



Acid-base Accounting Test as a Tool for Prediction of Mine Drainage Acid Risk at a Still Functional Mine Site: Case of DraaSfar Mine in Marrakech (Morocco)

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ABSTRACT

The release of heavy metals from functional mines can result in significant environmental pollution, posing a threat to both soil and water quality. Therefore, it is crucial to characterize the chemical and physical properties of mine tailings to assess the potential risk of toxic heavy metal mobility in the environment. In this study, the chemical forms of Cd, Cu, Pb, and Zn in the tailings of Draa Sfar mine in Marrakech (Morocco) were investigated using standard sequential extraction and Atomic Absorption Spectrophotometric techniques. Results indicate that mining residues exhibit a highly acidic pH (2.9), low carbonate content (0.5%), and high electrical conductivity (7.2 mS/cm) due to significant soluble salts, particularly sulfates (3.7%). Assessing metal speciation indicated Cd's high availability in short-term mobile fractions (42%), while Cu primarily associated with soluble and exchangeable fractions (32%). Pb showed strong association with long-term mobile fractions (61%), and Zn displayed an equal distribution among mobile and lithogenic fractions. Acid-base accounting test results reveal a lower Neutralization Potential (196.4t CaCO₃/kt) compared to Acid Potential (306.3t CaCO₃/kt), indicating a negative Net Neutralization Potential and suggesting potential acid mine drainage. This result demonstrates that DraaSfar mining residues possess a lower acid-consuming capacity, primarily driven by their carbonate content, compared to their acid-generating capacity. This finding indicates that these mining residues have the potential to become sources of acid mine drainage, and underscore the importance of implementing sustainable mining practices and mitigation strategies to reduce environmental impact.

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INTRODUCTION

Mining activities have been an essential part of human civilization, providing valuable resources for its economic development (Ruiz-Sánchez et al., 2023; Hosseinpour et al., 2022). However, the extraction and processing of minerals mainly disturbs and changes the topography of land; ultimately adversely affects the hydrogeologic conditions (Karbassi et al., 2014; Nabi et al., 2007) and often result in the generation of waste materials, including mine tailings, which can have significant environmental impacts if not properly managed, and consequently can pose a threat to human and animal health (Emmanuel et al, 2022; Zhang et al., 2020; Ettlér, 2016). The degree of impact depends on many factors, in particular, mining type and the size of the operation (Nasrabadi et al., 2009). One such mining site that has raised concerns is the

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Draa Sfar Mine, known for its production of various metals, including lead, zinc, and copper.

Mine tailings are typically composed of finely crushed rock, water, and chemicals used in the mining process (Nwaila et al., 2021), and are often stored in large containment areas or tailings dams (Schlesinger et al., 2022). These tailings can pose a risk to the environment and human health due to their potential to generate acid mine drainage (AMD), which is characterized by the release of acidic water containing toxic metals into the surrounding environment (Ruiz-Sánchez et al., 2023). AMD can have detrimental effects on aquatic ecosystems, soil quality, and human health, and it is a persistent environmental problem associated with many mining operations worldwide (Ilhwan et al., 2019).

Over the past few years, a growing recognition has emerged regarding the imperative to evaluate the potential occurrence of AMD stemming from mine tailings, constituting an integral component of conscientious mining protocols (Ilhwan et al., 2019). One common approach to evaluating the potential for AMD is the use of acid-base accounting (ABA) (Sobek et al., 2000), which involves quantifying the acid and base generating potential of mine materials based on their mineralogical composition (Skousen et al., 2022; Mafra et al., 2022; Miller et al., 2015). ABA provides a valuable tool for predicting the long-term environmental impact of mine tailings and helps in the development of appropriate management strategies to mitigate the potential effects of AMD.

The Draa Sfar Mine, located in a remote region with limited infrastructure and sensitive ecosystems, has been a subject of concern regarding the management and environmental impact of its mine tailings. Several studies have reported the presence of potentially acid-generating minerals in the mine tailings, raising questions about the potential for AMD and its impact on the local environment. Therefore, a comprehensive assessment of the acid-base accounting of the mine tailings from DraaSfar Mine is necessary to evaluate the potential for AMD and understand its environmental effects.

This article aims to assess the mineralogical composition of DraaSfar mine tailings and to provide a detailed assessment of their acid-base accounting, using established methodologies to quantify the acid and base generation potential of the studied tailings materials. Based on the results, sustainable mining practices and potential strategies to mitigate the environmental impact of these mine tailings may be proposed.

