

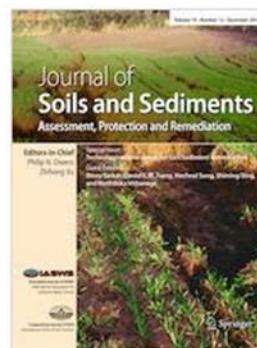


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Support 

Sources and distribution of arsenic in agricultural soils of Central Mexico

Gabriela A. Zanor¹  · María Gabriela García² · Luis Eduardo Venegas-Aguilera¹ · Adriana Saldaña-Robles³ · Noé Saldaña-Robles³ · Oscar A. Martínez-Jaime⁴ · José Jesús N. Segoviano-Garfias¹ · Luis F. Ramírez-Santoyo⁴

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Abstract

Purpose The concentrations and distribution of arsenic (As) in two different soil types (Vertisols and Entisols) of Central Mexico impacted by mine activities and irrigation with As-rich groundwater are analyzed in order to determine their impact on the soil quality, and to contribute reliable data that may help to assess the environmental risk that represents the progressive accumulation of As in the arable soils of Guanajuato.

Materials and methods Two Entisol and two Vertisol profiles located in the Guanajuato state (Mexico) were described and sampled from ~ 1.20-m-deep pits. Soils are irrigated with As-rich deep and shallow groundwaters that were sampled from irrigation boreholes. Additionally, a Vertisol profile located in a parcel not impacted by irrigation was sampled and used as a control soil. Minerals were identified by X-ray diffraction (XRD) and scanning electron microscopy (SEM) coupled with dispersive X-ray spectrometry (EDS). Geoaccumulation indexes (Igeo) were calculated to evaluate As enrichment with respect to a control soil and the Upper Continental Crust (UCC). Anions and cations of groundwater were analyzed by high-performance liquid chromatography (HPLC) and by inductively coupled plasma atomic emission spectroscopy (ICP-AES), respectively. As in soils was determined by ICP-AES.

Results and discussion Near total As concentrations are higher in Entisols (mean As value = 7.20 mg/kg) than in Vertisols (mean As = 1.02 mg/kg). As concentrations in the control soil are lower (0.34 to 0.70 mg/kg). The in-depth distribution of As in Vertisol profiles reveals that the higher As concentrations are found in the uppermost horizons (10 cm) and they tend to decrease with depth. In Entisols, As concentrations do not follow a vertical trend. Igeo values of As indicate moderate to heavy As contamination in Vertisols and moderate contamination in Entisols. SEM-EDS analyses revealed the presence of some potential As-bearing minerals such as magnetite and abundant Fe oxides and Ti-Fe coatings precipitated onto feldspar grains, particularly in Entisols.

Conclusions Irrigation of Vertisols with As-rich groundwater determines As concentrations in the uppermost horizons that exceed the natural background of the region (0.4 mg/kg). In depth, clay grain-sized particles inhibit the downward migration of As, while Fe oxides and organic matter scavenge As by adsorption. As concentrations in Entisols are higher, and the in-depth distribution of this element is controlled by periodic contributions of As-bearing minerals delivered from mine prospects located at the river's catchments.

Keywords As adsorption · As-rich irrigation water · Fe oxides · Entisols · Geoaccumulation index · Vertisols

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1 Introduction

Soil is a living, dynamic resource at the surface of the Earth (Lindbo et al. 2012) that produces biomass, regulates the hydrological cycle, fixes greenhouse gases, and acts as an environmental filter (Porta et al. 2010). In the last decades, urbanization, industrial, and agricultural activities have exerted a great impact on the soil resource all over the world. According to Hatfield et al. (2017), soil degradation is the reduction of the capacity of a soil to perform specific services, especially farming. Most of the inorganic contaminants in soils are heavy metals or potentially toxic trace elements (PTE), which are frequently in concentrations above the average values for world soils (Alloway 2013), affecting food security in most countries (Tóth et al. 2016). Arsenic (As) is one of the most hazardous PTE because it is highly toxic for human health (Sarkar and Paul 2016) and highly mobile in the environment. The presence of As in soils is greatly related to geological factors, such as rock weathering, presence of nearby mineral deposits, and volcanic activity (Mandal and Suzuki 2002). Therefore, its concentration in soils depends on the nature of the parent material. Background concentrations do not generally exceed 15 mg As/kg, although concentrations ranging from 0.2 to 40 mg As/kg have been reported worldwide (Walsh et al. 1977; Chen et al. 2002; Bundschuh et al. 2008). Anthropogenic sources, such as mine wastes and agrochemicals, may also contribute As to the soil environment (e.g., Jiang and Singh 1994; Nieva et al. 2016). In addition, the use of As-rich groundwater for irrigation can enhance the accumulation of this element in soils, resulting in concentrations far exceeding the background level or safety level for agriculture soils (Chi et al. 2018).

The mobilization of As in the edaphic system depends not only on the chemical conditions of the system (e.g., pH, redox potential, salinity), but also on soil properties, such as texture, organic matter content, and moisture (Yan-Chu 1994). Dissolved arsenic anionic species present in pore waters infiltrating throughout the soils can be scavenged by soil minerals and organic matter, by adsorption onto Fe and Mn (hydr)oxides, clay minerals, and organic matter (i.e., Goldberg 2002; Giménez et al. 2007; Hossain et al. 2008; Zeng et al. 2008; Garcia et al. 2014; Otero et al. 2016). Fe and Mn (hydr)oxides are the most effective As scavengers, usually present in soils in the form of thin coatings deposited onto silicates or as individual grains or nodules.

Agriculture (strawberries, lettuce, broccoli, barley, and goat milk) and cattle raising are the most important economic activities in the state of Guanajuato, contributing about 4.2% to the national gross domestic product (Instituto Nacional de Estadística y Geografía, INEGI). These primary activities, however, have caused degradation and chemical alteration of soils that resulted in the loss of fertility, increased salinity, compaction, and contamination (SEMARNAT 2015).

In Mexico, there is a great concern regarding the high levels of As determined in the water and soil resources (e.g., Razo et al. 2004; Armienta and Segovia 2008; Mar Camacho et al. 2011; Morales et al. 2016) due to both natural and anthropogenic causes. In the study area, for example, Hernández-Silva et al. (2005) reported total As concentrations in top soils and sediments of the Guanajuato River's drainage basin ranging from 0.009 to 110 mg/kg, and they attributed these levels to the presence of mineralized areas and mine activities at the river's catchments. A recent study by López-Pérez et al. (2017) reported total As concentrations in surface soils of the Guanajuato state, ranging from 0.01 to 14.90 mg/kg. Although these soils are usually irrigated with groundwater with mean As concentration of 0.33 mg/L (Rodríguez et al. 2016), the impact of this practice on the As levels of the irrigated soils has not been assessed so far.

In this work, the concentrations and distribution of As in two different soil types of Central Mexico impacted by mine activities and irrigation with As-rich groundwater are analyzed in order to determine their impact on the soil chemistry, and to contribute reliable data that may help to assess the environmental risk that represent the progressive accumulation of As in the arable soils of Guanajuato.

2 Materials and methods

2.1 Study area

The study area is located in the southwestern part of the state of Guanajuato, close to the city of Irapuato (Fig. 1). The region is located in a structural depression called Bajío Guanajuatense (Fig. 1), within the physiographic province of *Faja Volcánica Transmexicana* (Ferrari 2000). Volcanic landforms dominate the southern part of the study area with peaks of 2050 m a.s.l. and large intermontane valleys filled with modern sediments and soils. The lithology is dominated by Tertiary and Quaternary basalts and andesites, and some isolated outcrops of Tertiary lacustrine travertine. The main rivers of the region are the Guanajuato and Temascalito Rivers (Fig. 1). The Guanajuato River catchments are located in the Sierra de Guanajuato and the course runs 40 km downflow through the city of Irapuato and some other small towns and industries (see Fig. 1). For centuries, this river has received the discharge of urban effluents but also it has been impacted by the mining activity developed in its catchment area (Hernández-Silva et al. 2005; Miranda-Avilés et al. 2012).

