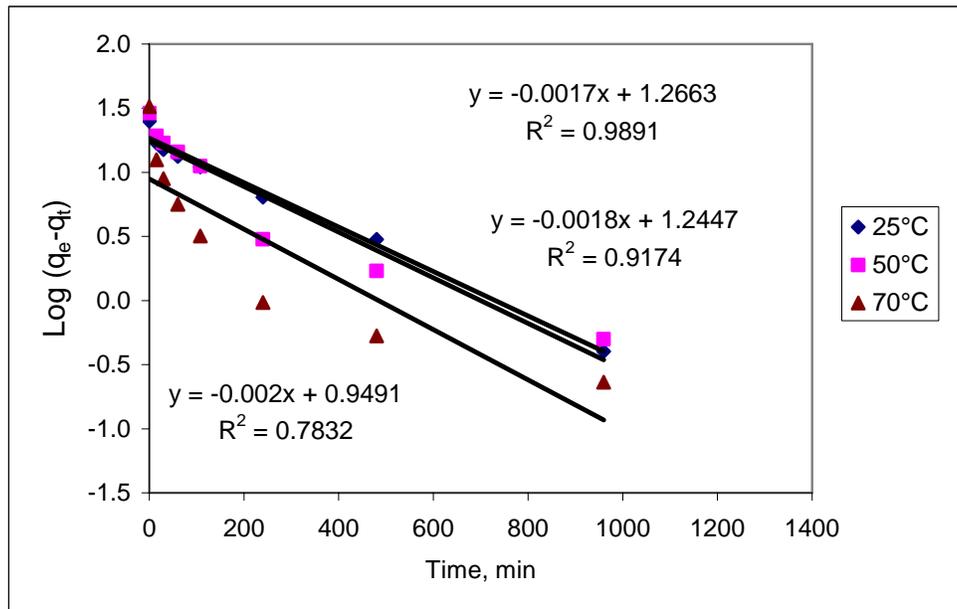


Fig. 3 The first-order kinetic plots for the adsorption of As(V) onto SnZP at various temperatures. Initial As(V) 160 mg dm^{-3} ; volume 20 cm^3 ; pH 2; sorbent dosage 0.1 g.

Table 1 First-order, second-order and intraparticle diffusion rate constants of As(V) sorption by SnZP resin.

Temp °C	q_e (exp) (mg g ⁻¹)	First-order rate equation			Second-order rate equation		Intraparticle diffusion rate equation		
		k_1 (min ⁻¹) x10 ⁻³	$q_{e(\text{theor})}$ (mg g ⁻¹)	r^2	k_2 (g mg ⁻¹ min ⁻¹) x10 ⁻³	$q_{e(\text{theor})}$ (mg g ⁻¹)	r^2	k_{id} (mg g ⁻¹ min ^{-1/2})	r^2
25	25.00	3.91	18.46	0.98	0.54	26.10	0.99	0.8825	0.99
50	29.00	4.14	17.57	0.91	0.66	30.03	0.99	1.2205	0.99
70	32.53	4.60	8.89	0.78	2.67	32.79	1.00	1.4057	0.96

Second-order Kinetics: Experimental data were also applied to the pseudo-second order kinetic model whose equation can be written as below:

$$t/q_t = 1/k_2q_e^2 + t/q_e \quad (3)$$

where k_2 (g mg⁻¹ min⁻¹) is the rate constant of pseudo-second order sorption reaction. Second-order kinetic is applicable if the plot of t/q_t versus t shows linearity and the value of sorption capacity at equilibrium, $q_{e(\text{theor})}$ close to experimental. Also this procedure is more likely to predict the behavior over whole range of sorption and is in agreement with chemical sorption being the rate-controlling step [10]. The plots of t/q_t versus t give a straight line as shown in Fig. 4. The rate constants (k_2), correlation coefficients of the plots together with the theoretical q_e values are given in Table 1. It is clear from these results that the correlation coefficient is high and experimental and theoretical q_e values are in accordance with each other. These results suggest that the sorption of As(V) on SnZP follows the second-order kinetic reaction [5]. Zeolite P with its porous structure and Sn(IV) as active ion present on its surface most probably behaves like a chelating exchanger. Therefore second-order chemical reaction kinetics is expected to be followed in the sorption processes.