MATERIAL AND METHODS

Description of the investigated site

The Draa Sfar mine which is located in 31° 42' 42.75" north latitude and 8° 8' 7.27" west longitude is the most important geologic and industrial feature in the area. It is positioned 10 km northwest of Marrakech city (Figure) and encompasses a pyrite deposit that gives rise to potential environmental concerns attributed to the discharge of tailings within the encompassing mining vicinity (Avila et al., 2012). The deposit at DraaSfar is approximated to house 10 million tons of ore characterized by a composition of 5.3 wt.% Zn, 2 wt.% Pb, and 0.3 wt.% Cu (Barkouch and Pineau, 2016). Predominantly, the ore bodies are constituted by pyrrhotite, constituting 70 to 95 vol.% of sulfides, although in zones depleted of Zn and Cu, it may account for 90 to 95 vol.% (Marcoux et al., 2008). Moreover, sphalerite (1 to 10 vol.%), galena (0.5 to 5 vol.%), and chalcopyrite (1 to 5 vol.%) are found in lesser proportions, with localized instances of deformed pyrite (constituting 2 to 3 vol.% of total sulfides), and arsenopyrite being the prevalent minor mineral (Marcoux et al., 2008).

The DraaSfar mine is positioned near the Tensift River and is situated in the vicinity of two rural communities, namely Tazakourte and Ouled Bouaicha, encompassing an approximate land area of 5790 hectares (Figure 2). Around 65% of this rural expanse is dedicated to agricultural cultivation (Elhaya et al., 2023). The climatic conditions within this region are characterized

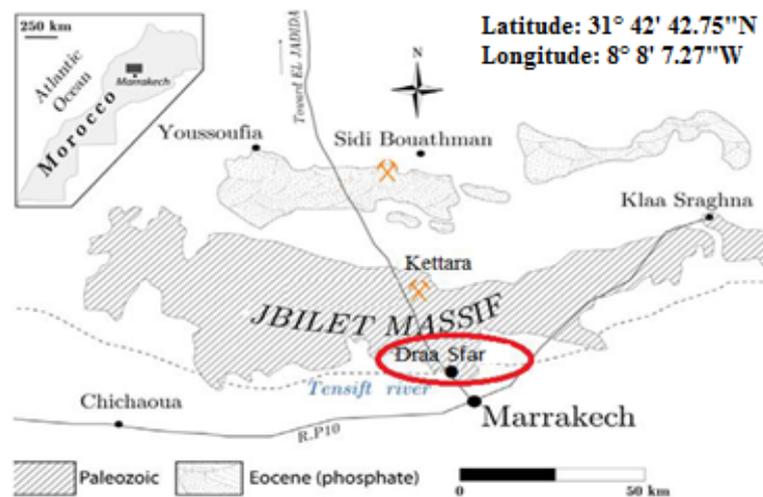


Fig. 1. Draa Sfar mine geographic situation in Marrakech Region.



Fig. 2. Satellite image (1.47 km) of the DraaSfar mining site and its mining residues.

by a Mediterranean influence, showcasing both arid and semi-arid attributes, with an average annual precipitation of 231 millimeters recorded over ten years. Temperature fluctuations are noteworthy, with mean readings ranging from 11.5°C in January to 36.8°C in July (Avila et al., 2012).

Sampling Description

To obtain a representative sample of Draa Sfar tailings, three sampling campaigns were conducted over three consecutive months. Four sampling points were carefully chosen from the tailings reservoir (dimensions: 155m/100m/13m) during each campaign (Figure 2). For the sampling process, a cylindrical stainless steel corer was employed. The collected tailings samples were then delicately homogenized and transferred into self-sealing polyethylene bags and subsequently transported to the laboratory (Abzalov, 2017). Upon arrival, these samples underwent a series of steps including air-drying, disaggregation, and sieving through meshes finer than either 2 mm or 100 µm. The <100 µm fraction of tailings was specifically chosen for sequential extraction and overall chemical analyses due to the well-established strong bonding of heavy metals with fine-grained soil components. On the other hand, the <2 mm fraction of tailings was utilized to ascertain parameters such as pH, electrical conductivity (EC), organic

matter (OM), organic carbon (OC), and carbonate content.

