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Assessment of Soil and Surface Water Quality in Makhat's Watershed (Taza Province, Morocco)

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ABSTRACT

Acid mine drainage (AMD) threats the soils and water bodies in the territories of the sulfide mining activities all over the world. Bouaazza's mine (NE Morocco) lead and sulfides exploitation resulted in the surface exposure of large superficies of acid discharges. The results of physicochemical and geochemical analysis indicate an acidic pH (3.0–7.0) of water samples, with concentrations of lead exceeding Moroccan standards (50 µg/L). The pH in soils is considered from slight up to moderate acidic (< 6.5), with low limestone content (< 6% of $CaCO_3$), and high lead and zinc values exceeding international standards (300 mg/kg). These results indicate the high health risk generated by the absence of environmental monitoring of the mining operations which threaten the water quality in the surrounding area.

Keywords: acid mine drainage, Bouaazza's mine, mining discharges, Makhat's watershed, pollution.

INTRODUCTION

Mining industry has positive impacts on the economy of countries through to raw materials it provides (Gałaś et al., 2021; Mancini & Sala, 2018). Despite its benefits, it's the environment that pays high price in front of the discharges left by its abandoned or current active mines. Indeed; the discharges impacts indicate a global scourge as few policies require site rehabilitation (Pelletier-Allard, 2014). Contamination and pollution of soil and water by high concentration of metals (Zinc, Lead, Iron and Arsenic) are among of the main problems of our time (Rezaie & Anderson, 2020). The majority of soils and waters exhibit metals which mainly have a geochemical origin (bedrock, weathering, etc.) (Gandhi & Sarkar, 2016; Kelepertzis et al., 2013; Kierczak et al., 2021). While in some sectors, anthropogenic activities (industrial, agricultural and mining) contribute to increase the total metal content in these environmental compartments (Briffa et al., 2020; Saleh et al., 2022).

In a Mediterranean climate with rainfall of 400 mm/year and a temperature of 9 °C to 32 °C, these sites constitutes a favorable environment for the production of sulfuric acid (H2SO4), decrease in pH, release the heavy metals in the medium and lead to an environmental contamination. It is the acidic mine drainage (AMD) (Lakrim et al. 2012). The contamination of water resources caused by acid mine drainage (AMD) processes remains the big problem with sulfide mining (Drapeau et al., 2021; Rivera et al., 2019): Acid mine drainage, phenomenon generated by mining activities; is a form of water pollution occurring when rain, runoff or streams come in contact with rocks rich in sulfur that are present in abandoned or currently active mines.

Despite the studies that have been made to deal with the impacts generated by this phenomenon (Ali, 2019; Coudert et al., 2019; Skousen et al., 2017; Itard & Bosc, 2001), AMD still causes negative impacts globally: In Colorado's San Juan Mountains (USA), site of the gold King blowout, an estimated 15 million gallons (57 million liters) of acid mine drainage per day are the results of 400 abandoned or inactive mine sites (Press, 2019). In China, the amount of coal mines has been reduced from more than 80,000 to about 5800 at the end of 2018 (China Coal Industry Association) (Zhang et al., 2019). These abounded mines have caused negative impacts on the environment by deteriorating the quality of water (Wang et al., 2021).

Morocco is a mining country thanks to the diversity of its resources (Phosphate, Zinc, Copper, Iron, etc.). It is the highest producer in Africa for Silver and Lead (Group Oxford Business, 2016). The mining sector provides a quarter of national exports and contributes to 10% of the national GDP and employs around 40,000 people (Akoudad, 2015). Although mining industry provides benefits to the country, abandoned or current active mines produce tones of discharges that have negative impacts on the environment. During the last 15 years, several studies were done to highlight the risks related to old mining activities which generate AMD (Amrani et al., 2020; Boularbah et al., 2006; Hakkou et al., 2008; Nfissi et al., 2017). The Kettara's pyrrhotite ore abandoned mine (Marrakech province) resulted a very large superficies of tailings that occupy an area of 16 ha. By geophysics surveys, the volume of its discharges was estimated around 462 400 m3. These results show the presence of fractured zones that constitute privileged drains for the transfer of the AMD to groundwater (Lghoul et al., 2014). The iron mining district of Nador which extends over an area of 35 km², shown pH results of water samples taken in situ is less than 3, reaches even 0.87 in some places (Lakrim et al., 2016).

