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Removal of Arsenic(V) from Aqueous Solutions by Using Natural Minerals†

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Abstract

The removal of arsenic(V) using four different natural minerals were evaluated. Parameters like contact time, pH, adsorbent dosages, and As(V) concentration were optimized. The kinetics of adsorption was observed to be fast and reached equilibrium within 2h. As(V) adsorption on studied minerals was dependent on pH and followed a pseudo-second-order reaction model. For kaolin, maximum adsorption was found at pH 5.0. Whereas, in case of other three minerals, a pH range of 6.0-7.0 was found to be the best for As(V) adsorption. The maximum adsorption capacity (Q) was calculated by fitting Langmuir equation to the adsorption isotherms obtained under a specified condition. From the slope of best fit, the Q values were calculated to be 2.07, 2.15, 1.95 and 0.86 mg As(V) /g of bauxite, wad, iron ore and kaolin, respectively. Desorption of As(V) from loaded materials was dependent on the type of leaching reagents used. Based on the results, it was found that among the studied natural minerals, wad was the best As(V) adsorbent.

Key words : Arsenic(V), Adsorption, Natural Mincrals, Desorption

1. INTRODUCTION

Arsenic is perhaps unique among the heavy metalloids and oxyanion forming elements due to its sensitivity to mobilization at the pH values typically found in ground water and under both oxidizing and reducing conditions. In aerobic conditions and at natural pH, As(V) (arsenate) is prevalent and exists as a monovalent ($H_2AsO_4^-$) or divalent ($HAsO_4^{2-}$) anion, whereas As(III) (arsenite), the most toxic form, exists as an uncharged ($H_3AsO_3^0$) or anionic species ($H_2AsO_3^-$) in a moderately reducing environment, specifically in anoxic conditions[1)].

Arsenic contaminated drinking water is a major health problem. It causes various diseases including cancer[2)]. Various treatment methods such as ion exchange, adsorption, ultra-filtration, reverse osmosis, and co-precipitation by metals (predominantly ferric chloride) followed by coagulation have been proposed and adopted for the removal of arsenic from aqueous media. Each of these methods has merits and demerits in terms of cost, material handling and need for skilled manpower[3)]. Adsorption and coagulation are two of the cheapest arsenic removal techniques and they usually employ the cationic metal salts of Fe(III) and Al(III). While flocculation is more effective, the use of solid adsorbents in removing arsenic compares favorably with conventional precipitation methods. In some flocculation treatments a large amount of salt must be added which introduces contaminants such as sulphate ions into the system, which requires subsequent treatment for removal. Moreover, the cost of the chemical reagents used in such treatments can limit their commercial applicability.

In comparison, solid adsorbents are easy to handle and are appropriate for use in rural conditions of India and Bangladesh, where maximum people are affected by arsenic contamination lives. In addition, such materials are ideal for one-time use requiring no regeneration. This is an important convenience, especially in areas which are remote, have no

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regeneration facility, or for communities where drinking water is usually handled by women and children who are not trained enough to handle regeneration. Recently, use of solid adsorbents particularly natural minerals have shown some alluring results in removal of arsenic from contaminated ground water. Of these, manganese wad[4], oxisol (high surface area mixed Fe/Al oxide) and its constituents[5], alumina[6] and natural iron ores [7] are the most often mentioned.

To be considered as a good adsorbent, a material is expected to combine a high arsenic adsorption capacity with a small arsenic release in further contact with aqueous phases. The stability of the arsenic loaded residues when disposed to the environment in the form of landfills or residue ponds is of great concern for the environmentalists[8]. The 'hazardous waste' classification is usually determined by the Toxicity Characteristic Leaching Procedure (TCLP) developed by The Environment Protection Agency of the United States [9]. However, the accuracy and validity of the TCLP for determination of the leachability of redox-sensitive compounds, such as arsenic species, are questionable[10]. It underestimates leachability of arsenic from solid residuals under real site conditions where leaching could be much more than by the TCLP. For example, the role of oxalate and citrate, which is commonly found in soil environment, is not considered.

