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Measurements of arsenite and arsenate contained in mining river waters and leached from contaminated sediments by sequential hydride generation flow injection analysis

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ABSTRACT

In this work, a new analytical method for gasifiable compounds based on sequential hydride generation flow injection analysis (SHGFIA) was applied to water analysis and leaching investigation. For water analysis, it was confirmed that 1 μg L^{-1} As(III) and As(V) were stable for a few days when EDTA was added in the sample waters. Dissolved As(III) and total arsenic (As(III) + As(V)) were converted to AsH_3 in neutral and acidic medium, respectively, to transfer to a miniature gas scrubber (100 μL in absorber volume). The collected arsenic was successively measured by flow analysis based on molybdenum blue chemistry. With this system, changes in As(III) and As(V) concentrations of water placed with arsenic-contaminated-sediment was monitored in near real time. From these data, kinetic analyses were carried out and kinetic constant was obtained from plot of $\ln\{(C^{\infty} - C)/C^{\infty}\}$ where C and C^{∞} were leached arsenic concentration and its final concentration, respectively. It was found that rate of As(III) leaching was much faster than that of As(V) while As(V) leached more in amount compared to As(III). In this work, it was demonstrated that kinetic investigation is also one of the important application of flow analysis. The SHGFIA system showed excellent performance for leaching analysis of arsenic with discrimination of As(III) and As(V).

1. Introduction

So far, most of flow systems have been used for determination of pollutant concentrations. Flow analysis is a useful tool not only for the chemical determination but also for process monitoring. Kinetic investigation and monitoring of leaching from sediment are new area of application for flow analysis. Miro and his colleagues proposed sequential analysis systems for measurement of heavy metal leaching [1–7]. Sediment sample was placed in a small column and different eluents were successively introduced and eluted heavy metal ions were analyzed by atomic absorption spectrometer (AAS) and inductively coupled plasma-mass spectrometer (ICP-MS). The proposed system is very attractive that heavy metals contained in sediment are determined with discrimination of their speciation. The eluents used were un-buffered salt solution such as $CaCl_2$, diluted HNO_3 and chelating agent such as EDTA for determination of cation-exchangeable, humic substances and non-labile species, respectively. Chomchoei et al. also proposed sequential in-line leaching analysis system using a micro column which contained 300 mg of sediment sample [8]. German group Jimoh et al. demonstrated in line leaching examination using in-line column leaching [9]. The leaching analysis was applied to Ni, Cu, Zn, Cd, Pb and Al ions by means of ICP-MS. In the present paper, on the other hand, leaching of arsenic is presented while there is no report on automated analysis of arsenic leaching to the best of our knowledge. In addition, leaching rate of arsenic is relatively slow. Therefore, sediment sample was mixed with water in a relatively large reactor and leaching water was taken periodically to measure arsenite As(III) and arsenate As(V) discriminately. Experimental water can be sampled to analyze from the leaching bath with a sequential system in certain intervals.

Several reports are presented for automated arsenic determinations. For examples, Australian group, Sakchai et al., proposed highly sensitive (limit of detection 0.3 μg L^{-1}) and rapid (150 h^{-1}) flow analysis based on chemiluminescence emission in the reaction with permanganate [10], and Hungary's group, Stefánka et al., developed flow injection method coupled with ICP-TOFMS [11]. Most of arsenic flow analyses recently reported are based on hydride generation with AAS [12–14] and atomic fluorescence spectroscopy [15] or with chromatographic separation [16,17]. In this work, we demonstrated that the flow system especially sequential system is useful for kinetic investigation of leaching from sediments. In the present paper, investigation results on arsenic leaching from contaminated sediments are presented. The