Bouaazza's mining activities are of local interest and characterized by limited volume operations which are not subject to strict governmental legislations. It is a small scale mine, operated by the locals and is situated in the middle of Makhat's watershed (Taza Province), it is a part of the NE of the Tazekka Variscan massif (Mesrar, 2013); characterized by a semi-arid climate (Naoura, 2012), cold in winter and hot in summer (Laaraj et al., 2020). The mineralization of Bouaazza's mine is embedded in Tazekka's shales. Its discharges are placed on the eastern slopes of Makhat's watershed, and are scattered over large superficies. Thus, the mineral present in these waste dumps generally galena (PbS), oxidizes in the presence of water (Moyo et al., 2019); creating AMD which indicates a pollution factor. The main objective of this investigation is to assess the quality of surface water and soil of Bouaazza's small scale operating mines and to identify the risk generated by its discharges.

MATERIALS AND METHODS

Geographical location and geology

The Dar Bouaazza deposit (Figure 1) is located in the North eastern part of Morocco, north of Bab Bou Idir; 12 km crow flies SSW of the town of Taza. It is part of the NE of the Tazekka Variscan massif, which is a Paleozoic buttonhole located SW of Taza at the limit between the tabular Middle Atlas and the folded Middle Atlas, formed by lower middle Paleozoic terrain structured in folds and scales of the NNE-SSW axis during the eovaric phase (Hoepffner, 1987). The main unit contains the lower Ordovician epimetamorphic shales of Tazekka, occupying the center and the east of the massif. The two veins of the deposit with NE-SW direction (60 °N) are embedded in its shales. They feature galena mineralization in a quartz gang, with sphalerite, pyrite, chalcopyrite and malachite. The traces of exploitation are located on eastern slopes of Makhat's watershed.

Sampling and analyses

The samples (6 water, 2 soils, 3 embankments) were taken in March 2021 from locations representative of the study area that are highly possible to be affected by pollution. For rivers, the primary sampling site was in the surface water layer (0–5 cm from the surface) at the center of the main flow. However, the top 1–2 cm of this surface layer was avoided so as not to collect floating dust, oil, etc. (Tun, 2005). The physicochemical parameters: temperature, pH and electrical conductivity were tested immediately after sampling as they will change during storage and

