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Arsenic, Sb and Bi contamination of soils, plants, waters and sediments in the vicinity of the Dalsung Cu–W mine in Korea

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Abstract

The objective of this study is to investigate environmental contamination derived from metalliferous mining activities. In the study area, the Dalsung Cu–W mine, soils, various crop plants, stream waters, sediments and particulates were sampled in and around the mine and analyzed for As, Sb and Bi by ICP-AES with a hydride generator. In addition, soil pH, cation exchange capacity, loss-on-ignition and soil texture were also measured. Concentrations of As, Sb and Bi in surface soils sampled in the mine dump sites averaged 2500, 54 and 436 μg g^{-1}, respectively. Relatively lower concentrations, however, were found in soils from alluvial and high land sites and household garden sites. Arsenic, Sb and Bi contents in plant samples varied depending upon their species and parts, with higher concentrations in spring onions, soybean leaves and perilla leaves and lower levels in red peppers, corn grains and jujube grains. These results confirm that elemental concentrations in plant leaves are much higher than those in plant grain. Elevated levels of As, Sb and Bi were also found in stream sediments sampled in the vicinity of the mine and decreased with distance from the mine. Concentrations in stream water samples ranged from 0.8 to 19.1 μg As l^{-1} and from 0.3 to 8.4 μg Bi l^{-1}; all the samples contained less than 1.0 μg Sb l^{-1}. Because of very low particulate loading at the time of sampling, the metal contents in particulates were very low (<8.2 μg As l^{-1},< 0.22 μg Sb l^{-1} and 2.8 μg Bi l^{-1}. This may be mainly due to the low solubility of those elements under moderately acidic and oxidizing conditions of the mining area. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

The metalloids, As, Sb and Bi are members of Group VA in the Periodic Table and are similar in geochemical behavior, commonly associated with non-ferrous deposits. Average concentrations of As, Sb and Bi in the Earth's crust are 1.5, 0.2 and 0.048 μg g^{-1}, respectively, and median values of the elemental composition of uncontaminated soils are 6.0 μg As g^{-1}, 1.0 μg Sb g^{-1} and 0.2 μg Bi g^{-1} (Bowen, 1979). Contaminated soils from var-

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ious sources, however, contain significant levels of the elements. The most important emission sources to the environment for the metalloids are the non-ferrous mining and smelting industry (Adriano, 1986). Various studies have been undertaken for As contamination derived from mining (Azcue and Nriagu, 1995; Loredo et al., 1999; Savage et al., 2000) and smelting (Manz and Castro, 1997). It was estimated that worldwide emission into soils in 1983 (median values) were 82×10^6 kg year^{-1} for As and 26×10^6 kg year^{-1} for Sb, and 10–20% of the As and most of the Sb were derived from mine tailings and smelter slags and wastes (Nriagu and Pacyna, 1988).

The concentrations of As, Sb and Bi in edible plants is generally low, often being close to the limit of detection, even when the crops are grown on contaminated land (O'Neill, 1990). However, some researches have found high contents of the elements in plants grown on contaminated soils from mining and smelting activities (Temple et al., 1977; Xu and Thornton, 1985; Li and Thornton, 1993; Loredo et al., 1999). In comparison with As, Sb and Bi concentrations in natural environment, relatively high contents of the elements were found in sediments and waters due to anthropogenic inputs including mineralization, industry, mining and smelting (Aston et al., 1975; Mok and Wai, 1990; Chatterjee et al., 1993).

The study area, the Dalsung Cu–W mine located in southeast Korea was one of the largest Cu and W mines, with production reaching a maximum in the 1960s when rates accounted approximately 5–15% of the total Cu and 3–5% of the total W outputs in the country. The study area is underlain by the Gyoungsang sedimentary formation (Middle-Upper Jurassic era) and the geology around the mine is greatly influenced by volcanic activity. The ore minerals are chalcopyrite ($CuFeS_2$) and wolframite ($(Fe,Mn)WO_4$) with a minor amount of arsenopyrite (FeAsS), bismuthinite (Bi_2S_3) and pyrite (FeS_2).

This present study examines the degree and extent of As, Sb and Bi contamination of soils, plants waters and sediments derived from metalliferous mining activities in Korea. This paper also discusses bioavailability of As, Sb and Bi and factors influencing uptake of the elements by crop plants.