The soil pH was measured with the aid of pH meter while EC was done with the use of electrode meter (Badmus et al., 2014). Organic carbon content of the tailings sample was measured by following Walkey and Black method (1934). 10 g of sieved sample was taken into a 500 ml wide mouthed Erlenmeyer flask. To this, 10 ml of 1 N $K_2Cr_2O_7$ was added and swirled the flask gently to disperse the soil in the solution. 20 ml of Concentrated H_2SO_4 was then added slowly followed by vigorous shaking for 1 min. Distilled water (200ml) was added to the flask and the suspension was filtered. Few drops of ophenanthroline indicator was then added to the filtered solution and titrated against 0.5 N $FeSO_4 \cdot H_2O$. Sample without tailings was kept as a blank. The amount of organic carbon in the soil sample was calculated by using the following formula:

Organic content (%)

$$= \frac{(\text{milliequivalents of } K_2Cr_2O_7 - \text{milliequivalents of } FeSO_4) \times 0.003 \times 100}{\text{Masse of tailings sample}}$$

% Organic Matter = % Organic Carbon 1.724.

Before analysis, the tailings samples were subjected to a process of homogenization. Subsequently, these homogenized samples were stored in hermetically sealed polyethylene bags at a temperature of 4°C until the commencement of the analysis process (Barkouch et al, 2015; Wufem et al., 2013).

For the determination of the overall concentration of heavy metals, the tailing samples underwent a process of acid digestion followed by analysis through atomic absorption spectroscopy. This acid digestion process encompassed the weighing of 1g of the dried sample and the addition of 3 ml of 70% HNO_3 , 6 ml of 37% HCl , and 3 ml of 48% HF (Barkouch et al, 2007). The sample was then subjected to digestion within a sand bath to facilitate the completion of the process. After digestion, the resulting sample solution underwent air-cooling and was subsequently diluted with deionized water in preparation for analysis through atomic absorption spectroscopy.

In order to analyze the composition of heavy metal species within the tailings samples, an optimized sequential extraction procedure was applied (Wufem et al., 2013). This sequential extraction process adopted a five-step methodology, with an additional sixth step introduced (Barkouch et al., 2015; Fuentes et al., 2004). The supplementary step involved the dissolution of the ultimate residue employing the same digestion procedure utilized for determining the total metal content (Avila et al., 2012; Gleyzes et al., 2002).

The soil sample underwent a process of fractionation to discern different forms of metal binding through a series of extraction techniques:

Water Soluble Fraction (F1): The initial step involved the extraction of the soil sample with 15 ml of deionized water for 2 hours to obtain the water-soluble fraction (F1).

Exchangeable Fraction (F2): Subsequently, the residue from the water-soluble fraction was subjected to extraction using 8 ml of 1M $MgCl_2$ (pH 7.0) for 1 hour, resulting in the exchangeable fraction (F2).

Carbonate-Bound Fraction (F3): Further extraction of the residue from the exchangeable fraction was carried out using 8 ml of 1M ammonium acetate (pH 5.0 with acetic acid) for 5 hours, leading to the carbonate-bound fraction (F3).

Fe-Mn Oxides-Bound Fraction (F4): The subsequent step entailed extraction of the residue from the carbonate-bound fraction utilizing 0.04 M $NH_2OH \cdot HCl$ in 25% (v/v) acetic acid at a temperature of 96°C for 6 hours, intermittently agitated, yielding the Fe-Mn oxides-bound fraction (F4).

Organic-Bound Fraction (F5): The residue from the Fe-Mn oxides-bound fraction

underwent extraction by employing 3 ml of 0.02 M HNO₃ and 5 ml of 30% H₂O₂ (pH 2 with HNO₃) at 85°C for 6 hours, intermittently agitated. After cooling, the addition of 5 ml of 3.2 M NH₄ acetate in 20% (v/v) HNO₃, followed by continuous agitation for 30 minutes, resulted in the organic-bound fraction (F5).

Residual Fraction (F6): Finally, the residue from the organic-bound fraction was subjected to digestion in a conical flask using 10 ml of 7M HNO₃ on a hot plate for 6 hours. Following evaporation, 1 ml of 2 M HNO₃ was introduced, and the residue was subsequently diluted to 10 ml using deionized water. A final wash of the residue with 10 ml of deionized water was conducted.

Each extraction step was followed by centrifugation at 4000 rpm for 30 minutes to facilitate separation. The resulting supernatants underwent filtration and subsequent analysis for heavy metals. The entirety of the experimentation was conducted utilizing reagent-grade chemicals and pure deionized water, with all plastic materials subjected to immersion in a 10% HNO₃ solution.