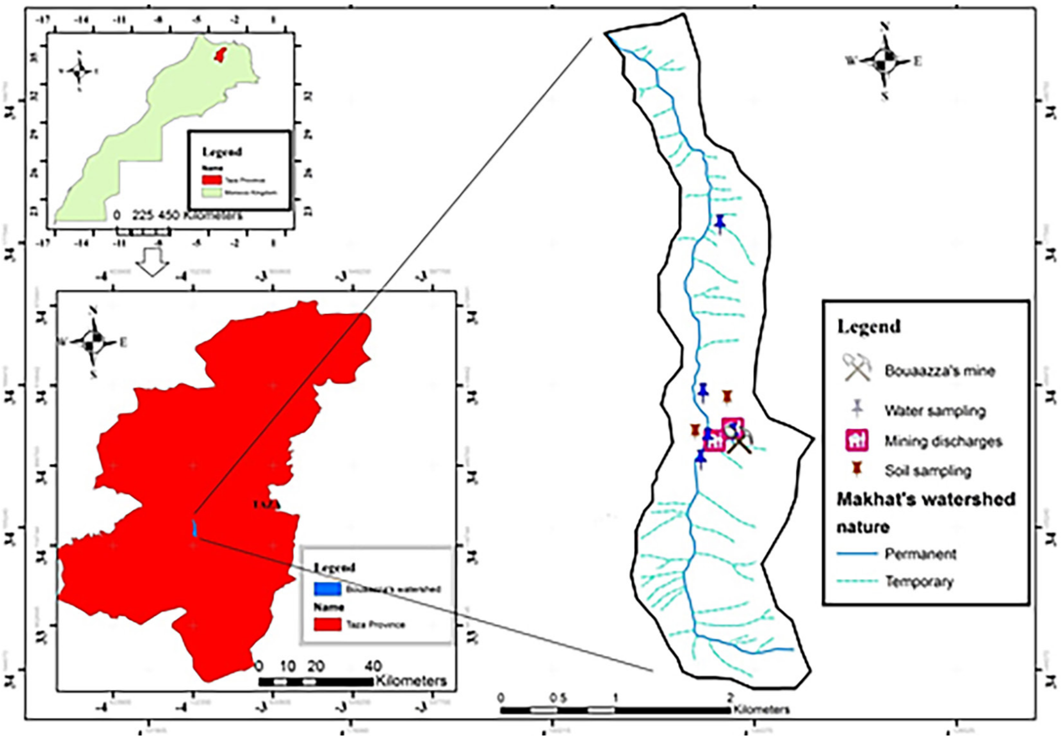

Figure 1. Study area location and sampling sites

transport (Galea, 2015), with a multi-parameter analyzer Type CONSORT – Model 835. To ensure reliable results, water samples were storage in clean polyethylene bottles. Soil samples were collected from 2 cultivated lands 6 inches deep in a 1-inch area with a trowel, and then stored in zip lock plastic bags. Samples were also taken from Bouaazza's mine embankments. All the samples were kept at a low temperature in the dark, and then transported to the Faculty of Science and Technology Fes's Laboratory.

Once in the Laboratory, soil samples were dried in ambient air, sieved to 2mm, and quartered by the cone method (Schumacher et al., 1990). Then, the following Physico-chemical analyzes were carried out: residual humidity (Norme NF ISO 11465, 1994); water pH (Norme NF ISO X31-103, 1988); electrical conductivity; determination of total organic matter by loss on ignition (Norme NF ISO 14235, 1998); determination of the contents of calcium carbonate ($CaCO_3$) using Bernard calcimeter method. For the geochemical analyzes, the determination of heavy metals of all the samples was carried out by inductively coupled plasma atomic emission spectroscopy (ICP-AES) at the Laboratory of the Innovation City of the University of Sidi Mohamed Ben Abdellah of Fez, Morocco which requires soil digestion to be analyzed, in this regard the triacid attack (Norme NF X31-147, 1996) was used to bring all the solid phases into solution without any residue remaining (total analysis). X-ray diffractometry (XRD) analyses were done in Technical Support Units for Scientific Research at the National Center for Scientific and Technical Research of Rabat, Morocco after well grounding and preserving soils and embankments samples.

RESULTS

Water physicochemical and geochemical parameters

pH

pH is a one of the most important factors: it affects the solubility and toxicity of chemicals and heavy metals in the water (PH of Water - Environmental Measurement Systems). According to the results shown in Figure 2, the pH values vary between 3 and 7. The impact generated by the mine wastes is clearly shown by the pH results of the samples S3 and S4 taken near the mine have less than 3,5. However, values greater than 6 are found in sites that are far from the embankments as S4 and S5 of the samples taken near the mine (less than 6).

Temperature

Temperature is important because of its influence on water chemistry. At high temperatures the rate of chemical reactions generally increases, as a result water can dissolve more minerals from surrounding rocks and will therefore have a higher electrical conductivity (Ammara Shoukat, Muhammad Shoukat Hussain, 2020). Figure 2a shows that the obtained results vary between 13.7 °C and 16 °C due to the period of sampling that was in winter, which influenced the temperature of the water, hence its physicochemical parameters.

