

ORIGINAL ARTICLE

Study of speciation of four Heavy metals in Agricultural soils around the abandoned mine of Sidi Bou Othmane - Marrakech (Morocco)

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ABSTRACT

Environmental pollution by heavy metals originated from abandoned mine tailings can become a very important source of contamination both in soil and water. Therefore, the chemical and physical characterization of tailings is important to assess the risk of potential environmental mobility of toxic trace metals that are contained in this kind of waste. The chemical forms of some heavy metals in soils and mine tailings around a Sidi Bou Othmane mine in north-west of Marrakech city (Morocco) was studied by determining soil Cd, Cu, Pb and Zn species using standard solvent extraction and Atomic Absorption Spectrophotometric techniques. The Chemical pools of the metals indicated that the metals were distributed into six fractions with most of the metals residing in the non-residual fractions, suggesting how readily the metals are released into the environment. Considering that the metals mostly occur in the most available forms, it is most likely that the metals must have been derived from a variety of human activities involving dredging, processing and use of metals and/or substances containing metal contaminants, specially mining activity that can pose a risk for the environment due to discharge of tailings all around this mine area.

Key words: heavy metal contamination, mining activity, sequential extraction, soils and tailings.

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INTRODUCTION

Environmental pollution, especially by chemicals, is one of the most effective factors in the destruction of the biosphere components. Among all chemical components, potentially toxic metals (PTMs) are believed to be of a specific ecological, biological and health significance.

In soils their forms are strongly determined by their origin and history. Although native metals are frequently in highly immobile forms, anthropogenic forms are often more reactive and thus are more available to plants. The soil is a dynamic system and any changes in environmental conditions, whether natural or anthropogenic, can alter the forms of PTMs, thereby affecting their behaviour in soil. The main controlling factors include degradation of organic compounds, change of pH, redox potential and ionic strength of the soil solution, solid and solution components and their relative concentrations and affinities for an element, and time [1,2,3,4].

The term PTMs includes essential (e.g., Cu, Mn, Se, Zn, Co) and nonessential (e.g., As, Hg, Cd, Pb) elements. The elements essential to healthy functioning and reproduction of microorganism, plants and animals are required in low concentrations and are also called micronutrients, but at high concentrations they may cause direct toxicity or reproductive effects for plants, animals, and humans [5]. Some elements are also non-essential and even low concentrations of these elements in the environment can cause toxicity to both plants and animals [1].

PTMs contamination is widespread, as they are non-biodegradable and therefore persist for long periods in aquatic as well as terrestrial environments [6]. It is generally accepted that the distribution, mobility,

bioavailability and toxicity of PTMs in soil depend not simply on their total concentrations but, critically, on their forms [7]. These may be soluble, readily exchangeable, complexed with organic matter, or hydrous oxides, substituted in stoichiometric compounds or occluded in mineral structures [8].

Mining is one of the industrial activities that causes the greatest and most persistent alterations in nature [9]. This activity affects relatively small areas, but could have a significant impact on the environment representing a potential danger to the health of human populations residing in the vicinity of these mining areas. The pre-existing ecosystems in mining areas become subjected to such disturbance that the most common consequences is their disappearance; the territories in the vicinity are very vulnerable and usually very much affected [10]. Following mine closure, the tailings are disposed onto surrounding soils, leading to their exposure to environmental factors [11]. The impact of mining activity on a given site is controlled by several factors, including the climate, mining methods, geological conditions, degree of mineralization of the tailings and whether the mine is active or abandoned [12].

The objective of this study is to assess the impact of mining activity on heavy metal levels in agricultural soils in the vicinity of Sidi Bou Othmane mine in Marrakech city, Morocco and comparing result with control site. This study will contribute to awareness of the potential impacts of pollutants and provide a key to effective management of surface soil quality in the mining area.

