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Arsenic removal from drinking water using activated red mud

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ABSTRACT Red mud is the by-product of alumina production, and in this study seawater-neutralised and activated red mud (called activated Bauxsol, AB) is used as an unconventional novel adsorbent for removing arsenic from water. The adsorption of arsenic onto AB is studied and the pH dependence of the process is investigated for both As(V) (arsenate) and As(III) (arsenite). It is found that the arsenic adsorption follows the Langmuir isotherm, and that the optimal pH for adsorption is 4.5 for arsenate and 8.5 for arsenite with roughly 100% and 53% removals, respectively. The linearized form of Langmuir isotherm is used to calculate the adsorption capacities at pH 7, and found as 39.8 for arsenate and 7.2 µmol g^{-1} for arsenite. The combined effects of competing anions *i.e.* phosphate, silicate, sulphate, and bicarbonate are also tested, and it is found that phosphate and silicate hinder arsenic removal more strongly in the presence of sulphate and bicarbonate, despite the fact that neither sulphate nor bicarbonate are strong suppressers at the concentrations found in natural waters. Consequently, this paper shows that AB can be a very efficient unconventional adsorbent for removing arsenate from water at near neutral pH values in the absence of competing anions, but in their presence of these anions sorptive capacity of AB decreases and therefore either the AB dosages must be increased or the pH values should be adjusted for favourable arsenate removal.

(Red mud, activated Bauxsol, arsenic adsorption, Langmuir isotherm, desorption)

INTRODUCTION

Arsenic (As) is a known carcinogen and chronic exposure of As is linked to serious health risks including cancers of the skin and internal organs, and cardiovascular and neurological effects [1, 2]. Soluble As found in natural waters can have many sources, including: weathering of arsenic bearing minerals, the discharge of arsenic waste from petroleum refining, glass melting, and the smelting of lead, copper, zinc, gold, silver and some other metallic ores; As-containing pesticides and fertilizers may also cause As release to the environment [3]. High concentrations of As in water and soil have been documented in many countries, including: Taiwan, Mongolia, Mexico, Chile, Argentina, Ghana, the UK, Greece, Spain, and the USA; but the most severe outbreaks of arsenic poisoning have been associated with groundwaters in the Ganges-Brahmaputra-Meghna (Bengal) Delta area of West Bengal, India and Bangladesh [2, 4].

Although organic As species can occur in natural water as a result of in situ biomethylation, and through the application of organoarsenical pesticides, only the more common inorganic forms are considered in this paper because organoarsenic compounds are only a very minor component of the overall As cycle [5]. As (III) (arsenite) and As (V) (arsenate) are common inorganic As species in natural waters and, of these, the arsenate anion is prevalent in aerobic conditions *i.e.* oxygen-rich surface waters, and arsenite is prevalent in reducing environments *i.e.* anoxic groundwater [6]. It is noted that arsenite exists as an uncharged species at pH values widely encountered in nature due to the fact that the pK_1 value of arsenite is 9.2, whereas arsenate can exist as arsenate anionic species. The toxicity of As depends on the oxidation state, and arsenite is notably more toxic than arsenate. Ionisation reactions of arsenous acid (H_3AsO_3) and arsenic acid (H_3AsO_4) are given in Table 1 (after [7]).

Humans are exposed to arsenic in many ways, but the most critical pathway is through drinking water. For example millions of people in the Bengal Delta area currently drink arsenic contaminated well water ($[As] \geq 0.05$ mg L^{-1}). As a result of the serious health problems associated with As exposure, As concentration guidelines for drinking water are strict and since 1993 the World Health Organization (WHO) has reduced