Electrical conductivity

The conductivity of water refers to the ability of water to conduct an electrical current depending on its salinity. It is important because it can tell how much dissolved substances, chemicals, and minerals are present in the water. Higher amounts of these impurities will lead to a higher conductivity (Sensorex, 2019). According to the results obtained in Figure 2b, the values are between 90 and 1000 µS/cm. The samples S3 and S4 taken around the mine have a more or less high conductivity (680 and 933 µS/cm), while the rest of the samples have values less than 260 µS/cm.

Heavy metals

Heavy metals are natural constituents of our environment, generally present in small amounts in natural aquatic environments (Aderinola et al., 2009). They are characterized by their high atomic mass and high density compared to water (Saleh, 2018). Industrial activities have raised natural concentrations causing serious environmental problems. The Figure 3 shows the distribution of heavy metal concentrations in the study area. All samples have contents less than 0.01 mg/L for As, Cu, Mo, Sn. However, samples S3 and S4 from the mine area indicate high concentrations for Fe (> 7 mg/L), Pb (> 0.1 mg/L), Zn (> 10 mg/L), Ni (> 0.4 mg/L). The rest of the samples give concentrations less than the ones found in S3 and S4 but still important for Pb (> 0.05 mg/L) and Zn (0.04 mg/L) which represents a toxicity in Moroccan standards, with the absence of Fe and Ni (<0.01 mg/L).

Figure 4 defines the grid of Makhat's watershed quality of surface water based on the decree of the ME-MPUPHE (2002), using the conventional colors created in its Article 8. The results show that water quality is bad since it has high concentrations of chemical elements and low pH, a part from site 5 highlighting a good quality of water with low concentrations of heavy metals, and pH under standards.

Soils

Physicochemical parameters

Table 1 represents the physicochemical parameters of two cultivated lands near the mine:

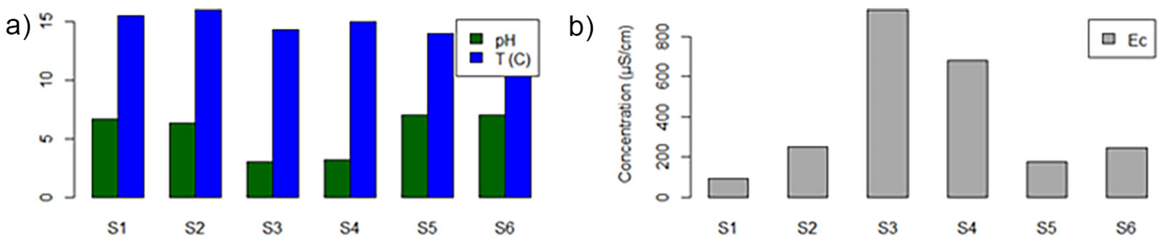

Figure 2. Surface water physicochemical parameters

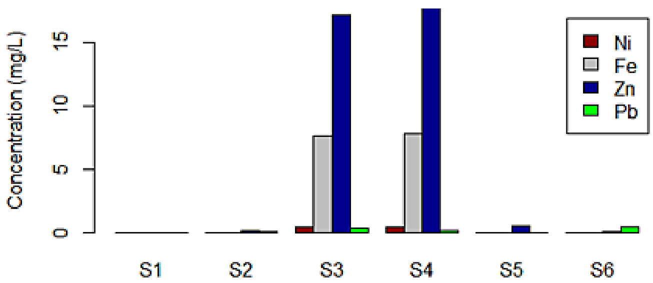

Figure 3. Surface water heavy metals results

Figure 4. Makhat's watershed surface water quality grid

Table 1. Physicochemical soil parameters

Soil samples	pH	EC (µs/cm)	$CaCO_3$ (%)	LOI (%)	Humidity (%)
S1	6	152	2	8.6	6
S2	6.5	124	5	10.1	13