MATERIALS AND METHODS

Site description

The abandoned Sidi Bou Othmane mine is localized at 30 km in the north of Marrakech, in the region of the Haouz district (Jbilet- Centrales) in southern Morocco (figure 1). Sidi Bou Othmane mine is located close to a rural district and surrounded by agricultural lands. Their exploitation started on 1953, treating 115 tons per day of mineral (0.5% Pb, 7.4% Zn and 6% pyrite) by flotation processes until its closure on 1980.

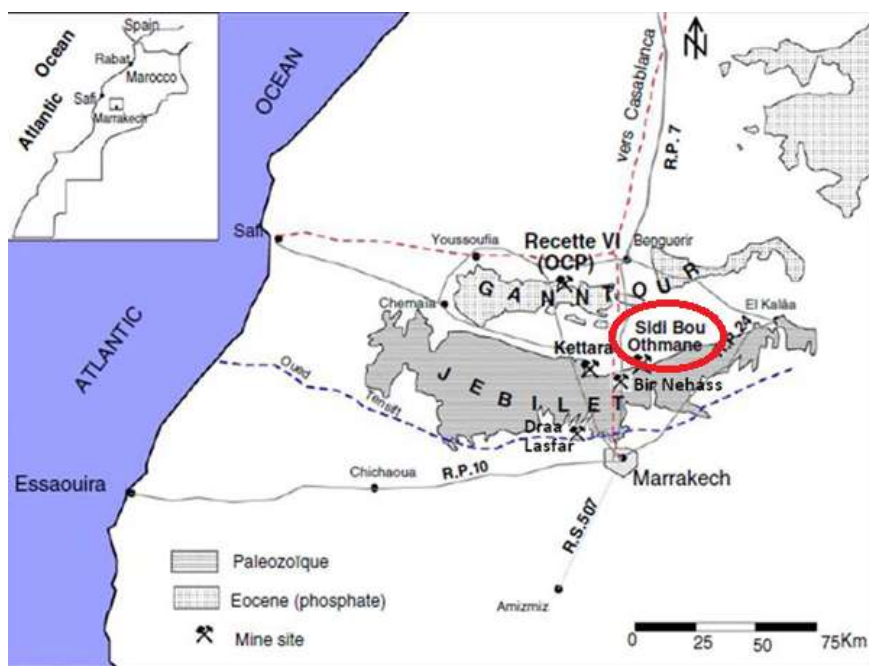


Figure 1: Geographic location of Sidi Bou Othmane mine.

Sampling Description

In order to assess the impact of the Sidi Bou Othmane mine on the surrounding environment, a total of 120 soil samples were collected in the vicinity of the mine covering 620 ha (figure 2). 3 representative background samples were taken at 2 km from the mining site in order to avoid mining contamination.

The soil samples from the various sampling sites (Sidi Bou Othmane rural center = RC, Sidi Bou Othmane agricultural soils = AS and Sidi Bou Othmane mine tailings = MT) were all taken at a depth of 0 to 20 cm each with the help of garden shovel cleaned with concentrated nitric acid. Five (5) samples were taken from each sampling site during 12 months. The soil samples were collected into plastic containers which had all been pre-cleaned with concentrated nitric acid. The reason was to remove any traces of heavy metal contaminant [13].