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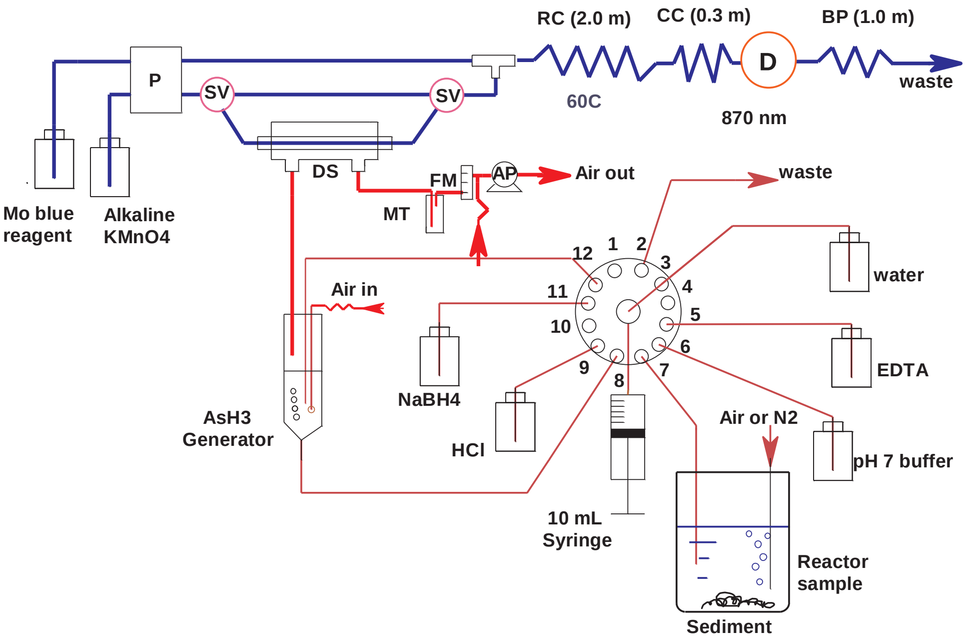

Fig. 1. Flow diagram for water and leaching analysis of arsenic. SV: three-way solenoid valves; DS: miniature diffusion scrubber; RC: reaction coil; CC: cooling coil; D: absorbance detector; BP: back pressure coil; MT: mist trap; FM: air flow meter; mAP: miniature air pump.

system used was based on hydride generation/collection and flow analysis utilizing molybdenum blue chemistry. In our laboratory, basics of hydride generation and effective collection of arsine was investigated earlier [18] and discriminative determination of arsenite and arsenate was demonstrated [19]. Dasgupta's group developed sequential system for discriminative vaporization of As(III) and As(V) and AsH_3 was detected by gas-phase chemiluminescence measurement [20]. In this work, the automated hydride generation with sequential treatment was coupled to the high sensitivity flow analysis of arsine gas. The sequential hydride generation flow injection analysis, SHGFIA, have been developed previously and applied to investigation of arsenic removal in our laboratory [21].

Here we demonstrate that our developed method is useful to monitor leaching of arsenic. Arsenite and arsenate were discriminatively determined in near-real time in the leaching test first time. Kinetic treatment method is also presented in this paper.

2. Experimental

2.1. Automated analysis of arsenite and arsenate

Arsenite and arsenate contained in all samples (river waters, sediment digested solutions and leaching examination) were determined SHGFIA which flow diagram is shown in Fig. 1. Sequential operation was performed by a syringe pump (Versa Pump 6 48K, 54021, Kloehn) equipped with a 12-port selection valves (V6 rotary valve, 24105, Kloehn) both were operated from a computer with a program platform of Kloehn Control. First, arsenite was determined as follows. Water sample (2 mL), 0.05 M ethylenediamine tetraacetate disodium salts (EDTA2Na, Dojindo, Kumamoto) (0.5 mL) and 1 M pH 7 phosphate buffer [1 M KH_2PO_4 (Nacalai) + 1 M Na_2HPO_4 (Nacalai), mixing ratio (3:4)] (1 mL) were taken by a 10-mL syringe and transferred into a hydride generator and 0.33 M $NaBH_4$ (Nacalai) was gradually introduced into the generator for 7 min at the rate of 1.4 mL min^{-1}. The formed AsH_3 was then transferred to an AsH_3 collector during the AsH_3 generation time and collected as arsenate into 100 μL of absorbing solution (15 mM $KMnO_4$ + 10 mM NaOH). Arsine collection scrubber comprised of polypropylene porous membrane tube (0.5 mm i.d. × 0.9 mm o.d. × 500 mm, inner volume ∼100 μL, Accurel PP, Membrana Wuppertal, Germany) in which the permanganate solution flew through. The collected arsenic as arsenate was then flowed to be mixed with molybdenum blue reagent solution [20 mM $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ (Nacalai)/1.8 M H_2SO_4 solution and 0.12 M ascorbic acid (Wako) solution were mixed before use with the mixing ratio 1:1] and absorbance of the reaction product was measured at 870 nm with an UV–VIS spectrophotometer (UV-1570 obtained from JASCO) equipped with a flow through cell to give a signal peak for arsenite. Total arsenic was measured in the same way but AsH_3 was generated with 9 M HCl for 4 min instead of the phosphate buffer for 7 min. Arsenate was determined by subtraction of the arsenite concentration from the total arsenic. Generations of AsH_3 from arsenite and total arsenic were repeated every 17 min automatically with the syringe pump, and measurement of collected arsenic was performed with the continuous flow system.