Table 1. Ionisation reactions of arsenic

Ionization of As (V)	
$H_3AsO_4 \rightarrow H^+ + H_2AsO_4^-$	$pK_1 = 2.2$
$H_2AsO_4^- \rightarrow H^+ + HAsO_4^{2-}$	$pK_2 = 7.1$
$HAsO_4^{2-} \rightarrow H^+ + AsO_4^{3-}$	$pK_3 = 11.5$
$HAsO_4^{2-} \rightarrow H^+ + AsO_4^{3-}$	$pK_3 = 11.5$
Ionization of As (III)	
$H_3AsO_3 \rightarrow H^+ + H_2AsO_3$	$pK_1 = 9.2$
$H_2AsO_3^- \rightarrow H^+ + HAsO_3^2$	$pK_2 = 12.3$

the recommended maximum arsenic concentration limit for drinking waters from 0.05 mg L^{-1} to 0.01 mg L^{-1} (≈0.133 µM) [8]. However, many countries still permit higher arsenic concentrations in drinking water, due to the high cost of treatment to lower concentrations. Conventional arsenic removal technologies usually rely on co-precipitation with ferric or aluminium hydroxides (*e.g.* [9], [10], [11]). Unfortunately, these methods are most suitable for large treatment plants and they result in the production of large amounts of sludge.

Red mud is a residue that remains when the Bayer process is used to extract alumina from bauxite. Because individual bauxites may differ in composition, and because slightly different operational procedures are employed at each refinery, the composition of the residues varies. However, all red muds contain abundant finely divided Fe-, Al- and Ti-oxides and oxyhydroxides, and untreated red mud contains large amounts of sodium hydroxide and sodium carbonate that leaves it highly caustic (the pH is usually >13.0). There are several possible ways to neutralise red mud, including adding acid, hard groundwaters, magnesium and calcium chloride salts, saline brines, or seawater.

Recently, the application of cheap and easily available materials *e.g.* red mud or seawater-neutralised red mud (Bauxsol), has been investigated as a way of cheaply and effectively removing arsenic from water [12, 13]. Altundogan *et al.* [12] investigated the possibility of using red mud and activated red mud (ARM) as a low-cost adsorbent to remove As from water. Their results indicated a moderate removal capacity at pH values suitable for potable water. Genç *et al.* [13] used seawater-neutralised red mud (Bauxsol) to remove arsenate from drinking water, and reported a much higher removal capacity than that obtained for red mud. Later, Genç-Fuhrman et al. [14] and Genç & Tjell 15] used a simple dissolution precipitation process [16] to prepare activated Bauxsol (AB) and found an even higher arsenate removal capacity using AB compared to Bauxsol. However they suggested investigating the process characteristics further particularly testing the adsorbent in combination with oxidation of arsenite to arsenate for effective removal, and evaluating the process efficiency in the multi-anion systems to realistically evaluate the operational capacity of AB.

Figure 1. SEM backscatter image of a polished mount representing a portion of a single Bauxsol grain showing the very fine particle mineral constituents.

MATERIALS AND METHODS

Activated Bauxsol (AB)

The red mud used in this study, obtained from the Queensland Alumina Ltd. Refinery, Gladstone, Australia, has been seawater-neutralised by adding sufficient seawater to the red mud to lower the pH of the suspension to less than about 9.0. Bauxsol is prepared by treating a red mud suspension with seawater, evaporatively concentrated seawater, calcium and magnesium-rich brines, calcium and magnesium chloride salts, or some combination of these alternatives, and stirring the mixture until the equilibrium pH is achieved [18]. The entire process takes about an hour and the fully neutralised Bauxsol has a pH of 8.6 ± 0.2, the Bauxsol can also be washed to remove excess porewater salts. The chemical composition and mineralogy (determined by X-ray fluorescence and X-ray diffraction studies) of the major components in the washed Bauxsol used in this study is given elsewhere [13]. The chemical composition of Bauxsol and AB (used in this study) is summarised in Table 2, where it can be seen that Bauxsol and AB are primarily composed of Fe- and Al-oxides. Texturally, Bauxsol is dominated by very fine particles and is fairly homogeneous; the texture is illustrated in Figure 1, which shows an SEM backscatter image. Detailed examination shows that the few coarse particles in Bauxsol are in fact aggregates of much finer, but compositionally similar, particles and that many of these finer particles are themselves aggregates of still finer particles [13].