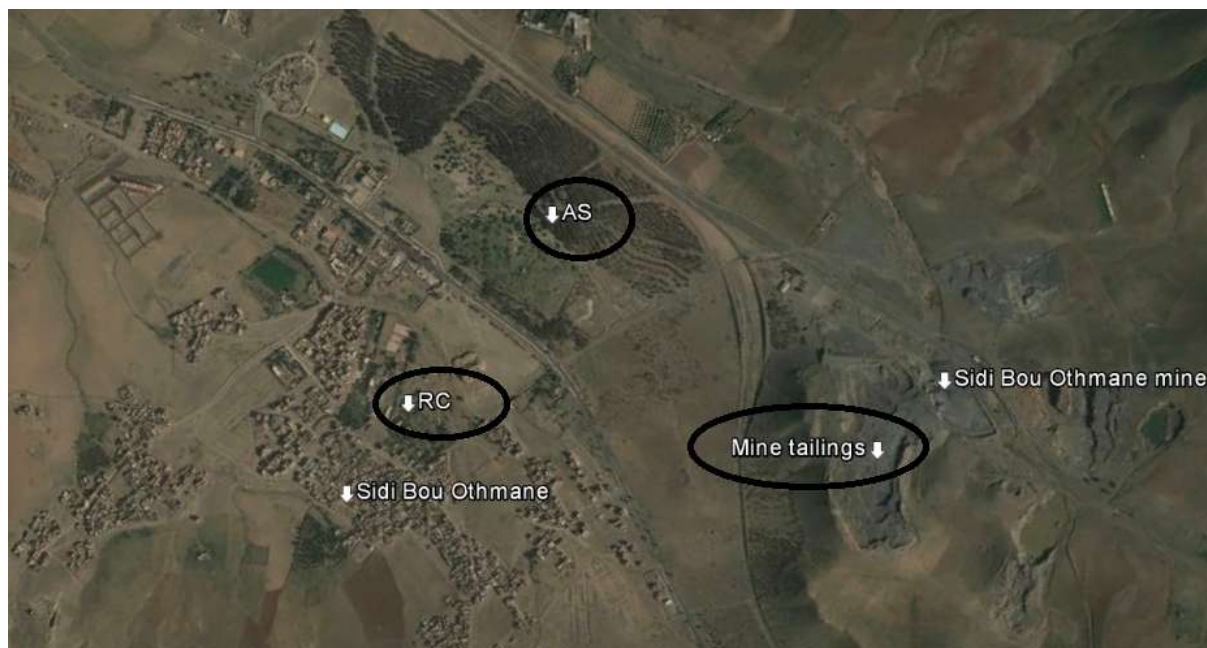


Figure 2: Geographic situation of Sidi Bou Othmane mine tailings, RC and AS.

Samples were taken every 50 meters from the upper 20 cm after removing the first layer of surface soil (2 cm) within an area of 100 cm².

The sampling of the tailings was carried out in the same tailing reservoir by using a specially designed cylindrical stainless steel corer, the average sample of each tailings reservoir is considered after mixture of the various subsamples.

After collection, the soil and tailing samples were carefully transferred to clean and dry self-sealing polyethylene bags and transported to laboratory.

After being air-dried, all samples were sieved through (<2mm or <100 µm) sieve.

The tailings samples from each zone were thoroughly mixed and homogenized by coning and quartering. Finally the soil and tailings samples were stored at 4°C in tightly sealed polyethylene bags until its analysis.

Due to the strong association and affinity of heavy metals with fine grained soil components, we used the <100 µm soil fraction for the sequential extraction and total acid digestion methods [14]. The <2 mm soil fraction was used to determine the pH, electrical conductivity (EC), Organic matter content (OM) and carbonate.

Sample analysis

The physical-chemical characterization consisted in the determination of the sample (soils and tailings) pH, electrical conductivity (EC), organic matter content (OM) and the carbonate content according to standard methods [15]. Total heavy metal concentration was determined by atomic absorption spectroscopy after digestion of the samples. The methodology followed for the digestion consisted in weighting 1g dry sample and adding 3 ml HNO₃ (70%), 6 ml HCl (37%) and 3 ml HF (48%). The analyzed sample was placed in a sand bath to complete the digestion. After digestion, the sample solution was allowed to air-cool and then diluted with deionized water. The optimized sequential extraction procedure was applied to assess heavy metal fractionation in the samples [16]. The sequential extraction was performed using five-step procedure. Additionally, a sixth step was added, which consisted of dissolving the final residue using the same digestion procedure for the total metal determination [17].