2.2. Sediment and water samples

Both sediment and water were collected from the river near an old mine (N 32°77′, E 131°36′) which was developed in the world war two for chemical warfare development. The water came out from the mining mouth and formed a river stream. Along the river, waters and sediments were sampled at eleven and three points, respectively (shown in Fig. 2). Sediments were collected at three places; the first sediment sample was taken just downstream of the closed mining (No. 4) and the second and third were 300 m (No. 6) and 3.5 km (No. 11) downstream, respectively.

Water samples were collected in plastic bottles and EDTA was added to be 1.25 mM to prevent change in the arsenic oxidation state. The next day, arsenite and arsenate were determined by SHGFIA.

Arsenic content in the sediments were determined by acid digestion followed by the standard method [22]. The each sediment sample (4 g) was digested with 15-mL HNO_3 and 15-mL doubly diluted H_2SO_4. Occasionally 10 mL of HNO_3 was added and finally 5 mL of HNO_3 and 3 mL of $HClO_4$ was added and heated until the liquid volume became 5 mL. After cooling, deionized water (50 mL)

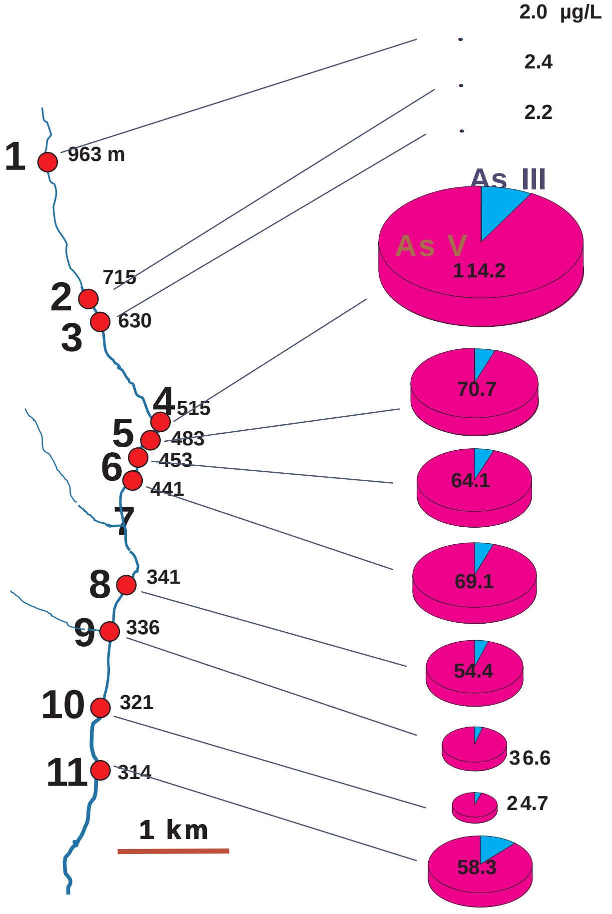

Fig. 2. Sampling points of water and sediments. Water samples were collected at 11 points shown as red circles. Numbers for the points indicate their altitudes. Size of circles in the right is proportional to the total arsenic concentration. Ratio of As(III) and As(V) is shown in the circle. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

was added and heated for 60 min again. The resulting suspensions were filtered with 5B filter paper and made up to be 100 mL. Arsenic content in the digested samples were determined by SHGFIA.

