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SORPTION OF ARSENIC (V) IONS BY SMZ: EFFECTS OF pH, TEMPERATURE AND IONIC STRENGTH V. SWARNKAR^{*}, N. AGARWAL and R. TOMAR

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ABSTRACT

We studied the ability of zeolite materials for sorption of arsenic from groundwater. We prepared three different prototypes of SMZ (surfactant modified zeolite) for arsenic sorption and eventually used one for further testing. Surface area and arsenic sorption capacity were analyzed for each fully prepared material. The major processes controlling As solubility in shallow ground water and other water sources are determining by effect of pH, Temperature and Ionic strength. Batch sorption kinetic and isotherm studies were conducted to compare and evaluate different types of sorbents As (V) ion sorption from aqueous media. Additionally, the effect of pH on sorption and the rate of sorption were determined for the SMZ material. All batch tests were performed using synthetic water based on the chemistry NM. Batch isotherms were performed on each SMZ material using arsenic concentrations that ranged from 0.1-0.5 N. Batch experiments were accompanied by small-scale experiments in the lab. The batch experiments showed that the SMZ has a maximum arsenic sorption capacity as calculated by the freundlich isotherm. The pH study showed that maximum arsenic sorption occurs at pH 7 and that sorption decreases below or above this pH value. The kinetic studies showed that 80% sorption is achieved. Arsenic sorption capacity varied greatly between the different types of newly created SMZ.

Key words: Sorption, As (V), Zeolites, pH, Temperature, Ionic strength.

INTRODUCTION

Arsenic is very widely distributed in nature with its abundance on earth is thought to be about 5 parts per million¹. In waters, it occurs in rivers, lakes, streams, groundwater and in the seas and oceans. Arsenic is not found in free state, it is largely found in combination with sulphur, oxygen and iron². The arsenic concentration of most potable waters seldom exceeds 10 ppb, although values as high as 100 ppb have been reported³. Exposure to arsenic may come from natural sources, from industrial sources or from food or beverages. Arsenic is increasingly being found in water in many parts of the world such as Bangladesh, Taiwan, Chile, West Bengal-India, Mexico, Argentina, Canada, Hungary and some parts of USA; Utah, Western Oregon and California. The arsenic polluted areas of the world can be geologically subdivided into areas made of sediments derived from water or volcanic rocks characterized by the presence of geysers, gold and uranium mining areas⁴. These elevated arsenic concentrations are mostly of natural origin. Most arsenic is used in the form of compounds of which As_2O_3 is the sole basic material. The largest consumers of arsenic trioxide are the USA, Malaysia and the UK⁵. The uses of arsenic are to make alloys mostly with lead, transistor and light-emitting diodes (LEDs) and also in wood preservatives as CCA (Chromated Copper

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Arsenate). The maximum contaminant level (MCL) for arsenic in drinking water was changed in 2001 by the EPA from 50 μ g/L to 10 μ g/L (EPA, 2001)⁶. The new drinking water regulations is estimated to affect about 3000 municipal water systems and 11 million people in the United States (EPA, 2000)⁷. Additionally, millions of people worldwide consume water with arsenic concentrations above 10 μ g/L (WHO, 1993)⁸. Arsenic can concentrate in the liver, kidneys, and skin by ingestion of groundwater over many years⁹. Several studies have found dose-response relations between arsenic consumption in drinking water and cancers of the bladder, kidneys, skin, lungs, and cancers of the prostate and liver in males¹⁰. Arsenic exists in groundwater predominantly as inorganic arsenite, As (III) (H₃AsO₃, H₂AsO₃⁻¹, HAsO₃⁻², AsO₃⁻³) and arsenate, As (V) (H₃AsO⁴, H₂AsO⁻¹, HAsO², AsO⁻³)¹¹. Arsenate and arsenite are thought to elicit acute toxicity via different mechanisms where arsenate mimicks phosphate and interfering with ATP production in the mitochondria while arsenite binds to and inactivates sulfhydryl-containing enzymes¹². Long term exposure to arsenic and its compounds can cause cancer where the inhalation can result in lung cancer and if swallowed, cancer is likely to develop in the bladder, kidneys, liver and lungs¹³. A recent study on arsenic toxicity have shown that exposure through drinking water has directed effect on the early stages of life when the brain is usually most vulnerable¹⁴. Another study by Tofail et al.¹⁵ have shown that exposure to contaminated drinking water during pregnancy is associated with low birth weight and fetal loss and there is concern that the infants' growth may be affected¹⁵. Since arsenic is well known as toxic and carcinogenic that affects many people around the world, the Safe Drinking Water Act requires Environmental Protection Agency (EPA) to revise the existing 50 ppb standard for arsenic in drinking water. Studies using X-ray adsorption spectroscopy showed that products after arsenic adsorption consisted of inner-sphere As (III) and As (V) mono- and bi-dentate complexes between arsenic species and ferric hydroxides¹⁶⁻²¹.