Neutralization Potential (NP) measurement:

To assess the Neutralization Potential (NP), approximately 0.5g of ground sample is taken. Subsequently, 25 ml of 0.1N HCl is added, and the mixture is vigorously stirred for 6 hours to ensure complete neutralization of any carbonate phases. The mixture is then titrated using 0.1N NaOH, with phenolphthalein serving as the color indicator. This titration enables the determination of the volume of excess HCl. The discrepancy between the initially added volume of HCl and the volume remaining in excess within the sample is employed to determine the volume of HCl that has counteracted the carbonates. Utilizing this volume and the chemical reaction outlined below, we calculate the quantity of carbonates present in the sample.



RESULTS AND DISCUSSION

The results of measurements of tailing grain size, pH, electrical conductivity (EC), organic matter (OM), organic carbon (OC), and calcium carbonate (CaCO₃) content for the former DraaSfar tailing mine are displayed in Table 1.

Based on the obtained results, it appears that these mining residues exhibit a highly acidic pH (pH = 2.9) due to the low value of total carbonates content, around 0.5%. These results showed also a high electrical conductivity of tailings (7.2 ms/cm). This is attributed to the substantial presence of total soluble salts, especially with sulfates accounting for 3.7%. The organic matter content in these wastes is 6.2%, while the organic carbon (OC) content is 3.6%. The low OC values observed in various soils are associated with a reduced rate of degradation by microbial-mediated processes due to heavy metal contamination. This phenomenon may also be linked to the limited absorbability of organic compounds on negatively charged quartz surfaces (Xilong et al., 2018), which are prevalent in the solid tailings of the DraaSfar mine. Additionally, the constant leaching action of rainfall could contribute to the low OC percentage in these soils.

Electrical conductivity (EC) measurements revealed consistently high values, accounting for 7.2mS/cm. It was observed that tailings with the most acidic pH values generally exhibited higher EC values. This correlation between low pH and high EC values can be explained by the presence of sulfur ions, which contribute to increased EC levels (Zhuoyi et al., 2023).

Regarding carbonate content, it can be inferred that these residues with a low pH of 2.9 have low levels of calcium carbonate. In this context, acidic residue samples, associated with moderate organic matter (OM) content and the presence of low values of carbonates contribute to the release of heavy metals within these residue samples.

Nevertheless, the total concentrations of metallic elements in the examined residues

Table 1. Textural and physicochemical characteristics of the Drâa Sfar mine tailings.

| Texture | | |
|-----------------------------------|-------------------------------|----------------|
| Coarse Fraction (>2mm) | Gravel and stones % | 21.2 ± 2.1 |
| | Clay% | 1.2 ± 0.3 |
| Fin fraction (<2mm) | Fin silt % | 4.5 ± 1.2 |
| | Coarse silt % | 17.4 ± 1.2 |
| | Fin sand% | 47.4 ± 3.7 |
| | Coarse sand% | 26.2 ± 2.1 |
| Physicochemical parameters | | |
| | pH | 2.9 ± 0.2 |
| | EC (ms/cm) | 7.2 ± 0.2 |
| | Total soluble salts(meq/100g) | 36.4 ± 2.1 |
| | Cl-% | 0.7 ± 0.1 |
| | SO ₄ ²⁻ | 3.7 ± 0.3 |
| | OC% | 3.6 ± 0.8 |
| | OM% | 6.2 ± 1.2 |
| | CaCO ₃ (mg/g) | 177.1 ± 15.2 |
| | CEC (meq/100g) | 7.8 ± 1.2 |
| Metallic trace elements | | |
| | Cd (mg/kg) | 143.2 ± 12.1 |
| | Cu (mg/kg) | 929.1 ± 88.5 |
| | Pb(mg/kg) | 3381.3 ± 507.1 |
| | Zn (mg/kg) | 2848.1 ± 460.3 |

display do not provide sufficient information regarding their potential hazardous impact on the environment, as their availability, mobility and ecotoxicity are strongly dependent on their specific chemical forms or binding. Metallic trace elements can be distributed across various components of mine tailings and can form different associations with them (Aikpokpodion et al., 2013; Barkouch et al, 2015). Assessing the speciation of Metallic trace elements is valuable for evaluating potential toxic effects. Figure 2 displays the percentages of each of the studied metallic elements in the various fractions F1 to F6.

Results showed that cadmium appears to be highly available, with over 42% of the total metal content being associated with the short-term mobile fraction (F1 and F2). The long-term mobile fraction (F3, F4, and F5) hosts more than 35% of the total metal. Finally, the non-mobile fraction (F6) sequesters over 18%.