Historical annual mean rainfall averages 693 mm and monthly mean temperature varies from 14 °C in January (winter season) to 21 °C in July (summer season; period 1978–2015). Maximum precipitation concentrates in the period June–September while the driest and hottest period occurs during March–May (Fig. 2). Total annual evaporation reaches

1964 mm/year and water budget is negative in all months of the year, except in July (+9.15 mm).

2.2 Soil sampling

Soil samples were collected from four soil profiles (1.20 m depth \times 1.50 m width \times 1.80 m length) dug in farmland areas at Irapuato municipality (Fig. 1). Samples were collected every 0.20 m in each profile. Soil profiles were sampled in different geomorphological environments at different altitudes where specific edaphogenetic processes have influenced soil characteristics. Clayed soils (ARC; 1730 m a.s.l.) are located in a tectonic depression (graben) characterized by thick fine-grained soil sequences, whereas sandy soils (ARE; 1753 m a.s.l.) are located in the sandy alluvial plain of Guanajuato River. Additionally, a control soil profile (CO; 1780 m a.s.l.) was dug in a parcel next to the ARC profiles (Fig. 1), with natural vegetation and which is not impacted by irrigation. All the sampled soils are affected by artificial irrigation using groundwater pumped from nearby boreholes.

Soils were described following the Soil Taxonomy classification (Soil Survey Staff 2014) for the identification of soil orders and suborders. The profiles ARC-1 and ARC-2 (Fig. 1) were dug in cultivated fields irrigated with deep groundwater (120–250 mbs; wells P1-P5) while the profiles ARE-1 and ARE-2 (Fig. 1) were located in fields irrigated with shallow groundwater (30 mbs; well P6).

All samples were collected using a plastic shovel in order to avoid metal contamination, double bagged in plastic bags, and transported to the laboratory. Before analysis, samples were dried at room temperature for 1 week, ground with a rubber hammer, and sieved through a 10 mesh; the fraction <2 mm was used for determinations and experiments carried out in this work.

2.3 Physical properties and chemical parameters of soils

Soil physical properties were determined following the Mexican Norm NOM-021-SEMARNAT-2000 (SEMARNAT 2000) for soil fertility.

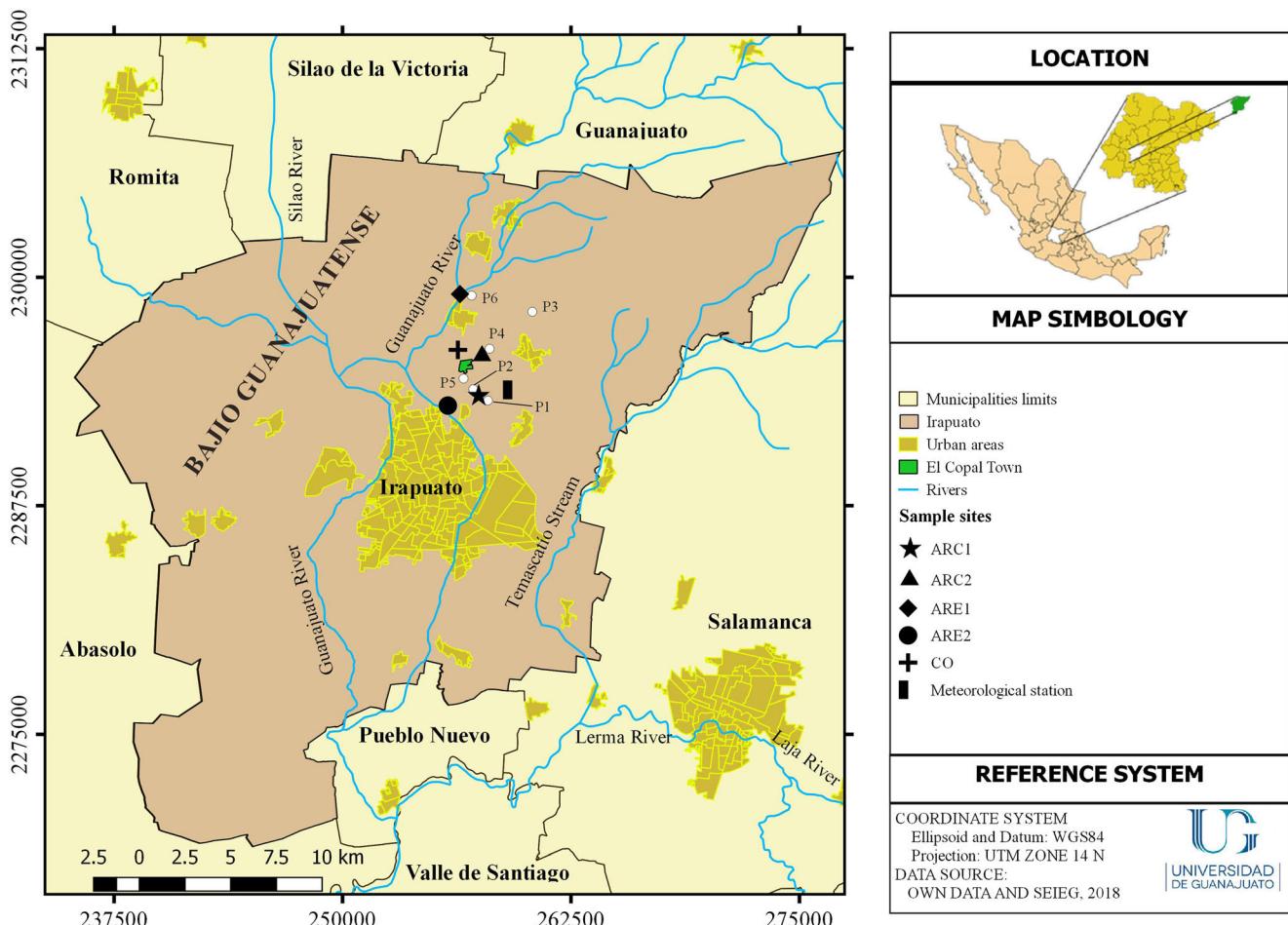


Fig. 1 Map showing soil sampling points and sampled water wells. The meteorological station El Copal is also shown in the study area

The clay fraction was quantified using the Bouyoucos method. Paste pH and EC were measured in a 1:2 soil:water paste using a potentiometer Conductronic PC45. Organic matter (OM) was determined by loss on ignition (LOI; Heiri et al. 2001). The concentration of carbonate was determined in triplicate using a Bernard calcimeter (50% HCl and 0.2 g of soil). Available phosphorous concentrations were calculated applying the Olsen method by colorimetry, using sodium bicarbonate and reading at 882 nm in a spectrophotometer.

For Fe and As measurements, soil samples were digested following the USEPA methods 3051A and 3050B, respectively (USEPA 1996, 2007). For the determination of Fe concentrations, ~0.5 g of the soil sample was treated with 9 mL of trace metal grade HNO₃ (69%, Fisher Scientific) and 3 mL of reactive grade HCl (37.4%, Fermont). Samples were digested using a microwave oven CEM (MARSX 230/60 model), operating at two ramps: first at 120 °C for 5 min and then at 175 °C for 5 min. For As determinations, ~2.0 g of soil sample was suspended in 10 mL of trace metal grade HNO₃ (69%, Fisher Scientific) and heated at 95 °C to a final volume of 5 mL. Total Fe and As concentrations were quantified by ICP-AES (Thermo Icap 6500 Duo) at the Centro de Innovación Aplicada en Tecnologías Competitivas (CIATEC, CONACYT) according to the method 6010C recommended by USEPA (2000). The detection limit for As was 0.025 mg/kg, and for Fe, 5 mg/kg. The accuracy of the results for Fe and As was checked against measurement of an internal blank and three calibration standards.

