

FULL PAPER

Evaluation of metal pollution in agricultural soil near industrial landfill in Ghazaout, Algeria

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The objective of this study was to assess the damage to soils used for agriculture and natural vegetation to study the risks of contamination and the mobility of heavy metals (Pb, Cd, Zn, etc.). The extractions carried out on the ground showed total levels above the limit standards, thus generating contamination by cadmium to a lesser extent by zinc, arsenic and within the standards for cobalt, copper, chromium, nickel and finally the major elements. As for cultivated plants, the study indicated a potential ecological risk of grapes contaminated in particular by chromium, then by zinc and finally by copper, thus leading to potential health risks. All the results obtained can contribute to understanding the effects affecting agricultural soils and food plants in the city of Ghazaout.

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KEYWORDS

Industrial discharges; cadmium; zinc; contamination.

Introduction

Increasing concentrations of Potentially Toxic Elements (PTEs) including metals and metalloids in the environment pose a serious threat to human, animal and plant health. For example, the frequently reported "blood lead" incident [1], "rice with cadmium" [2] and "vegetables contaminated with heavy metals" [3] that are all associated with PTE pollution. In addition, PTEs may pollute the air through wind erosion [4] as well as surface and underground water bodies through surface runoff or deep percolation [5].

Heavy metals are highly toxic substances due to their high concentrations; they are not biodegradable and have a low capacity for mobilization in the environment. Therefore, they cause soil and water pollution, as well as toxic, genotoxic, teratogenic and mutagenic effects on living organisms, causing endocrine

and neurological disorders even at low concentrations [6,7].

Since the last century, the development of industrial activity (metallurgical and chemical industries) and agricultural activity (pesticides, fertilisers, etc.) has sometimes led to intensive inputs of these elements, which become pollutants for the environment. Even at low concentrations, they can constitute a serious public health problem because of their toxicity and their bio-accumulative nature [8].

The first threshold values for hyperaccumulation of trace elements were determined as follows: 1000 mg / kg for Ni, Co, Cu, Cr, Pb and > 10000 mg / kg for Zn and Mn [9]. A new updated and revised proposal for thresholds considers a plant a candidate for hyperaccumulator if its dry matter of aboveground tissue contains more than 100 mg / kg Se, Cd and Tl, 300 mg / kg Cu, Co, Cr,

1000 mg / kg Ni and Pb, 3000 mg / kg Zn and 10000 mg / kg Mn [10].

Due to heavy metal toxicity, soils contaminated with these metals constitute an environmental problem that requires an effective and affordable solution.

In order to study the risks of accumulation, contamination and mobility of heavy metals (Pb, Cd, Zn, etc.) in cultivated food plants, near the ALZINC industrial landfill, we selected a soil sample whose culture is vine.

The objective of this study was therefore to determine the impact of heavy metals from agricultural soil to the roots, then the aerial parts of grapes using a multidisciplinary approach. In our study, we applied commonly used protocols, such as sampling methods, physico-chemical characteristics, soil dissolution, which made it possible to determine the total content of metallic elements contained in the samples in order to better understand and explain the behavior of trace elements in soil and grapes through measuring these metals by ICP-AES.

Another reason to address this study was that in Algeria only industrial environments (aquatic and atmospheric) have been considered so far. On the other hand, the behavior of heavy and major metals in agricultural soils and grapes in this study was considered for the first time in the city of ghazaout-Algeria.

Materials and methods

Origin of samples

The samples selected for this study were three soils from an agricultural site located near industrial discharges from the ALZINC plant and also near solid and liquid domestic waste (Figure 1). For this, a plot of land whose crop is grapes was chosen.

Sample I (soil R): an area of approximately 1.5ha; Slope: 2-6%, where the grapes are grown.



FIGURE 1 Study area: sample collection plots

Sampling

For an analysis, characterization or diagnosis, the date of soil sampling was not considered significant over the course of a year. However, after adding fertilizer, we should avoid taking samples until the layer has found sufficient homogeneity and, to this end, it is recommended to wait 1 to 3 months after adding fertilizer, soil conditioner, or cooked mineral and 6 months after adding any other amendment (Figure 2).

