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Characterization of arsenic leaching in paddy field soil

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Abstract

This paper examines the arsenic leaching characteristics in the paddy field soils. Samples were collected from actual paddy field of Bangladesh which was irrigated by groundwater. Soil samples were tested by sequential extraction procedure (0.1N HCl and 0.1N NaOH) and pH leaching test. Results indicated that about 22-57% of the total arsenic in the samples were 0.1 N NaOH extractable arsenic which seemed to be strongly bound with amorphous iron hydroxides. Leached arsenic concentrations were high in high alkaline pH conditions. Comparing the extraction results with pH leaching test it was observed that leached arsenic in low pH was from the 0.1N HCl extractable arsenic fractions which might be sorbed weakly on the soil surface. At high alkaline conditions (pH 13) the leached arsenic concentrations were as much as 6-10 times higher than at low pH (pH 1) which indicated that strongly bound arsenic would be leached at high alkaline and under low redox conditions.

Keywords: acid-alkali extraction, arsenic contamination, soil arsenic leaching, redox potential, paddy soil

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1. Introduction

Arsenic (As) contamination of groundwater is a major public-health concern in Bangladesh and elsewhere (BGS, 2001; IARC, 2004; Smith et al., 2000). Chronic toxicity of arsenic in humans from arsenic contaminated drinking-water occurs in 61 of 64 districts in Bangladesh, affecting millions of people (BGS and DPHE, 2001). The maximum permissible level of arsenic in drinking-water recommended by the World Health Organization (WHO) is 10 μg/L and, in Bangladesh, it has been adjusted to 50 μg/L by the local authorities (Smith et al., 2000). Being located in the downstream of the mighty rivers, the Ganges and the Brahmaputra a part of the sediments carried is deposited in the flood plains of Bangladesh every year (Ahmed, 2000). Arsenic release from subsurface minerals and sediments is the main cause of groundwater arsenic contamination (Chowdhury et al., 2000).

Where shallow groundwater is contaminated, it is likely that arsenic is present in bioavailable forms in soil and in irrigation water (Ravenscroft et al., 2009). With the increased use of groundwater through irrigation-pumps during the 1990s, arsenic contamination of water at different depths of soil surface was observed (IARC, 2004; Uddin and Khan, 2004). Later in the 2000s, the use of water from both shallow and deep tubewells for irrigation of agricultural lands began, particularly during the dry season (Zahidul, 2003). In all likelihood, the use of arsenic contaminated water for irrigation may have contributed substantially to the spread of arsenic in the top soil-crop-food (Huq and Naidu, 2005; Meharg and Rahman, 2003).

Arsenic can occur in the groundwater in the +3 and +5 oxidation state, namely arsenite [As(III)] and arsenate [As(V)]. As(V) is the predominant species under atmospheric or more oxidizing environment in the pH range of 6–9. It exists predominantly as oxyanions, namely, $H_2AsO_4^-$ or $H_2AsO_4^{2-}$, while As(III) is thermodynamically stable and exists predominantly as H_3AsO_3 or $HAsO_2$ under mildly reducing conditions (Fendorf et al., 1997; Manning et al., 1998; Raven et al., 1998). As(III) is the most common species in anaerobic groundwater and is more toxic than As(V) (Pontius, 1994). Among the species, As(V) binds more strongly with the metal oxides of Fe compared to As(III) species. However, the binding mechanism depends on the various environmental conditions such as pH and redox potential. As(V) adsorbed more efficiently at low pH, while adsorption affinity of As(III) is higher at high pH (Masscheleyn et al., 1991; Yang et al., 2002).

It is generally recognized that total arsenic concentration in the soil is not a good indicator of potential mobility and leaching. Sequential extraction method is widely used to fractionate metals (Ahnstrom and Parker, 1999; Qiao et al., 2003; Tessier et al., 1979). Several extraction methods were used by researchers but were not compared with the effects of important factors such as pH and redox potential. The aim of this study was to evaluate the leaching characteristics of arsenic in the paddy field soil. Accordingly, sequential extraction procedure was employed to determine the major, operationally defined arsenic binding phases. Effect of pH was also investigated separately by batch experiments and compared with extracted fractions.