2. Materials and methods

2.1. Soils

Surface soils (0–15 cm) were taken at the mine dump sites, alluvial and high land sites and household garden sites, and a nearby control area underlying the same geology in the mine but without mining activities. The samples were collected with a 2.5-cm diameter hand auger (a stainless steel screw) and a hand spade. Each sample comprised a composite of nine subsamples taken across a $1\times1\text{-m}^2$. The samples were air-dried at 25 °C for 72 h and then disaggregated, sieved to −10 mesh or <2 mm and ground to a fine powder in a 'Tema' mill. This finely milled soil (−80 mesh or <180 μm) was used for chemical analysis and the <2-mm fraction was used for measuring pH (sample:de-ionized water = 1:2.5), organic matter content using the loss-on-ignition method (Ball, 1964), cation exchange capacity and soil texture analysis (Hesse, 1971). Simultaneous determinations of As, Sb and Bi concentrations in soils and sediments were determined by ICP-AES (model no. ARL 34000) using a hydride generation method after wet ashing with magnesium nitrate ($Mg(NO_3)_2$). The generation method involved the reduction of these elements with sodium tetrahydroborate to give As, Sb and Bi hydrides, which were then swept into the ICP-AES source. First, 0.25-g samples of dried, milled soils and sediments were weighed into 50-ml clean numbered beakers, and 1 ml of saturated magnesium nitrate solution and 1 ml nitric acid added and mixed thoroughly with the samples. The beakers were placed in a furnace and heated at 450 °C for 6 h. After cooling the beakers were taken from the furnace and 5 ml of concentrated hydrochloric acid was added. The beakers were then covered with clingfilm and placed on a rocking machine for 12 h. After removing the clingfilm, 5 ml of freshly prepared 0.2% (w/v) potassium iodide solution was added and the beakers shaken gently by hand. The solutions were decanted into polystyrene test tubes and analyzed by ICP-AES with a hydride generator (Thompson and Walsh, 1988).

2.2. Crop plants

Various crop plants including corn grain (*Zea mays*), jujube grain (*Zizyphus jujuba*), perilla leaves (*P. frutescens var. japonica*), red pepper (*Capsicum annuum*), soybean leaves (*Glycine max*) and spring (Welsh) onions (*Allium cepa*) were sampled in and around the mine. Similar materials were also collected at a nearby control area. The samples were thoroughly washed in deionized water and dried in a clean room at room temperature (20 °C) for 5 days. The dried samples were milled to a fine powder in a 'Cyclotec 90' sample mill, digested in fuming nitric acid and magnesium nitrate and analyzed by ICP-AES as for soils (Thompson and Walsh, 1988).

2.3. Stream sediments, waters and particulates

Sediment and water samples were taken up to 1.5 km downstream from the mine at 50–150-m intervals. The sediments were sampled using a stainless steel trowel and the preparation and analysis of the samples were the same as those for soils. Water samples were filtered with a hand-pump water sampler using 0.45-μm membrane filter paper (4.5 cm diameter). After immediate acidification ($pH < 2$) with concentrated HCl, the sample was stored in a cooling box (<4 °C). A 5-ml of freshly prepared 0.2% (w/v) potassium iodide solution were added to 5 ml of the water samples and directly analyzed by ICP-AES with a hydride generator for As, Sb and Bi. The digestion procedure for filter paper samples with particulate matter was similar to that for plants. The sampling locations of soils, plants, sediments and waters are shown in Fig. 1.

A rigorous quality control program was implemented, which included reagent blanks (average values 0.0001 $\mu g\ ml^{-1}$ for As, Sb and Bi), replicate samples, in-house reference materials (HRM1 and 2 for soils and sediments, HRM12 and 14 for plants) and certified international reference materials (SRM2711 for soils and sediments and SRM1573 for plants) (Ramsey et al., 1987). The precision and bias of chemical analysis were less than 10%.