The actual adsorption of arsenic is thought to be controlled by two processes: generation of iron hydroxide corrosion sites and the ability of arsenic to reach adsorption sites via diffusion^{22,23}. Surfactant-modified zeolite (SMZ) is a proposed medium for arsenic removal that has about 50 % has shown promise in other remediation schemes. Surfactant-modified zeolite (SMZ) refers to a zeolite that has been altered by attaching long surfactant molecules to the surface. These surfactant molecules consist of a non-polar carbon chain and a polar head group (a quaternary amine); the surfactant forms a bilayer on the zeolite surface such that two surfactant molecules are attracted to one another through their non-polar tail groups and the polar head groups are exposed. The surfactant-modified zeolite (SMZ) contained within the material has been shown by Bowman²⁴ to effectively remove pathogens, chlorinated hydrocarbons, chromate, and organics in oilfield waters. Sullivan et al.²⁵ have also shown that SMZ is effective in removing arsenic from soil leachates. SMZ is effective in removing and chemically reducing contaminants such as chromate^{26,27}. This study is aim to determine sorption behaviour of surfactant modified Erionite, Cowlesite and Willhendersonite for As (V) sorption, using different parameters pH, kinetic, Thermodynamic and sorption isotherm and characterized the materials to determine their structure and crystallinity by X-Ray, DTA/TGA and FTIR techniques.

EXPERIMENTAL

Materials and method

Sodium arsenate (Na₃AsO₄) used for anion exchange in zeolites was procured from HIMEDIA. Sodium nitrate, potassium nitrate, sulpheric acid and any alcohol were supplied by Qualigions, calcium nitrate and aluminium nitrat, ammounim molybdate and hydrazinium sulphate were perchased by RENKEM, CDH, HIMEDIA and CDH, respectively. All reagents were analytical grade and the solutions were prepared in DDIW.

Surfactant modification

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The surfactant used is hexadeyl trimethyl ammonium (HDTMA) from Qualigens, A cationic surfactant that has bromide as the balancing counterion. The synthesized zeolites erionite, cowlesite and willhendersonite zeolites 5.0 g were loaded with cationic surfactant i.e. HDTMA (0.25 g). These mixture were equilibrated centrifuged at 150 rpm and the supernatant solution was removed. The zeolites were washed with two portions of distilled water and then air-dried. The supernatants for the initial modification and subsequent was analyzed for HDTMA concentration and the results showed that the target HDTMA loading in the minerals was achieved.

Arsenic sorption

Stock solution of 0.1 N were made from Na₃AsO₄.

Sorption isotherms were conducted by batch experiments at 25° C, pH 7.0. The experiments were carried out in 150 mL stopper conical flasks containing 50 mL of arsenic (Sodium arsenate (Na₃AsO₄.12H₂O; AR) solutions with concentrations 0.1 N and sorbent 0.1 g analyzed the effects of pH, the equilibrium sorption of arsenate was measured at different pH levels from 1 to 9 by setting the initial arsenic concentration at 0.1 N with sorbent 0.1 g. The initial pH of the solution was adjusted by 0.1 M HCl and NaOH solutions. The flasks were shaken at 180 rpm in a shaker for 24 h and then the suspension was passed through a membrane filter for analysis. Arsenic was analyzed on an UV-visible spectrometer (systronic) (experiments were repeated three times, and all of the data are the average of the three). Kinetic study was consulted reserve (+3). Kinetic studies were carried out for different time periods, using buffer solution pH 7, in some hours time intervals (1, 3, 5, 7 and 24 hours). Each experiment was duplicated under identical conditions. The amount of sorption at equilibrium, q_e (mg/L), was calculated by –

$$q_e = \frac{V(C_o - C_e)}{W}$$

where C_o and C_e (mgL⁻¹) are the liquid-phase concentrations of zeolites at initial and equilibrium, respectively, V is the volume of the solutions L and W is the mass of adsorbent used (g).