2. Materials and Methods

2.1 Sampling site and collection procedure

Soil samples were collected from different paddy fields in Khulna, southwestern region of Bangladesh. Fig.1 shows the map of the sampling sites and is indicated as K-1, K-2, K-3, and K-4. The tubewells of these sites showed high concentration of arsenic (81-96% yielded water with more than 50 μg As/L), and the vast majority of farmers used contaminated groundwater for irrigation. Surface samples (0~20 cm) were taken from three sampling points of each site. Moreover, samples were taken from one sampling point of irrigation channel indicated as K-IC. Moisture content (MC) of the soil samples was measured in the laboratory of Khulna University of Engineering and Technology (KUET), Bangladesh on the same day of the collection. Samples were air-dried and homogenized by mixing and brought back to Japan packing with polyethylene bags. Air- dried soil samples were ground and passed through a 0.5 mm sieve.

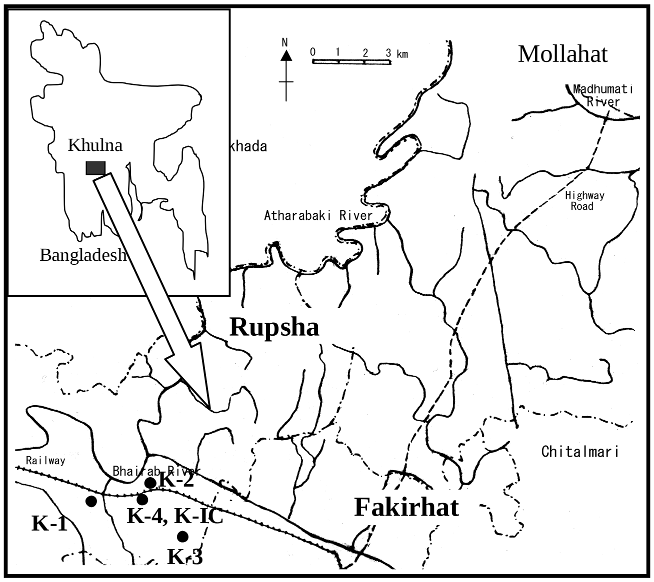

Fig. 1. Geographical locations of sampling site

2.2 Sequential extraction procedure

A two steps extraction procedure was employed sequentially to determine the association of arsenic in the soil samples (Table 1). Reagent grade 0.1N HCl were used for extracting easily soluble, carbonates, Mn oxides and very amorphous Fe oxhydroxides phase and 0.1N NaOH was used to extract metal associated phase (Nakajima and Okubo, 2003). In the extraction experiments, 1.0 g of oven dried soil sample was mixed with 50 ml of 0.1N HCl solution in 100 ml Teflon bottle and shaken for 3 mins at 200 rpm. The HCl extract was obtained by filtration through 1μm pore filter paper (Advantec, No 5C, Japan). The residue of the filtration with the filter paper was remixed with 100 ml of 0.1N NaOH solution and was shaken for 17h at 140 rpm. The NaOH extract was obtained by centrifuge and filtration through 1μm pore filter paper (Advantec, No 5C, Japan). The extract samples were acidified with HNO_3 and analyzed for As and Fe. Residual fraction (Res.) was determined as the difference between total and extracted fractions. Extraction experiments were carried out in the room temperature and triplicate samples were used to ensure the reproducibility.

Table 1
Sequential extraction procedure

Step	Extraction fluid	L/S ratio (ml/g)	Duration	Target phase	References
1	0.1N HCl	50:1	3 mins	Easily soluble, Carbonates, Mn oxides, and very amorphous Fe hydroxides	Nakajima *et al.* 2003 (applied for BAP)
2	0.1N NaOH	100:1	17h	Amorphous Fe hydroxides	Nakajima *et al.* 2003 (applied for BAP)
3	Residual	-	-	Crystalline Fe oxides	-

2.3 pH batch leaching test

Controlled pH experiments were conducted using de-ionized water (DW) as extraction fluid to simulate neutral, low mineralized runoff water. In the pH experiments, 1.0 g of oven dried soil sample (K-4 and K-IC) was mixed with 100 ml of DW in Teflon bottle. pH of the mixed liquor was adjusted to 1.0, 3.0, 5.0, 7.0, 9.0, 11.0 and 13.0 by adding $NaHCO_3$. Then it was shaken for 24h at 140 rpm. The redox potential was also measured at different pH. After shaking the mixed liquor in the bottles was filtered through No.5C filter paper. Percentages of the dissolved arsenic were estimated. pH leaching experiments were carried out in the room temperature and triplicate samples were used to ensure the reproducibility.