- soil pH is very important due to the fact that it regulates plant nutrient availability by controlling the chemical forms of the different nutrients and also influences their chemical reactions (Oshunsanya, 2018). The results indicate that pH values are between 6 and 6.8; these values are considerate to be moderately acidic (5.6–6.0) to slightly acidic (6.1–6.5) according to the USDA National Resources Conservation Service, which shows the impact generated by the mine since most agricultural crops perform optimally around soil pH 7.0 (neutral);
- soil electrical conductivity indicates the amount of salts present in the soil; it is an indirect measurement that connects with several soils physical and chemical properties (Patel, 2015). EC values fond are 152 and 124 µS/cm; these values are between 0 and 2000 µS/cm, as a result the soils in the study area are non-saline (Shirokova, 2000);
- soil moisture is the water stored in the soil and is affected by precipitation, temperature, soil characteristics, and more; it is a measure of soil health (SSSA, 2022). The results show that the humidity values are 6 and 13%, this variation is explained by the difference in texture for each soil;
- soil organic matter is any material produced originally by living organisms (plant or animal) that is returned to the soil and goes through the decomposition process (Hüppi et al., 2015). Loss on ignition (LOI) measures the weight of a dried soil before and after burning away its organic matter. LOI measurements obtained are 8.6 and 10.1%; these values are between 3 and 15% which reflects soils are classified as mineral soils with organics (Day and Everett, 1972);
- one of the most important factors is the amount of carbonate; it influences the physical, chemical and biological properties of soils (pH, sorption-desorption, precipitation-dissolution processes etc.) (FAO, 2020). The obtained values show that the amounts of $CaCO_3$ in soils are 2 and 5%; these values show that soils are very low in limestone, which coincides with the results found in pH.

Geochemical parameters

Figure 5 indicates the distribution of heavy metals in the studied soil in the vicinity of the mine by ICP-AES, these values are well presented in Table 2 in comparison with international standards:

- the results show that the obtained values for Cu, Fe, Mo, Ni, and Sn are lower than international standards, hence the absence of toxicity regarding these elements;
- the obtained values for As show that they exceed the standards for the Netherlands (> 4.5 mg/kg) but are lower than the Austrian standards (< 50 mg/kg);
- for Pb and Zn, the obtained results exceed the standards with very high concentrations, which indicate the impact generated by mine wastes in the vicinity of the study area.

Embankments

Figure 6 shows the levels of heavy metals retained by ICP-AES in the ramblings present near the galleries of Bouaazza's mine. The results

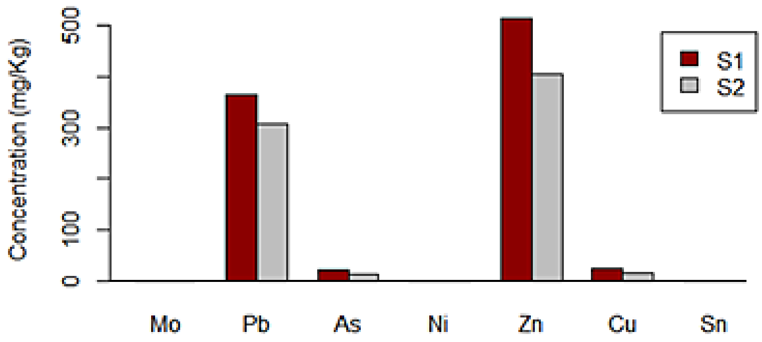

Figure 5. Soil heavy metals results

Table 2. Descriptive statistics of heavy metals in soil samples and the international maximum allowable standards

Heavy metal (mg/kg)	As	Cu	Fe	Mo	Ni	Pb	Sn	Zn
S1	19.8	25.05	8378.4	0.03	0.03	363.3	0.03	513.8
S2	13.5	15.78	5065.5	0.03	0.03	308.1	0.03	404.9
Netherlands standard	4.5	3.5	-	253	2.6	55	34	16
Austria standard	50	100	-	-	100	100	-	300
The European Union standard	-	140	-	-	75	300	-	300
France standard	-	100	30000	-	50	100	-	90

indicate a predominance of Pb in all the samples with values exceeding 30000 mg/kg. Cu shows a remarkable presence with values varying between 904 and 15000 mg/kg (Figure 6a). Fe and Zn are also present with contents of 422 to 630 mg/kg and 253 to 310 mg/kg, As and Ni show fairly low concentrations, ranging from 14.7 to 19.22 and 3.9 to 4.2 mg/kg (Figure 6b). The results highlight the absence of Mo and Sn with concentrations less than 0.3 mg/kg (standards).