3. Results and discussion

3.1. Physical and chemical properties of soils, sediments and waters

Properties of soils, sediments and waters are summarized in Table 1. It is well known that most soils in Korea have a slightly low pH, are very sandy, have lower organic matter content and a low cation exchange capacity (Kim, 1985). Soils in the study area share similar characteristics. Because of the weathering of sulphide minerals, pyrite and pyrrhotite from mine waste materials, relatively lower pH values were found in the mine dump soils, with the range of 3.0–5.7. Average soil pH values from the other sites are close to the average Korean soil pH of 5.7 reported by Kim (1985). In comparison with other sites, the mine dump soils contain low organic matter content (average of 3.2%) ($P < 0.05$). These lower organic matter contents in the soils are possibly due to the paucity of vegetation on the soils, resulting from the poor conditions for plant growth (low soil pH, unbalanced nutrition and a high sand fraction). This condition also contributes to the low cation exchange capacity (CEC), with a minimum value of 3 meq/100 g. However, the CEC of cultivated soils (garden and control soils) is higher than that of uncultivated soils (mine dump and alluvial and high land soils) ($P < 0.05$), possibly due to continuous soil management for agricultural land usage. According to USDA (1975), the mine dump soils are classified as sand and the other soils are classified as loamy sand and/or sandy loam.

Similar range of pH values were found in the sediment and water samples, with the ranges of 3.0–6.7 and 3.2–5.5, respectively (Table 1). These acidic conditions are mainly due to the weathering of sulphides existing in mine waste materials and sediments and waters sampled up to 1.5 km downstream also had low pH values (4.5–5.5). The organic matter contents and cation exchange capacity of sediments are similar to those of soils sampled in the immediate vicinity of the mine dump sites, resulting from directly influence on

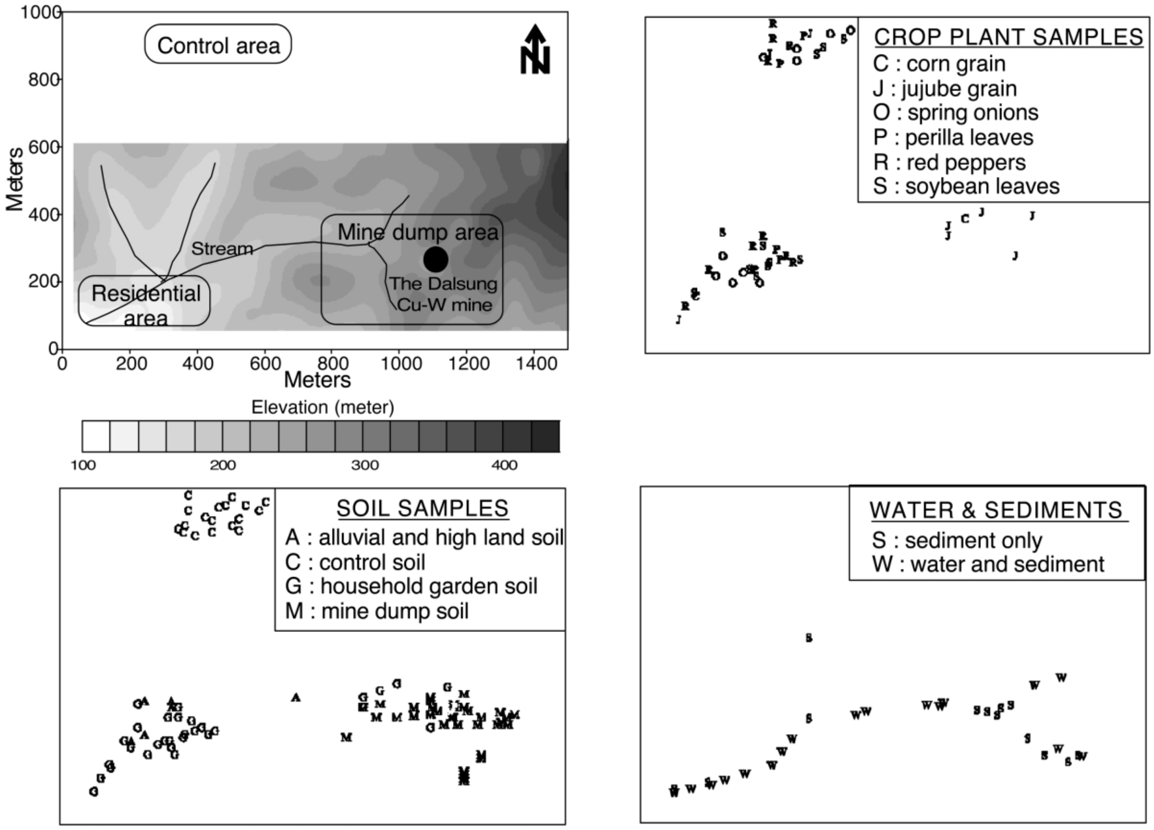

Fig. 1. Sampling locations of soils, crop plants, sediments and waters in the study area.

the movement of the waste materials downstream the mine dump sites.