(F1) Water Soluble: Soil sample extracted with 15 ml of deionized water for 2 hours.

(F2) Exchangeable: The residue from water soluble fraction was extracted with 8 ml of 1M MgCl₂ (pH 7.0) for 1 hour.

(F3) Carbonate-Bound: The residue from exchangeable fraction was extracted with 8 ml of 1M Ammonium acetate (adjusted to pH 5.0 with Acetic acid) for 5 hours.

(F4) Fe-Mn Oxides-Bound: The residue from carbonate fraction was extracted with 0.04M NH₂ OH. HCl in 25% (v/v) Acetic acid at 96°C with occasional agitation for 6 hours.

(F5) Organic-Bound: The residue from Fe-Mn oxides bound fraction was extracted with 3 ml of 0.02M Nitric acid and 5 ml of 30% H₂O₂ (adjusted to pH 2 with HNO₃) was added and the mixture heated to 85°C

for 3 hours, with occasional agitation. A second 3 ml aliquot of 30% H₂O₂ (pH 2 with HNO₃) was added and the mixture heated again to 85°C for 3 hours with intermittent agitation.

After cooling, 5 ml of 3.2M NH OAc in 20% (v/v) HNO₃ was added and the samples was made up to 20 ml with deionized water and agitated continuously for 30min.

(F6) Residual: The residue from organic fraction after drying was digested in a conical flask with 10 ml of 7M HNO on a hot plate for 6 hours. After evaporation, 1 ml of 2M HNO₃ was added and the residue after dissolution was diluted to 10 ml. the residue was washed with 10 ml of deionized water. After each successive extraction separation was done by centrifuging at 4000 rpm for 30 min. The supernatants were filtered and analyzed for heavy metals.

Quality Assurance: All chemicals used were of reagent grade and pure deionized water was used throughout the experimentation. All plastic ware soaked in 10% HNO₃.

RESULTS AND DISCUSSION

Table 1 presents the estimated results of physicochemical properties of soil samples obtained from the two studied areas (RC and AS).

Table 1: Mean values (%) of the grain-size analysis of different soils and tailings in Sidi Bou Othmane region.

	RC	AS	MT	Background soil
clay	14.9 ± 3.2	12.7 ± 4.4	29.1 ± 3.1	15.4 ± 2.5
Fine silt	19.8 ± 2.7	21.2 ± 2.1	13.5 ± 2.7	19.6 ± 3.7
Coarse silt	13.9 ± 1.2	11.7 ± 2.1	13.8 ± 1.5	11.1 ± 1.2
Fine sand	21.1 ± 3.4	23.9 ± 3.4	19.7 ± 3.1	25.2 ± 0.7
Coarse sand	22.5 ± 3.5	26.4 ± 3.8	23.9 ± 2.8	25.1 ± 0.4

Table 2: Geochemical characteristics of different soils and tailings in Sidi Bou Othmane region

Parameters	RC	AS	MT	Background soil
pH	6.6 ± 0.3	7.2 ± 0.3	4.2 ± 0.5	7.8 ± 0.2
E.C (μS.cm ⁻¹)	1698.4 ± 42.4	1824.5 ± 54.1	7573.9 ± 164.2	958.4 ± 82.1
OM (%)	4.3 ± 1.0	4.8 ± 0.7	3.2 ± 1.7	4.3 ± 0.8
OCC (%)	2.5 ± 0.6	3.7 ± 0.4	2.6 ± 1.0	2.9 ± 0.5
CaCO ₃ (mg.g ⁻¹)	158.7 ± 32.4	124.8 ± 27.8	162.1 ± 21.4	134.9 ± 17.4
S %	1.2	1.9	4.2	0.3
Cl-	< 0.1	< 0.1	< 0.1	< 0.1

Table 3: Mean Concentrations of heavy metals in different soils in Sidi Bou Othmane region.