Characterization techniques

The crystalline phase identification of the surfactant modified zeolites (SMZ-E, SMZ-C, SMZ-W) were determined by XRD (X- ray diffraction technique) analysis using Cu K α radiation at KV and 2Q range of 5⁰ to 70° with a scan time of 1 step/ second. FTIR spectra of HDTMA modified zeolites were recorded on a FTIR spectrometer (NICOLET 410 Spectrometer). Thermogravimetric analysis was performed on perking elmer (Pyris diamond) in the temperature range of 25°C-900°C for zeolitic products with a heating rate of 10°C min⁻¹ under flowing air.

Kinetic Study

In order to accomplish the kinetic study the effect of contact time experiments of CrO_4^{2-} , AsO_4^{3-} and SeO_4^{2-} sorption by the surfactant modified erionite were carried out with 0.1 N concentration, ranging from 1 hour to 24 hours. The initial concentration and metal concentrations at preset time intervals. This metal oxoanion is seen that the equilibrium time of seven hours is sufficient, since maximum sorption is attained during this period.

Desorption studies

Desorption behaviour of loaded analogue of modified zeolites was studied by equilibrating loaded analogue of modified zeolite with distilled water, NaOH (0.01 M) pH ~12, and 0.28 M Na₂CO₃/0.5 M NaOH pH ~13. Each mixture was equilibrating at 25°C for 24 hour under the following conditions.

- (i) The whole specimen was exposed to leachant so that full surface of the specimen remained in contact with it.
- (ii) Sealed polypropylene bottles were used as leachant or buffer containers.

The technique of extraction efficiency studied has been developed by Zhohui Li^{22} . Leaching rate was studied for AsO_4^{3-} oxoanion from loaded modified zeolite minerals and has been carried out with a view to explore the possibilities of its application for long term disposal of these toxic metal oxoanion.

RESULTS AND DISCUSSION

XRD

Analogues of modified Erionite, Cowlesite and Willhendersonite with different degrees of crystallinity and crystal size (determined by XRD) were obtained in the experiments. Powder X-Ray diffraction patterns of these analogues synthesized from media of NaOH concentration and with sodium silicate as the silica source are shown in Fig. 1(a,b,c). In all three cases, the degree of crystallinity is very high. Powder X-Ray diffraction pattern for all the three materials were recorded at room temperature on PW 3064 diffractometer. Samples were scanned using CuK α radiation of wavelength (λ) = 1.54060 Å and the diffractograms were recorded in the range $2\theta = 5^{\circ} - 70^{\circ}$ at the scanning speed of 1 step/ second^{177,178}. Powder X-Ray diffraction (XRD) patterns were taken after different hydrothermal crystallization periods. While using the silica source and maintaining the same temperature and stirring conditions as given in Fig. 1(a,b,c). It shows sharp peaks which indicate the crystalline nature of synthesized materials.