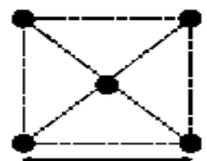


FIGURE 2 Sampling method

Transport

The samples are transported in such a way as to minimize changes in the water content of the soil and are kept in the dark with free access. A loosely closed polythene bag is usually sufficient. Once in the laboratory, the soils were treated as soon as possible after their sampling and after removing vegetation, stones and impurities. Then, the soils were spread out to facilitate the sieving operation.

Physico-chemical characterizations

Sample preparations before analysis

Intermediate sampling was done according to the technique of quarters in order to minimize the risk of error on the composition of the soils related to their heterogeneity and to obtain the mass of residues necessary for the analyses and tests (Figure 3). Particles larger than 2 mm were rejected. Thanks to this manipulation, the error related to sampling did not exceed 10%. The resulting soil residue is thus prepared for chemical treatment [11].

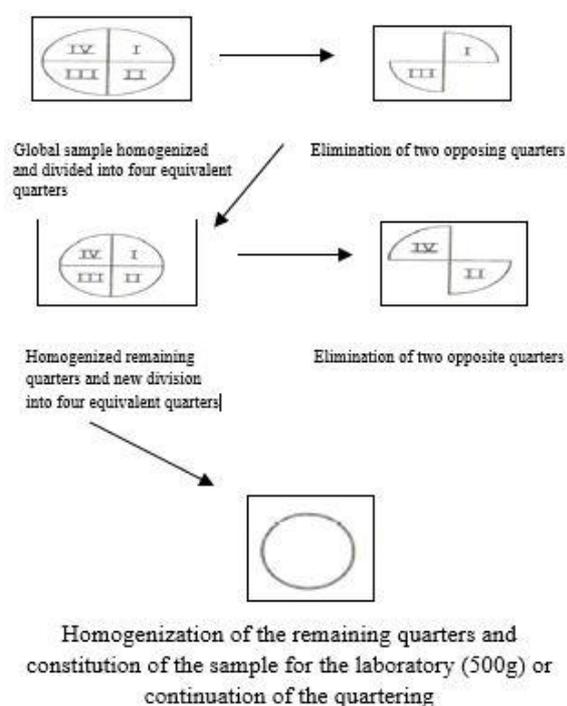


FIGURE 3 Constitution of the laboratory sample (principle of quartering)

Particle size analysis of soils

The particle size was determined by following the NF X31-107 75 standard in the civil engineering soil analysis laboratory-University of Tlemcen [11].

Determination of residual humidity

Residual moisture is defined as the mass lost after drying at 105 °C of a sample whose mass is constant at 40 °C. Its measurement makes it

possible to determine the dry mass of a soil sample. The residual humidity H is expressed in mass percentage and is calculated by the following relationship:

$$H = (m_0 - m_1) \times 100 / m_0$$

where m_0 signifies the initial mass of sample dried at 40 °C and m_1 is its final mass, after drying at 105 °C and cooling [11].

Particle size analysis of soils

The particle size was determined by following the NF X31-107 75 standard in the civil engineering soil analysis laboratory-University of Tlemcen [11].

Determination of pH

10 g of soil was suspended in 50 mL of distilled water, stirred for 1 hour on a stirring plate, then left to rest for 2 hours. The pH was then measured using a Radiometer M220 type pH meter [11].

Determination of carbonate content

We took 0.5 g from the soil, crushed it and sieved it to 0.1 mm. The determination of the carbonate content was made by Bernard calcimeter [11].

Determination of organic matter (OM)

The soil was calcined at a temperature of 500°C in an oven for 5 hours. The organic matter content represents the difference between the two masses (before-after calcination); the content then was reported at 100 g of the initial sample [11].

$$\% M_0 = (M_0 / M_1) * 100$$

where M_0 stands for the initial mass of sample dried at 40 °C, in grams and M_1 signifies final mass, after calcination at 500 °C, in grams. 100 g for each dry soil was sieved at 2 mm.