2.4 Index of Geoaccumulation

The Index of Geoaccumulation (Igeo) proposed by Müller (1969) was calculated in the four soil profiles, using the following equation:

$$I_{\text{geo}} = \ln \frac{C_n}{1.5 B_n}$$

where C_n is the As concentration in the samples and B_n is the As background value in the Upper Continental Crust (UCC; 4.8 mg/kg; Rudnick and Gao 2003). 1.5 is a constant that considers natural fluctuations in the content of a given element and small influences by anthropogenic sources. For ARC soils, the Igeo calculations were also performed considering As background value (B_n), the As concentration measured in each horizon of the control soil (CO; mean = 0.44 mg/kg). The Igeo values determined in the studied soils were compared with the six Igeo classes established by Müller (1969), who classified the soils according to their contamination degree, from class 0 (Igeo ≤ 0) corresponding to uncontaminated soils to class 6 (5 < Igeo), representing extremely contaminated soils.

2.5 X-ray diffraction and SEM/EDS

Minerals in soil samples were identified by X-ray diffraction (DRX) and scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDS) measures at the Laboratorio de Investigación y Caracterización de Minerales (LICAMM, Universidad de Guanajuato). The XRD analyses were performed with a Rigaku Ultima IV Diffractometer with CuKα radiation, operating at 30 mA and 30 kV. The XRD data were obtained for random samples in the 2 range from 2 to 80° (step size 0.02; 3 s/step) with a detection limit of 1%. SEM/EDS measurements were performed in a HITACHI TM-1000 equipment coupled with an Oxford dispersive X-ray spectrometry (EDS) detector at C-GEO (UNAM). Mineral observations were performed in total samples gently disaggregated with a pestle and mortar and in a fraction separated with a magnet.

2.6 Water sampling and chemical analysis

Six water samples were collected from irrigation boreholes located in the study area during February–March 2016 (dry season; Fig. 1). Field determinations consisted of pH, electrical conductivity (EC), total dissolved solids (TDS), temperature (T), and oxidation reduction potential (ORP). All in situ parameters were measured using a Hanna HI98194 multi-parameter probe. Water samples for total alkalinity were stored in polyethylene bottles (500 mL) and refrigerated at 4 °C. Total alkalinity was determined by titration using 0.1 N H₂SO₄ and methyl orange as end point indicator (Mexican Standard NMX-AA-036-SCFI-2001; Secretaría de Economía 2001).

Immediately after collection, samples were filtered through 0.22-μm cellulose acetate membrane filters (Millipore Corp.) and divided into two aliquots. The filtration equipment was repeatedly rinsed with sample water prior to filtration. Aliquots used for major cations and trace element determinations (50 mL) were acidified to pH < 2 with ultrapure HNO₃ (> 99.999%, redistilled) and stored in pre-cleaned polyethylene bottles. An aliquot of 100 mL was stored in polyethylene bottles, without acidifying, at 4 °C for the determination of major anions.

Chemical analyses were performed at Centro de Geociencias, C-GEO (Universidad Autónoma de México, UNAM). Anions (Cl⁻, SO₄²⁻, F⁻, NO₃⁻) were measured by high-resolution liquid chromatography (HPLC), while cations (Ca²⁺, K⁺, Mg²⁺, Na⁺) and As were determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES), following the method 6010C of the USEPA. Deionized water (18 MΩcm Milli-Q, Millipore Corp.) was used for all solutions and dilutions. Detection limit for As was 0.002 mg/L.

3 Results

3.1 Soil characteristics and classification

ARC and CO soils are classified as Vertisols (Typic Haplusterts), according to the Soil Taxonomy classification. Vertisols are characterized by the presence of high contents of clays that lead to pronounced changes in the soil volume with variations in moisture. Cracks that open and close periodically and evidences of soil movement in the form of slickensides are also typical features of Vertisols.

Two horizons were recognized in the soil profile ARC1 (Fig. 2). The uppermost 30-cm-thick Ap horizon presents granular structure and consists of dark brown clays which contain abundant traces of roots and crops (especially, corn, barley, and wheat). High moisture, well-developed blocky structures, and abundance of slickensides, indicative of expansive clays (smectite group), characterize the bottom layer Bss-t (30–120 cm). Wet soil consistency resulted sticky (plastic) while dry soil consistency was very hard. The soil profile ARC2 shows a similar sequence but more horizons were identified (Fig. 2). The Ap horizon (0–30 cm) shows root debris (2.5 mm diameter), occasional fine surficial cracks, and a

granular structure. A/B layer (30–60 cm) represents the transition between the surficial organic-rich layer and the underlying mineral layer. Increasing contents of clays and moisture are observed in the Bt1 horizon (50–70 cm) located immediately below. Slickensides were also observed but in smaller proportion than in the ARC1 profile. The bottom Bt2 horizon (70–120 cm) was characterized by a sticky consistency when wet and a well-developed subangular blocky structure. Four horizons were identified in the soil profile CO (Fig. 3). The uppermost A horizon shows abundance of roots (2–5 mm diameter) and bioturbation (leaves debris and coprolites). The underlying A/B layer shows root remains and a slightly developed laminar structure. Scarce slickensides are also recognized in this transition horizon. Below, the Bt horizon is characterized by the presence of abundant slickensides, high moisture, and a very hard consistency when dry. The bottom horizon B/C contains rock fragments from the catchment area (basalts and andesites), as relics of the parental material of this soil.

According to the Soil Taxonomy classification, ARE soils are Entisols (Typic Ustifluvents), characterized by intense reddish-orange color, dominance of mineral soil materials, low OM content, absence of distinctive pedogenic horizons,