In the present paper, four different natural minerals i.e., bauxite, wad, kaolin and iron ore, abundantly found in Eastern part of India, were tested to remove As(V) from aqueous solutions to evaluate the efficiency of adsorption. The work reports adsorption of As(V) as a function of contact time, pH, adsorbent dosages, and adsorbate concentration. Also, the desorption of arsenic from the As(V) loaded adsorbents was tested using four different leaching reagents i.e. deionised water, 0.1M acetate, 0.1M oxalate and 0.1M citrate.

2. MATERIALS AND METHODS

2.1. Natural mineral sample characterization

In the present study four different natural minerals i.e., bauxite, wad, kaolin and iron ore, collected from Eastern part of India, were used. The samples were powdered and sieved, and the –75 μm fractions were washed with distilled water by mixing at 1:5 solid/liquid (w/v) ratios for 15 min and then filtered and dried at room temperature. These materials were used for all further experiments. The chemical compositions of the samples were determined by wet chemical methods. The mineralogical compositions of the samples were carried out by combining X-ray diffraction analysis (Rigaku, Model- RH 200, Japan) and wet chemical analysis. The surface area was determined by BET method using five-point surface N_2 (Micromeritics, Model- TRISTAR, V 5.02, USA). Silica content was determined by weight loss after treatment with hydrofluoric acid. Total iron was determined by titration with potassium permanganate. Density was measured by a PRECISA analytical balance fitted with density measurement kit (Model XT 202A, Switzerland). Other metal analysis was carried out by ICP-AES (Jobin Yvon, JY-38) after acid digestion.

2.2. Adsorption study

1000 mg/L As(V) stock solution was prepared from $Na_2HAsO_4 \cdot 7H_2O$ salt (Merck, Germany), which was subsequently used to prepare experimental solutions of specified concentrations. Adsorption studies were carried out in 250 mL glass conical flasks. Necessary adjustment of pH was done before the addition of adsorbent by adding either HCl or NaOH. All experiments were carried out at room temperature (25 ± 1) °C. After introducing the arsenic bearing solution of required strength and the natural mineral samples, the flasks were capped tightly and immersed into the temperature controlled water bath and then shaken for required time at the rate of 150 ± 10 cycles/ min with a mechanical shaker (Julabo-SW-20C). After the required time, the suspension was centrifuged and filtered through a 0.2 μm membrane filter paper. The arsenic concentration in aqueous solutions was determined by using ICP-AES. Experiments were performed in duplicate and the mean values were considered. In order to ascertain the reproducibility of results, a group of experiments were repeated a number of times and the results were found to vary within ± 5%.

2.3. Leaching study

The As(V) loaded solids were washed with deionised water, and dried at room temperature and used for leaching study. The concentration of As(V) in the solid was determined by the acid digestion of the residue followed by analyzing arsenic in the solution. For each leaching experiment, 5g of dried As-loaded solids were added to 100 mL (solid/liquid (w/v) 1:20) of the extractant of appropriate concentration. The pH of the leaching solution was maintained at 5.0 0.1. The leaching reagents used were deionised water (DW), 0.1M oxalate, 0.1M citrate and 0.1M acetate. The suspension was shaken for 18h followed by filtration and arsenic analysis as described above.

2.4. TCLP test

The extraction fluid consisted of a buffered acetic acid (0.1M) solution at pH 4.93, prepared by adding 64.3 mL of 1N NaOH and 5.7 mL of glacial acetic acid to water, and diluting to a final volume of 1L. Other leaching procedures were same as maintained earlier. As per TCLP leaching specification, a solid is classified as non-toxic for arsenic levels in the filtrate below 5 mg/L; otherwise the solid is classified as toxic[9)].

3. RESULTS AND DISCUSSION

3.1. Characterization of the minerals

Physico-chemical characteristics of all the four natural minerals are presented in Table 1. Gibbsite was found to be the major mineral phase in the bauxite sample, followed by silicates, kaolinite and goethite. Major mineral phases identified in wad were goethite, hematite, and gibbsite. Iron ore contained both hematite and goethite in equal proportions. As expected, major constituents of kaolin were SiO_2 and Al_2O_3. Surface area of wad was found to be maximum (76.7 m^2/g) among the four minerals studied.