	RC	AS	MT	Background soil
Cd (mg/kg)	1.1 ± 0.7	2.2 ± 0.2	143.1 ± 11.8	0.2 ± 0.0
Cu (mg/kg)	227.8 ± 225.3	330.5 ± 22.8	928.8 ± 88.5	40.7 ± 0.7
Pb (mg/kg)	184.0 ± 27.1	255.3 ± 24.0	3381.0 ± 507.1	11.8 ± 1.4
Zn (mg/kg)	648.0 ± 174.3	890.5 ± 101.0	2847.8 ± 460.3	133.9 ± 2.0

Textural characteristics of the studied soils are shown in table 1 according to the classification of Shepard [18]. These results showed that coarse sand (2.0-1.0 mm) and fine sand (MS 0.250-0.125 mm) were dominant fractions of all agricultural soils samples, with range from 22.5 to 26.4% and 21.9 to 23.1% in RC and AS respectively. Fine silt (0.002 - 0.0063 mm) is represented with 19.8 to 21.2% in RC and AS respectively.

pH, EC and carbonate (CaCo3) content are geochemical soil characteristics able to provide sufficient information to understand the soils capacity to retain heavy metal pollutants [19].

Numerical values on pH, EC and CaCO₃ for each analyzed sample are estimated in Table 2.

Results obtained for the soil pH measurements revealed that, in general, all sampled points presented a slightly acidic to neutral pH ranging from 6.6 to 7.2, lower than the background soil with the exception of a very acidic sample corresponding to the tailing sample with a pH 4.2.

These soil pH variations seemed to be related to heterogeneous deposits of sulfidic residues generated by mine tailings at the surroundings of the studied mine which can cause a decrease of the pH by corresponding oxidation and formation of sulfuric acid (0.9 to 1.2% of S).

EC showed more variability than the pH, with EC values ranging from 1698.5 to 1824.4 $\mu\text{S}/\text{cm}$. These obtained values of EC are significantly higher than for the background samples which indicate an increasing salinity gradient and consequently high amounts of labile ions close to the mine area. Mine tailings constitute a hot spot with an EC of 7573.9 $\mu\text{S}/\text{cm}$. This high value is mainly due to high amounts of metals present in this area.

Among the factors influencing the accumulation of metals, particle size played a significant role. Fine Grained soils often show higher concentrations of nutrients due to their greater surface-to-volume ratio and enrichment of organic matter (OM) [20].

Mean organic matter contents in studied soils were in the range of 4.3 in RC and 4.8% dw in AS. These results can be justified by the anthropogenic contribution; the discharge of domestic sewage at the Sidi Bou Othmane region was an important source of OM at this mining zone. High values of organic content were due to agricultural activities in the vicinity of the mine.

The organic carbon content (OCC) ranged from 2.5% dw in RC to 3.7 % dw in AS.

This parameter increased in AS soils corresponding to a decrease of the soil grain size. The highest organic carbon contents occurred at the soils that had the lowest sand contents and the highest silt and clay contents (table 1).

Table 3 shows the estimate results of total concentrations for the following elements in soils: Cd, Cu, Pb and Zn. All the results are expressed in mg/kg.

This total metal concentration obtained after a strong acid digestion does not provide sufficient information of its potential hazardous effects on environment because the mobility and eco-toxicity of heavy metals depend strongly on their specific chemical forms or binding. Consequently, these are the parameters that have to be determined, rather than the total element contents, in order to assess toxic effects.

Heavy metals may be distributed among many components of the soil and may be associated with them in different ways [21]. The nature of this association is known as speciation. The chemical form of heavy metals in soil is of great significance in determining the potential bioavailability and translocation of the metals to other environmental compartments like water, plant and microorganisms when physicochemical conditions are favorable [22].

For tailings, overall, cadmium (figure 3) seems to be very available with more than 53% of total metal bound to the short-term mobile fraction (F1 and F2). The long-term mobile fraction (F3, F4 and F5) homes more than 32% of total metal. However, the immobilized fraction (F6) finally traps about 15%.