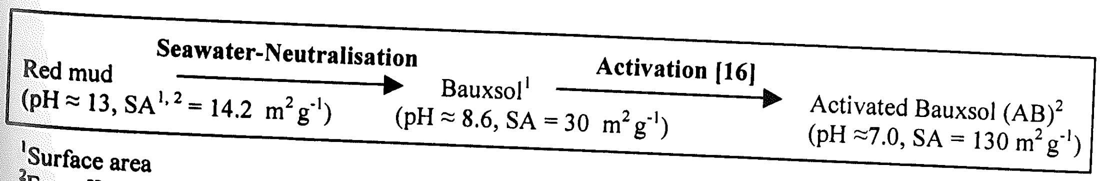
In this study, AB is prepared using the combined acid and heat treatment method of Pratt and Christoverson [16], which involves refluxing Bauxsol in HCl, adding ammonia for complete precipitation, filtering, washing with deionised water, and calcining at 500 ^{0}C for two hours. Details of this method along with more data on the characteristics of AB are also given elsewhere [14, 15]. The simple scheme for the AB production from red mud is given in Figure 2, where it can be seen that the seawater neutralization decreases the pH of the red mud and increases the surface area, similarly the activation further neutralizes the Bauxsol and increases the BET-N_2 surface area from 30 to 130 m^2 g. Note that the adsorption is s surface process; therefore the increase in the surface area is a favourable adsorption condition.

Standard solutions

Arsenate and arsenite stock solutions were prepared by dissolving $Na_2HAsO_4.7H_2O$ and $NaAsO_2$, in water to a concentration of 1 g L^{-1} As. Secondary stock solutions of 100 and 10 mg L^{-1} As were prepared weekly, and used to prepare As test solutions with concentrations between 0.05 mg L^{-1} (0.67 μM), and 14.60 mg L^{-1} (194.4 μM). Silicate, phosphate, sulphate, and bicarbonate test solutions were prepared from the sodium salts of the anionic species; *i.e.* $Na_2SiO_3.5H_2O$, NaH_2PO_4, Na_2SO_4, and $NaHCO_3$ and were used to evaluate the effects of the competing anions.

Table 2. Chemical composition of Bauxsol and Activated Bauxsol (AB).

Constituent	% (w/w)		Constituent	% (w/w)	
	Bauxsol	**AB**		**Bauxsol**	**AB**
Fe_2O_3	34.05	46.55	Na_2O	2.74	0.5
Al_2O_3	25.45	26.51	MgO	1.86	0.5
SiO_2	17.06	17.82	K_20	0.20	0.4
TiO_2	4.90	7.0	P_20_5	0.15	0.4
CaO	3.69	0.7	MnO	0.04	0.08



^{1}Surface area
^{2}From []

Figure 2. Simple scheme of preparing activated Bauxsol (AB) from red mud.

Sorption and desorption experiments

Duplicate batches were examined to prepare adsorption and desorption curves, and to investigate the characteristics of the adsorption process *i.e.* pH dependence of the adsorption and the combined effect of the anions. All chemicals used were reagent-grade and were used without additional purification. A matrix electrolyte of 0.01 M NaCl, prepared by dissolving reagent grade NaCl in deionised water, was used for all sorption experiments. Solutions of 50 mL having different initial As concentrations were added to100 mL inert plastic bottles (PE), and samples were taken from each bottle to represent the initial As concentrations prior to adding AB at each adsorbent dose. The reaction pH of AB is 7.0 ± 0.1, and most of the adsorption studies were carried out at this pH. For other tests the pHs of the samples were adjusted by addition of 2 M HCl or NaOH solutions as required. The flasks were then capped tightly and shaken with a mechanical shaker at room temperature (23 ± 1°C) for 3 h for arsenate and 8 h for arsenite, as these times are previously reported as the pseudo-equilibrium times for the adsorption [14]. After 3 or 8 h, the pH was measured and is reported in this study as the pH of the experiment. Note that the addition of AB to the solution results in minor pH changes within the pseudo-equilibrium time, thus results are reported to ± 0.1 pH units. In the desorption tests arsenate loaded AB was leached using deionised water, and the pH was adjusted to the desired value by adding 2M HCl or NaOH as required. All glassware and PE containers used in the tests were acid-washed for at least 12 h before each experiment using 6% HNO_3, and then washed with deionised water four times and dried.