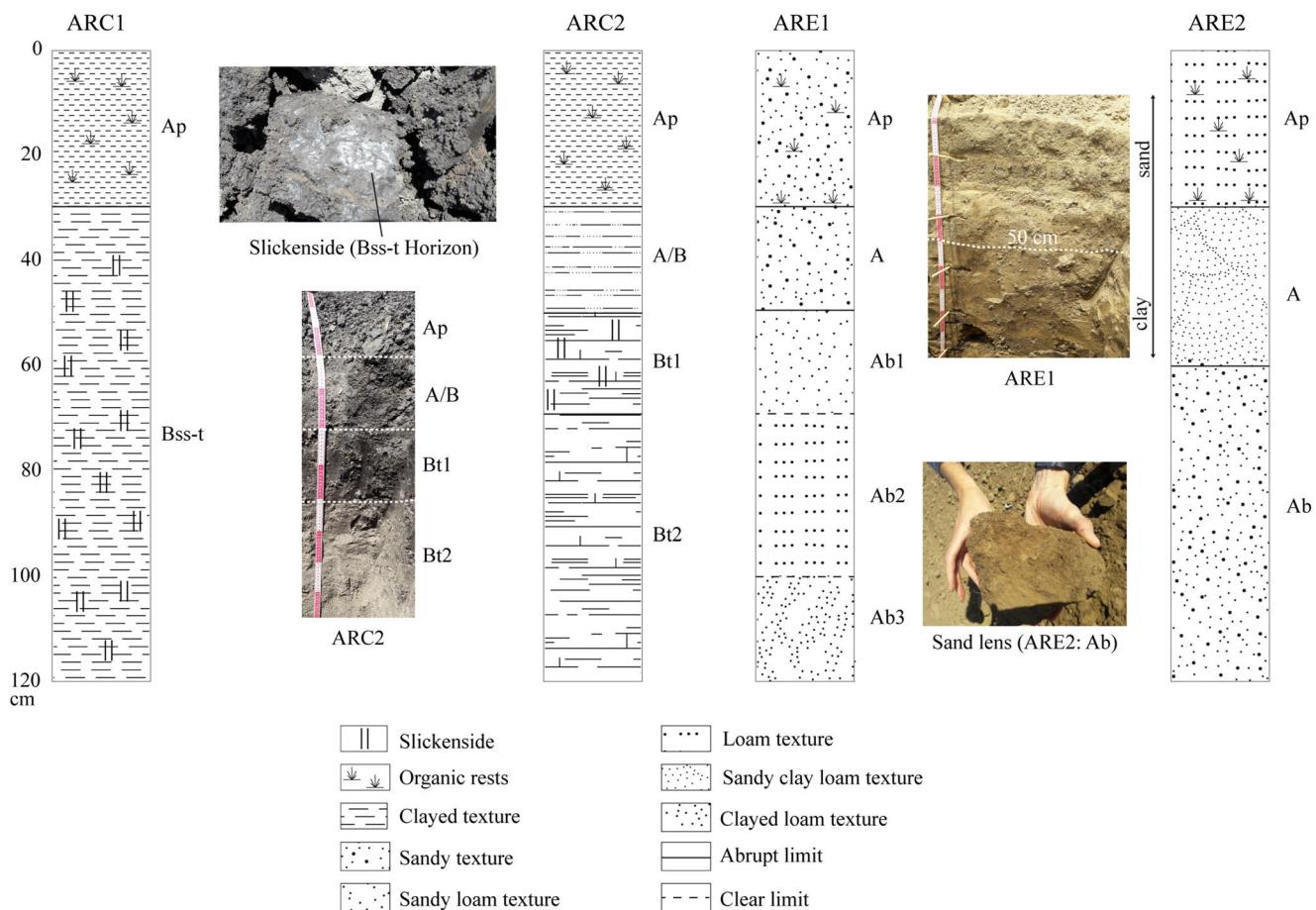


Fig. 2 Soil profiles and main horizon characteristics

and a sandy-clayed stratified structure (Fig. 2). A remarkable feature in the studied Entisols is the presence of Ab-type horizon that indicates the alternation of soil layers and fluvial deposits. The sequence of layers in the studied Entisols (ARE profiles), from the top to the bottom, is Ap, A, Ab1, Ab2, and Ab3 (ARE1), and Ap, A, Ab (ARE2). Ap horizons (0–30 cm) were identified in both ARE profiles and are characterized by the presence of root and crop debris (mainly corn and peanut). In the ARE1 soil, the A layer (30–50 cm) shows scarce organic debris, was slightly wet at the moment of sampling, and presents a weakly developed stratified structure. The Ab1 horizon (50–70 cm) shows a distinctive sandy loam texture and a weakly to moderately developed granular structure. It is separated from the horizon underneath by a net limit. The Ab2 horizon (70–100 cm) shows loam texture and a moderately laminar structure. Finally, the Ab3 horizon (100–120 cm depth) presents clayed-loam texture and a weakly developed laminar structure. Wet soil consistency in all Ab layers was friable. The ARE2 soil consists of an A horizon (30–60 cm) slightly wet and that contains no root debris. Texture is sandy clay loam and the structure is weakly laminar (Fig. 2). The limit with the underlying Ab horizon (60–120 cm) was abrupt. The Ab horizon shows a sandy texture and a fine stratification. Dry soil consistency was loose and wet soil consistency resulted very friable.

3.2 Soil physical and chemical parameters

The physical and chemical parameters measured in the studied soil profiles are reported in Table 1 while the in-depth variations of these variables in both types of soils are depicted in Fig. 4.

Grain size in ARC soils is markedly finer than in ARE soils (Table 1). While in ARC soils the clay content is about 66%, in ARE soils, the mean clay content is approximately 15%. As seen in Fig. 4, the clay contents in ARC soils increase with depth and the proportions are nearly identical in both profiles. On the contrary, in the two studied ARE soils, the observed trends differ in the two analyzed soil profiles. In ARE1, the clay content slightly increases with depth, whereas in the ARE2 profile, the trend is the opposite (Fig. 4). Clay contents in CO soil vary from 60 to 70%, increasing slightly with depth (Fig. 3).

Another remarkable difference among the study profiles is evidenced through the EC values. As shown in Table 1, the mean EC values recorded in the horizons of ARC1 and ARC2 profiles (mean = 1.05 dS/m) are about one order of magnitude higher than those of the ARE soils (mean = 0.22 dS/m). Soils with EC values lower than 1.0 dS/m are classified as non-saline while EC values between 1.0 and 2.0 dS/m correspond to slightly saline soils (Mexican Standard NOM-021-SEMARNAT-2000). It can be observed that EC values increase with depth in both ARC soil profiles (Fig. 4). On the