3.2. Effect of ionic strength

Evaluation of the effect of changes in ionic strength on adsorption behavior is one method of inferring

Table 1. Physico-chemical and mineralogical composition of the studied natural minerals.

Chemical composition, % (w/w)				
Major constituents	Bauxite	Wad	Kaolin	Iron ore
Al_2O_3	78.6	19.4	36	6.3
Fe_2O_3	1.52	33.24	2.5	76.2
SiO_2	8.5	8.12	43.8	4.23
CaO+ MgO	0.32	0.33	1.3	2.6
K_2O+ Na_2O	0.19	0.35	2.5	
MnO_2	0.74	25.8		0.6
Loss on ignition (LOI) (1100°C)	9.13	12.46	13.9	5.7
Major mineralogical composition, (%) w/w				
Al hydrate phase (Gibbsite)	79.8	22	39.5	3.4
Iron phase (Hematite and Goethite)	3.7	43	1.6	82.5
Kaolinite	5.4	2.3	18.5	-
Other silicates (mica, quartz etc.)	11.1	7.0	37.2	5.8
Physical properties				
Bulk density (g/cm^3)	2.56	1.13	2.61	3.3
Porosity (%)	21.7	66.0	31.0	18.0
Surface area (m^2/g)	24.13	76.7	33	13.2
PZC	6.92	7.34	4.8	7.1

adsorption mechanism. Inner-sphere and outer-sphere mechanisms are distinguishable indirectly by the effect of ionic strength on degree of adsorption. McBride[11)] indicated that ions that form outer-sphere surface complexes (for example, selenate) show decreasing adsorption with increasing solution ionic strength. Anions that adsorb by inner-sphere association show little ionic strength dependence (for example, selenite) or show increasing adsorption with increasing solution ionic strength (example, borate and phosphate). Greater ion adsorption with increasing ionic strength is due to the higher activity of the counter ions in solution available to compensate the surface charge generated by specific ion adsorption.

The effect of ionic strength on As(V) adsorption using different minerals were studied at pH 6.0, adsorbent 40 g/L, As(V) 10 mg/L and varying ionic strength from 0.001 to 0.1M NaCl (fig not shown). It was found that As(V) adsorption was almost independent of the ionic strength in case of all the adsorbents. Such a weak ionic strength dependence suggests that As(V) may be adsorbed by strongly bonding chemical bonds, i.e., a largely covalent bond forming inner-sphere complexes with little competitive adsorption of counter anions. The formation of an inner-sphere complex with surface coordination[12)] can be explained by the equation:

$$= S\text{-}OH + L^- + H^+ \longleftrightarrow S\text{-}L + H_2O \qquad (1)$$

where S-OH is a surface hydroxyl group and S-L is the adsorbed species. As reported in the literature, As(V) adsorption on amorphous Fe oxide and Al oxide[13)] showed very little ionic strength dependence as a function of solution pH, suggesting an inner-sphere adsorption mechanism. Here, similar adsorption mechanism for As(V) adsorption on studied minerals may be expected since, those are primarily a mixture of Fe- and Al-oxides. So, all further experiments were carried out at 0.001M ionic strength.

3.3. Kinetics of adsorption

To better understand the kinetics of adsorption, the effect of contact time on As(V) adsorption was investigated by taking samples at various time intervals and measuring the residual As(V) as a function of time (Fig. 1). Under the conditions used, the adsorption of As(V) increased with time and attained equilibrium within 2h for all the natural minerals. With further increasing time up to 3h, there was small increase in adsorption. So, all further studies were carried out for 3h contact time to facilitate maximum possible adsorption. In case of bauxite, the residual As(V) concentration decreased from 10 mg/L to 2.35 mg/L after 3h. In case of wad, and iron ore the residual As(V) concentration decreased to 2.1 and 2.7 mg/L, respectively, from an initial 10 mg/L at the same time. Whereas, for kaolin, the residual As(V) concentration was as high as 5.2 mg/L. Pierce and Moore[14)] stipulated that, adsorption solely due to electrostatic processes are rapid, in order of seconds. Since in the present study, adsorption is not so rapid, it indicates a specific adsorption or formation of a chemical bond between the As(V) and the studied adsorbents. The adsorption of arsenic from liquid to solid can be considered a reversible process with equilibrium between the two phases. In that case, the kinetic data obtained can also be applied to Lagergren's rate equation:

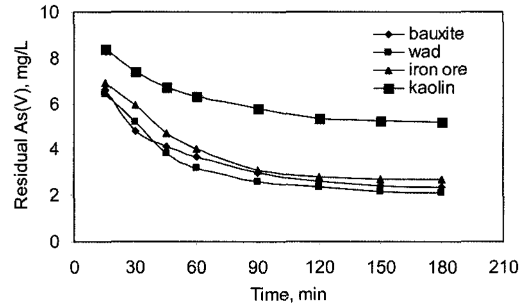

Fig. 1. Effect of time on As(V) adsorption (conditions: As(V)=10 mg/L, adsorbent=40 g/L, pH=6.0).

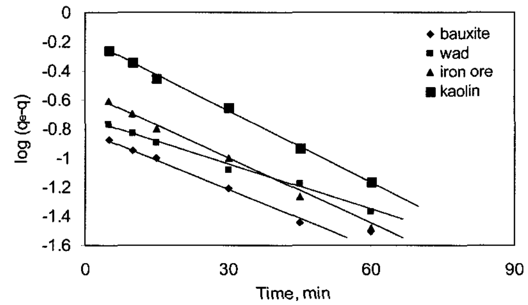

Fig. 2. Lagergren first-order kinetic plot at pH 6.0.

$$\log(q_e - q) = \log q_e - k_{ad}.t/\ 2.303 \qquad (2)$$

where q_e is the amount of As(V) adsorbed at equilibrium (mg/g), q is the amount adsorbed at time t (mg/g), and k_{ad} is the adsorption rate constant. The plot of log (q_e-q) versus time is related to the nature of the process, and any linear fit indicates that the system has first-order reaction characteristics. As shown in the Fig. 2, all the plots were fitted to the model up to 60 min of the adsorption process with correlation coefficients of 0.97-0.99. However, the experimental data considerably deviate from the theoretical data after this period. This showed that adsorption could be described by the Lagergren's first-order equation only at the beginning of the process. In order to find a more reliable description of the kinetics, a pseudo-second-order kinetic equation was applied to the adsorption data[15]:

$$t/q_t = 1/kq_e^2 + (1/q_e)t \qquad (3)$$

where q_e is the amount of As(V) adsorbed at equilibrium (mg As(V)/g adsorbent), q_t is the amount adsorbed at any time t (mg As(V)/g adsorbent), and k is the pseudo-second-order rate constant. The fitting validity for this model was checked by a linear plot of t/q_t against t with slope and intercept equal to $1/q_e$ and $1/kq_e^2$, respectively. As shown in Fig. 3, all the plots were linear over the whole range of experimental time with an excellent correlation coefficients of >0.99 and the second order rate constants, k of 0.016, 0.019, 0.014 and 0.006 (g/ mg.min) for bauxite, wad, iron ore and kaolin, respectively. Calculated k and q_e values from pseudo-second-order plots also correlated well with initial As(V) concentration. This showed that, the kinetics of the As(V) adsorption on the adsorbents can best be explained by pseudo-second-order model for the total reaction time.