Fig. 1(a): XRD diffractogram of hydrothermally synthesized E-SMZ



Fig. 1(b): XRD diffractogram of hydrothermally synthesized C-SMZ



Fig. 1(c): XRD diffractogram of hydrothermally synthesized W-SMZ

FTIR

Zeolite is found to exhibit a typical infrared spectroscopic patterns. To ensure quantitative analysis, the samples were mixed with KBr at a fixed ratio (1%). The same amount of fixed powder was used to prepare the petllets for FTIR. The spectra can be grouped in two classes. The first is due to internal vibrations of the TO₄ tetrahedrons which is the primary unit of structure and is not sensitive to other structural vibrations. It is found is the range 950-1250 cm⁻¹ and 420-500 cm⁻¹. Strongest vibration at 950-1250 cm⁻¹ is arranged to the T-O stretching and the next strongest band at 420-500 cm⁻¹ is assigned to the T-O bending mode. The stretching modes are sensitive to the Si-Al composition of the framework and may shift to a lower frequency, while the bending mode may be related to the linkages between tetrahedral. (Two bands around 3300 cm⁻¹ and 2800-2900 cm⁻¹ appeared in the SMZ indicating asymmetric and symmetric stretching vibrations of –CH₂ of alky chair and band at about 1400-1500 cm⁻¹ was arranged to vibration of trimethyl ammonium quaternary group [CN (CH₃)₃⁺]. The FTIR spectra of SMZ is presented in Fig. 2(a,b,c) and values in Table 1. The major peak for SMZ are observed at 0.956.69, 470.63, 586.36, 542.00, 3423.65, 1631.78 cm⁻¹. While are comparable with peaks of modified materials except for additional peak observed near 280 cm⁻¹. Indicating asymmetric and symmetric and band at about 1900-1500 cm⁻¹ was assigned to vibration of peak of with the peaks of trimethyl ammonium quaternary group CN (CH₃)⁺.

Table 1: FTIR frequencies for different Zeolite samples

Sample	Peak-1 995 cm ⁻¹	Peak-2 460 cm ⁻¹	Peak-3 660 cm ⁻¹	Peak-4 550 cm ⁻¹	Peak-5 3400 cm ⁻¹	Peak-6 1645 cm ⁻¹
FRI	945.126	470.63	586.36	542.00	3385.07	1625.99
COW	656.69	443.63	586.336	562.00	34233.65	1631.78
WILL	945.126	470.63	586.36	542.00	3385.07	1625.99

Frequency reported in the literature

Table 2: Weight loss of ERI,	COW, WIL and their SMZ from	TGA measurements
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Sample	Weight loss at low temp. w% (a)	Weight loss at high temp. w% (b)	(a + b) w%
ERI	13.4	21.8	35.2
COW	11.8	18.6	30.4
WIL	6.2	16.1	22.3
ERI-SMZ	10.4	20.0	30.4
COW-SMZ	9.9	20.6	30.5
WIL-SMZ	9.7	21.5	31.2



Fig. 2a: FTIR Spectrum of surfactant modified gel Erionite



Fig. 2b: FTIR Spectrum of surfactant modified gel Cowlesite



Fig. 2c: FTIR Spectrum of surfactant modified gel Willhendersonite

DTA /TGA

Thermal analysis of the prepared material was carried out with a heating rate of 10°C/meter to determine the thermal behavior of the prepared zeolite. DTA /TGA thermo grams obtained for zeolite (ERI, COW, WIL) and their SMZ are shown in Fig. 3, where it can be seen that all exhibit a continuous weight loss upon heating from room temperature to 200°C. This weight loss is associated with the dehydration of physically sorbed water. All the materials are found to be structurally stable up to 100°C. The total weight loss of synthesized zeolite and their SMZ are presented in table.





Fig. 3a: TGA/DTA graph of surfactant modified Erionite



Fig. 3b: TGA/DTA graph of surfactant modified Cowlesite



Fig. 3c: TGA/DTA graph of surfactant modified Willhendersonite

Effect of Contact time

The time dependence sorption of As (V) on E-SMZ, C-SMZ & W-SMZ Na₃AsO₄ solutions with 0.1 gm of sorbents. The equibration time was varied from 1-24 hours and results are shown in Fig 4. It is seen that the % age sorption increases with increases in equilibrium time. Equilibrium sorption was sorbed at 7 minits beyond which there is no further increase in the percentage sorption. Therefore 7 hrs equilibration time was considered sufficient for the sorption of As (V) on sorbent materials and was employed for all the subsequent experiments. The short equilibration time suggests that the sorption process is a surface phenomenon.