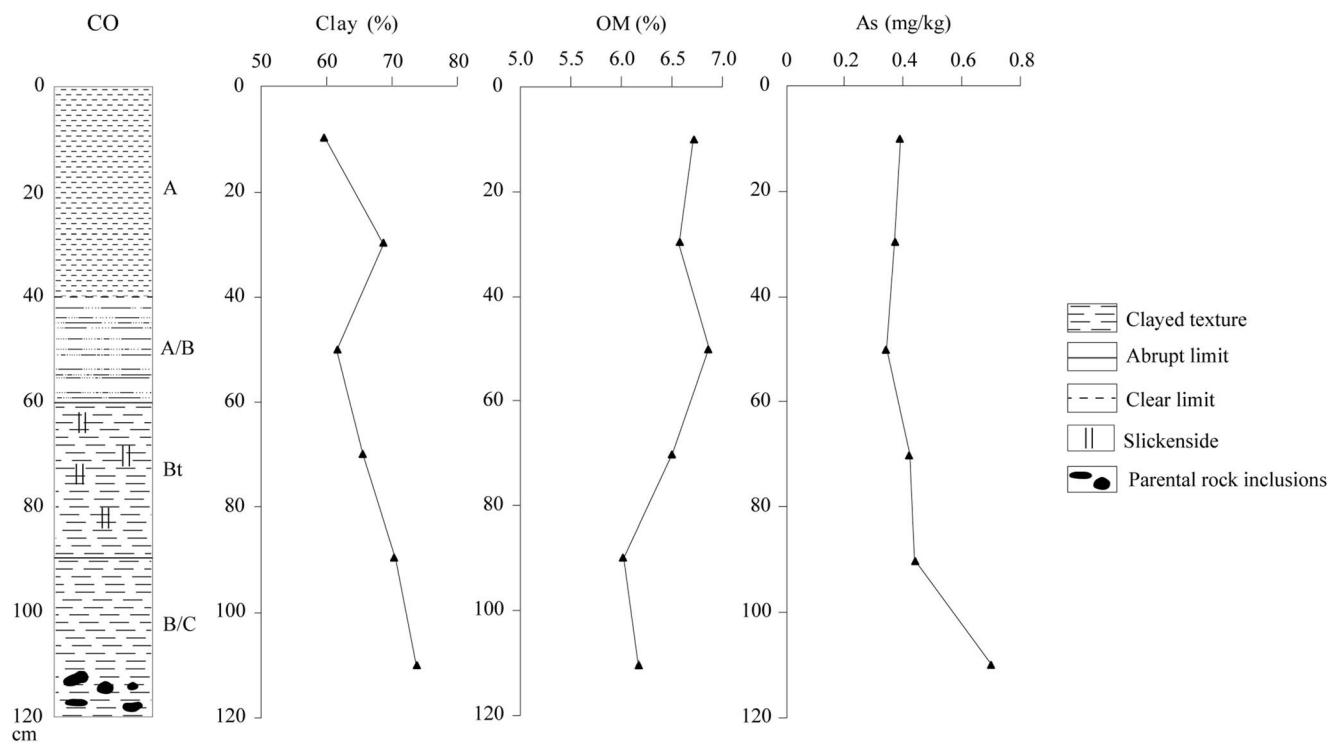
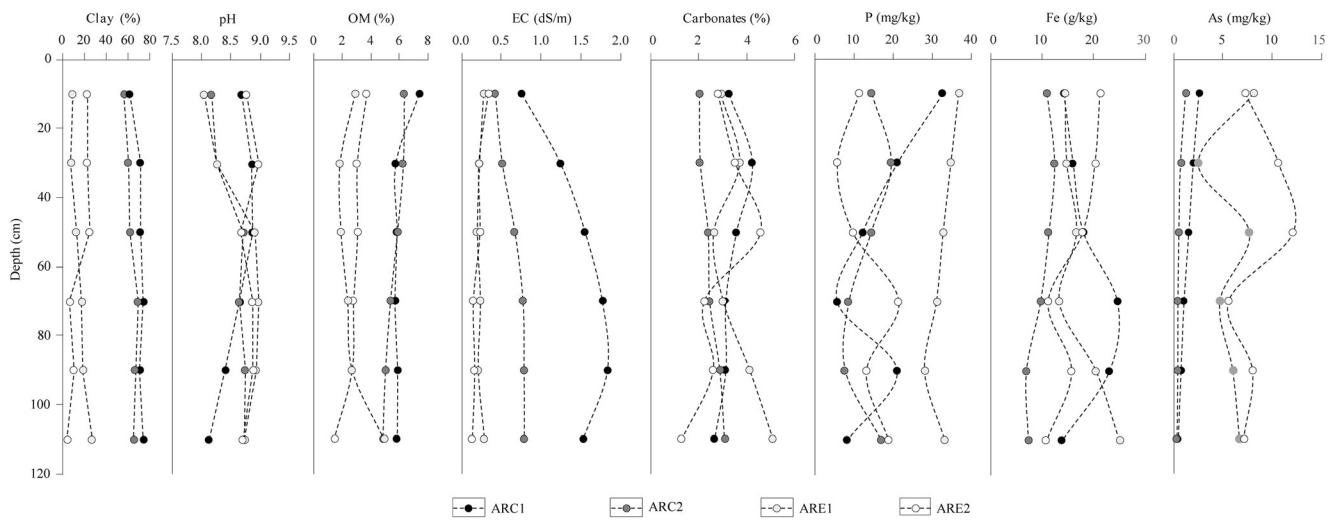


Fig. 3 Control soil profile showing main horizons and variations in clay, organic matter (OM), and As contents

Table 1 Physical and chemical variables determined in ARC and ARE soil profiles. The location of profiles is presented in Fig. 1. OM organic matter, EC electrical conductivity, Carb carbonates

Depth (cm)	Clay (%)	pH	OM (%)	EC (dS/m)	Carb (%)	P (mg/kg)	Fe (g/kg)	As (mg/kg)
ARC1								
0–20	60.96	8.68	7.41	0.75	3.27	32.60	14.20	2.58
20–40	70.96	8.86	5.76	1.24	4.23	21.10	16.01	2.01
40–60	70.96	8.87	5.84	1.55	3.57	12.20	18.04	1.55
60–80	73.96	8.65	5.69	1.76	3.13	5.77	24.69	1.06
80–100	70.68	8.4	5.87	1.83	3.13	21.00	23.11	0.76
100–120	73.68	8.13	5.85	1.52	2.67	8.36	13.86	0.46
ARC2								
0–20	55.96	8.17	6.32	0.42	2.03	14.5	10.97	1.28
20–40	59.96	8.26	6.26	0.51	2.07	19.40	12.37	0.74
40–60	60.96	8.71	5.86	0.66	2.4	14.30	11.31	0.56
60–80	68.96	8.64	5.39	0.76	2.47	8.66	9.68	0.46
80–100	66.96	8.74	5.06	0.78	2.90	7.76	6.95	0.45
100–120	64.96	8.72	4.87	0.78	3.10	16.90	7.32	0.36
ARE1								
0–20	9.68	8.05	2.89	0.27	2.93	36.70	14.41	8.10
20–40	7.96	8.27	1.85	0.22	3.70	124.00	14.76	2.50
40–60	12.96	8.90	1.91	0.19	2.63	32.80	16.64	7.70
60–80	17.96	8.97	2.38	0.23	3.03	31.10	13.28	4.70
80–100	18.96	8.93	2.70	0.21	4.13	28.10	20.50	6.00
100–120	26.96	8.74	4.94	0.29	5.10	33.20	25.05	6.70
ARE2								
0–20	22.96	8.76	3.69	0.34	2.80	11.30	21.33	7.30
20–40	22.68	8.97	3.04	0.22	3.53	5.87	20.36	10.60
40–60	24.68	8.67	3.06	0.23	4.57	9.75	17.93	12.10
60–80	6.68	8.87	2.79	0.15	2.23	21.40	11.13	5.61
80–100	10.68	8.88	2.68	0.16	2.60	13.20	15.76	8.00
100–120	4.68	8.70	1.45	0.13	1.30	18.80	10.84	7.10

**Fig. 4** Vertical variations of physical and chemical properties in ARC (Vertisols) and ARE (Entisols) soil profiles. Black circle: ARC1; dark gray circle: ARC2; light gray circle: ARE1; white circle: ARE2

other hand, EC values in the ARE soil profiles remain constant with depth and are nearly identical in both profiles.

pH values are alkaline in all samples, ranging from 8.0 to 9.0 (Table 1). In soil profiles ARC2 and ARE1, pH increases below ~50 cm depth, while in the soil profile ARC1, the opposite trend is observed. In the soil profile ARE2, pH values remain nearly constant with depth (Table 1 and Fig. 4).

According to the Mexican Standard, the contents of OM in the Vertisol soils are considered high (3.6–6.0%), while in the Entisol soils, the contents are medium (1.6–3.5%) to high (3.6–6.0%). The mean OM content in the ARC profiles is 5.85%, while in ARE soils, the mean OM content is 2.78%. As it is seen in Fig. 4, OM contents in ARC profiles tend to decrease with depth. In the case of the ARE1 profile, the highest OM content was measured in the bottom layer (100–

120 cm depth), but in general, the variability of this parameter with depth is rather limited in all the studied profiles (Fig. 4).

The content of carbonate is similar in all the studied soils, ranging between ~1.30 to ~4.50% (Table 1). The highest carbonate content was recorded in the bottom layer of the soil profile ARE1 (5.10%). No specific trends with depth have been observed in the studied profiles (Fig. 4).

The soil profile ARE1 shows the highest contents of P (36.70 mg/kg), which are rather constant throughout the entire profile (Fig. 4). In the remaining profiles, the content of P varies between ~10 and 30 mg/kg (Table 1). Similarly, the contents of Fe vary from 7000 to 25,000 mg/kg along the soil profiles, but no clear trends with depth are observed (Fig. 4).