Arsenic Measurements

After centrifuging batches for 30 min at 4200 rpm, the supernatants were analysed for As using an on-line hydride generation unit on a Perkin-Elmer 5000 atomic absorption spectrophotometer. The system was calibrated using dilutions prepared from a commercially available 1 g L^{-1} standard As solution. For hydride generation 3% $NaBH_4$ (prepared in 1% NaOH) and 1.5% HCl solutions were reacted with the samples except for arsenite determinations, which used 1% $NaBH_4$ (prepared in 0.1% NaOH) and 0.1% HCl with the pH kept at 5 to minimise arsenite oxidation to arsenate. After trying various combinations and checking the precision and sensitivity of the measurements, these analytical conditions were found to give the best results for separate arsenate and arsenite determinations. In the hydride generation unit, the glass reaction vessel and the quartz cell were continuously purged with nitrogen gas to eliminate the interference from air at 194.3 nm. The detection limit was 2 μg L^{-1}. Soluble phosphate and silicate concentrations were determined using the ascorbic acid and molybdate-blue methods [19]. All samples were analysed within two days and analysis of duplicates (or triplicates) indicated a precision better than ± 5%.

RESULTS AND DISCUSSION

Effect of the pH

The pH dependence of arsenate and arsenite sorption onto AB is investigated at pH values ranging from 4.5 to 12.5 using fixed solution concentrations, adsorbent dosages, and contact times, as fine grained compounds in the AB (*e.g.* Fe- and Al-oxides and hydroxides) exhibit a pH dependent surface charge. The corresponding adsorption curves are presented in Figures 3 and 4, which shows that sorption is favoured at pH values below 7 for arsenate, and at 8.5 for arsenite. No further increase in arsenate adsorption capacity is observed below about pH 7 in Figure 4 because all arsenate in the solution is already adsorbed, although not all the available adsorption sites are occupied.

Arsenate adsorption on AB suggests a ligand-based adsorption process; *i.e.* there is a decrease in anion sorption at higher pHs due to the increasing negative charge on particle surfaces, because OH^- is competing for the available adsorption sites. The data obtained in this study are in agreement with the arsenate and arsenite sorption results obtained elsewhere. For example, the adsorption of arsenate on metal oxides and oxyhydroxides is reported to increase at lower pH values and to decrease gradually at higher pH values as in Arai *et al.* [21]. The optimal adsorption pH value (*i.e.* where the maximum sorption occurs) for arsenite is reported to be between 7.0 and 8.5 by Altundogan *et al.* [12] and Manning & Goldberg [22].

Adsorption isotherms

Adsorption isotherms relate the amount of a substance (the adsorbate) that can attach to a surface (the adsorbent) to its concentration in a solution at a fixed temperature. The experimental