2.3. *Leaching examinations*

Before leaching examination, the sediment samples were washed with water on an 80-mesh sieve to remove fine particles. Then, the sediments were dried at 135 °C for 3 h to remove adsorbed water in air for aerobic experiments or with introducing N_2 gas at a flow rate of 500 mL min^{-1} for anaerobic experiments. Thus treated sediment sample (100 g) was added into 800 mL of synthetic ground water which contained Ca^{2+} 100 mg L^{-1}, bicarbonate 100 mg L^{-1}, silicate 20 mg L^{-1}, nitrate 2 mg L^{-1}, and phosphate 3 mg L^{-1}, and pH was adjusted to be 9.0 by addition of 0.1 M Na_2CO_3. Air or N_2 was purged through the synthetic groundwater at the flow rate of 20 mL min^{-1} since 3 h before the addition of sediment to make the water condition aerobic or anaerobic. Thus, the sediment and water were treated with air and N_2 beforehand for aerobic and anaerobic experiments, respectively. Leached arsenite and total arsenic were measured by SHGFIA. Dissolved oxygen, redox potential and pH of the water was monitored concurrently with DO meter and ion meter (OM-14 and F-53, both from Horiba, Kyoto, Japan).

For comparison examination, water samples were collected in the same timing of SHGFIA sampling and filtered. 5 mL of water sample was taken into volumetric flask and made up to 50 mL after addition of 0.5 mL of nitric acid and 100 µL of 0.5 mg L^{-1} yttrium as an internal standard. Arsenic was measured by ICP-MS (Element, Thermo Fisher Scientific) to compare with SHGFIA results.

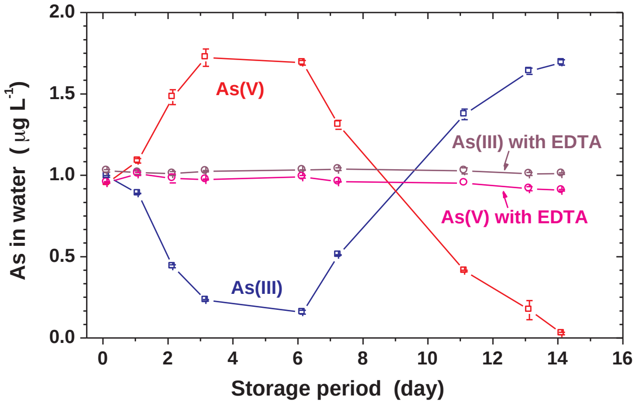

Fig. 3. Change in oxidation state of arsenic during storage with EDTA, phosphoric acid and without the stabilizer. Examination was performed using natural water with addition of As to be 1 µg L^{-1} and kept at room temperature.

3. Results and discussion

3.1. *Arsenic contents in waters and sediments*

First, As(III) and As(V) contained in water samples were measured which were collected at 11 points along the river. The arsenic levels in different oxidation states were measured by the previous flow system [19] which has been developed for field analysis. On site measurement is important for arsenic analysis to distinguish the oxidation state because arsenic oxidation state changes during the transferring and storage. Thus we had measured the arsenic in the field to obtain depth profile of oxidation state of dam lake water. However, arsenic oxidation states could be kept with addition of EDTA [23,24]. Fig. 3 shows that the oxidation state changed dramatically in the storage while the concentrations of As(III) and As(V) were constant for a few days when EDTA was added into the natural water samples even for such low concentrations. On the contrary, when the water was not treated with EDTA, As(III) was oxidized to As(V) probably due to dissolved oxygen, and then the formed As(V) was gradually converted back to As(III) after 3 days with being anaerobic. Finally the all arsenic was three-valent in two weeks. While Gallagher et al. [24] showed the changes in As concentration of groundwater which contained As initially from 19.3 to 511 µg L^{-1}, changes in arsenic oxidation state and effect of EDTA in storage are shown for such very low concentration natural water (1 µg L^{-1}) in this work.