The contents of As in the study soil profiles range between 0.36 and 12.10 mg/kg (Table 1 and Fig. 4). As concentrations

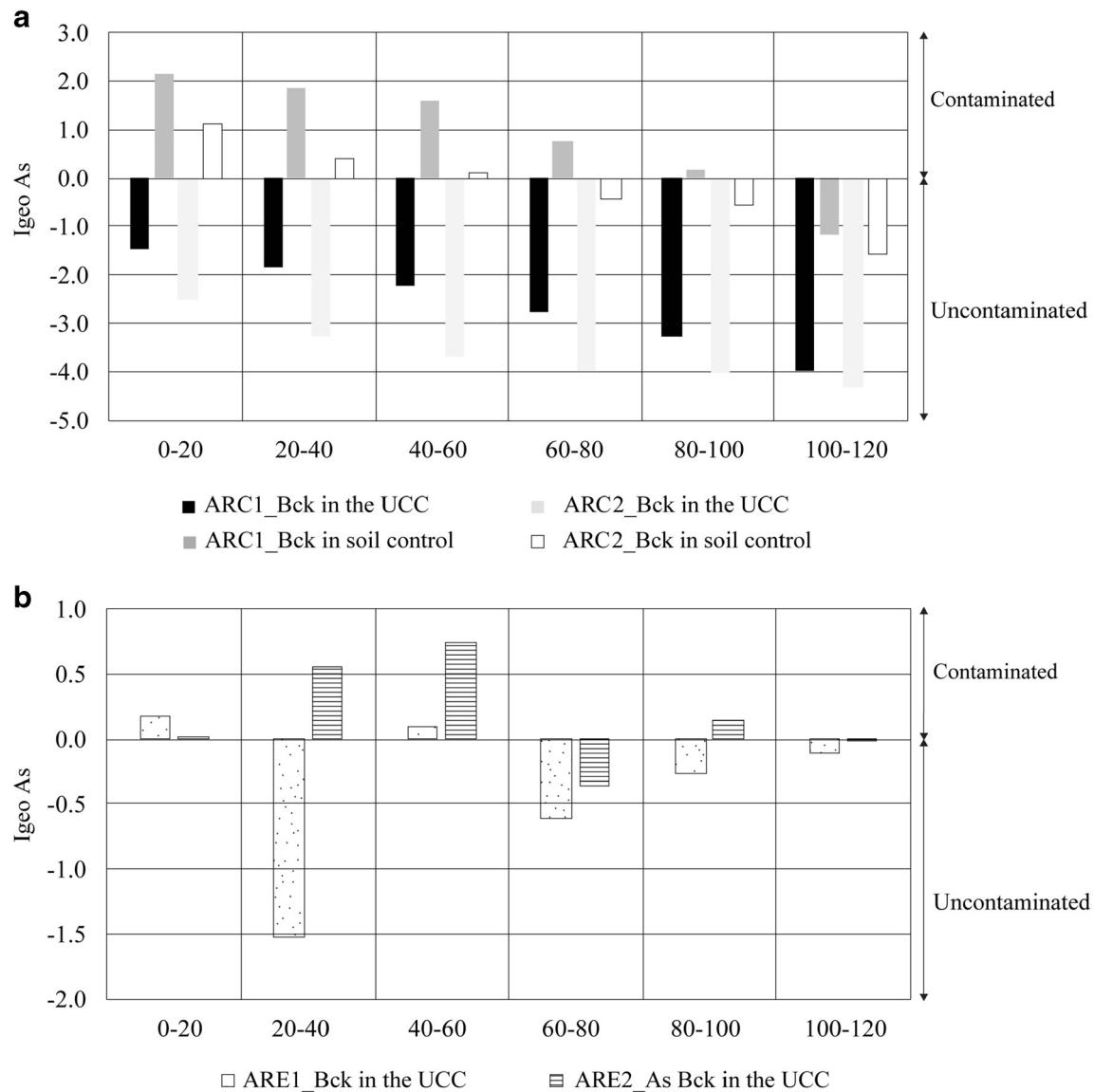


Fig. 5 Igeo values for As in soil profiles. *Bck* background, *UCC* Upper Continental Crust

in the control soil are the lowest, varying from 0.34 mg/kg (40–60 cm) to 0.70 mg/kg (100–120 cm; mean = 0.44 mg/kg; Fig. 3). In general, near total As concentrations are noticeably higher in the sandy profiles (ARE, mean As value = 7.20 mg/kg) than in the clayed profiles (ARC, mean As value = 1.02 mg/kg). None of these values exceed the Mexican maximum As concentration guideline for agricultural soils (22 mg/kg). The in-depth distribution of As in profiles of clayed soils reveals that the higher As concentrations are found in the uppermost horizons (10 cm) and they tend to decrease with depth (Fig. 4). In the sandy soils, the variability of the As concentrations downwards is not the same in the two

analyzed profiles. While in the ARE2 soil profile, the highest As concentration was measured at 50 cm depth, and in the ARE1 profile, maximum As concentrations are found in the uppermost layer, as in the case of the clayed soils (see Fig. 4).

3.3 Geoaccumulation index

I_{geo} values calculated with mean As concentration in the UCC (4.8 mg/kg) indicate that Vertisols are nearly uncontaminated (class 0; Fig. 5). On the contrary, I_{geo} values calculated with every As concentration of each horizon of the control soil in the