Intraparticle diffusion: Intraparticle diffusion model is expressed with the equation given by Weber and Morris [12]:

$$q_t = k_{id}t^{1/2} \quad (4)$$

where q_t is the amount of metal ions adsorbed at time t (mg g⁻¹) and k_{id} is the intraparticle diffusion rate constant (mg g⁻¹ min^{-1/2}). Plots of q_t versus $t^{1/2}$ are shown in Fig. 5. As it can be seen, there is no linear distribution of the points. The plot has an initial linear range followed by a plateau. The linear portion is attributed to the intraparticle diffusion and the plateau portion represents the equilibrium. The intraparticle diffusion constant, k_{id} was calculated from the slope of the linear portions of the curves and given in Table 1. The values of k_{id} increase as the temperature increases. The result showed that intraparticle diffusion, may play an important role as a rate determining step in the sorption process. However, because of the deviation of the curves from the origin and non linear distribution of the plots, intraparticle diffusion cannot be accepted as the only rate determining step for the sorption of As(V) onto SnZP resin [13].

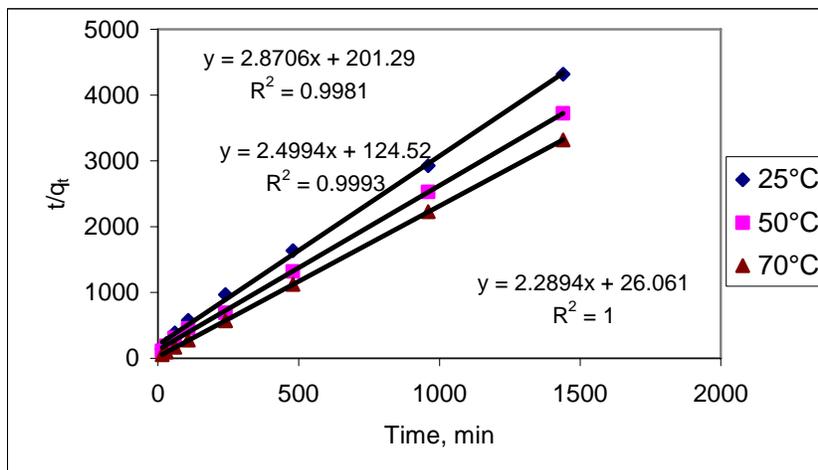


Fig. 4. Pseudo-second-order kinetic plots for the adsorption of As(V) onto SnZP at various temperatures. Initial As(V) 160 mg dm⁻³; volume 20 cm³; pH 2; sorbent dosage 0.1 g.