3.4. Effect of initial pH

The pH of a solution often determines the extent of adsorption. Particularly, oxide and hydroxide mineral surfaces in contact with water are electrically charged because of ionization of MOH functional group ('M' represent a metal ion at the solid surface). The magnitude and sign of the surface charge vary with the solution pH. Generally, low pH is favorable for uptake of anions due to protonated surfaces, while, cations find large degree of adsorption at a higher pH range due to deprotonated surfaces. Adsorption of As(V) on natural minerals is strongly pH dependent, as has been commonly observed for As(V) adsorption on oxide surfaces[16]. Fig. 4 shows the effect of initial pH values (4.0-9.0) on the efficiency of As(V) adsorption with initial As(V) concentration of 10 mg/L and adsorbent dosages of 40 g/L. For kaolin, maximum adsorption was found at pH 5.0. With further increasing pH the As(V) adsorption decreased steadily. This can be explained by the fact that, the oxygen atoms present on the clay surface interact with water in the acidic medium forming some aqua complexes[17], which result in positive charge on the clay surface resulting better As(V) adsorption at lower pH. As the pH increases, the clay surface becomes more and more negatively charged preventing As(V) anions from approaching the surface and explains the decreasing extend of adsorption at higher pH.

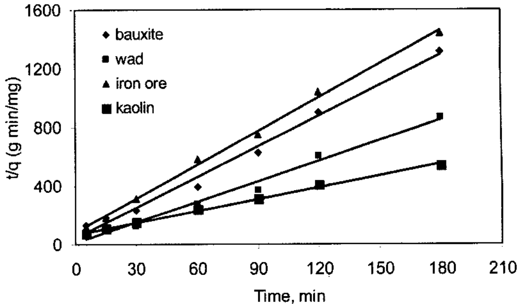

Fig. 3. Pseudo-second-order kinetic plots for As(V) adsorption (conditions: As(V)=10 mg/L, adsorbent =40 g/L, pH=7.0)

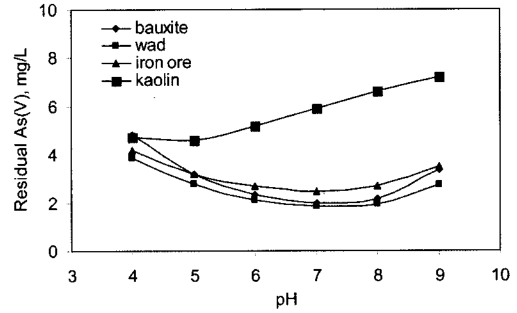

Fig. 4. The pH dependence for As(V) adsorption (conditions: As(V)=10 mg/L, adsorbent=40 g/L, contact time=3 h)

Whereas, in case of other three minerals a pH range of 6.0 - 7.0 was found to be the best for As(V) adsorption. As can be seen, residual As(V) concentration goes through a minimum 2.0, 1.85 and 2.5 mg/L for bauxite, wad and iron ore, respectively at pH 7.0. Below and above this pH range residual As(V) concentration increased, indicating a decrease in the efficiency of As(V) adsorption. It is a great advantage that these adsorbents could be operated at a neutral pH without sacrificing the adsorption capacity. There was significant difference between initial pH and final pH values of the mixtures. This pH change may be due to the buffering nature of the natural mineral samples. The adsorption of As(V) on to hydroxylated mineral surface can be described by a ligand exchange reaction mechanism, that involves the exchange of an aqueous ligand for a surface hydroxyl group resulting in the formation of an inner sphere complex, which causes an increase in solution pH. However, in the higher pH medium, the acid dissociation dominates which causes a decrease in bulk solution pH[6].

As(V) adsorption can also be explained on the basis of nonspecific adsorption. Nonspecific adsorption involves the coulombic interaction, and is mainly limited to pH-dependent sites below pH_{pzc} (the pH value at which surface charge is zero) of the adsorbent. The pH_{pzc} are 6.92, 7.34, 7.1, and 4.8 for bauxite, wad, iron ore and kaolin, respectively. An increase in pH above these values make the adsorbent surfaces more negatively charged resulting in electrostatic repulsion, marked by a decrease in adsorption. Lower pH enhances net positive charge on these surfaces resulting in electrostatic attraction with negatively charged anions. However, this hypothesis could not explain; why adsorption decreased at lower acidic pH values as found in our study. So, columbic interaction is assumed to play a limited role in the adsorption process. It can be concluded that, ligand exchange mechanism dominates in the adsorption of As(V) onto natural minerals. The adsorption mechanism is however quite complicated, which requires further studies.