3.2. Arsenic, Sb and Bi concentrations in soils

The 'total' As, Sb and Bi concentrations in surface soil samples are shown in Table 2. Significant As, Sb and Bi concentrations were found in the mine dump sites due to mineralization associated with those metalloids, with the average values of 2500, 54 and 436 μg g^{-1}, respectively. These levels are tens to hundreds times higher than those in world average soils. In comparison with soils from a nearby control soils, relatively higher concentrations were also found in soils sampled in the alluvial and high land sites and household garden sites where were strongly influenced by the mining activities. In Korea, the guideline for As of 6.0 μg g^{-1} (extracted by 1 M HCl) in agricultural land has set in Korea (DoE Korea, 1996). Because the concentrations of As in soil samples were determined by extraction using standard magnesium nitrate solution, it is not easy to convert to As contents extracted by 1 M HCl. However, it can be expected that soils in the study area are highly contaminated by the mining activities and large area in the vicinity of the mine would be prevented from agricultural land usage due to contamination of As.

3.3. Arsenic, Sb and Bi concentrations in crop plants

It is generally accepted that concentrations in edible plants grown on uncontaminated or unmi-

Table 1
Physical and chemical properties of soils, sediments and waters

Sample type		pH	LOI (%)	CEC (meq/100 g)	Size fraction (%)		
					Sand	Silt	Clay
Mine dump soils	Range	3.0–5.7	0.4–6.9	3.3–18.4	70–90	1–15	0.2–16
($N=28$)	M±S.D.	$4.1^{a}\pm0.7$	$3.2^{a}\pm1.4$	$11.4^{a}\pm3.4$	$86^{a}\pm6.9$	$9^{a}\pm3.7$	$5.7^{a}\pm3.7$
Alluvial and high land soils	Range	4.2–6.5	6.8–9.4	15.5–21	63–66	20–23	11–14
($N=6$)	M±S.D.	$5.3^{b}\pm0.8$	$7.9^{b}\pm0.9$	$17.4^{b}\pm1.9$	$66^{b}\pm2.4$	$22^{b}\pm1.2$	$12^{b}\pm1.2$
Household garden soils	Range	4.1–7.7	4.6–14	15–46.7	56–80	13–35	0–17
($N=30$)	M±S.D.	$5.3^{b}\pm0.8$	$7.9^{b}\pm0.9$	$23^{c}\pm10.3$	$70^{c}\pm5.7$	$20^{b}\pm5.1$	$11^{b}\pm3.7$
Control site soils	Range	4.9–6.6	5.3–9.3	13–53	55–74	16–41	2–16
($N=15$)	M±S.D.	$5.6^{b}\pm0.5$	$6.7^{c}\pm1.0$	$30\ ^{c}\pm14.3$	$65^{b}\pm4.4$	$23^{b}\pm5.7$	$13^{b}\pm3.4$
Sediments	Range	3.0–6.7	1.9–6.9	6.9–16	–	–	–
($N=29$)	M±S.D.	4.1±0.7	3.7±0.9	11.8±2.1	–	–	–
Waters	Range	3.2–5.5	–	–	–	–	–
($N=17$)	M±S.D.	3.8±0.5	–	–	–	–	–

LOI, loss-on-ignition (%); CEC, cation exchange capacity (meq/100 g); M±S.D., mean±standard deviation; *N*, numbers of sample. Where mean values share the same letter suffix for a given soil property, there is no statistically significant difference between the sampling sites at $P<0.05$. Where mean values have a different letter suffix for a given soil property, a statistically significant difference between the sampling sites occurs at $P<0.05$.