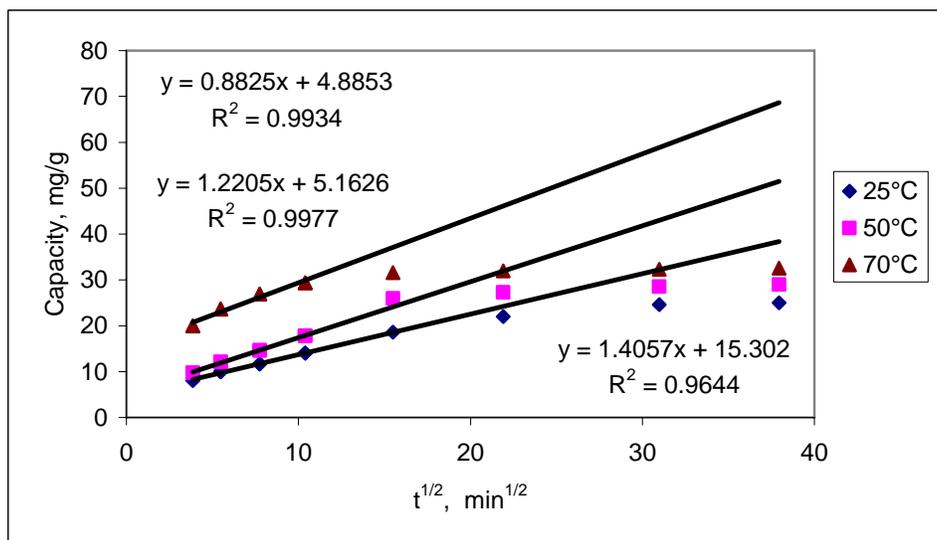


Fig. 5. Intra-particle diffusion plots for sorption of As(V) onto CeZP at various temperatures. Initial As(V) 160 mg dm⁻³; volume 20 cm³; pH 2; sorbent dosage 0.1 g.

Sorption isotherms

The effect of initial concentrations on As(V) uptake was investigated by varying the initial concentrations of the arsenic anion at optimum pH value and 16 h of equilibration time. As it can be seen from Fig. 6, the sorption capacities were increased with increasing the initial concentrations and reached a plateau showing the maximum sorption capacity of the resin. The increase in loading capacity of the sorbent with relation to the As(V) concentration can be explained with the high driving force for mass transfer. The analysis of the isotherm data is important to develop an equation which accurately represents the results and which could be used for further design purposes [14]. The experimental data obtained from the effect of initial concentration on sorption capacity were evaluated with the three sorption models which are Langmuir, Freundlich and D-R models. The linearized forms of the equations representing the models were used.

Langmuir isotherm: Langmuir isotherm which models the monolayer coverage of the sorbent surface assumes that sorption occurs at specific homogeneous sorption sites within the sorbent and intermolecular forces decrease rapidly

with the distance from the sorption surface. The model also based on the assumption that all the sorption sites are energetically identical and sorption occurs on a structurally homogeneous sorbent. Linearized form of the Langmuir equation based on those assumptions is given as [15]:

$$C_e/q_e = 1/bQ^\circ + C_e/Q^\circ \quad (5)$$

where q_e is the amount of solute sorbed on the surface of the sorbent (mg g^{-1}), C_e is the equilibrium As(V) concentration in the solution (mg dm^{-3}), Q° is the Langmuir constant of maximum sorption capacity at monolayer coverage (mg g^{-1}) and b is the Langmuir sorption constant which relate to binding constant ($\text{dm}^3 \text{mg}^{-1}$). Fig. 7 shows that the plots of C_e/q_e versus C_e give a straight line. The values of the Langmuir constants, Q° and b were calculated from the slope and the intercept of the plots, respectively and are given in Table 2.

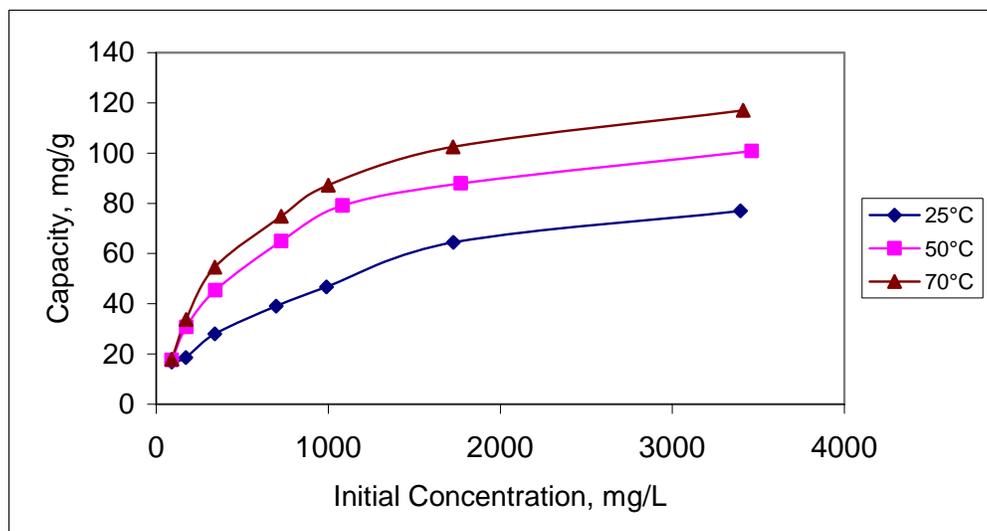


Fig. 6. Effect of initial concentration on As(V) sorption on SnZP at various temperatures. Volume 20 cm^3 ; pH 2; sorbent dosage 0.1 g.