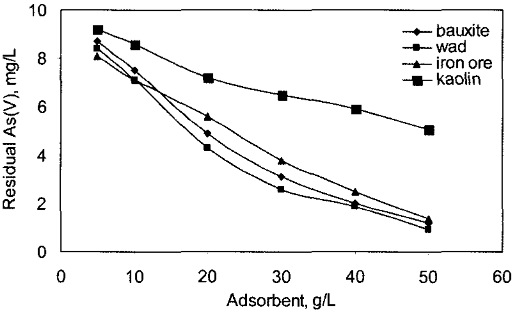

Fig. 5. Residual As(V) as a function of adsorbent amount (conditions: pH=7.0, contact time=3 h, As(V)=10 mg/L).

3.5. Effect of adsorbent dosage and initial As(V) concentration

The effect of the adsorbent dosage (5 to 50 g/L) and initial As(V) concentration (2 to 10 mg/L) on the As(V) removal efficiency has been studied. A typical example is presented in Fig. 5 showing 10 mg/L initial As(V) concentration and varying amount of adsorbent dosage from 5 to 50 g/L at pH 7.0 and agitation time of 3h. The extent of residual As(V) decreased as more and more of the adsorbent was added. For example, when 50 g/L adsorbent was added, residual As(V) concentration decreased to 1.2, 0.95, 1.4 and 5.05 mg/L for bauxite, wad, iron ore and kaolin respectively. As expected, availability of increasing number of exchangeable sites ensured enhanced uptake of As(V). On the other hand, with increasing As(V) concentration at a fixed adsorbent amount the adsorption efficiency decreased (figure not shown), because the number of active adsorption sites is not enough to accommodate enough adsorbate ions.

3.6. Adsorption isotherms

As discussed in the previous section 3.5 and Fig. 5, the adsorption of As(V) was found to be concentration dependent. So, the distribution of As(V) between the two phases can be expressed in terms of adsorption isotherm. The experimental data obtained were applied to linear forms of Langmuir (Eq. 4) and Freundlich (Eq. 5) isotherms.

$$C_e/q_e = 1/(bQ) + C_e/Q \qquad (4)$$

where C_e (mmol/L) is the equilibrium concen-tration of adsorbate in the solution, Q denotes the number of mols of solute adsorbed per unit weight of adsorbent in forming a monolayer on the adsorbent surface, b is the Langmuir constant related to binding energy.

$$\ln q_e = \ln K + 1/n \ln C_e \quad (5)$$

where q_e is quantity of As(V) adsorbed at equilibrium (mmol/g), K is a constant which is a measure of sorption capacity and 1/n is a measure of adsorption density. Table 2 summarizes the results of the adsorption isotherm parameters. The results indicate that the Langmuir isotherm model (Correlation Coefficient (r^2) = 0.92-0.98) can describe the isotherm better than the Freundlich isotherm model (r^2 = 0.82-0.90), which is mostly applicable to non-specific adsorption on heterogeneous solid surfaces. As shown in the table, the Langmuir isotherm for As(V) was characterized by a strong adsorption bond energy as indicated by the constant "b", typical characteristics of a monolayer adsorption[18]. The bond energy, b, has values of 32.8 (kaolin) to 41.66 (bauxite) and the large values show that the equilibrium,

Natural mineral (solid phase)+As(V) (aqueous phase) = Natural mineral...As(V)

is shifted predominantly to the right side, i.e., towards the formation of the adsorbate-adsorbent complex. The largest value for bauxite indicate that the interactions were the strongest between As(V) and bauxite.

Table 2. Langmuir and Freundlich isotherm constants for the adsorbents (adsorbent = 40 g/L, pH = 7.0, contact time = 3h).