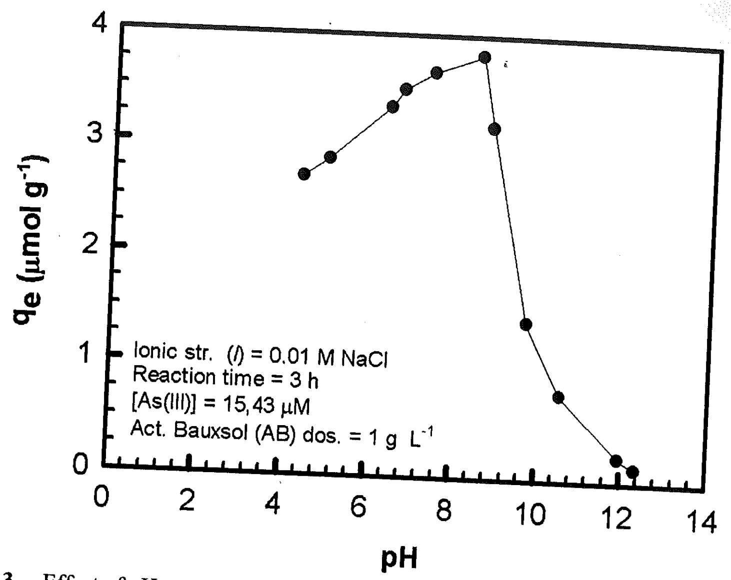


Figure 3. Effect of pH on arsenite adsorption using activated Bauxsol (AB) at room temperature, [22].

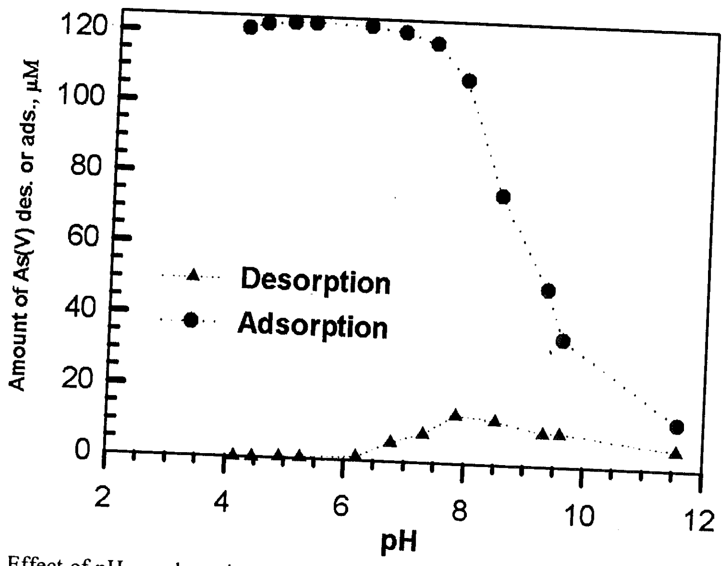


Figure 4. Effect of pH on adsorption and desorption of arsenate using the activated Bauxsol (AB) with reaction conditions: Ionic strength (*I*) 0.01 M NaCl, reaction time = 3 h, the AB or the spent AB dosage = 5 gL^{-1}, and initial arsenate concentration (C_0) = 123.1 μM.

data obtained for arsenate and arsenite are applied to the Langmuir isotherm,

$$q_e = \frac{(Q_0 b C_e)}{(1 + bC_e)}, \qquad (1)$$

and to its linearized forms:

$$\frac{C_e}{q_e} = \frac{1}{(Q_0 b)} + \frac{C_e}{Q_0}, \text{ or}$$

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0 C_e} \qquad (2)$$

Here C_e is the equilibrium concentration in μM, q_e is the amount adsorbed at equilibrium in μmol g^{-1}, and *b*, *K* and *n* are isotherm constants. Langmuir isotherms for arsenate and arsenite are given in Figure 5. The Langmuir isotherms (with high correlation coefficients determined by linear regression) fit the adsorption data better than the Freundlich isotherm (data not shown). The linearized forms of the Langmuir isotherm are used to calculate the adsorption capacity, which is determined to be 39.8 and 7.2 μmol g^{-1} for arsenate and arsenite, respectively. It is noted that detailed information on the Langmuir and the Freundlich adsorption constants of AB is reported elsewhere [20]. In this study, at pH 7.0 ± 0.1 the calculated uptake efficiencies are 96% to 99% for arsenate, and 13% to 53% for arsenite using 5 g L^{-1} AB. Thus it is suggested that arsenate is adsorbed by AB in appreciable quantities, whereas the capacity is significantly lower for arsenite. Similarly, metal adsorption onto red mud using the Langmuir model, in which adsorption is limited by surface saturation (Langmuir modelling is useful for predicting the experimental saturation capacity of a sorbent), can be efficiently applied using monodendate surface complexes despite the heterogeneous nature of the adsorbent [12, 23, 24]. Comparison of adsorption capacities for AB with published data show that it competes favourably with goethite, gibbsite, hematite, activated bauxite, and activated alumina [25].