Freundlich isotherm: Freundlich equation is derived to model the multilayer sorption on heterogeneous surfaces. The Freundlich model is formulated as [5]:

$$q_e = K_F C_e^{1/n} \quad (6)$$

where q_e is the equilibrium solute concentration on the sorbent (mg g^{-1}), C_e is the equilibrium concentration of the solute (mg dm^{-3}), K_F is the Freundlich constant (mg g^{-1}) which indicates the sorption capacity and n is the heterogeneity factor which represents the bond distribution. A linear form of the Freundlich equation can be obtained by taking logarithms of the equation:

$$\log q_e = \log K_F + 1/n \log C_e \quad (7)$$

According to this equation the plot of the $\log q_e$ versus $\log C_e$ gives a straight line and the values of K_F and n can be calculated from the intercept and the slope of this straight line, respectively. Experimental data obtained from the effect of initial concentrations to the sorption of As(V) on SnZP was evaluated by applying this equation and the plots of $\log q_e$ versus $\log C_e$ gave a straight as shown in Fig. 8. The calculated related constants are given in Table 2. On comparison of the r^2 values given in Table 2, we can conclude that Langmuir equation gave a better fit to the experimental data than the Freundlich equation. This result also predicts the homogeneity of the sorption sites on SnZP. The Langmuir constant Q° indicates the maximum sorption capacity of the sorbent. As it can be seen from Table 2, Q° value for As(V) sorption was found to be 88.33 mg g^{-1} . This sorption capacity is comparable with some of those reported metal loaded types of sorbent [16-19] but very much higher compared to the sorption by hydrous tin oxide [5].

The Langmuir constant b , which indicate the binding constant for As(V) sorption on SnZP, was found to be $51.00 \text{ dm}^3 \text{ mg}^{-1}$.

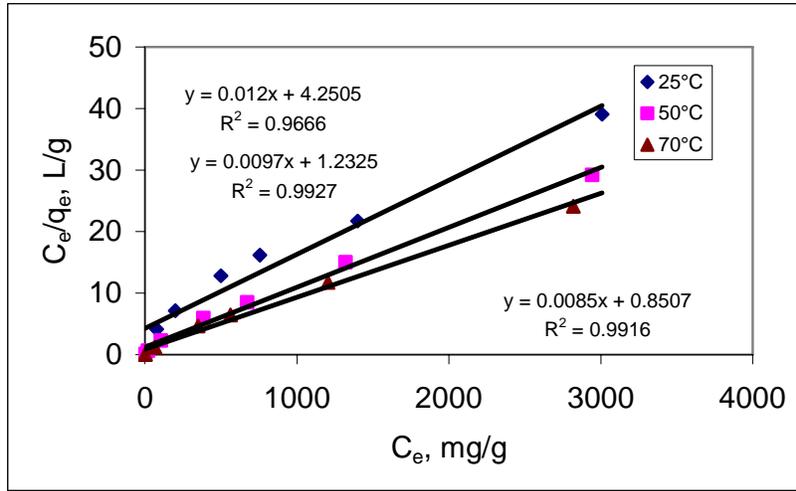


Fig. 7. Langmuir plots for the sorption of As(V) onto CeZP at various temperatures.