Determination of the cation exchange capacity (CEC)

The exchange capacity is a measure of the quantity of ions likely to be retained by exchange on a solid, in the presence of an excess of exchanger ions in solution. This capacity can be either cationic (CEC) or anionic (CEA), depending on whether the surface of the medium is negatively or positively charged. Determining the CEC of a solid consists of measuring a parameter characterizing a state of equilibrium between the soil sample and a given experimental environment. The values obtained are a function of the medium, namely saturating cation, pH, ionic strength, presence of other ions, ..., as well as the conditions of realization influencing the yield of the exchange reactions. CEC is initially linked to the presence of clay, organic matter and at the same time to heavy metals [12].

Total content of major elements and metallic trace elements (Total Extraction Or/Mineralization) in soil

Pre-treatment of samples

The samples were homogenized and quartered (Figure 4), then dried at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$. The samples were then sieved at 2mm (elimination of plants, plastic, glasses, pebbles, ...).

Homogenization was carried out by grinding a 0.315 mm or/315 μm sieve to increase the contact surface between the metals and the reagents.

Principle

The dosage consists, initially, of dissolving the elements with a mixture of hydrochloric and nitric acid in a 3 V/V ratio (the mineralization phase). The elements were then assayed by ICP-AES.

Operating mode

The samples were mineralized by acid digestion with aqua regia. Each sample was

the subject of 3 test portions of $0.5\text{ g} \pm 5\text{ mg}$. Each catch was placed in a glass Erlenmeyer flask where it received 2-3 mL of demineralized water to obtain a boat product, followed by addition of 7.5 mL of hydrochloric acid (37% (m/m), density 1.19 g/mL, Merck Supra pure max Hg 0.005 ppm) and 2.5 mL of nitric acid (65% (m/m), $\rho=1.38\text{ g/mL}$, Merck Supra pure max Hg 0.005 ppm). The mixture was sealed and left at room temperature for 12 hours. The mineralization was then concentrated by boiling for 2 hours. After cooling, and placing a filter on ashless filter paper, the volume obtained was adjusted to 25 mL with distilled water.

Plants

Sampling

Grape samples were taken a few days before harvesting. The plants at the edge of the paths are spaced out so as to limit bias due to dust deposits. These samples were transported in polypropylene bags.

Sample preparation

We cut and eliminated the ends, as well as the damaged leaves of the grapes, then washed them twice with tap water.

Then, we cut them with a stainless steel knife in slices applied on the stems of the grapes. Finally, they were dried in an oven at a temperature not to allow it exceed $50\text{ }^{\circ}\text{C}$.

Grinding

All samples were crushed using an oscillating tungsten carbide ring mill and sieved at 315 μm .

Storage

Once these operations were carried out, the samples were placed in inert bottles, then stored in opaque barrels and stored away from dust. The samples conditioned were

ready for mineralization and chemical determination of metals.

Calcination

Protocol

200 to 300 mg \pm 5 mg of vegetable powder was weighed into a porcelain capsule. The capsule was placed in a muffle furnace. The best results were obtained by keeping the oven at 300 °C until the carbon ceased to glow. The oven temperature then rose to 400-450 °C. The mineralization time was variable; it depended on the nature of the material to obtain friable white ashes.

The oxidation of the last traces of organic material was then carried out by adding 1 to 2 mL of HNO₃ (1N) after cooling the capsule. It was evaporated to dry on a hot plate or in a sand bath and placed in the oven at 400 °C for one hour.

Mineralization

Operating mode

Dissolution for the determination of heavy metals was carried out by acid attack of a

quantity of 0.5 g of plant sample per 10 mL of aqua regia (7.5 r= 1.38 g/mL, pure Merck Supra max Hg 0.005 ppm, by a volume ratio (3/1). The whole was then brought to the boil on a hotplate for 3 hours using reflux heating. Cooling was followed by filtering into a (25 \pm 0.1) mL volumetric flask, then it was completed with distilled water to the mark.

Instrumental analysis of metallic elements in soil and plants

The determination of metals was carried out by plasma atomic emission spectroscopy by inductive coupling.

The expression of the results in mg/kg relative to the dry matter (Ms) is given by the following formula :

$$C(\text{sample}) (\text{mg/kg}) = C_1 \cdot V \cdot D / \text{Test sample mass}$$

C₁: (sample) (mg/l), V: V(mineralization),

D: dilution

Results and discussion

The particle size distribution of the different fractions obtained is given in Table 1.