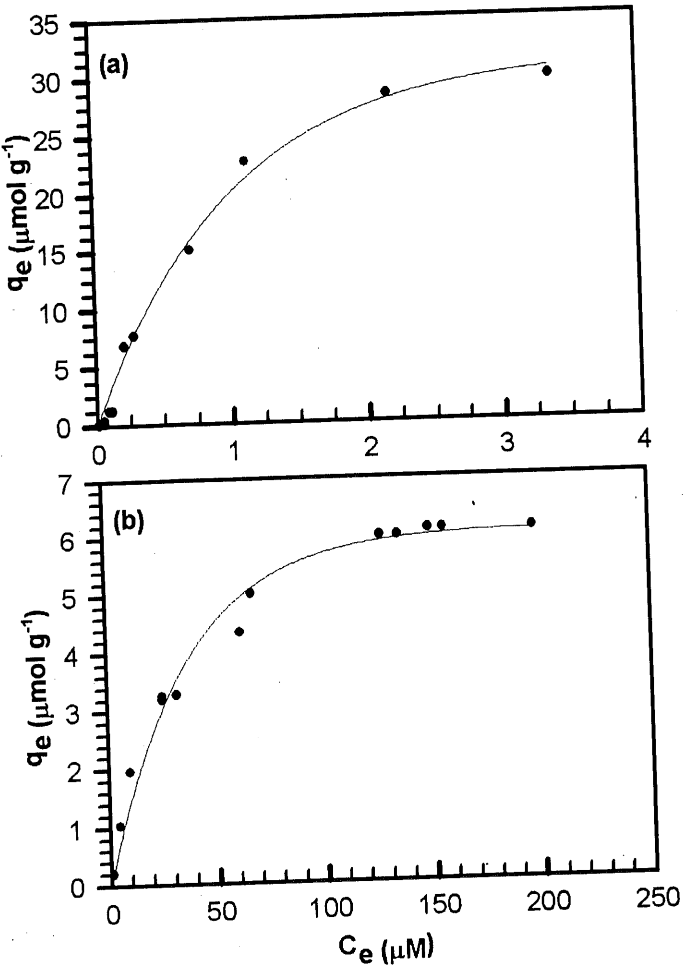


Figure 5. Langmuir isotherms for (a) As(V) and (b) As(III) adsorption on activated Bauxsol (AB) at room temperature with reaction conditions: Initial As conc. range = 0.4 -157 μM, pH = 7.0 ± 0.1, reaction time = 3 h for arsenate and 8 h for arsenite , AB dosage 5 g L^{-1} and ionic strength (*I*) = 0.01 M NaCl.

Desorption

The reversibility of arsenate sorption on AB is studied at several pH values. The investigation is carried out using the spent AB and deionized water, and following the same experimental procedure used for the adsorption experiments. The amount of arsenate (in µM) desorbed as a function of changing solution pH is presented in Figure 4 which shows that the maximum amount of desorption takes place at pH 8. However, detailed results are presented in Genç-Fuhrman *et al.* [20], indicate that the optimum desorption (~40%) is taking place at pH 11.56. This apparent discrepancy may be due to the fact that more arsenate is adsorbed at pH 8 than that at pH 11.56, and thus more arsenate can be desorbed. The low reversibility of arsenate adsorption using AB indicates that the arsenate binding mechanism is not simple ion exchange, but may involve chemisorption, which is the result of much stronger forces (comparable to those leading to the formation of chemical compounds) [20]. The arsenate binding mechanisms are currently under further investigation.