The principal component analysis (Table 3) allowed defining the relationships between the concentrations of heavy metals in the embankments. The results show that lead and iron has an excellent correlation with a factor of 0.99. The pairs Fe-Zn and Pb-Zn indicate plausible correlations with factors of 0.53 and 0.58, they are causal and can be established between them in pyrite, galena, sphalerite, and in other sulphurous structures depending on the crystallization conditions. The results show also excellent correlations for As-Cu, As-Ni, Ni-Sn, Cu-Sn and Cu-Ni pairs with variance factors ranging from 0.82 to 0.99. X-ray scattering phenomenon is used in XRD technique to elucidate the crystal structure of crystalline/semi crystalline materials, with scattering of X-rays by periodic array of atoms giving rise to definite diffraction patterns that bestow a qualitative image of atomic arrangements within the crystal lattice (Rajeswari et al., 2020). Figure 7 represent the results carried out by XRD, they confirm that the embankments contains minerals such as cerusite ($PbCO_3$), chlorite-serpentine ($(Mg, Al)_6$ $(Si, Al)_4$ O_{10} $(OH)_8$), high concentrations of galena (PbS), muscovite-3 \ ITT \ RG ($((K, Na)$ $(Al, Mg, Fe)_2$ $(Si_{3.1} Al_{0.9})$ O_{10} $(OH)_2)$), and quartz (SiO_2) which is

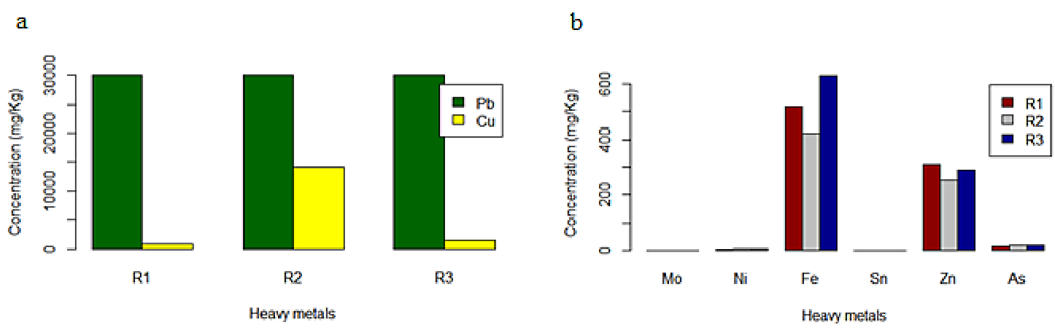

Figure 6. Embankments heavy metals results

Table 3. Major elements content correlation matrix

	As	Cu	Fe	Ni	Pb	Zn	Mo	Sn
As	1							
Cu	0.82	1						
Fe	-0.35	-0.82	1					
Ni	0.99	0.87	-0.44	1				
Pb	-0.28	-0.78	0.99	-0.39	1			
Zn	-0.96	-0.94	0.57	-0.99	0.53	1		
Mo	0.60	0.05	0.55	0.51	0.59	-0.37	1	
Sn	0.89	0.98	-0.73	0.95	-0.67	-0.99	0.18	1