3. Analytical Methods

Arsenic standard stock solution of internal standard (1000 ppm), HCl (35%), HNO_3 (60%), H_2SO_4 (97%) and NaOH (96%) were purchased from Nacalai tesque. Inc. Kyoto, Japan. Iron, solution (1000 ppm) and $NaHCO_3$ (99.5%) were obtained from Wako Pure Chemical Industries Ltd. Japan. Fresh calibration standards were prepared by diluting analytical standards in 5% nitric acid. pH and Oxidation reduction potential (ORP) was measured by UC-23 Digital pH/ORP meter (CKC) and ORP was converted to Eh. Organic matter (OM) was determined by the percentage of weight loss after ignition (600^{o}C for 1 hr). Total concentration of arsenic (T-As) and iron (T-Fe) were measured after hot acid digestion with H_2SO_4 and HNO_3. T-As, HCl-As and NaOH-As were determined by graphite furnace atomic absorption spectrometry (GF-AAS) (Hitachi Z2700). T-Fe, HCl-Fe and NaOH-Fe were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES) (Seiko SPS 4000).

4. Results and Discussion

4.1 Soil Characterization

In the current study, MC, OM, T-Fe and T-As in soil of different sites varied (Table 2). Soil pH indicated that almost all site samples were moderately alkaline with the exception of K-IC that had neutral or moderately acidic soil. MC showed almost uniform distribution in almost all the sites. It varied from 25% to 42% and paddy field K-3 and K-4 covered with water during wet season had higher MC value. OM content ranged between 4.4 and 7.1% with K-IC had higher values. T-Fe ranged from 21 to 33 mg/g. T-Fe contents were high seemed to be due to the influence of irrigation water. T-As ranged from 9.0 to 19.3 µg/g except K-IC which had high concentration (60µg/g). Average arsenic content in Bangladesh soil is less than 10 µg/g (Meharg and Rahman, 2003)**.** As compared to average value T-As contents in some paddy field soil were high because of arsenic accumulation during irrigation. K-3 and K-4 containing high T-As among the sites seemed to be influenced by the irrigation groundwater and subsequent rise and fall in groundwater level during wet and dry season. The high concentration of T-As in irrigation channel (K-IC) seemed to be caused by oxidation of Fe (II) to Fe (III) during irrigation and Fe hydroxide particles are formed which bind arsenic partly or completely and settles down in the channel.

Table 2
Characteristics of soil samples (mean ± SD)

Site Name	pH	MC (%)	OM (%)	T-Fe (mg/g)	T-As (μg/g)
K-1	8.56 ± 0.04	30.4 ±1.1	5.66 ± 0.10	22.0 ± 3.51	13.6 ± 5.30
K-2	7.66 ± 0.17	24.9 ±3.9	4.40 ± 0.22	23.8 ± 10.2	9.00 ± 1.12
K-3	7.74 ± 0.29	41.2 ±11	6.03 ± 0.66	21.2 ± 1.36	16.4 ± 4.80
K-4	7.40 ± 0.14	41.7 ±3.2	6.71 ± 0.31	33.3 ± 5.11	19.3 ± 0.96
K-IC[¶]	6.81	24.9	7.08	21.2	60.9

Table 3
Acid-alkali extracted amount of soil samples (mean ± SD)