Combined effect of competing anions

The effect of competing anions *i.e.* phosphate, silicate, sulphate and bicarbonate on the arsenate removal using AB has previously been tested in a binary ion systems [14-15]. These studies have shown that the arsenate removal using AB is an effective water treatment option although the competing anions, especially phosphate and silicate, suppress the removal. In the present study the combined effects of silicate and phosphate; and of silicate, phosphate, bicarbonate and sulphate are studied. The motivation is that the competitive adsorption in a multi-component mixture is common, and it may not be equivalent to the sum of the constituent mono-component adsorptions. Therefore in the present study, experiments are carried out in multi-anion systems. Source water compositions are given in Table 3. The results are recorded in Figures 6 and 7, where the percentage arsenate removal is plotted against the arsenate concentration in the solution after the removal. The results in Figure 6 show that the percentage arsenate removal decreases to ~10% in the presence of phosphate, silicate, sulphate, and bicarbonate compared to ~25% in the presence of phosphate or silicate alone, indicating that that even though bicarbonate and sulphate do not have a large effect when present alone [15], they can cause significant cumulative suppression of As adsorption when present with phosphate and silicate. Here, it should be noted that the experiments differ slightly in that although both experiments start with the same pH of 6.9, the end pH is about 7.2 for the phosphate and silicate system, compared to about 6.9 for the combined anion system in Figure 6. However, according to Figure 4, which shows the effect of pH on adsorption, small changes in pH should not affect adsorption within this pH range. Therefore it is clear that the effect of anions in a single anion system cannot be directly applied to a multi-anion system.

Figure 7 shows the adsorption data for arsenate in another multi-adsorbent system with pH 8.5; we note that groundwater with such a high pH is not uncommon (*e.g.* as reported for Hungary in (26)). In this test, the low adsorption can be attributed to the combined effects of high pH and the presence of competing anions. In such a system the pH should be decreased to at least to 7 and /or the adsorbent dosage would need to be increased for efficient arsenate removal using AB.

The individual and combined effects of phosphate, silicate, sulphate, and bicarbonate have also been studied elsewhere [11, 15, 27, 28]. In particular, Genç & Tjell [15] studied the effect of the anions on the arsenate removal using AB in binary systems, and report a decreasing order of adsorption suppression on a molar basis as phosphate > silicate > sulphate > bicarbonate. It is apparent that phosphate competes with

Table 3. Water compositions

Anion	**Anion concentrations, µM**	
	Sample 1	**Sample 2**
Arsenate	1.5 – 42.4	1.5 – 42.4
Phosphate	38.0	59.9
Silicate	723	685
Sulphate	260	0
Bicarbonate	4098	0

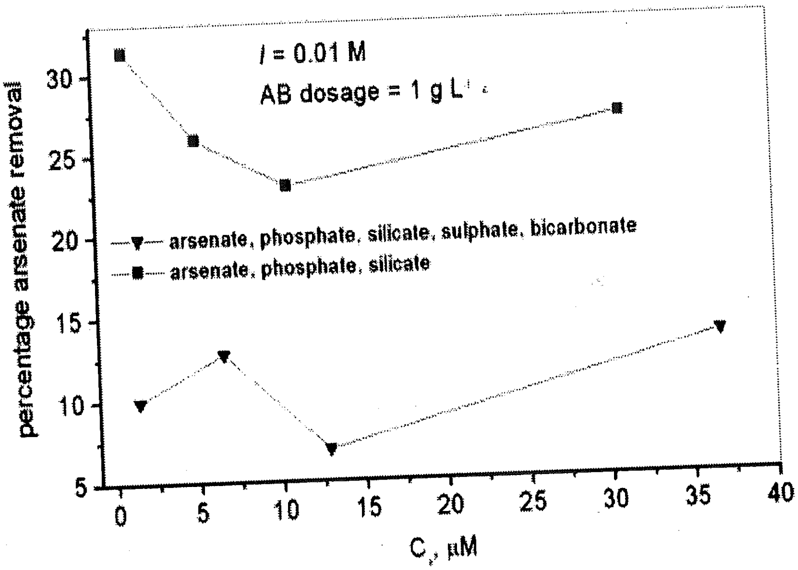


Figure 6. Arsenate adsorption onto activated Bauxsol (AB) in the multi adsorbent systems at room temperature with reaction conditions: Reaction time = 3 h, pH= 7.0 ± 0.1. The water composition of each sample is given in Table 3.