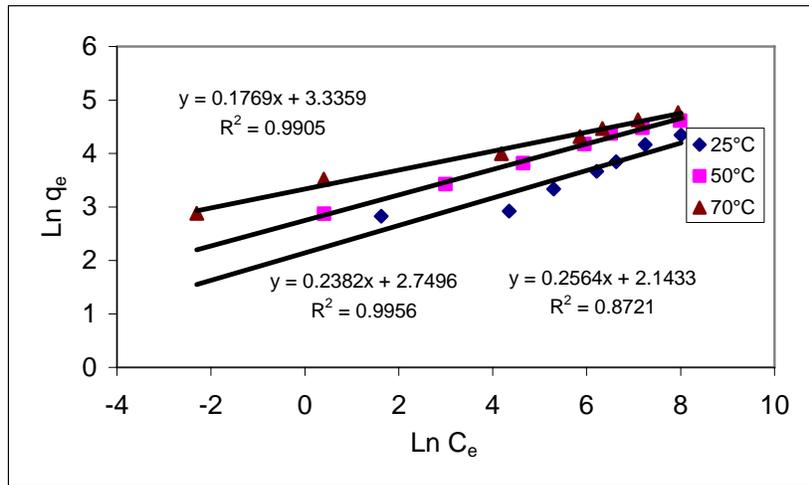


Fig. 8. Freundlich plots for sorption of As(V) onto CeZP at various temperatures.

Table 2. Langmuir, Freundlich and D-R isotherm constants of As(V) sorption SnZP

Temp °C	Langmuir isotherm			D-R isotherm			
	Q° (mg g ⁻¹)	b (dm ³ mg ⁻¹) x10 ⁻³	r^2	q_m (mg g ⁻¹)	k (mol ² kJ ⁻²)	E (kJ mol ⁻¹)	r^2
25	83.33	51.00	0.97	69.09	3.00x10 ⁻⁹	12.91	0.76
50	103.09	11.95	0.99	108.39	3.00x10 ⁻⁹	12.91	0.97
70	117.64	4.31	0.99	111.62	2.00x10 ⁻⁹	15.81	0.97
Temp °C	Freundlich isotherm						
	K_F (mg g ⁻¹)	n	r^2				
25	2.39	3.90	0.87				
50	15.63	4.20	0.99				
70	28.10	5.65	0.99				

D-R isotherms: Langmuir and Freundlich isotherms do not give any idea about sorption mechanism but the former describes sorption on a single type of sorption sites. In this respect the D-R isotherm is an analogue of Langmuir type but it is more general because it does not assume a homogeneous surface or constant sorption potential [20]. The linearized D-R isotherm equation can be written as:

$$\ln q = \ln q_m - k\varepsilon^2 \quad (8)$$

where ε (Polanyi potential) is $[RT \ln(1 + (1/C_e))]$, q is the amount of solute adsorbed per unit weight of sorbent (mol g⁻¹), k is a constant related to the sorption energy (mol² kJ⁻²) and q_m is the sorption capacity (mol g⁻¹). The plots of $\ln q$ versus ε^2 (Fig. 9) gives a straight line curve. The values of q_m and k were calculated from the intercept and slope of the straight line and presented in Table 2. The free energy of sorption, E (kJ mol⁻¹) was calculated from the k values using the equation:

$$E = (-2k)^{-0.5} \quad (9)$$

The magnitude of free energy E is useful for estimating the type of sorption process. The free energy between 8 and 16 kJ mol⁻¹ indicates sorption process can be explained by ion exchange process [20]. In this study the E value is calculated to be 12.91 kJ mol⁻¹. Therefore it is possible to assume that sorption mechanism of As(V) on SnZP is an ion-exchange process [5].