Site name	HCl-As (μg/g)	NaOH-As (μg/g)	Res-As (μg/g)	HCl-Fe (mg/g)	NaOH-Fe (mg/g)
K-1	0.06 ± 0.01 (0.50%)	2.78 ±0.31 (22.3%)	10.8±5.4 (77.2%)	0.23 ± 0.11 (1.07%)	0.15± 0.24 (0.81%)
K-2	0.24 ± 0.04 (2.7%)	1.97 ±0.26 (22.2%)	6.79±1.1 (75.1%)	0.11 ± 0.04 (0.56%)	0.06± 0.04 (0.36%)
K-3	7.80 ± 2.60 (47.4%)	7.73 ±1.92 (47.6%)	0.88±0.90 (5.0%)	0.20 ± 0.13 (0.98%)	0.07± 0.01 (0.33%)
K-4	2.67 ± 0.88 (13.8%)	10.5 ±2.10 (54.6%)	6.08±1.2 (31.6%)	0.18 ± 0.10 (0.52%)	0.14± 0.01 (0.44%)
K-IC[¶]	4.57 (7.5%)	34.8 (57.1%)	21.5 (35.4%)	1.03 (4.86%)	0.19 (0.90%)

4.2 Sequential extraction results

Table 3 summarized the results from the sequential extraction procedure of the samples. Considerable variation occurred between sites in the amount of arsenic present in extracted fractions**.** In spite of the variation, some general trends were apparent. The acid extractable fraction (HCl-As) was small and accounted for <15% of the T-As except surface layer of K-3 had higher proportion of this fraction 47.4% (7.80 ± 2.6 μg/g). At most of the sites high proportions of T-As were extracted in the NaOH fraction (22-57%). Amount of HCl-As was lower than that of NaOH-As in almost all the samples. HCl-As was bound with carbonate, Mn oxides and very amorphous Fe oxyhydrooxides fraction. The fraction of arsenic more than HCl-As in the soil samples was step 2 (strongly bound with amorphous Fe hydroxides) fractions. Available arsenic, which is considered as weakly bounded and easily releasable, is defined here as resulting from first step and part of step two of the sequential analysis.

In all the samples, the majority of Fe was liberated in the 0.1N HCl extraction steps, which leached about 0.5-4.9% of the total Fe (Table 2). The sequential extraction results on Fe association indicated that almost 95% Fe as amorphous or crystalline oxides. Therefore, in the case of arsenic extraction from the soil surface, arsenic seemed to be sorbed with the Fe. The presence of amorphous and crystalline Fe oxides and arsenic association with them indicated adsorption was the major mechanism for arsenic trapping in the soil (Chatain et al., 2005).

Sequential extraction can provide some insight into the mechanism of arsenic mobilization. On the basis of sequential extraction results, the arsenic extracted through first step was low (except for K-3). This result suggested that arsenic extracted in the first step seemed to be related to some type of adsorption reaction since Fe was concurrently released. Studies illustrated that Fe(III) oxide surface has a high affinity for As(V) capable of forming inner-sphere bidenate, binuclear As(V)-Fe(III) (*Sun and Doner, 1996*). As(III) species also can strongly bond to the Fe(III) oxides surface and form an inner sphere adsorption configuration similar to As(V) (Bayard et al., 2006). In the extraction step with 0.1N NaOH low percentage of Fe dissolution was occurred (0.29-0.81%). This is because of the ligand displacement reaction of hydroxyl ion with arsenic species and high pH conditions that can prevent re-adsorption of arsenic because predominant negatively charged crystalline oxides do not attract the negatively charged oxyanions.

4.3 *Effect of pH and redox potential on arsenic release*

Figure 2 presents the constant pH leaching experiments for arsenic. Eh of the experimental set-up was not controlled as any gas was purged. Moreover, the redox conditions attained in the experiments at different pH leaching test is shown in Fig. 2. A small change of pH was observed during 24h agitation. The leached amount of arsenic was generally much lower than the total metal concentrations and exhibits strong pH dependence. Similar pH dependent arsenic leaching profiles were observed in both soil samples (K-4 and K-IC). With an increase in pH from 1 to 5 the total dissolved arsenic concentrations decreased from 28.1±1.1 μg/L to 16.4±1.0 μg/L for K-IC and from 13.8 ± 0.5 μg/L to 6.82 ± 1.1 μg/L for K-4. Dissolved arsenic concentration was increased at pH 5 (26.6 ± 2.6 μg/L and 15.9 ± 1.5 μg/L for K-IC and K-4, respectively) and dramatically increased at pH 13 (269 ± 29.6 μg/L and 88.3 ± 1.2 μg/L for K-IC and K-4, respectively). Maximum arsenic leaching was observed at pH 13.