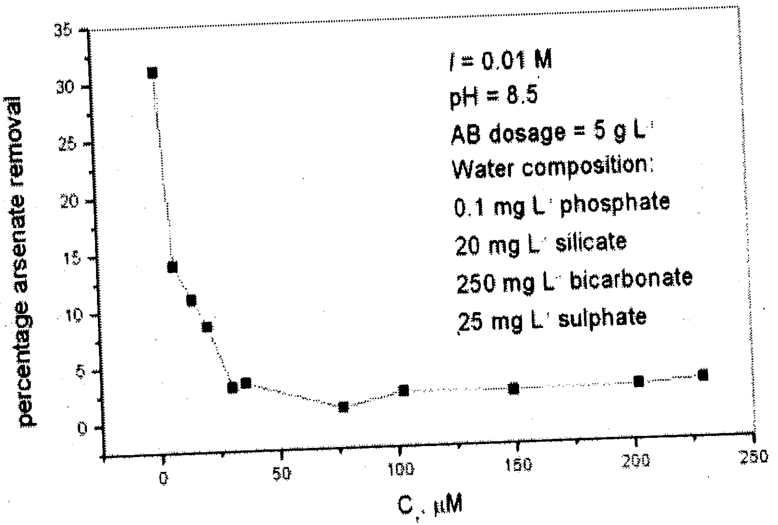


Figure 7. Arsenate adsorption onto activated Bauxsol (AB) in the artificial groundwater sample at room temperature with reaction time of 3h, and initial arsenate concentration range = 2 -240 µM.

dissolved arsenate for the available adsorption sites on AB most effectively as these two anions have rather similar chemical properties (*i.e.* pK_a). In the present study, although all observed suppression can be attributed to the anion competition for the available sorption sites, it is also possible that other factors *e.g.* insufficient reaction time may have influenced the adsorption as well (note that the pseudo-equilibrium times used in the present study are measured at the absence of the competing anions in [14]).

CONCLUSIONS

Arsenic adsorption onto activated Bauxsol (AB) is investigated in batch experiments by preparing adsorption isotherms and by testing the effect of the pH on the process efficiency. The combined effects of the anions (*i.e.* phosphate, silicate, sulphate and bicarbonate) have previously been reported to suppress As removal in binary systems is also investigated. The results demonstrate that within the pH range studied optimum removal is achieved at pH 4.5 for arsenate (roughly 100% removal) and pH 8.5 for arsenite (53% removal), respectively. It is noted that although the optimum arsenate removal is observed at the acidic pH values, arsenate can also be efficiently removed at circum-neutral pH values, but arsenite needs to be oxidised to arsenate prior to the treatment for optimum removal. Furthermore, it has been previously reported that using AB does not contaminate the water and the spent AB passes the toxicity characteristics leaching procedure (TCLP) test [14].

The combined effects of anions are tested and it is found that the combined presence of phosphate and silicate or that of phosphate, silicate, bicarbonate and sulphate significantly hinders the removal efficiency using AB. Of these, the presence of phosphate and silicate in the source water hinder the arsenic removal more strongly when they coexist with sulphate and bicarbonate in the source water, despite the fact that neither sulphate nor bicarbonate are strong individual suppressors at the concentrations found in natural waters.

In the light of the foregoing discussion, it is suggested that the AB used in this study has a high potential for use as an unconventional adsorbent that is comparable to commonly used pure adsorbents for arsenic removal (*e.g.* Fe- and Al- hydroxides), and it may well be suitable for use in drinking water treatment. However, the effect of the competing anions needs to be considered when designing practical systems, and further experimental work is required to elucidate the process characteristics under both batch and continuous flow conditions. A detailed cost analysis will also be necessary for full evaluation of the application potential.

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