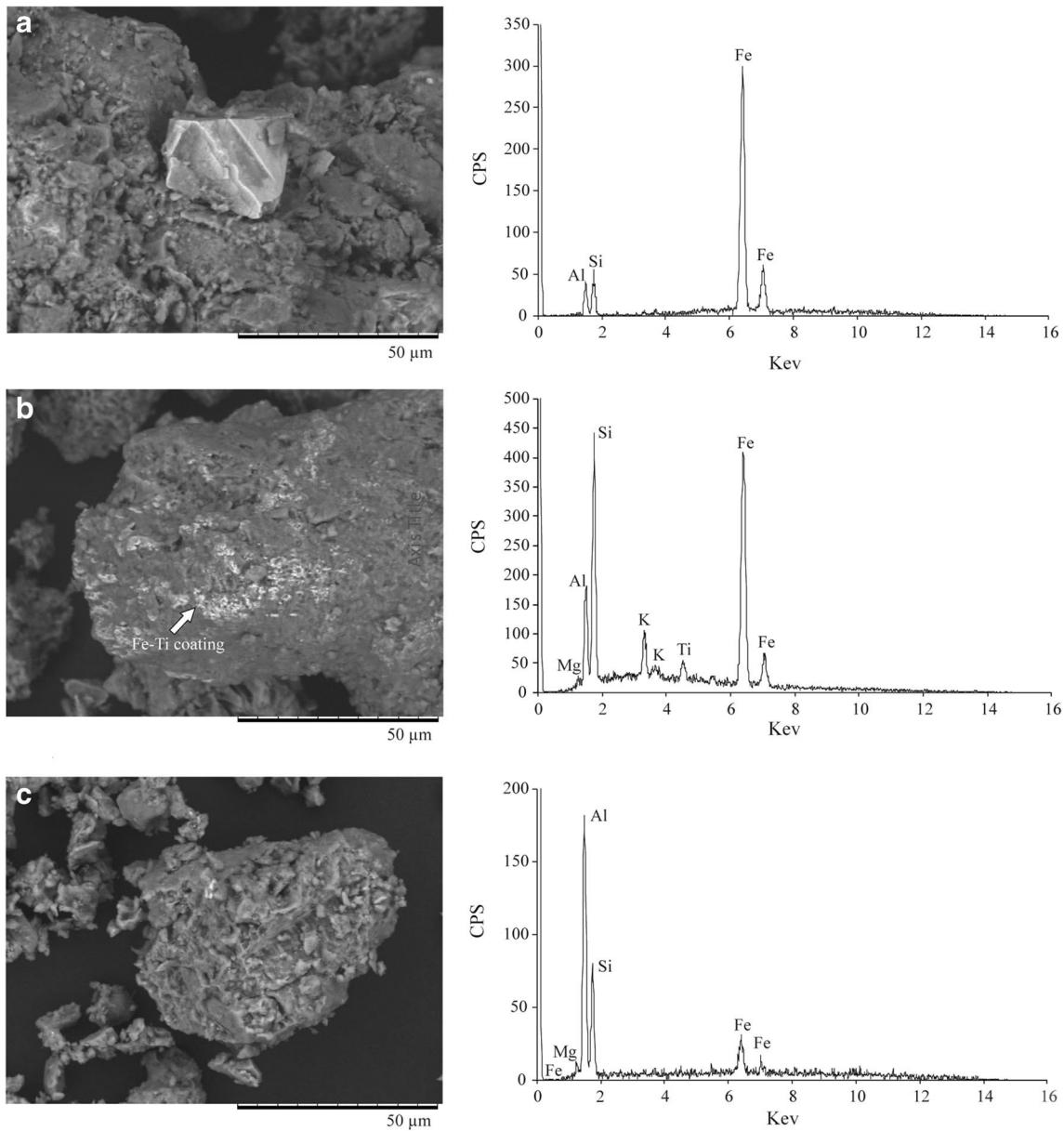


Fig. 6 SEM images and associated EDS spectrums for ARC (Vertisols) and ARE (Entisols) soil profiles. **a** Subangular magnetite (ARE1 100–120 cm). **b** Fe-Ti coating on feldspar surface (ARE1 100–120 cm). **c** Altered mineral grain coated with Fe oxides (ARE1 0–20 cm)

study area indicate that most samples in the upper 60 cm of the profiles are slightly to highly contaminated (classes 1, 2, and 3; Fig. 5). In the uppermost horizons (0–20 cm), Igeo values are the highest ($\text{ARC}_1 = 2.1$, class 3; and $\text{ARC}_2 = 1.1$, class 2). Conversely, Igeo values for ARE2 correspond to classes 0 and 1 (nearly uncontaminated to moderately contaminated respectively). According to the Igeo values calculated in the Entisols, these soils are nearly uncontaminated regarding their As contents (class 0 and class 1, Fig. 5). In these soils, Igeo values do not show any vertical distribution pattern along the profile.

3.4 Mineralogical composition

DRX analyses show that silicates such as quartz and plagioclases are the main primary minerals while the most frequent minor minerals are cristobalite and montmorillonite.

Due to the affinity of As for Fe-bearing minerals, SEM/EDS analysis was carried out in the fraction of minerals magnetically separated from the soil samples. The separation showed a greater abundance of magnetic minerals in Entisols than in Vertisols. In all cases, magnetic minerals mostly consist of subangular grains of magnetite (Fig. 6a) and abundant Fe oxides and Ti-Fe coatings precipitated onto feldspar grains (Fig. 6b). SEM/EDS observations in the bulk fraction of the soils mostly revealed the presence of altered quartz and feldspar grains frequently coated with clays and Fe oxides (Fig. 6c).

3.5 Groundwater major composition

The physicochemical parameters and the concentration of major ions and selected trace elements measured in the groundwater samples are shown in Table 2 and Fig. 7. The highest temperature was recorded in the deepest well (P1: 46.64 °C; 250 mbs) and the lowest temperature in the shallowest well (P6: 24.40 °C; 30 mbs). Slightly acidic to neutral pH values prevail in the analyzed samples, with pH values ranging between 6.74 and 7.67. EC varies from 558 to 1627, while TDS concentrations fluctuated between 279 and 817 mg/L. Oxidizing conditions prevail in both the deep and shallow aquifers as indicated by the positive ORP values measured in the samples. The lowest ORP and DO values were determined in the sample with the highest temperature.

The major chemical composition is dominated by HCO_3^- , Ca^{2+} , and Na^+ ions (Fig. 7a). According to the major ion composition, most groundwater samples are of the sodium bicarbonate type, while the samples collected from the shallower aquifers are of the calcium-bicarbonate type. As a general trend, the chemical composition of groundwater tends to evolve from the calcium-bicarbonate type to the Na-bicarbonate type in the flow direction.

Samples	Water level (m)	Depth (m)	pH	EC (µS/cm)	TDS (mg/L)	DO (mg/L)	ORP (mV)	Ca^{2+} mg/L	K^+ mg/L	Mg^{2+} mg/L	Na^+ mg/L	F^- mg/L	Cl^- mg/L	NO_3^- mg/L	SO_4^{2-} mg/L	TA mg/L	As mg/L	
P1 (ARC)	90	± 250	46.64	6.74	1627	817	1.56	57.90	59.63	11.70	6.77	279.70	2.72	87.11	5.44	315.20	396.80	0.18
P2 (ARC)	90	± 250	42.32	7.10	1033	517	2.55	69.00	24.03	7.45	2.30	194.00	2.90	37.44	9.92	141.13	325.00	0.14
P3 (ARC)	90	± 250	34.67	7.43	933	467	2.68	64.50	19.21	9.58	6.07	237.70	3.87	49.47	7.82	172.91	262.10	0.20
P4 (ARC)	80	± 250	32.72	7.67	670	334	3.73	57.10	22.52	7.39	3.12	123.10	1.80	18.01	12.81	66.75	258.20	0.08
P5 (ARC)	60	120	30.42	7.22	558	279	4.80	123.00	49.94	9.53	7.83	58.60	0.78	15.97	11.14	51.52	258.20	0.06
P6 (ARE)	9	30	24.40	7.30	1210	605	4.75	77.80	141.10	9.51	32.80	86.23	0.44	56.13	2.50	179.93	446.80	0.04

The concentrations of arsenic exceed the recommended guideline value for drinking waters set by the Mexican and international regulations (0.025 and 0.010 mg/L, respectively; NOM-127-SSA1-1994, Semarnat 1994 and WHO 2004) in all the analyzed samples, and 50% of the samples are above the limit of As for irrigation water (FAO 0.10 mg/L). A remarkable feature is that the highest As concentrations were determined in the deepest and hottest samples (Fig. 7b).

4 Discussion

In the study area, two main types of agricultural soils were identified. Vertisols (ARC 1 and ARC2 profiles) were formed in a topographical basin called *Bajío Guanajuatense* (Fig. 1), where a tectonic depression (graben) formed by extensional faults (Ferrari 2000) favored the formation of thick and mature soil sequences. The parental rocks of these soils include tertiary and quaternary andesites and basalts, while isolated outcrops of tertiary travertines of lacustrine origin (Nieto-Samaniego et al. 2012) also contribute with natural inputs to the soils. In contrast, Entisols (ARE1 and ARE2 profiles) are associated with the history of periodical flooding and fluctuations of the sediment load of the Guanajuato River, which led to the formation of younger and more immature soil columns.