In soils, cadmium was mainly present in the labile phase (water soluble, exchangeable and carbonate-bound fractions) with more than 57% in all the stations: The exchangeable fraction represents about 37% of the total metal in the two areas, while the carbonate fraction presents 19 and 25% of the total metal respectively in the RC and AS soils. The residual fraction does not exceed 13% of the total metal in the two areas.

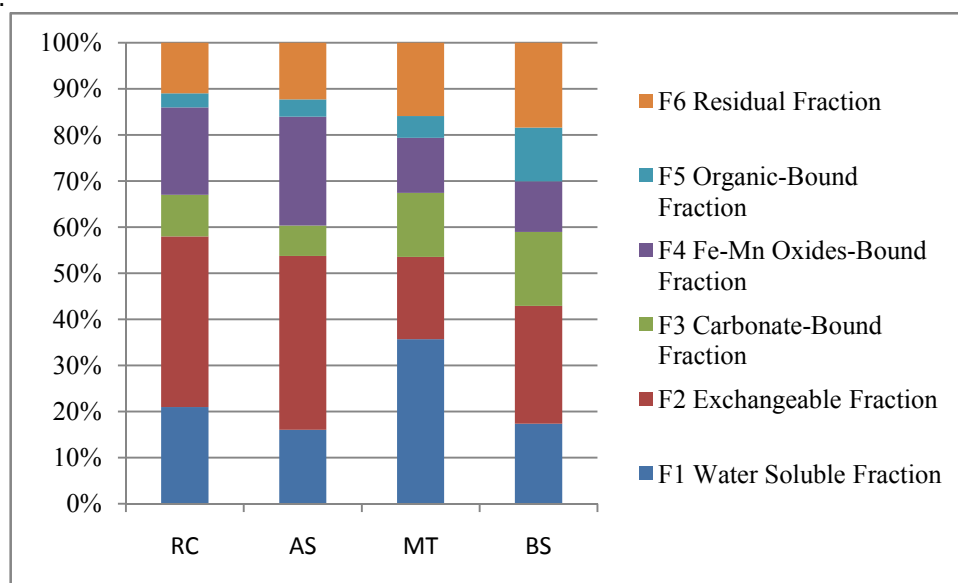


Figure 3: Relative distribution of cadmium among six fractions (F1-F6) of studied samples.

Copper in tailings (figure 4) is essentially bound to soluble (F1) and exchangeable fractions (F2) with more than 45% of the total metal content. Copper is also widely present (23%) in the residual fraction

(F6). The oxidizable (F3), acid-soluble (F4) and reducible fractions (F5) are represented by small percentages in these tailings; they trap respectively 13, 7 and 10%.

Copper presents almost the same distribution in different fractions of studied soils. It is widely abundant respectively in RC and AS in the organic form (33-37%) followed by residual (23-26%) and carbonate fractions (19-23%). The soluble and exchangeable fractions poorly represented in these soils.