Thermodynamics of the sorption

The experiment on the effect of temperature on sorption of As(V) onto SnZP resin was carried out at 25, 50 and 70°C. The initial concentrations of As(V) was taken as 160 mg dm⁻³. It was observed that the distribution coefficient values, K_D , increased with increase in temperature and that shows the endothermic nature of the sorption process. Thermodynamic parameters like Gibbs's free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) were estimated using the following equations:

$$\log K_D = \Delta S^{\circ}/2.303R - \Delta H^{\circ}/2.303RT \quad (10)$$

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \quad (11)$$

where K_D is the distribution coefficient (cm³ g⁻¹), and R is gas constant (8.3145 kJ mol⁻¹ K⁻¹). Using equation (10) the values of ΔH° and ΔS° were calculated from the slope and intercept of the $\log K_D$ versus $1/T$ plots (Fig.10) and listed in Table 3. The ΔH° value was found to be +83.86 kJ mol⁻¹. The positive value of ΔH° confirms to endothermic nature of the sorption. The ΔG° values were calculated at different temperatures using equation (11) and listed in Table 3. Negative values of ΔG° indicates the spontaneous nature of the reaction, which is favored and getting easier at higher temperatures. The value of ΔS° was found to be positive due to the exchange of the As(V) ions with more mobile ions present on the exchanger. In the case of physisorption which may also contribute to the total sorption process can cause

increase in entropy because the release of the water molecules from the hydrated ions and from the surface of the zeolite.

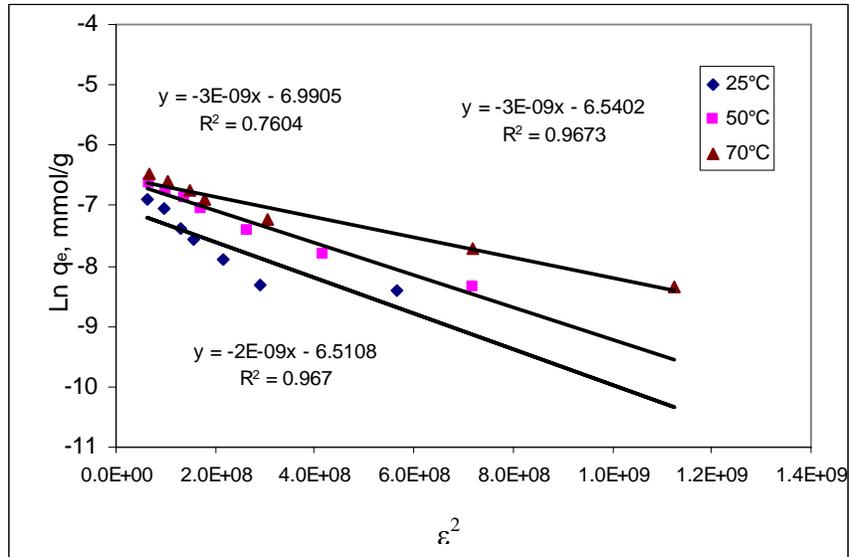


Fig. 9 D-R plots for sorption of As(V) onto SeZP at various temperatures.