Fig. 4: Effect of time on sorption AsO₄²⁻ by surfactant E-SMZ, C-SMZ & W-SMZ

Sorption thermodynamic study

Isotherm study

Oxyanions are sorbed from aqueous system onto surface of modified zeolite by a wide variety of mechanism. The operative mechanism for any particular reactions depends on the nature and properties of the active sites on the material surface, the bulk chemistry of the system e.g. pH, hydration status, exchangeable inorganic cations and nature of the sorbate. The sorption isotherm is generally used for the design of sorption system. A sorption isotherm was measured using the batch equilibrium method. Freundlich isotherm model is applied in present sets of the equilibrium data for sorption of metal oxoaions CrO₄²⁻, AsO₄³⁻ and SeO₄²⁻ by surfactant modified zeolite at 25°C temperature. Freundlich plot give R² values near about 0.9998 for all the sorbents (Table 3 and Fig. 5) indicating linear relationship between log C_{ads} and log C_{eq} . The sorption is expressed by freundlich equation. $C_{ads} = KCeq^{1/n}$ Where C_{ads} is the sorbed amount of metal oxoanions, Ceq is the equilibrium concentration of metal oxoanion in solution and K & n are the constants which represents sorption capacity and intensity of the sorption. Freundlich exponent 1/n provides information about surface heterogeneity and surface affinity for the solute 1/n values are computed from slopes of the plots -3.8141, -3.8013 & -3.7729. The graph of Cads verses Ceq gives a straight line (Fig. 5) for the sorption of metal oxoanions by modified Erionite, Cowlesite and Willhendersonite minerals. The linear graph for these metal oxoanion support the applicability of freundlich isotherm model in case of present study. Sorption data of metal oxoanions on modified zeolite minerals fit well in the freundlich isotherm and the values at different temperatures is observed to be linear indicating the applicability of freundlich isotherm model to the sorption of metal oxoanions on these modified zeolite minerals.

Matriala	Conc ⁿ of metal		25°C	
used	oxoanions N	log C _{ads} (meq/g)	log C _{eq} . (meq/L)	log K
	0.01	1.0294	1.5944	
	0.02	1.5353	1.8176	
E-SMZ	0.03	1.8076	1.9335	1.8643
	0.04	1.9626	1.9581	
	0.05	1.7545	1.8693	
	0.01	1.3049	1.4750	
	0.02	1.7356	1.6590	
C-SMZ	0.03	1.9845	1.6661	1.8556
	0.04	1.2920	1.5844	
	0.05	1.6426	1.3474	
	0.01	1.3355	1.4526	
	0.02	1.7650	1.6212	
W-SMZ	0.03	1.9850	1.7276	1.8208
	0.04	1.8527	1.7753	
	0.05	$\overline{1}$.7078	1.7324	

Fable 3: Sorption isotherm for AsO 4	[*] sorption on E-SMZ,	C-SMZ &	W-SMZ
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Fig. 5(a): Freundlich sorption isotherm of AsO₄³⁻ on E-SMZ at 25°C



Fig. 5(b): Freundlich sorption isotherm of AsO₄³⁻ on C-SMZ at 25°C



Fig. 5(c): Freundlich sorption isotherm of AsO₄³⁻ on W-SMZ at 25°C

Thermodynamic parameters

The variations in the extent of sorption with respect to temperature has been explained on the basis of thermodynamic parameters viz, changes is standard free energy (G°), Enthalpy (H^{0}) and entropy (S^{0}). There were calculated by using the following Equations :

$$\Delta G^{0} = -2.303 \text{RT} \log \text{Kc}$$

$$\Delta H^{o} = 2.303 R \left[\frac{T_{1}T_{2}}{T_{2} - T_{1}} \right] \log \frac{Kc_{2}}{Kc_{1}}$$

$$\Delta S^{0} = \frac{\Delta H^{0} - \Delta G^{0}}{T}$$

Where R is the gas constants, T is the temperature on the absolute scale and Kc₁ and Kc₂ are the equilibrium constants at temperature T, T₁ and T₂, respectively (Fig. 6). It is obvious from that the negative and small values of free energy change (ΔG°) are an indications of spontaneous nature of sorption process. The negative values of standard enthalpy change (ΔH°) at different temperature are indicating of the endothermic nature of the sorption process. The negative values of (ΔS°) suggest the decreased randomness at the solid, solutions interface during the sorption of the As (V) onto surfactant modified zeolites.