neralized soils are 0.01–1.5 μg As g^{-1}, 0.0001–0.2 μg Sb g^{-1} and <0.06 μg Bi g^{-1} based on dry weight (DW) (Bowen, 1979). It is reported that the levels of As in the plants do not directly reflect the As concentrations in soils and the relative order and magnitude of plant As accumulation remained constant mainly due to low bioavailability of the element (O'Neill, 1990). Xu and Thornton (1985) reported that concentrations of As ($\mu g\ g^{-1}$, DW) in vegetables grown on contaminated soils by mining and smelting activities in Cornwall, UK averaged 0.85 in lettuce, 0.21 in carrot, 0.20 in onions, 0.04 in pea and bean. Li and Thornton (1993) reported that most of herbage grown on mining and smelting areas in the UK contained 0.03–4.86 μg As g^{-1}, <0.02–0.45 μg Sb g^{-1} and <0.01–0.18 μg Bi g^{-1} (DW). Table 3 shows a summary of As, Sb and Bi concentrations in crop plants sampled in the study area. Although all the samples contained within normal range of As, Sb and Bi reported by Bowen (1979), spring onions and some soybean leaves contain relatively higher As, Sb and Bi concentrations compared with edible crops grown on uncontaminated soils. This study confirmed that plant leaves (soybean leaves, spring onions and perilla leaves) have higher As, Sb and Bi concentrations than plant grain or fruit (corn grain, red pepper and jujube grain) ($P<0.05$). Thus, As, Sb and Bi concentrations in the crop plats sampled decrease in the order: spring onions > soybean leaves > perilla leaves ≈ red peppers > corn grain ≈ jujube grain.

Table 2
Arsenic, Sb and Bi concentrations in surface soils (0–15 cm depth) in the study area ($\mu g\ g^{-1}$)

Sample type	As		Sb		Bi	
	Range	M±S.D.	Range	M±S.D.	Range	M±S.D.
Mine dump soils	539–9380	$2500^{a}\pm1944$	6–165	$54^{a}\pm33$	42–1510	$436^{a}\pm358$
Alluvial and high land	52.9–127	$96.4^{b}\pm30$	2.0–3.7	$3.0^{b}\pm0.6$	$3.8^{b}\pm19.0$	$13.3^{b}\pm6.3$
Household garden soils	58–788	$133^{b}\pm130$	1.4–12.6	$32^{b}\pm2.0$	4–186	$21^{b}\pm32$
Control site soils	5.1–25.3	$13.6^{c}\pm5.4$	0.9–1.5	$1.1^{c}\pm0.3$	0.9^{c}–1.8	1.4±0.3

See Table 1 for explanation of abbreviation.

Table 3
Arsenic, Sb and Bi concentrations in crop plants in the study area ($\mu g\ g^{-1}$, DW)

Sample type	Location	*N*	As		Sb		Bi	
			Range	M±S.D.	Range	M±S.D.	Range	M±S.D.
Corn grain	Mining site	3	0.03–0.06	0.04±0.02	<0.01		0.01–0.02	0.01±0.01
	Control site	1	<0.01		<0.01		0.03	
Red pepper	Mining site	6	0.03–0.12	0.07±0.03	<0.01		0.01–0.02	0.01±0.01
	Control site	4	0.01–0.03	0.01±0.02	<0.01		0.01–0.04	0.02±0.02
Soybean leaves	Mining site	8	0.31–1.92	0.75±0.50	0.01–0.04	0.02±0.01	0.03–0.13	0.06±0.04
	Control site	3	0.08–0.17	0.11±0.05	0.01–0.03	0.02±0.01	0.02–0.08	0.04±0.03
Spring onions	Mining site	4	2.38–3.10	2.78±0.31	0.01–0.04	0.04±0.07	0.05–0.42	0.25±0.19
	Control site	2	0.35–1.08	0.72±0.52	<0.01		0.06–0.25	0.16±0.13
Jujube grain	Mining site	6	0.02–0.05	0.03±0.01	<0.01		0.01–0.04	0.02±0.02
	Control site	2	0.03–0.07	0.05±0.03	0.01–0.05	0.03±0.02	0.01–0.02	0.01±0.01
Perilla leaves	Mining site	3	0.54–1.18	0.81±0.33	<0.01		0.04–0.11	0.08±0.04
	Control site	2	0.15–0.19	0.17±0.03	<0.01		0.05–0.05	0.05±0

See Table 1 for explanation of abbreviation.