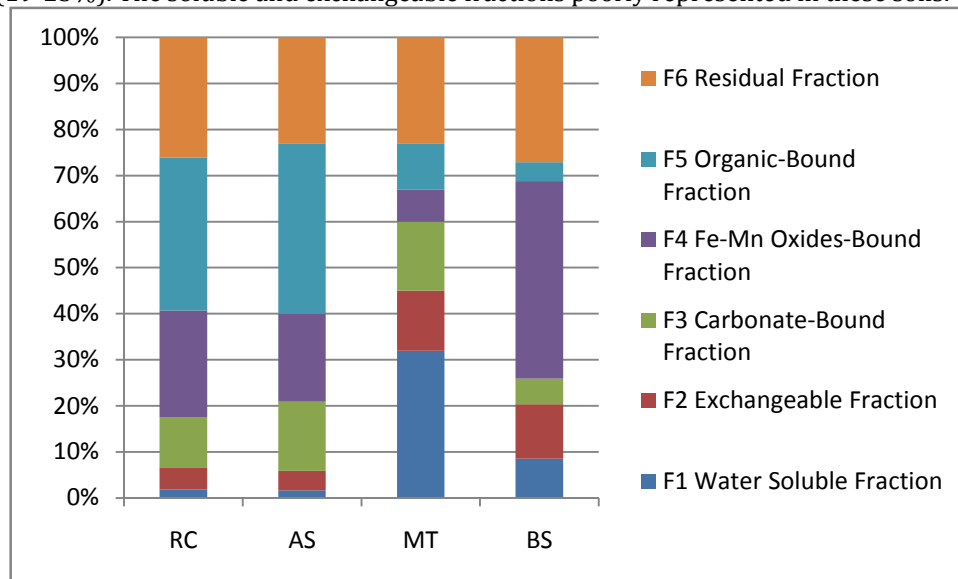


Figure 4: Relative distribution of copper among six fractions (F1-F6) of studied samples.

The high Cu concentrations in organic phase in the studied soils can be justify by that copper is characterized by high complex constant organic matter thus it can be hypothesized that Cu is bound to labile organic matter such as lipids, proteins, and carbohydrates. The relatively high percentage of Cu in the residue is likely due to the fact that Cu is easily chemisorbed on or incorporated in clay minerals (Pickering, 1989).

Lead distribution (figure 5) in tailings is characterized by a strong dominance of this metal bound to long-term mobile fraction (F3, F4 and F5), with more than 64% of the total metal. The short-term mobile fraction (F1 and F2) represents about 24%. The remaining fraction (F6) traps about 12% of the total metal.

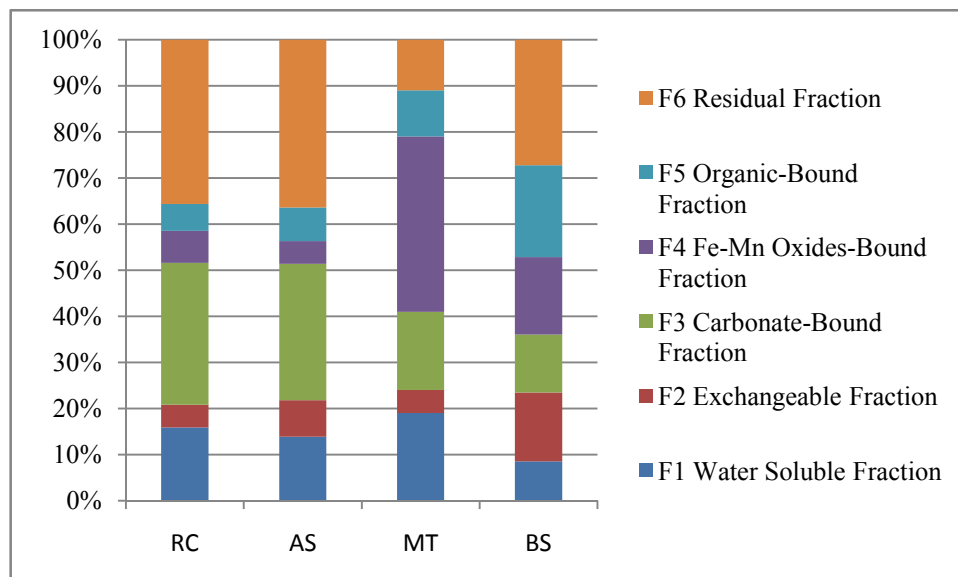


Figure 5: Relative distribution of lead among six fractions (F1-F6) of studied samples.

For studied soils, the lead distribution is the same. Indeed, after the residual fraction (about 37%), Pb is located in the long-term mobile fraction (F3, F4 and F5) with more than 47%. However, the soluble and exchangeable fractions do not exceed 16% in the two studied areas.