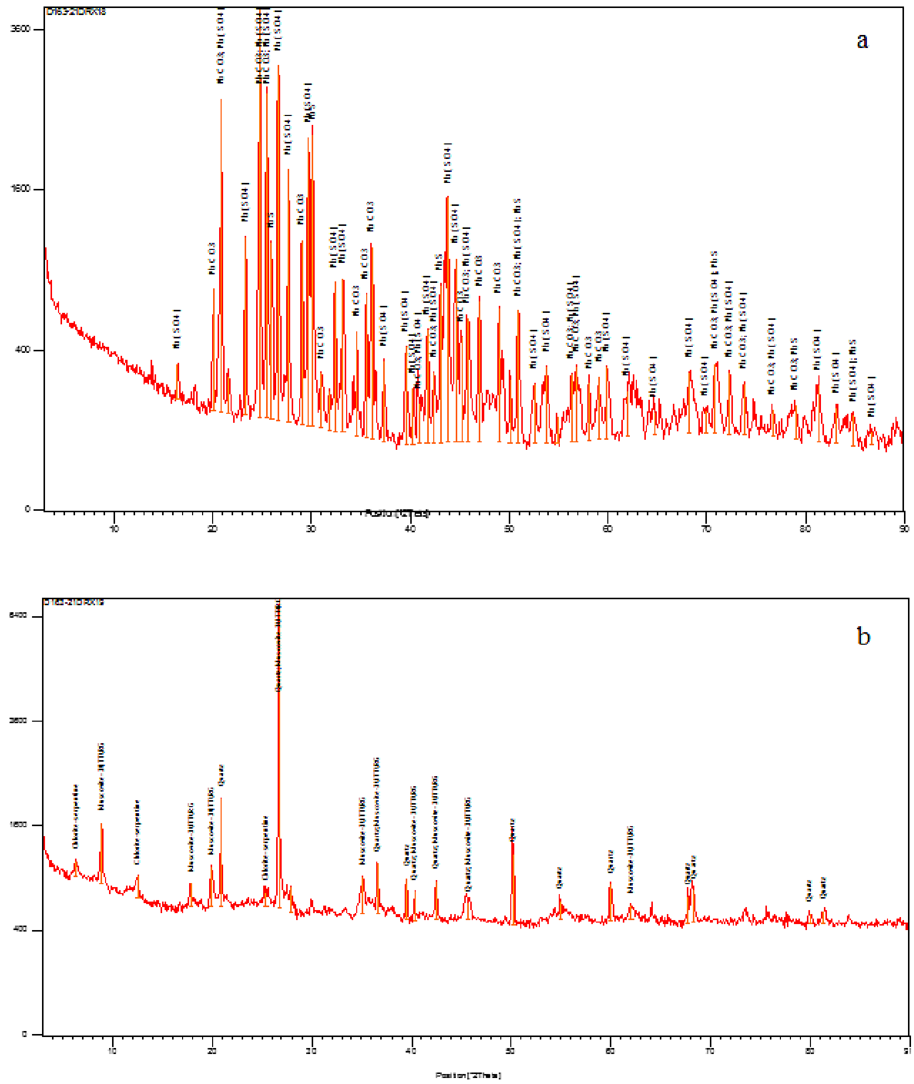

Figure 7. XRD embankments results, (a) Neoformation minerals and (b) Basement minerals Shales

available in all samples. Moreover, the comparison of the diffractograms of the samples indicates the presence of net spectra relating to the primary mineralization (quartz, galena, etc.), and others bad identified which are probably related to the subsistence of friable and poorly crystallized minerals.