According to the above results, sampling and measurements were performed with EDTA addition in the river water analyses. The results of water measurements are shown in Fig. 2. Upstream of the mining, the arsenic levels were around 2 µg L^{-1}. On the other hand, the concentration was over 100 µg L^{-1} at the point just next to the mining, which water was flowing out from the mouth of mining (sample No. 4). With going downstream from the mining, arsenic level decreased even in a few hundred meters. In further downstream, arsenic level went down to 25 µg L^{-1}. Dissolved arsenic might deposit to the river sediments and the contaminated water was diluted with water from branches of the river. Unexpectedly, however, the arsenic concentration increased to be double of the former at the 11th sampling point. The last point was in a sediment dam and water stream was on the deposited small sands. Probably, arsenic was leached from the deposited sands. Another interesting thing was that the ratio of As(III)/total As was twice

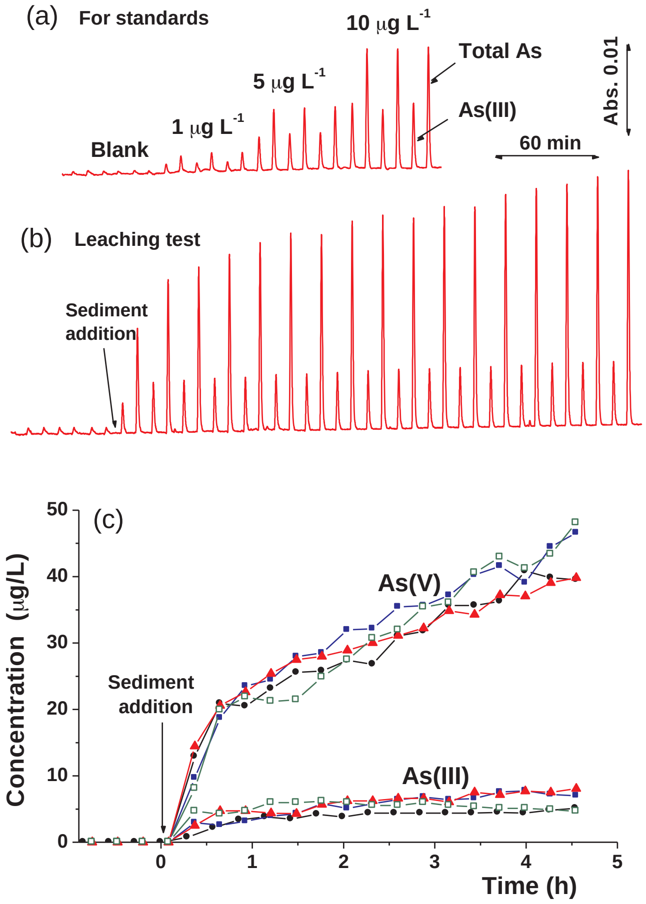

Fig. 4. Response signals of the SHGFIA system for standard solutions of 0, 1, 5 and 10 μg L^{-1} As(III) + As(V) (a) and for monitoring of leached arsenic from sediment No. 6 in aerobic condition (b). Calculated concentrations of leached As(III) and As(V) are shown in panel (c) obtained by repeating three experiments for sediment 6 in aerobic condition.

(11.6%) of those of the other waters (4.15–5.52% at No. 5–10). From the odor at the last sampling point, water and sediment was supposed to be in reducing condition. To investigate these unexpected results, sediments were collected at three points along the river and brought back to our laboratory. The sediments were washed with water before leaching examination. Arsenic content in the sediments were 218–476 mg kg^{-1} and downed to 102–450 mg kg^{-1} by washing with water. The decrease in sediment arsenic was corresponding to adsorbed arsenic and fine particles which contained high concentration of arsenic.