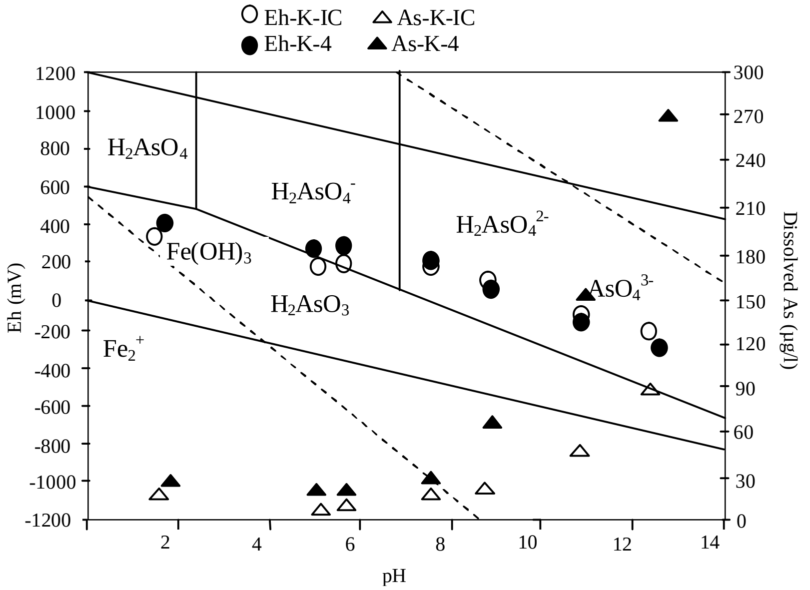

Fig. 2. Effect of Eh and pH on the release of As from soil sample K-IC and K-4.

The predicted effect of redox potential (Eh) and pH on the oxidation state of As is shown in Fig. 2 (Bayard et al., 2006). At pH 1 and Eh of 370-410 mV As(III) is the major dissolved species. On the other hand, Eh was decreased with increasing pH and As(V) was the major dissolved specie at pH 5 to 13 in both samples. The Eh-pH diagram illustrated that total arsenic released from the sample at pH 5 to 13 was the dissolved As(V) (Fig. 2). Although the speciation of leached arsenic was not carried out in this study, however, Ito et al. (Ito et al., 2001) showed that at pH ≥ 5.0 and relatively high Eh (>100 mV) conditions most of the leached arsenic was As(V). This can be explained by the pH and Eh dependent adsorption characteristics of As(V) onto the oxides surface (Evans, 1989; Masscheleyn et al., 1991). The decreasing positive surface charge of the oxides with increasing pH facilitated the desorption of As(V).

Figure 3 presents the constant pH leaching experiments for Fe. With an increase in pH from 1 to 7 Fe concentrations decreased from 41.7 ± 4.1 mg/L to 0.23 ± 0.07 mg/L for K-IC. At pH 9 and 11 it increased to 0.53± 0.22 mg/L and 0.90 ± 0.03 mg/L and again decreased to 0.45 ± 0.02 mg/L at pH 13. For S-3 Fe concentrations decreased from 32.2 ± 0.15 mg/L to 0.67 ± 0.0 mg/L with an increase in pH from 1 to 5. At pH 7 it increased to 2.24 ± 0.01 mg/L and slightly decreased to 1.68 ± 0.0 mg/L at pH 9. Fe concentrations again increased to 6.42 ± 0.28 mg/L at pH 11 and dramatically decreased to 0.16 ± 0.01 mg/L at pH 13. Maximum Fe leaching was observed at pH 1.

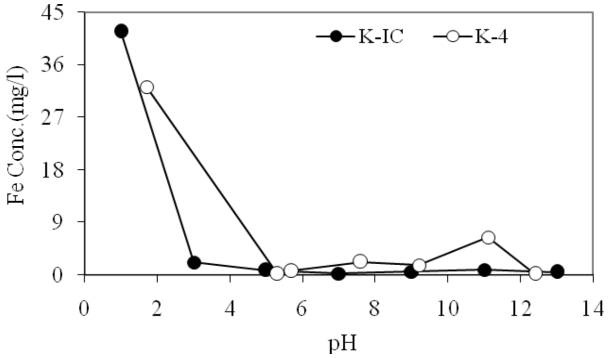

Fig. 3. Effect of pH on the release of Fe from soil sample K-IC and K-4.