The relatively high percentage of Pb in reducible phase is in agreement with the known ability of amorphous Fe–Mn oxides to scavenge Pb from solution (Yusuf, 2007). Thus, a high element percentage in the reducible fraction is a hazard for the aquatic environment because Fe and Mn species can be reduced into the porewaters during early diagenesis by microbially mediated redox reactions [23]. Dissolution will also release Pb associated with oxide phases to the porewater possibly to the overlying water column and to benthic biota (Kabala and Singh, 2001). The major sources of Pb are from intensive human activities, including agriculture in the drainage basin [24]. In addition, a substantial contribution from the factories located in the upstream of the Tensift river dealing with Pb play a vital role in soils contamination by heavy metals as referred by [25].

Zinc distribution (figure 6) in the tailings shows an equal distribution of this metal in the three fractions with 32% in the short-term fraction (F1 and F2), 34% in the long-term mobile fraction (F3, F4 and F5) and 34% of the total zinc content in the lithogenic fraction (F6).

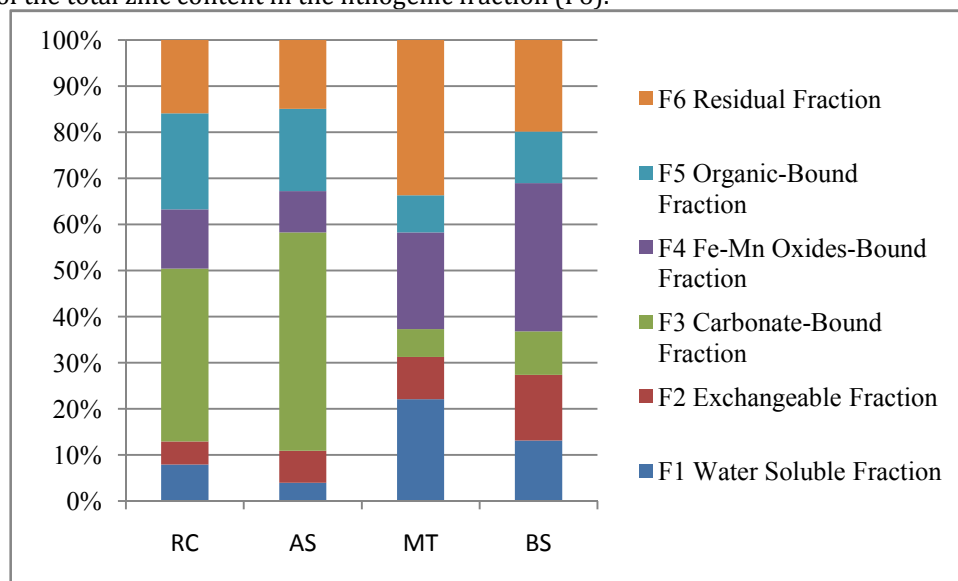


Figure 6: Relative distribution of zinc among six fractions (F1-F6) of studied samples.

Zinc distribution in the studied soils is almost the same. Indeed, after the reducible fraction representing between 37 and 48% of total metal. This result can be justified by the high stability constants of Zn oxides. Iron oxides adsorb considerable quantities of Zn and these oxides may also occlude Zn in the lattice structures [26].

The carbonate, residual and organic fractions oscillate between 13, 16 and 21%.

CONCLUSION

Fractionation of the metals species in soils around the abandoned Sidi Bou Othmane mine shows higher percentages of the metals residing in the non-residual fractions. This suggests potential bioavailability of cadmium, copper, lead, and zinc in the soils around the Sidi Bou Othmane mine, indicative of their anthropogenic source. The knowledge of the chemical speciation of metals in the soil may assist in determining the behaviour of the metals since they occur in different forms in the soil, and may influence their mobility and bioavailability. The rapidly mobilizable metals in the soil indicate the potential health hazards of cultivated agricultural lands around this mine.

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