The results of the copper speciation study indicate that it is primarily associated with the soluble (F1) and exchangeable (F2) fractions, accounting for over 32% of the total copper content in these tailings. Copper is also present in a high amount (31%) in the residual fraction (F6). The (F3), (F4) and (F5) fractions are represented by low percentages in these residues, trapping 7%, 11%, and 13%, respectively.

The distribution of lead is characterized by a strong association of the metal with the long-term mobile fractions (F3, F4, and F5), constituting over 61% of the total lead content in the mining residues. The short-term mobile fraction, comprising the soluble (F1) and exchangeable (F2) fractions, accounts for approximately 23%. The residual fraction (F6) sequesters around 18% of the total metal.

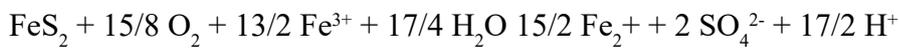
The zinc speciation demonstrates an equal distribution of the metal in the three fractions. Consequently, it is represented by 31%, 34%, and 35% of the total zinc content in the short-term

mobile, long-term mobile, and lithogenic fractions (F6), respectively.

In the studied mining residues, it appears that trace elements can be available in the waste. This availability is linked to the physicochemical characteristics of these tailings. Indeed, in these residues, the availability of trace metals, which involves the transfer of metals from the most stable phases to the soluble and exchangeable phases (Dang et al., 2002), is likely associated with their acidic pH (2.9). This parameter is the most determining factor affecting the solubility of metallic trace elements (MTEs) (Yapi et al., 2021; El Gharmali, 2005). Acidification promotes metal solubilization through proton exchange, dissolution of soluble salts, or even destruction of retention phases. Positive correlations have been observed between increasing pH and the retention of metals by mining residues (Nuttall and Younger, 2000). These authors demonstrated that maintaining the pH above 6 through the addition of limestone is necessary. In fact, the carbonate fraction constitutes one of the solid phases controlling the process of metal retention through adsorption (Madrid and Diaz-Barientos, 1992), thereby reducing metals solubility and their diffusion in water. Thus, lime is used to neutralize and treat mining residues to minimize the solubility of mineral micropollutants (Banks et al., 1997; Iribar et al., 2000).

Due to their constituents, the waste dumps from the surveyed mining site can generate Acid Mine Drainage (AMD). These effluents, generated during wet periods, could potentially serve as a source of metal pollution for the surrounding surface water resources (Moreno-González et al, 2022; Muniruzzaman et al, 2020).

The mechanism of acid production (acidogenesis) has been extensively studied (Gray, 1998; Caboi et al., 1999; Stromberg and Banwart, 1999; Nordstrom et al., 2000; Williams and Smith, 2000). In addition to the production of high concentrations of sulfates and metals, this mechanism leads to the precipitation of ferric iron with the formation of hydroxides. This process can be illustrated by the oxidation reactions of a common sulfide mineral, pyrite (FeS₂). The general outline of the mechanism is summarized by the following reaction:



Several control techniques for the AMD process have been developed. Some of them, used for the production's prediction of these effluents, are generally based on procedures involving

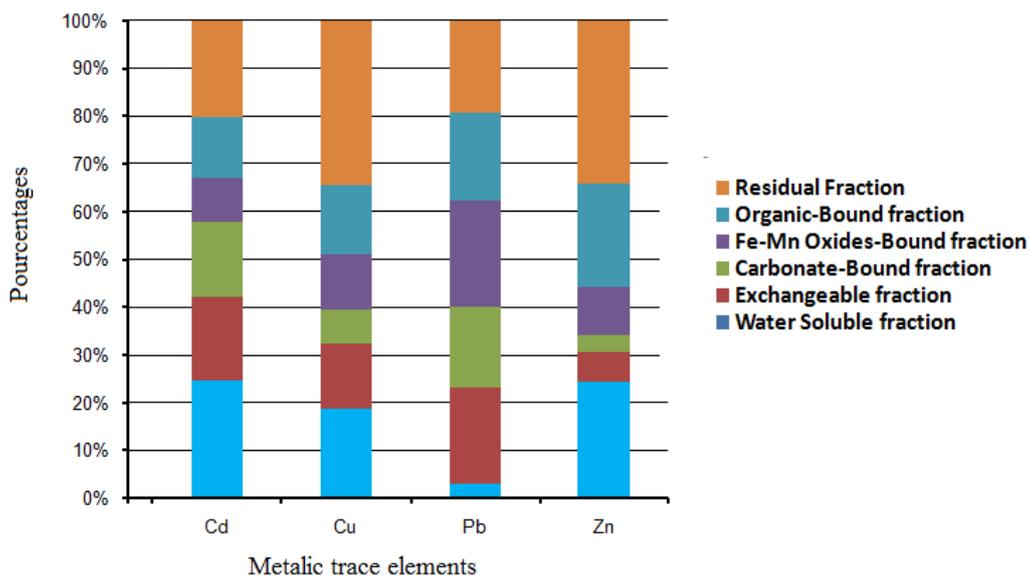


Fig. 3. Speciation of four metallic trace elements in the Drâa Sfar mine tailings.