3.4. Soil–plant relationship

Concentrations of As, Sb and Bi in crop plants can be influenced by various factors including total or extractable contents of the elements in soils, soil pH, organic matter contents of soils, soil texture, plant species, plant part and age and climatic condition. Concentrations of As, Sb and Bi in soils, whether total or extractable, are the most important factors affecting elemental concentrations and uptake by plants. Although the extent of the uptake by plants varies depending upon various factors, it is generally agreed that concentrations of the elements in plants increase with increasing those in soils (Adriano, 1986; O’Neill, 1990; Li and Thornton, 1993). In study area, statistically significant correlation of As concentrations in plants to those in soils was found in red pepper and spring onions ($P<0.05$). However, no considerable relationship between Sb and Bi concentrations in plants and soils were found in plant samples due to little variation of the elemental contents in plants sampled. In general, the bioavailability of the As, Sb and Bi is affected by variation of soil pH. Although As toxicity effects on plants increase under more acid conditions when As-binding species become more soluble, the uptake of As by plants may be increased on higher pH soils (O’Neill, 1990). In this study, there is no significant relationship between concentrations of As, Sb and Bi and soil pH. Other soil factors (organic matter contents, cation exchange capacity, soil texture) can give some influence on the elemental uptake by the crop plants sampled in the study area. However, an individually significant factor affecting the availability of the As, Sb and Bi in crop plants were not found in this study. Thus, it can be concluded that the uptake of the elements by the plants can be controlled by the combination of various factors.

3.5. Arsenic, Sb and Bi concentrations in stream sediments and waters

Concentrations of As, Sb and Bi in stream sediments, waters and particulates are summarized in Table 4. Although the concentrations in the sediments are widely ranged, most of the samples contain elevated levels of the elements, with average values of 1110 μg As g^{-1}, 22 μg Sb g^{-1} and 161 μg Bi g^{-1}. These levels are tens to several hundred times higher than those in uncontaminated sediments and are derived from the movement of mine waste materials scattered in the vicinity of the mine. In comparison with sediments, very low concentrations of As, Sb and Bi were found in stream waters, containing <20 μg As l^{-1}, <1 μg Sb l^{-1} and <8.4 μg Bi g^{-1} (Table 3). This may be due to the low solubility of the elements under the acidic (pH 3–4) and oxidizing (400–500 mV)

Table 4
Arsenic, Sb and Bi concentrations in sediments, waters and particulates in the study area

Sample type	*N*	As		Sb		Bi	
		Range	M±S.D.	Range	M±S.D.	Range	M±S.D.
Sediments ($\mu g\ g^{-1}$)	29	119–2400	1110±474	0.4–42	22±10	9–462	161±94
Waters ($\mu g\ l^{-1}$)	17	1–50	21±10	<1		<1	
Particulates ($\mu g\ l^{-1}$)	17	0.3–8.2	2.3±2.4	0.01–0.69	0.10±0.17	0.09–2.78	0.58±0.73

See Table 1 for explanation of abbreviation.

conditions. Thus, considerable amount of As, Sb and Bi in the stream sediments may be resulting from the precipitation of the elements on the stream. Masscheleyn et al. (1991) also reported that As solubility is low at low water pH and under oxidizing conditions.

3.6. Distribution patterns of As, Sb and Bi in soils, sediments and waters

Scatter diagrams for As, Sb and Bi in soil, sediment and water samples are shown in Fig. 2. Soils taken in the mine dump site contain relatively higher As, Sb and Bi concentrations. Jung (1995) estimated the total quantities of As, Sb and Bi present in the mine dump sites (0–15 cm depth) to be 8640, 160 and 1080 kg, respectively. Large amount of those elements may be continuously dispersed downstream and downslope from the mines by a clastic movement through wind and water. Enrichment of the elements was also found in stream sediments sampled in the immediate vicinity of the mine and decreased gradually with distance from the mine. However, some sediment samples taken at 1 km away from the mine also contained high levels of the elements due to a clastic movement by water. In comparison with the concentrations in sediment samples, very low contents of the elements were found in stream water samples, even taken in the vicinity of the mine. This may be mainly due to very low solubility of the elements under moderately acidic and reducing conditions of the mine.