DISCUSSION

The hydrodynamic processes which control the circulation of water in various environments (Liu, 2018), with the intervention of several hydrochemical phenomena, in particular of a mineralogical nature; dissolution of minerals and release of chemical elements in the waters (Nganje et al., 2017). This process plays an important role in the surface water composition. The obtained results by XRD indicated that Bouaazza's embankments contains a wide variety of minerals, with high concentrations of galena (PbS), which was confirmed by the ICP-AES values that indicated a predominance of Pb in all the samples with values exceeding 30000 mg/Kg, and the presence of other heavy metals (Cu: > 14000 mg/kg, Fe: >500 mg/kg, Zn: > 300 mg/kg). These discharges influenced the physicochemical and geochemical composition of the soil near the mine, which was given in the results of soil samples which indicated very low concentrations of $CaCO_3$ regarding the pH values that are moderately to slightly acidic, and with the results obtained for As, Pb and Zn that exceeded international standards. Similar soil heavy metal pollution is documented from Pb/Zn smelting regions in China that have resulted mean values of heavy metals for Pb, Zn, As and Cd 2123 mg/kg, 4110 mg/kg, 63.9 mg/kg and 502 mg/kg respectively, exceeding the soil background values in China (Pb: 26.0 mg/kg, Zn: 74.2 mg/kg, Cd: 0.097 mg/kg and As: 11.2 mg/kg) (Luo et al., 2023) data on heavy metal pollution in soils resulting from Pb/Zn smelting (published in the last 10 years. The investigation of the heavy metal toxicity of tailings and soils collected from the polymetallic mine (Zn, Pb, Cu and Ag) located in the south of Morocco, resulted in high soil Zn (up to 87000 mg/kg) and Pb (up to 29800 mg/kg) values (Boularbah et al., 2006); validating the link between high concentrations of heavy metals in tailings and the toxicity in the soils.

The presence of heavy metals in Bouaazza's embankments affected also the quality of Makhat's watershed, which was confirmed by the results of the physicochemical parameters which gave low pH values especially for the samples taken from the sites in the vicinity of the mine (<3). However the conductivity was very low with values reaching 933 µS/cm. These results are explained by the geological nature of the study area based on the shales of Tazekka, and the absence of limestone and dolomite which was confirmed by Bernard calcimeter method. The main heavy metals found present in the surface water are Pb and Zn. All samples exceeded Moroccan standards of Pb in the natural waters (>50 µg/L). Makhat's fresh water values are higher than the obtained for the Biała Przemsza River in Poland impacted by the local ore mining industry (Jabłońska-Czapla et al., 2016) total contents of metals and metalloids were researched in the water and bottom sediment samples from the Biała Przemsza River. The samples were collected monthly in 2014 at five sampling points along the river. The research helped to determine correlations between the parameters and components of the water environment (metals/metalloids, cations/anions, pH, Eh, conductivity, carbon (TOC, IC, TC. Also Makhat's Pb values are higher than the drainages of both former Pb–Zn mines of Aouli and Mibladen (Iavazzo et al., 2012), which contribute into the Moulouya tributary (Morocco) generating potential risk to human health.

CONCLUSIONS

The recent situation of Makhat's watershed surface water shows an unfavorable environmental impact of past mining activities generated by Bouaazza's mine. Generally, pH values are acidic reaching 3.2. The highest concentrations, especially of Pb, with values exceeding Moroccan standards (> 50 µg/L) are recorded in the water samples collected near the mine. These concentrations are caused by the presence of the embankments with wide variety of minerals confirmed by XRD results. The results of this analysis have indicated the presence of galena (PbS), cerusite ($PbCO_3$), and anglesite ($PbSO_4$), in winter these minerals dissolve and release high concentrations of Pb, resulted by the ICP-AES values the embankments with concentrations exceeding 30000 mg/Kg, and the presence of other heavy metals (Cu: >14000 mg/kg, Fe: >500 mg/kg, Zn: > 300 mg/kg). The presence of high concentrations of Pb can generate dangers to riparian that are using these waters for different purposes: domestic use, watering livestock and irrigating. The impact on agricultural soil was confirmed by the physicochemical and geochemical analyses which shown that the pH of soil samples is moderately to slightly acidic (< 6.5), caused by the low concentrations of $CaCO_3$ that maintain the neutralization of the soil. The ICP-AES results have shown values for Pb and Zn exceeding international standards (>300 mg/kg).

The presence of contamination generated by acid mine drainage from Bouaazza's mine in Makhat's watershed is validated. Waters and soils are exposed due to the lack of pollution monitoring during the historical mining operations and the semi-arid conditions. These results warn for the health risk of water, soils and food nexus caused by acid mine drainage of the abandoned mines in Morocco.

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