3.2. *Leaching test with SHGFIA*

Leaching characteristics of the sediments sampled along the mining river were examined by SHGFIA. Examples of raw data are shown in Fig. 4. The top panel (a) shows responses for standard solutions containing 0, 1, 5 and 10 μg L^{-1} of As(III) and As(V). The data for low concentrations of arsenic indicate that the SHGFIA system had enough ability to measure arsenic level of sample waters with distinguishing As(III) and As(V). For the same sample, the first peak was corresponding to As(III) and the second peak was for total As (As(III) + As(V)). Limits of detection (LODs) obtained from three times deviation of blank signals were 0.02 and 0.03 μg L^{-1} for As(III) and total As, respectively. Calibration curve was straight from 1 μg L^{-1} to 0.5 mg L^{-1}.

The panel (b) shows real signals obtained during the leaching test where sediment was added into water and As(III) and As(V) were monitored before and after the sediment addition. After the sediment addition, peaks appeared and their intensities gradually increased with the time. Peaks for As(III) were saturated in a short period of time and concentrations did not increase much while peak for total As continued increasing. Interferences from coexisting species are not large as shown previously and effects from heavy metal ions can be reduced by addition of EDTA [19,20]. Iron and copper ions were the species needed attention of interference, and phosphate, silicate, carbonate, sulfate, cyanide, magnesium, calcium, ammonium, nickel ions did not interfere. To make sure that there are few effects from these ions, iron, copper and sulfide ions contained in leaching sample solutions was analyzed by AAS, and they were below limits of detection of AAS (0.1 mg L^{-1}). These metals do not exist as free ions in neutral solution due to large formation constants for hydroxide complex. The content of iron was not high enough to interfere our arsenic analysis. Sulfide could not be detected by the fluorometric method using fluorescence mercuric acetate [25].

Plot of As(III) and As(V) concentrations against the leaching time obtained with the sediment No. 6 in aerobic condition is shown in the panel (c). The measurement was repeated four times and the each run was performed for 4.5 h. It can be seen that repeatability of the leaching examination was good and this method was suitable to investigate leaching property of sediment. Concentration of As(III) reached constant in 30 min to be ~5 μg L^{-1} in those experiments. On the other hand, As(V) concentration continued increasing and the As(V) level reached ~40 μg L^{-1} in the end of experiments. It may suggest that leaching of As(III) was faster and most of As(III) contained in the sediment consumed in a short period of time. It was probably due to As(III) is 5–10 times more soluble than As(V) [26]. In this way interesting results for As(III) and As(V) with relatively good time resolution while so far only total As was monitored in hours or days [27].

The pH of sample solution was adjusted to be 9 with addition of 0.1 M Na_2CO_3. Effect of pH was tested preliminary and it was found that leaching was higher in weekly alkaline solution compared to neutral or slightly acidic solution. The adsorption affinity of arsenate is higher at low pH and for arsenite at higher pH values [28].

Comparison examination was also carried out with ICP-MS as mentioned in Section 2. Correlation of data by this method and ICP-MS in μg L^{-1} was expressed as:

$$\text{SHGFIA data} = 0.840\,\text{ICP-MS data} + 0.002 \quad (R\,0.973)\ \text{for total As.}$$

Particularly, agreements were obtained for comparison analysis ($n = 10$), as the value of the slope being 0.840 approaching unity and it can be said that the present method was reliable in the leaching analysis. ICP-MS showed slightly larger data compared to SHGFIA data probably due to solid small particles contained in leaching samples.

3.3. *Kinetic consideration of the leaching*

In this sub-section, kinetic treatment of arsenic leaching is discussed. We assume that the leaching was pseudo first order reaction from the sediment with the rate constant k. Accordingly, the rate of leaching namely decreasing rate of arsenic concentration of sediment $-(dC_{\text{sed}}/dt)$ can be expressed as Eq. (1).

$$-\frac{dC_{\text{sed}}}{dt} = \frac{dC_{\text{sol}}}{dt}\frac{W}{V} = kC_{\text{sed}} \tag{1}$$

The term C_{sed} is leachable arsenic concentration in sediment in μg kg^{-1}, C_{sol} is leached arsenic concentration in solution in μg L^{-1}, W is weight of sediment in kg and V is volume of solution in L. Eq. (1) can be changed to Eq. (2) with using initial sediment concentration

Table 1
Leaching results obtained in aerobic and anaerobic conditions.