The levels of As in Entisols are higher than the values determined in Vertisols and they show remarkable variations in depth (Fig. 4). The slight trend of increasing As concentrations with increasing Fe, OM, or clay contents in Entisols could likely indicate the presence of adsorbed As onto Fe oxide coatings, clay minerals, or humic compounds present in the soils (Fig. 4). The presence of As as an impurity within Fe oxide lattices such as magnetite, ilmenite, or hematite (i.e., Sun et al. 2016; Coker et al. 2006) not necessarily could be reflected in good Fe-As correlations, as many other Fe-bearing minerals that contain no As are also present in the soils. In view of all these factors, the main As-bearing phases in Entisols are considered to be the Fe oxides extensively identified by SEM/EDS. In-depth variations in the As concentrations also suggest that the contributions of these minerals have occurred in pulses associated with the historical variations of the Guanajuato River discharges. Due to the presence of polymetallic sulfide ores in the river's catchments, it is possible that As released by the oxidative dissolution of sulfides is then adsorbed and/or co-precipitated onto poor crystalline Fe (III) oxyhydroxides, ubiquitously distributed as mineral coatings and discrete particles in river sediments. These secondary Fe oxide particles are delivered downflow and accumulated in the riverbanks where they were later affected by the edaphic processes. Because these minerals are

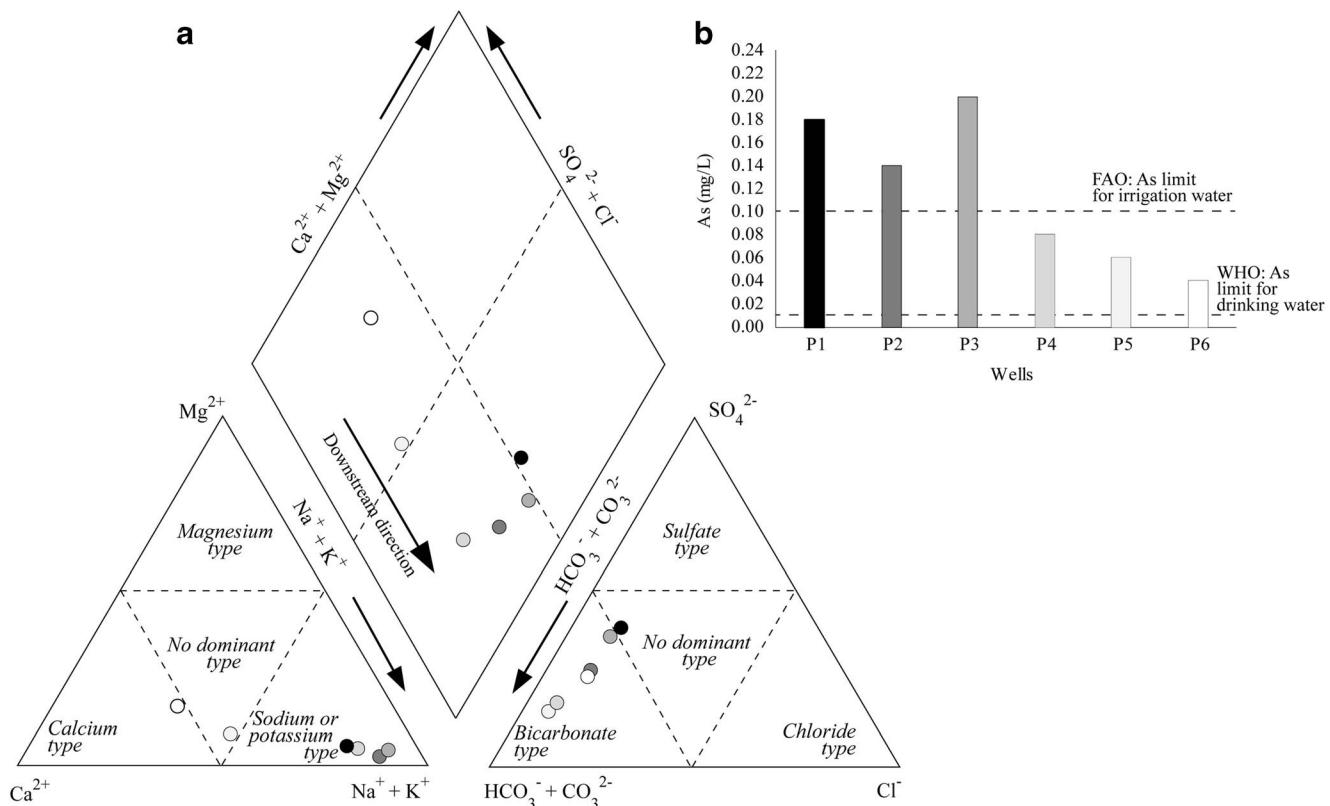


Fig. 7 **a** Piper diagrams illustrating water composition types in groundwaters used for irrigation in studied soils (P1-P6). **b** As concentrations (mg/L) in the different groundwater wells (P1-P6). Black arrow indicates flow direction. The As limit for drinking water and for irrigation water is also shown

rather stable during weathering (i.e., Keimowitz et al. 2005; He et al. 2010), they remain unaltered during the edaphic processes, and therefore, As associated with them is not released to the pore water.

Unlike Entisols, the contents of As in Vertisols are remarkably lower and nearly constant along the soil profile. Highest As concentrations were only determined in the uppermost A horizons in both Vertisol profiles, while in depth, As concentrations are low and nearly constant. Both Vertisols and Entisols are currently irrigated with groundwater pumped from deep (P1-P5; Vertisols) and shallow wells (P6; Entisols; Table 2 and Fig. 7). The source of As in deep groundwater had been attributed to the weathering of the rhyolite rocks that host the main aquifers, enhanced by the circulation of warm to hot geothermal waters (25–50 °C; Morales et al. 2015). The observed distribution of As concentrations in Vertisols can be associated with the irrigation with As-rich groundwaters that would favor the accumulation of As in the uppermost horizons where the element is likely scavenged by adsorption onto Fe oxides and organic matter (Bhattacharya et al. 1997; Giménez et al. 2007; Arco-Lázaro et al. 2016). Due to the dominance of clay-sized horizons in Vertisols, the infiltration of irrigation waters is limited, leading to a gradual increase in the As concentrations in the uppermost layers of the soils, while in the underlying layers, the As concentrations are close to the regional background level. The regional As background level of Vertisols in the studied area is considered to be ~0.4 mg/kg, which corresponds to the mean As concentration of the control soil profile (Fig. 3). In addition, the Igeo values of the Vertisol samples indicate that their uppermost horizons are moderately to highly contaminated with As. Conversely, the impact of irrigation with As-rich waters is not that evident in Entisols, likely because the amount of As accumulated in the soil that derives from irrigation water is much lower (about one order of magnitude) than the bulk As concentrations of these soils. Besides, the in-depth distribution of As is highly dependent on the presence of As-bearing Fe oxides delivered from the mine district (DMG), located in the Guanajuato River's catchments.

5 Conclusions

Agriculture has been an important economic activity in the state of Guanajuato since the middle of the last century. In this region, crops are irrigated with As-rich groundwaters pumped from shallow and deep aquifers, which represent a potential risk due to the accumulation of As in soil horizons and its introduction into the food chain through uptake by the crops.

The accumulation and distribution of As across soil profiles in the study area revealed that irrigation with As-rich groundwater has mostly affected As concentrations in the uppermost horizons of Vertisols, which show As concentrations

significantly exceeding the natural background (~0.4 mg As/kg) of the region. Among the main physical and chemical properties measured in these soils, the clay grain-sized fraction and the contents of Fe and OM seem to play a major role on the As distribution across the soil profiles. A/B and B horizons, richer in clay-sized particles, inhibit the downward migration of As, while Fe oxides formed as a product of mineral weathering during the soil formation and organic matter likely scavenge As by adsorption.

In Entisols, formed at the Guanajuato riverbanks, As concentrations are higher than the UCC background level and also than the concentrations measured in Vertisols. The distribution of As across the soil profiles seems to be mostly controlled by periodic contributions of As-bearing Fe oxides delivered from mine prospects located at the river's catchments. Because the concentration of As in the soil-forming minerals is relatively high, the impact of irrigation with As-rich groundwaters is not that evident, as in the case of Vertisols.

The results of this work suggest that the use of As-rich groundwater for irrigation in the study area should be limited in the future as they are producing a noticeable increase in the As concentrations of the agricultural soils. In addition, mine activities at the Guanajuato River's catchments also play an indirect control on the contents of As measured in the soils formed at the river's terraces by providing As-bearing Fe oxide particles from the upstream mineralized areas.

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