The results of the physico-chemical properties are listed in Table 2

TABLE 1 Results of granulometry by sieving soils

Sample	Sieve mesh in (mm)	Mass of sieves in (g)		Cumulated screens (100 - (Rn/ Ps)) in (%)
		Partials (Ri)	Cumulative (Rn)	
Soil R	5	5,63	5,63	99,44
	3,15	4,51	10,14	98,99
	2	11,63	21,76	97,82
	1	37,89	59,65	94,03
	0.5	17,97	77,62	92,24
	0,2	200,81	278,44	72,16
	0,1	665,32	943,76	5,62
	0,083	31,89	975,65	2,44
	<0.083 (fund)	22,35	998,00	0,20

TABLE 2 Physico-chemical characteristics of the soil

Identification of the land	Residual humidity	Layers (cm)	pH	CEC (C+mole/litre)	Carbonates (g/kg)	MO (%)
Soil R	11.96	0-20	8.07	30.29	4.56	2.86

CEC symbolizes Cation exchange capacity, expressed in (C+mole/litre)*, MO signifies Organic matter, expressed in % relative to the raw sample, and CaCO₃ represents Carbon carbonate, expressed in % relative to the raw sample.

The results obtained from the analysis of the total contents of major metals and heavy metals by extraction with aqua regia of the soil, the industrial landfill and the various organs of the grape are given in Tables 3 and 4 and represented as histograms in Figures 4-13.

TABLE 3 The average total contents (g/kg on dry matter) of major metals in the different organs of grape

	Ca	K	Mg	Na	Fe
Sheets	12,23	2,00	0,82	1,35	0,21
Fruits	3,07	1,20	0,78	1,12	0,04
Stems	2,75	2,01	5,08	1,51	0,05
Roots	3,37	2,02	1,25	0,68	0,14
Soil R	201,30	19,52	21,37	4,31	11,32
Industrial dump	8,59	1,44	0,92	1,00	6,13

TABLE 4 The average total contents (mg/kg on dry matter) of heavy metals in the different organs of the grape

	Pb	Cd	Cu	Zn	Ni	Mn	Co	Cr	As
Sheets	7,15	3,20	20,13	361,77	3,04	39,40	0,38	4,20	0,02
Fruits	3,92	0,06	10,00	71,13	2,66	7,52	2,01	0,78	0,67
Stems	0,95	0,89	7,51	107,75	0,94	8,22	0,01	2,59	0,30
Roots	0,33	0,67	4,14	343,15	8,39	7,53	1,90	26,09	31,10
Soil R	40,31	13,15	18,31	307,21	13,02	503,30	3,02	68,04	42,11

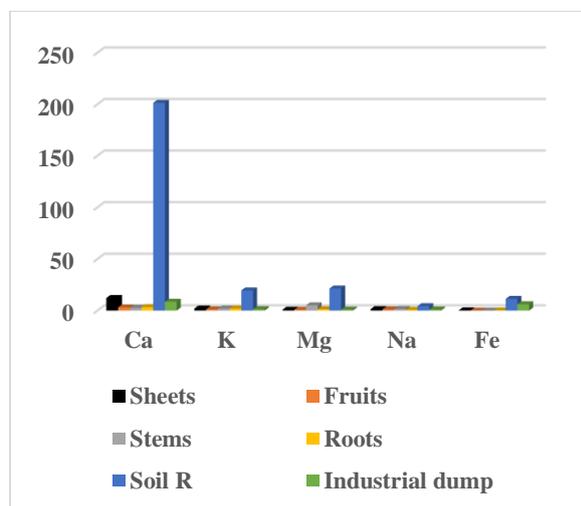


FIGURE 4 The total average contents of major metals (g/Kg on dry matter) in different parts of the grapes, in the soil and in the industrial waste