Adsorbent	Langmuir constants			Freundlich constants		
	Q	b	R^2	n	K	R^2
Bauxite	0.028	41.66	0.92	1.78	0.025	0.87
Wad	0.029	35.22	0.98	1.49	0.037	0.85
Iron ore	0.026	36.3	0.95	1.61	0.045	0.90
Kaolin	0.012	32.8	0.96	1.58	0.033	0.82

Table 3. Effect of different leaching reagents on As(V) leaching from As(V)-loaded samples.

	As(V) in loaded samples (mg/g)	Leaching solutions	Leached As(V), mg/L	Leached As(V), %
Bauxite	2.07	0.1M Oxalate	9.8	9.5
		0.1M Citrate	12.4	12
		TCLP	4.6	4.4
		DW	0.3	0.29
Wad	2.15	0.1M Oxalate	6.6	6.2
		0.1M Citrate	9.2	8.6
		TCLP	2.5	2.32
		DW	0.3	0.3
Iron ore	1.95	0.1M Oxalate	17.5	17.9
		0.1M Citrate	4.6	4.7
		TCLP	2.4	2.5
		DW	0.25	0.25
Kaolin	0.86	0.1M Oxalate	6.6	15
		0.1M Citrate	9.8	22.8
		TCLP	2.2	5.1
		DW	0.3	0.70

The maximum adsorption capacity (Q) was calculated by fitting Langmuir equation to the adsorption isotherms obtained under a specified condition. From the slope of best fit, the Q values were calculated to be 2.07, 2.15, 1.95 and 0.86 mg As(V) /g bauxite, wad, iron ore and kaolin, respectively. This shows that among the four natural minerals wad is the best As(V) adsorbent, followed by bauxite, iron ore and kaolin.

3.7. Desorption study

Desorption of arsenic and consequent migration to the environment has received relatively little attention compared to determination of adsorption capacity and mechanism. In the present study, desorption of As(V) loaded with different natural minerals were tested using deionised water, 0.1M acetate, 0.1M oxalate and 0.1M citrate. Following their specific affinities discussed before, the loaded solids employed in this investigation presented arsenic contents varying from 0.86 to 2.15 mg/g. In the case of the TCLP tests pH was buffered at 4.93 with the acetate solution. For the others, pH (un-buffered) varied within 5.0 0.1 units. As per the TCLP leaching specification, 5 mg/L is the maximum allowable concentration of arsenic in leached solution. Table 3 indicates the average amount of arsenic leached, in duplicate tests. The results show that in several cases the leached solution exceeded the TCLP leaching specification. As high as 17.5 mg/L (17.9%) arsenic was leached to the solution when 0.1M oxalate was used as a leaching reagent with As(V) loaded iron ore. In general, the effect of citrate was more when aluminium was present in the leached material. On the other hand, more arsenic was leached in presence of oxalate when iron was predominant in leached material. In case of DW, negligible amount of As(V) was leached into the solution. That shows the relative potential hazards of arsenic loaded materials when mismanaged in the presence of determined organic leachants.

4. CONCLUSIONS

In the present study four different natural minerals namely, bauxite, wad, iron ore and kaolin collected from Eastern part of India were tested for their effectiveness as As(V) adsorbent. The general conclusions from the study are listed below:

1. The kinetics of adsorption was observed to be fast and reached equilibrium within 2h.
2. It was found that As(V) adsorption was almost independent of the ionic strength in the range 0.001 to 0.1M NaCl.
3. As(V) adsorption was pH dependent. For kaolin, maximum adsorption was found at pH 5.0. Whereas, in case of other three minerals, pH range of 6.0-7.0 was found to be the best for As(V) adsorption.
4. The kinetics of the As(V) adsorption on studied natural minerals followed a pseudo-second-order model for the total reaction time.
5. The adsorption process followed Langmuir adsorption isotherm and based on the same the maximum adsorption capacity (Q) was calculated to be 2.07, 2.15, 1.95 and 0.86 mg As(V) /g of bauxite, wad, iron ore and kaolin, respectively.
6. Desorption of As(V) from loaded materials was dependent on type of leaching reagents used.

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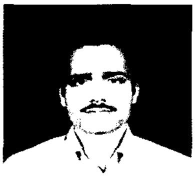

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