4. Conclusions

Soils sampled in the study area were characterized by low pH (3.0–5.7), low organic matter contents (<1–7%), a normal range of cation exchange capacity (3–18 meq/100 g) and high fraction of sand (70–97%). Soils and waste materials sampled from the mine dumps average 2500 μg As g^{-1}, 54 μg Sb g^{-1} and 436 μg Bi g^{-1} which are tens to several hundreds times greater than the worldwide average concentrations in soils.

Although concentrations of As, Sb and Bi in crop plants vary with their species, relatively higher values are found in plant leaves and lower in grain and fruit. Thus, in this present study, concentrations of the elements in crop plant samples decrease in the order: spring onions > soybean leaves > perilla leaves ≈ red peppers > corn grain ≈ jujube grain. The accumulation ratios (defined as elemental concentration in plant divided by that in soil) are very low due to the low bioavailability of the elements in plants.

Elevated levels of the As, Sb and Bi are found in stream sediments, especially taken in the vicinity of the mine. These concentrations gradually decrease with distance from the mine. In comparison with the concentrations in the sediments, very low contents of the element are found in stream waters, even taken in the vicinity of the mine. This may be mainly due to the low solubility of those elements under moderately acidic and oxidizing conditions of the mining area.

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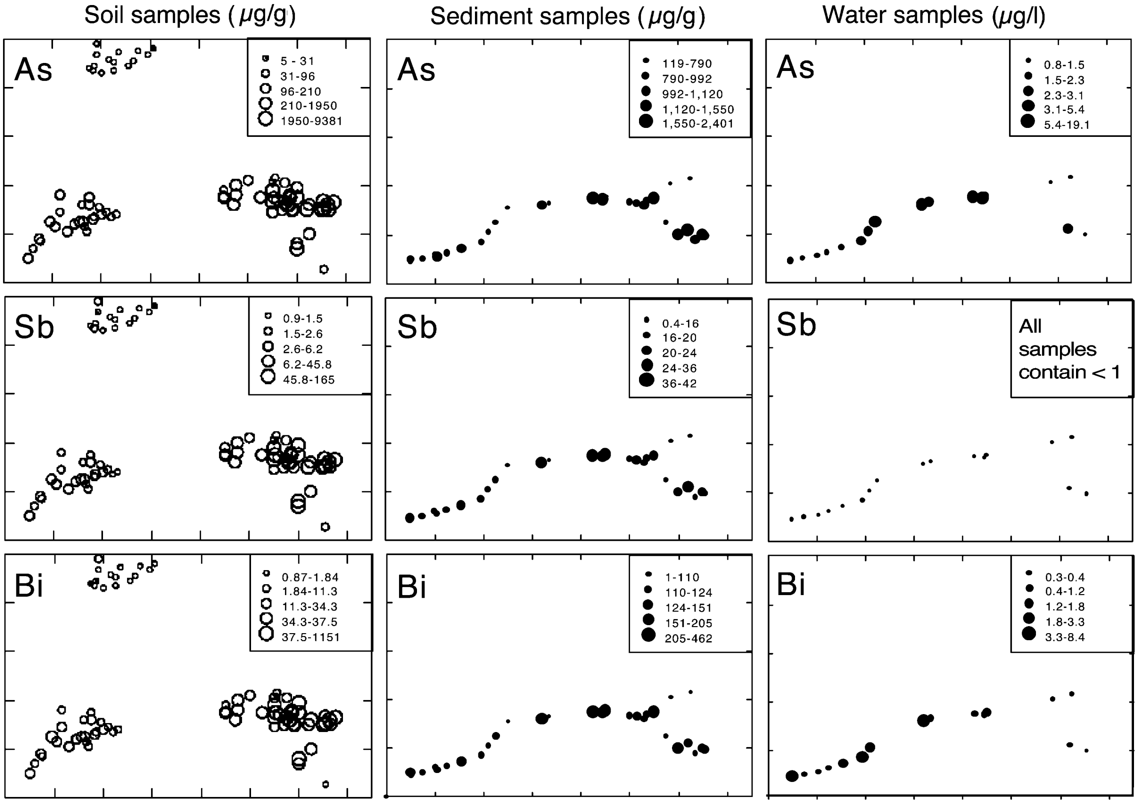

Fig. 2. Diagrams for As, Sb and Bi concentrations in soils, sediments and waters in the study area.

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