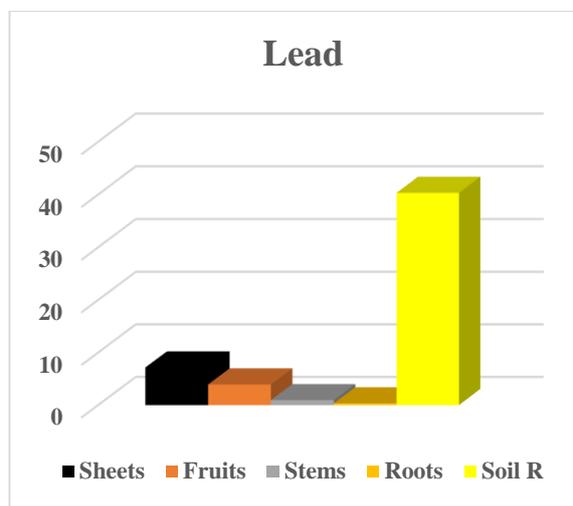


FIGURE 5 The average total contents (mg/Kg on dry matter) of lead in the different parts of the grape and its nourishing soil (R)

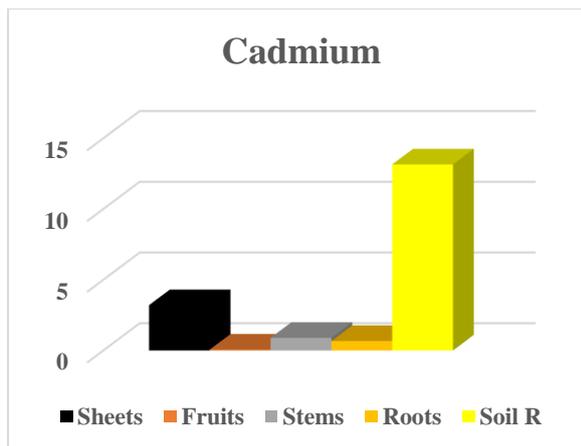


FIGURE 6 The average total contents (mg/Kg on dry matter) of cadmium in the different parts of the grape and its nourishing soil (R)

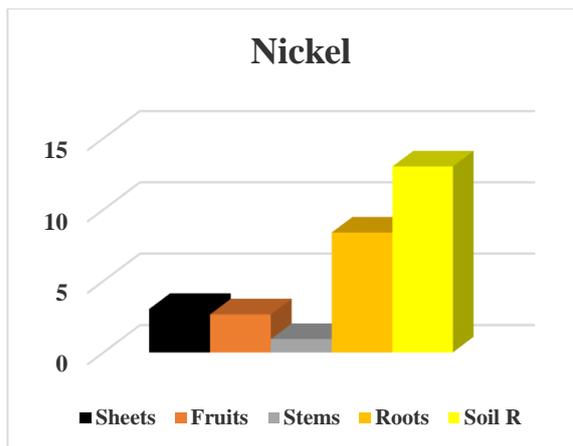


FIGURE 9 The average total contents (mg/Kg on dry matter) of Nickel in the different parts of the grape and its nourishing soil (R)

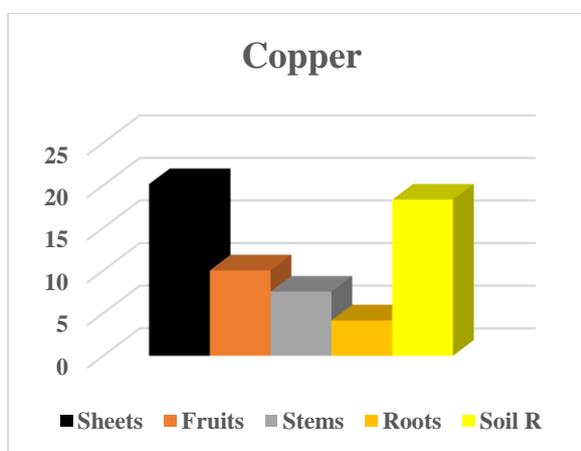


FIGURE 7 The average total contents (mg/Kg on dry matter) of copper in the different parts of the grape and its nourishing soil (R)