The amount of arsenic and Fe leached from the soil sample can be considered as caused by either dissolution of less resistant minerals or sorption/desorption mechanisms which are influenced by pH. The dissolved arsenic concentrations at pH 1 (2.8 and 1.4 µg/g for K-IC and K-4, respectively) was about 61% and 56% of acid extractable (carbonate) phases, indicating either release of weakly sorbed As on ferric hydroxides or dissolution of carbonates driven by cation exchange (Bayard et al., 2006; Masscheleyn et al., 1991). With an increase of pH to 5 and highly aerobic redox conditions dissolved Fe could be oxidized to ferric hydroxides and co-precipitated with arsenic resulting in lower concentration in dissolved arsenic. With an increase in pH beyond pH 5, about 11 % of the total arsenic was leached at pH 9 in both soil samples, whereas about 44% of total arsenic was leached at pH 13. By contrast, about 8.2% to 9.7% of the total Fe was released at pH 1 and about 1.0%-0.04% of total Fe was leached at highly alkaline conditions (pH 13) in both samples. The increase of dissolved arsenic with pH can be attributed to the replace of hydroxyl ions with arsenic on the ferric hydroxides sorption site, resulting the desorption of arsenic oxyanions (Fendorf et al., 1997; Price and Moore, 1982; Yang et al., 2002).

4.4 Comparison of As Release at Different Extraction Conditions

The concentrations of arsenic of K-IC and K-4 soil samples extracted at different pH conditions were compared with the concentrations measured by sequential extraction analysis (Fig. 4). Percentage of arsenic extracted at pH 3 and pH 7 (2.94% and 4.36% for K-IC and 2.71% and 7.77% for K-4, respectively) was lower than the 0.1 N HCl-As. Extractable arsenic (28.3% and 46.8% for K-IC and 23.8% and 43.0% for K-4, respectively) at pH 11 and pH 13 (highly alkaline conditions) was larger than 0.1 N HCl-As but smaller than 0.1 N NaOH-As (at pH 11) and located nearly 0.1 N NaOH-As (at pH 13). These results indicated that at high alkaline conditions, strongly bonded arsenic would be released in the environment. Therefore, this laboratory experiments suggested maintaining non-alkaline and high redox conditions for minimum arsenic solubility and mobilization from soil.

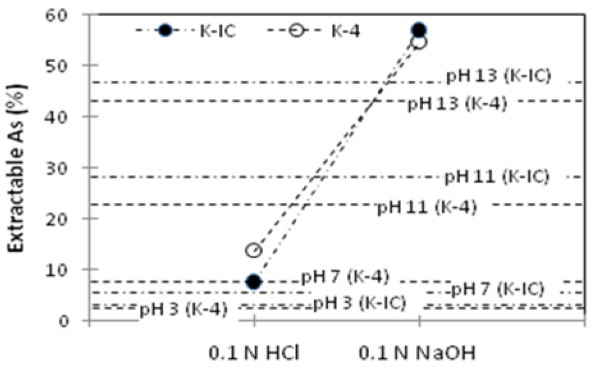

Fig. 4. Comparison of As release at different pH conditions and by sequential extraction on soil samples K-IC and K-4.

5. Conclusions

Paddy field soil samples contained Fe and arsenic was subjected to sequential extraction procedure and batch leaching test at different pH. Characterization of soil samples using sequential extraction procedure indicated the arsenic associated either with the amorphous iron oxyhydroxides or with crystalline iron oxides (goethite or hematite). pH batch leaching studies showed a strong dependence of pH and Eh on arsenic leaching from the soil sample. Arsenic leaching was strongly correlated with that of Fe at low pH, occurred either dissolution of the amorphous Fe-oxyhydroxides bearing phase or the less resistant carbonate phase in the acidic region. Arsenic leaching was maximum in the alkaline conditions indicated desorption of arsenic in high pH and low Eh conditions.

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