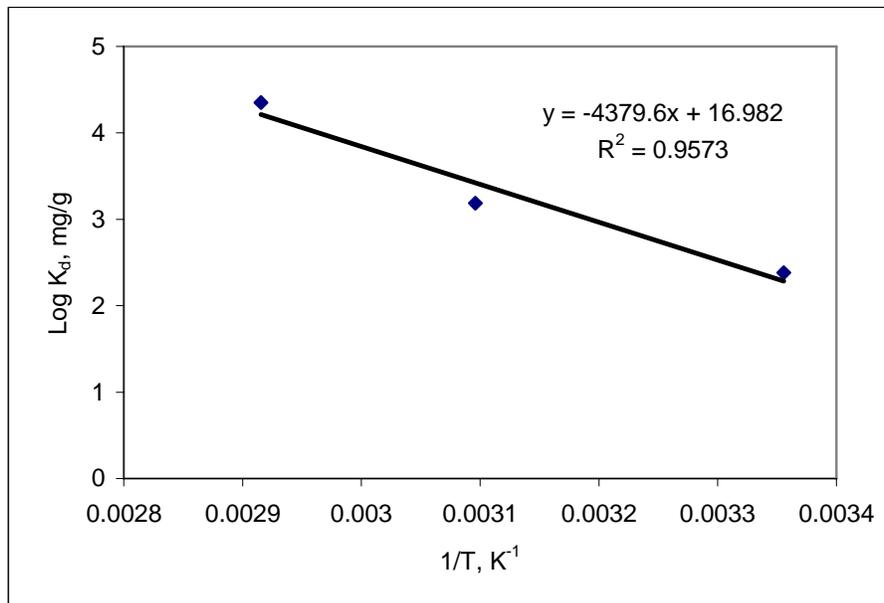


Fig. 10. Estimation of thermodynamic parameters for the sorption As(V) onto SnZP

Table 3 Thermodynamic properties of As(V) sorption onto SnZP

Tem °C	ΔH° kJ mol ⁻¹	ΔS° J mol ⁻¹	ΔG° kJ mol ⁻¹
25.0	8.36	325.16	-13.04
50.0			-21.17
70.0			-27.67

Sorption selectivity of SnZP for arsenate

In many cases samples such as surface water and industrial wastewater contain various kinds of ions which are not necessary to be removed. It is therefore required for a sorbent to have a high selectivity only toward hazardous ion to prolong its life. Fig. 11 shows the sorption behavior of SnZP resin for arsenate in the presence of common anions. The figure shows that phosphate reduced the arsenic sorption capacity while sulphate, chloride, nitrate and carbonate did not affect the arsenic removal significantly.

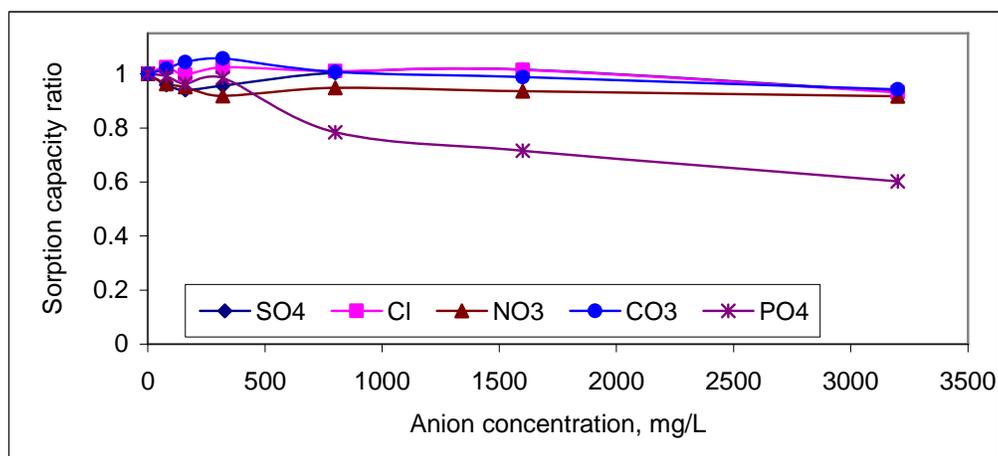


Fig. 11 Selectivity of SnZP resin for sorption of As(V) in the presence of various competing ions.

Conclusion

Sodium in Zeolite P can be exchanged with Sn(IV) at room temperature and used as sorbent for removal of a basic As(V) from aqueous solutions. The following results were obtained:

1. Batch studies suggest that the sorption of As(V) by CeZP occurred in acidic solutions at pH range 1-4.
2. The sorption obey the Langmuir model with the maximum sorption capacity, Q° was 83.33 mg g⁻¹ at 25°C and increased to 117.64 mg g⁻¹ at 70°C.
3. The mechanism of the As(V)-CeZP interaction is thus likely to be ion exchange process and interaction between negative arsenic ion and positively Sn(IV) metal ion site in exchange zeolite.
4. As(V) sorption onto CeZP follows the pseudo-second-order kinetic and intraparticle diffusion models.
5. The thermodynamic parameters obtained from isotherm data confirm the sorption was ion exchange in nature, spontaneous and endothermic process.
6. Phosphate reduced the arsenic sorption capacity while sulphate, chloride, nitrate and carbonate did not affect the arsenic removal significantly

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