Sediment No.	Sediment As (mg kg^{-1})	Aerobic				Anaerobic			
		As(III)		As(V)		As(III)		As(V)	
		Elution[a] (μg L^{-1})	k (h^{-1})	Elution (μg L^{-1})	k (h^{-1})	Elution (μg L^{-1})	k (h^{-1})	Elution (μg L^{-1})	k (h^{-1})
4 (course)	102	0.83	0.554	20.6	0.118	0.77	0.964	16.3	0.157
6 (middle)	450	5.09	0.740	39.5	0.116	8.71	0.497	49.4	0.153
11 (fine)	142	19.8	1.15	26.2	0.117	18.1	0.402	45.1	0.142

[a] Elutions are concentrations at 4.5 h leaching.

C^0_{sed}. The C_{sed} is expressed as subtraction of leached amount from the initial concentration C^0_{sed} and Eq. (1) can be changed to Eq. (2).

$$\frac{dC_{\text{sol}}}{dt}\frac{W}{V} = k\left(C^0_{\text{sed}} - C_{\text{sol}}\frac{W}{V}\right) \tag{2}$$

Both sides are divided by W/V and we obtain Eq. (3).

$$\frac{dC_{\text{sol}}}{dt} = k\left(C^0_{\text{sed}}\frac{V}{W} - C_{\text{sol}}\right) \tag{3}$$

Eq. (3) shows the rate of increase in solution concentration, which can be measured in the leaching experiments. Eq. (3) can be converted in integrated form. First, $C^0_{\text{sed}}(V/W)$ can be rewritten as C^{∞}_{sol} and we obtain Eq. (4).

$$\frac{dC_{\text{sol}}}{C^{\infty}_{\text{sol}} - C_{\text{sol}}} = k\,dt \tag{4}$$

Integrated form:

$$\ln(C^{\infty}_{\text{sol}} - C_{\text{sol}}) = \ln\, C^{\infty}_{\text{sol}} - kt \tag{5}$$

or it can be expressed as:

$$\ln\frac{C^{\infty}_{\text{sol}} - C_{\text{sol}}}{C^{\infty}_{\text{sol}}} = -kt \tag{6}$$

The term C^{∞}_{sol} can be estimated from the $C_{\text{sol}} - t$ curve obtained in leaching examination. The concentrations of As(III) and As(V) were almost constant from 24 to 72 h as shown in Fig. 5, and 24 h data was taken as C^{∞}_{sol} value. Log scale plot is shown in the bottom panel of Fig. 5: $\ln\{(C^{\infty}_{\text{sol}} - C_{\text{sol}})/C^{\infty}_{\text{sol}}\}$ was plotted against t. Good linearity was observed in the first 1 h for both As(III) and As(V).

From the first 1 h data of the four runs (Fig. 4c), the rate constant k was obtained as 0.740 ± 0.166 for As(III) and 0.116 ± 0.007 for As(V). It can be said that leaching of As(III) was four times faster than that of As(V). However, basically the leaching amount of As(III) was much smaller than that of As(V). It can be said that the leachable As(V) is dominant in the sediment compared to As(III) content.

3.4. Leaching characteristics with different sediments and different conditions

Leaching was investigated with different sediments sampled along the river. Anaerobic and aerobic conditions were tested for the leaching and solution concentration at 4.5 h leaching and kinetic constants obtained as described in the previous sub-section are shown in Table 1. Among the samples, sediment sample No. 6, which contained higher amount of arsenic (450 mg kg^{-1}), gave leached arsenic as 5.1 μg L^{-1} As(III) and 39.5 μg L^{-1} As(V) under aerobic condition. Under aerobic from the sediment sample No. 4, desorption of arsenic was low because sediment physical properties was almost pebble-stone and adsorbed less amount of arsenic, as a result the minerals dissolution and desorption of arsenic was also low. In the case of sediment sample No. 11, which was in reduced condition in the sampling situation, desorption of As(III) was significant compared to the other sediment. From the sediment sample No. 4 arsenic was leached as 1.0 μg L^{-1} As(III) and 20.6 μg L^{-1} As(V) whereas from the sediment sample No. 11 arsenic was leached as 19.8 μg L^{-1} As(III) and 26.2 μg L^{-1} As(V), respectively. The finer sediment sampled in anaerobic condition released inorganic arsenic significantly in the leaching examination compared to the other sediment samples. In any cases, leached arsenic in 4.5 h was ∼3% of sediment arsenic. From this information, the most of arsenic was to be in insoluble form.