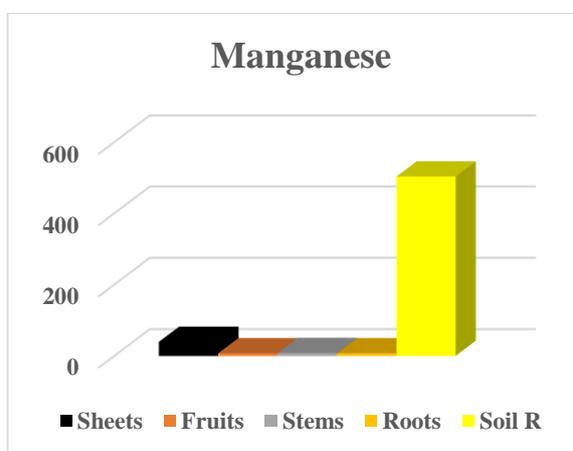


FIGURE 10 The average total contents (mg/Kg on dry matter) of Manganese in the different parts of the grape and its nourishing soil (R)

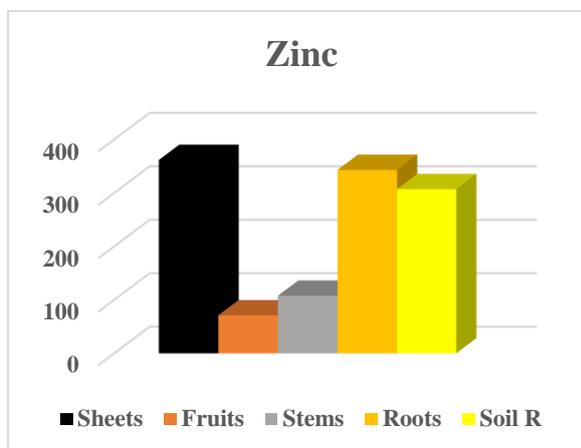


FIGURE 8 The average total contents (mg/Kg on dry matter) of Zinc in the different parts of the grape and its nourishing soil (R)

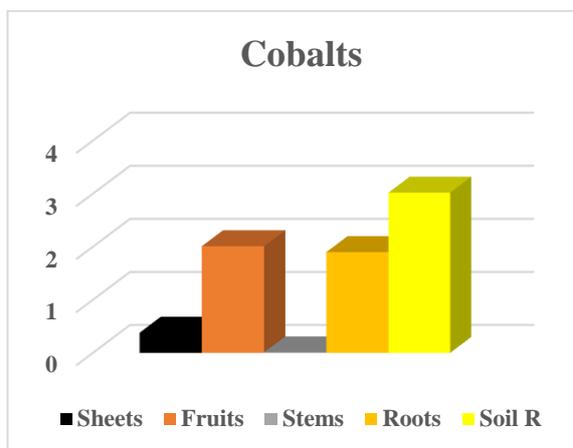


FIGURE 11 The average total contents (mg/Kg on dry matter) of cobalt in the different parts of the grape and its nourishing soil (R)

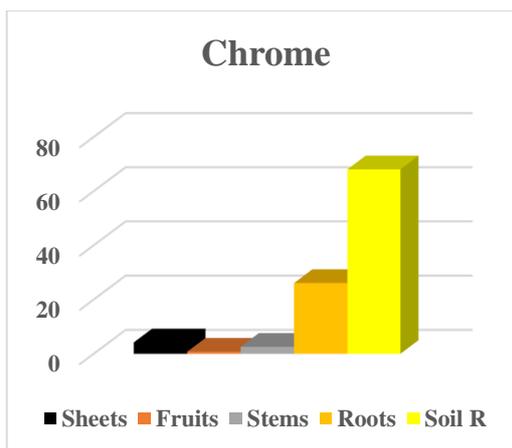


FIGURE 12 The average total contents (mg/Kg on dry matter) of chrome in the different parts of the grape and its nourishing soil (R)