Fig. 6: Vant's Hoff Plot of ln kc Vs 1/T for As (V) sorption on modified zeolites

Influence of pH

The sorption of As (V) is highly depended on pH of the solutions that effects the surfaces charge of the sorbent degree of ionization and speciation of the sorbate species. The pH dependence study of As (V) sorption (0.1 N) on SMZ carried out using 50 mL of buffer solutions of different pH covering a range of 1 to 9, using 0.1 g of sorbent. The result obtained are shown in Fig. 7. The % age sorption gradually increases up to pH 7. The sorption % age of the metal ions by the SMZ increases gradually with on increases in pH values. Arsenic exhibits different types of pH dependent equilibrium in aqueous solutions. As the pH is shifted, the equilibrium will also shift, the As (V) species occurs mainly in the form of H₂AsO₄ in the pH range between 3 and 6, while a divalent anion HAsO₄²⁻ dominates at higher pH values (such as between pH 8 and pH 11). In the intermediate region which is in the pH range between 6 & 8 both species coexist with one another. Thus it is evident that the sorption of arsenate by SMZ is pH dependent. This effect was

attributed to the electrostatic interactions between the anions of the As (V) in the solution and the surface change of the SMZ.

	Extractants	Desorption %	
	E-the storts	Descention 0/	_
Equilibration Time: 24 hrs.			
Table 4: Desorption behavior	of metal oxonions loaded on	surfactant modified Erionite mi	neral

Material used	Extractants	Desorption %
	Distilled water	
	0.1 M NaOH	0.7
E-SMZ	0.01 M NaOH	17.6
	0.28 M Na ₂ CO ₃ /0.5 M	19.4
	NaOH	67.5
	Distilled water	
	0.1 M NaOH	1.6
C-SMZ	0.01 M NaOH	21.6
	0.28 M Na ₂ Co ₃ /0.5 M	40.3
	NaOH	58.4
	Distilled water	
	0.1 M NaOH	4.5
W-SMZ	0.1 M NaOH	35.6
	0.28 M Na ₂ CO ₃ /0.5 M	52.4
	NaOH	61.9



Fig. 7: Effect of pH of the solution on sorption of AsO₄²⁻ by surfactant E-SMZ, C-SMZ and W SMZ

Desorption behaviour

The present desorption studies have been performed with Arsenate saturated powder sample in the presence of different extracts and distilled water. Loaded modified erionite, cowlesite and willhendersonite (100 mg) sorbed with respective metal oxoanions, AsO_4^{3-} was placed in sealed polypropylene bottles with

25 mL of buffer solution NaOH (0.01M) pH \sim 12, NaOH (0.1 M) pH \sim 12 and 0.28 m Na₂CO₃/ 0.5 M NaOH pH \sim 13 and distilled water (Fig. 8). The bottles were placed in reciprocating water bath shaker at room temperature. After the completion of produced leaching time, the solution was centrifuged and the concentration of metal oxoanion leached out from modified erionite, cowlesite and willhendersonite mineral into the leachant is then estimated spectrophotometrically.



Fig. 8: Desorption behavior of AsO₄³⁻ loaded on E-SMZ, C-SMZ and W-SMZ

CONCLUSION

The purpose of this work was to study the feasibility of Arsenate removal from aqueous solutions by sorption onto surfactant modified Erionite, Cowlesite and Willhendersonite. It could be assumed that modifying zeolites could enable zeolites to sorb Arsenate and lead to considerable decrease in its concentration. The data reported here showed that surface modified zeolites are effective sorbent for removing Arsenate from aquatic solution. The sorption capacity of zeolites were higher than the reported values for zeolites and the sorption ability was enhanced by surface modification, which enables zeolites to sorb As (V). Equilibrium studies were conducted for the sorption of As (V) from aqueous solutions onto surface modified zeolite. pH was the most important factor in As (V) sorption, and other factors had little effect. The equilibrium data have been analyzed using Freundlich isotherm models and the results revealed that Freundlich model is in good agreement with experimental results. Results showed that surface modified zeolites (E-SMZ, C-SMZ and W-SMZ) are efficient tool in removing As (V) from natural water containing As (V) ion in concentrations lower than 90 µg/L under optimum conditions.

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