The leaching of arsenic concentration in anaerobic is more than leached in aerobic condition. In anaerobic condition, reductive dissolution of metal (Fe, Al, Mn) oxides are more which combine with arsenic, therefore leaching of arsenic also more than in aerobic. This indicates that anaerobic condition enhances the leaching of arsenic from the sediments. Both in aerobic and anaerobic condition the same sediment samples were used for the leaching experiments. Under anaerobic condition from the sediment sample No. 6, 8.71 μg L^{-1} As(III) and 49.4 μg L^{-1} As(V) were obtained, and their levels were more than leached under aerobic condition (5.09 μg L^{-1} As(III) and 39.5 μg L^{-1} As(V)). Sediment sample No. 11 sampled from in reduced condition may be As(III) and As(V) co-precipitated/adsorbed with metals oxides. This is why in the both conditions As(III) was leached from the sediment sample No. 11 more than other two sediment samples.

While amounts of elution of As(V) were higher than those of As(III), kinetic constants for As(V) were lower than As(III). Desorbable As(III) leached quickly compared to As(V).

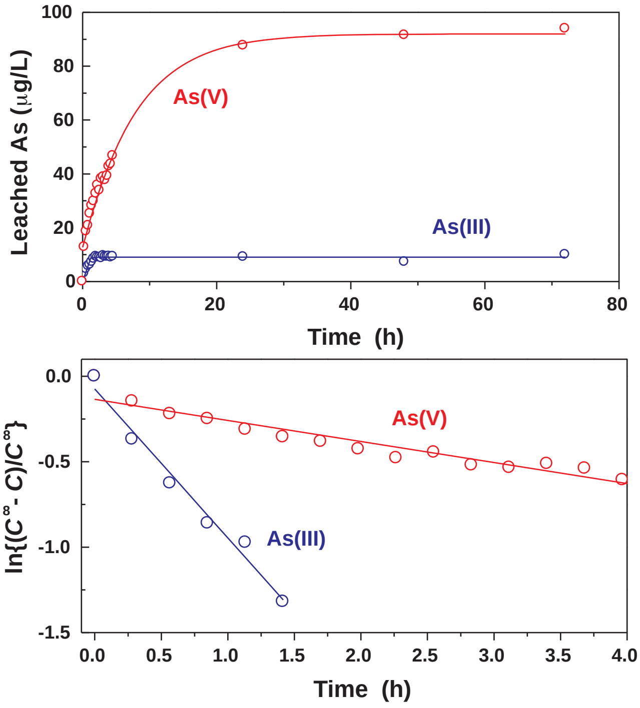

Fig. 5. Long term change in leached As(III) and As(V) (top) and log scale plot of the leached arsenic (bottom) obtained with sample No. 6 in aerobic condition.

4. Conclusions

The SHGFIA is useful tool for analysis of arsenic contained in water and analysis of arsenic leaching from contaminated sediments. For water analysis, addition of EDTA is effective to keep the oxidation state of inorganic arsenic and to prevent the loss of arsenic during the storage. The concentrations of As(III) and As(V) are stable with EDTA for a few days at room temperature even for low concentration samples such as 1 μg L^{-1}. SHGFIA can determine As(III) and As(V) (As(total)–As(III)) automatically and can be used for kinetic analysis. Theoretical treatment was investigated, and the kinetic constants for leaching of As(III) and As(V) are available to obtain from the increase in solution concentration and final concentration of the each arsenic. Leaching of As(III) is much faster than that of As(V) while final concentration of As(III) is lower than As(V). As described above, SHGFIA is useful for leaching analysis and detailed investigation of leaching mechanism is expected with this system, for examples, effect of diverse ions (e.g. phosphate), ionic strength and pH, etc.

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