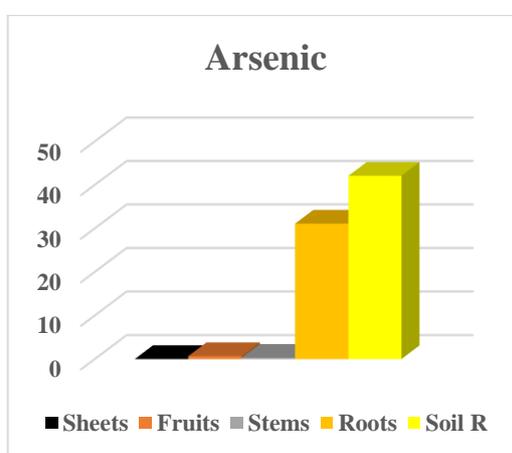


FIGURE 13 The average total contents (mg/Kg on dry matter) of Arsenic the different parts of the grape and its nourishing soil (R)

Granulometry

The soil has a dominant composition by the 0.1mm fraction of 66%, followed by 19% the particles of size 0.2 mm; it is said that the soil is mainly composed of aggregates of size between [0.1–0.2], so it is fine sandy soil, which influences the availability and mobility of metals in soils.

The physico-chemical parameters

We noticed that a residual humidity was quite average. The uptake of ETM by plants increases mainly with an increase in the residual moisture value.

The pH is another important factor influencing the solubility and the speciation of the metal and therefore its toxicity; when the pH decreases by one unit, the concentration of free metal cations increases by about a factor of 2 in the soil solution and hence enhances phytoextraction [13,14].

The soil collected has a remarkable basicity. Changes in pH could in particular result from biological activity and an increase in the CO₂ content.

Carbonates are found in large quantities, which explains the basicity of the soil studied.

The percentage of organic matter (OM) seems very low.

The cation exchange capacity is also an important characteristic of a solid matrix, corresponding to the quantity of saturating cations fixed by the soil in a buffered medium. Our soil is highly ion exchanger; the estimated CEC is higher compared with the average values announced by the literature by 0.6 meq/g (60 meq/100 g) for a mineral soil and 2 meq/g (200 meq/100 g) for organic soil. Indeed, humic compounds have a particular affinity for this Ca⁺² cation; the adsorption of Ca⁺² in the presence of organic matter is favored [15].

The cation exchange capacity includes all the exchangeable ions in this soil (Ca⁺², Na⁺, Mg⁺², K⁺).

Major elements in agricultural soil

In the absence of Algerian standards relating to the reference contents in the soil, we adopted a comparative approach with respect to the references noted in the bibliography and supplemented by the European standards to interpret our results [16].

To interpret our results, we reported these results in mass percentage and are grouped in the Table 5 below.

The presence or absence of soil micronutrients and macronutrients determine the viability of plants on a particular soil. This necessitated the need to

determine the levels of some important nutrients in the study area, Mg and K, which are the important macronutrients for plant growth.

TABLE 5 Mass rate (%) of major elements in agricultural soil

Ca ²⁺	K ⁺	Mg ⁺²	Na ⁺	Fe
78,08	7,57	8,29	1,67	4,39

The calcium ion (Ca²⁺) is found in predominant quantity for the studied soil, representing a mass percentage of (78.08-79.06)%.

The quantities of Ca²⁺, K⁺, Na⁺ ions as well as the quantities of iron and magnesium are greater than those of the discharge from the ALZINC plant (Table 3), the origin of which is essentially the main anthropogenic sources, due to traffic and from the erosion of urban materials. Potassium is found in fertilizers, it also comes from the combustion of biomass and vegetation and sodium originating from urban sources that emit into the atmosphere.

Heavy metals in soil

All soil samples were taken at varying distances from the Alzinc dump. The lead, copper, nickel, cobalt, manganese and chromium contents are below the soil toxicity threshold [17] and the standards for agricultural soils [16]. So, they are within the standards, which have no negative effects or symptoms on the environment (soils, plants, etc.), so they do not present any danger.

Cadmium levels greatly exceed the toxicity threshold of agricultural soils. In addition, the zinc and arsenic levels are slightly higher than the normal threshold of French standards [18]. There is therefore contamination by cadmium to a lesser extent by zinc and arsenic. This contamination is of an anthropogenic nature, which poses a problem in the medium and long term.

Knowing the total levels of heavy metals is necessary, but not sufficient to assess their

mobility and the resulting environmental risks. The prediction of these risks is closely linked to the physico-chemical forms in which the metals are present, i.e. to their speciation, the study of the speciation of pollutants such as zinc, cadmium, arsenic provides information on their interactions with the solid phase and on the bonding forces with it, and therefore on their mobility as the potential danger for the environment being directly linked to the mobility of the polluting elements.