Table 3. Predicting parameters of AMD production by Draa Sfar mine residues

| Parameters | Draa Sfar mine |
|------------------------------|----------------|
| % S (sulfates) | 2,7 ± 0,4 |
| % S (total) | 12,5 ± 3,2 |
| PN (t CaCO ₃ /Kt) | 196,4 ± 32,1 |
| PA (t CaCO ₃ /Kt) | 306,3 ± 72,5 |
| PNN | - 109,9 |
| PN/PA | 0,64 |

the measurement of acid-forming compounds (sulfides) and acid-consuming substances in the substrate (rocks, residues, etc.) (Thuong et al, 2023; Masindi et al, 2022; Sheoran et al, 2010). Among these methods, an acid-base titration approach known as Acid-Base Accounting (ABA) was employed in this study (Skousen, 2017, Bouzahzah et al, 2015).

This method enables the calculation of the Net Neutralization Potential (NNP), derived as the variance between the Neutralization Potential (NP) and the Acid Potential (AP). These parameters are ascertained through the analysis of neutralizing minerals and sulfur in their diverse forms, respectively. The units for these parameters are expressed in tons of CaCO₃ per 1000 tons (1KT) of dry substrate (CaCO₃/Ton of substrate). NNP assumes negative values signifying the potential for acid drainage, as does a NP/AP ratio below 1 (Huaman et al, 2023; Nguyen et al, 2023; Plaza et al, 2017; Moo et al, 2015; Hageman et al, 2015).

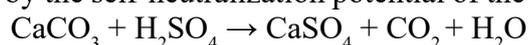
Acid Potential (AP) Measurement

The acid potential (AP) of studied samples corresponds to the consumption of sulfur in the form of available sulfide while regenerating sulfuric acid. The quantity of liberated acid can be expressed as follows (Belzile et al., 2000):

$$Q(\text{H}_2\text{SO}_4)\% = \% \text{ S (Sulfide)} \times [\text{PM}(\text{H}_2\text{SO}_4)/\text{PMm}(\text{S})]$$

$$\% \text{ S (Sulfide)} = \% \text{ S (total)} - \% \text{ S (Sulfate)}; \% \text{ S (Sulfate)} = \% \text{ (Sulfate)}/3.$$

This acidity is neutralized by the self-neutralization potential of the sample, according to the chemical reaction:



This is a one-to-one molar reaction. By converting the quantity of acid into carbonate equivalents, the formula becomes:

$$\text{AP} = \% \text{ S (sulfide)} \times [\text{PM}(\text{CaCO}_3)/\text{PM}(\text{S})].$$

Since AP is expressed in tons of CaCO₃ per 1000 tons of the sample, the formula becomes:

$$\text{AP} = \% \text{ S (sulfide)} \times 31.25 \text{ tons CaCO}_3 \text{ per } 1,000 \text{ tons of substrate.}$$

The results obtained in table 3 show that the Neutralization Potential (NP) of the residues (196.4t CaCO₃/kt) is lower than the Acid Potential (AP), which is approximately 306.3t CaCO₃/kt. This outcome leads to the conclusion that the acid-consuming capacity of these mine wastes, primarily based on their carbonate content (chemical neutralization components), is low compared to their acid-generating capacity. Consequently, the Net Neutralization Potential (NNP) of these mine residues is negative, and their NP/AP ratio is less than 1. Therefore, they are potential sources of acid mine drainage.

CONCLUSION

The Marrakech region is subject to metal pollution which mainly comes from water and open-air deposited residues from the DraaSfar mine located at 13 km northwest of Marrakech. The study conducted was able to demonstrate that mineral micropollutants found in the mine residues of DrâaSfar mine are abundant in the soluble and exchangeable fractions. The

application of the ABA method highlighted the acid-generating potential of these mine residues, capable of causing acid drainage. This, through a pH decrease, promotes the solubilization and release of Trace Metal Elements into the surrounding ecosystems.

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The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and / or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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