Variations in the levels of the plant species (grapes)

To verify the character of metal accumulation in barley, an extraction of heavy metals by aqua regia at the level of the roots and the aerial parts was carried out and to interpret our results we compared with the normal and critical thresholds of the plants.

The results of the major metallic element contents detected in the vegetation show the facts presented as follows:

The major metallic element in the plant species (grapes)

Calcium and potassium are concentrated more in the leaves than in the other organs; moreover, magnesium and potassium are mainly distributed respectively in the roots and the fruits, which are within the norms. On the other hand, the iron levels are below those of the standards, so iron deficiencies are recorded in the grapes.

The heavy metals in the plant species (grapes)

Lead, cadmium, manganese, zinc and copper are mainly concentrated in the leaves. In addition, nickel, chromium and arsenic are mainly concentrated in the roots. On the other hand, cobalt concentrates preferentially in the fruits than in the other organs.

It is noted that the chromium contents greatly exceed the critical threshold of the

plants [17]. Notable chromium toxicity is reported in the roots of the raisin. This toxicity is due to the distance that is close to the industrial landfill of Alzinc.

While the zinc and copper levels are within the critical threshold for plants, a strong accumulation of copper and zinc in the leaves is deduced, which presents a real danger. In terms of toxicity, we found that zinc presents a great danger, which obliged us to report it urgently to agriculture. Grapes contaminated with zinc are close to rejection (discharge).

Consequently, the levels of cadmium, cobalt and arsenic greatly exceed the normal threshold for plants [19], so there is a high accumulation of these heavy metals in the grapes. Cadmium is the metal of most concern in terms of contamination throughout the food chain. It easily accumulates in plants and animals because it replaces Zn in some enzymes. Moreover, it is more mobile in soils and more absorbed by plants than other metals such as Cu and Pb [20-21]. As a result, the likelihood of it being transferred from the soil to the plant and then to humans is greater. Symptoms in the man were bone disorders and kidney dysfunction. [22].

In addition, the lead and nickel contents are within the standards.

On the other hand, the manganese and copper contents particularly in the stems and roots are below the normal thresholds for plants. Manganese and copper deficiencies (in stems and roots) are recorded in grapes.

Grapes accumulate metals in the order $Cr > Zn > Cu$. Criteria were reported for a plant to be categorized as a hyperaccumulator, that is, a plant with exceptional ability to concentrate heavy metals in their leaves.

Conclusion

This research work has provided access to detailed knowledge on the fate of metal pollutants in agricultural soils and grapes.

He also warned us of the danger that man may experience. Due to lack of data on the

geochemical background of Algerian soils, it was difficult for us to differentiate with certainty the share of metals of endogenous nature from that of anthropic origin. Nevertheless, the high levels observed on the surface would probably be an argument in favor of an anthropogenic contribution.

Indeed, if these total levels give no information on the mobility of metals in soils or on their bioavailability, but only the expression of a potential danger of a soil that could present a threat to plants, it is more speciation which allows us to assess the fate of metal pollutants and an approach to risk.

The study of the transfer and accumulation of heavy metals in the various organs of the grape shows a fairly significant accumulation of chromium, then of zinc and finally of copper in the roots and leaves. This accumulation is due to the remoteness, which is close to the Alzinc industrial landfill.

This interesting result represents a novelty, which is reported for the first time in the city of Ghazaout-Algeria.

Furthermore, it was interesting to be able to compare the results relating to these sites with those obtained on other sites in order to consider a generalization of the trends observed and also to be able to propose practical measures to control or attenuate the effects of this pollution.

It would also be interesting to continue this study with a phenomenon modeling approach, which would make it possible to estimate the risks of migration of pollutants or the accumulation capacities within the different phases according to variations in multiple conditions (mixture of reagents,...) and on a larger time scale, by limiting experiments to the laboratory. The use of methods allowing the monitoring of the evolution of speciation, in conjunction with these experiments, would make it possible to better support the conclusions.

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