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Mineral and Heavy Metal Contents in Soils Downstream of the Ankavia ANTALAHA Watershed

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ABSTRACT : The study of mineral elements contained in soils downstream of the Ankavia watershed remains a relevant research topic related to groundwater consumption in Madagascar, concerning both its quality and quantity. Very few people have benefited from drinking water, which is necessary to assess its potential impact on public health. This study aims to determine and compare the mineral element contents in soils. The analysis was carried out at the Nuclear Physics and Environmental Physics Laboratory of the University of Antananarivo (Madagascar) using energy dispersive X-ray fluorescence spectrometry. Several categories of mineral elements were identified in the samples: essential nutrients (potassium, calcium, iron, zinc, copper), as well as potentially undesirable elements such as titanium, vanadium, nickel, lead, and bromine. Concentrations vary significantly depending on the sampling point. This study highlights the presence of trace heavy metals in some soils, highlighting the importance of vigilance on the part of water consumers and regulatory authorities.

KEYWORDS: Soil, ED-XRF, mineral elements, well water, SAVA Region.

I. INTRODUCTION

In Madagascar, 90 % of the population lives in rural areas **[2]**. Most of them are farmers, we can say that the soils are mainly the source of life for the inhabitants **[3]**. The soil is the loose surface layer of the Earth's surface [9]. Its thickness can range from a few tens of centimeters to several meters **[6]**. It results from the degradation of organic matter of plant origin coming from the surface **[8]** and mineral matter coming from the alteration of the parent rock which supports it **[7]**. A large number of Fokontany in the basin use well water and especially surface water for their daily activities. However, concerns about the quality of the basin's soils have led to a demand for available data on the distribution of mineral elements. This prompted us to take the initiative to conduct this research.

II. METHODOLOGY

1. Study Area

The study area is concentrated in the Ankavia watershed in the Antalaha District. The Ankavia River is the main water supply for the city of Antalaha, which had a population of 282,158 in 2020 (INSTAT 2020). Samples were collected in three areas: the commune of Marifinaritra, the commune of Antombana, and the commune of Antalaha.

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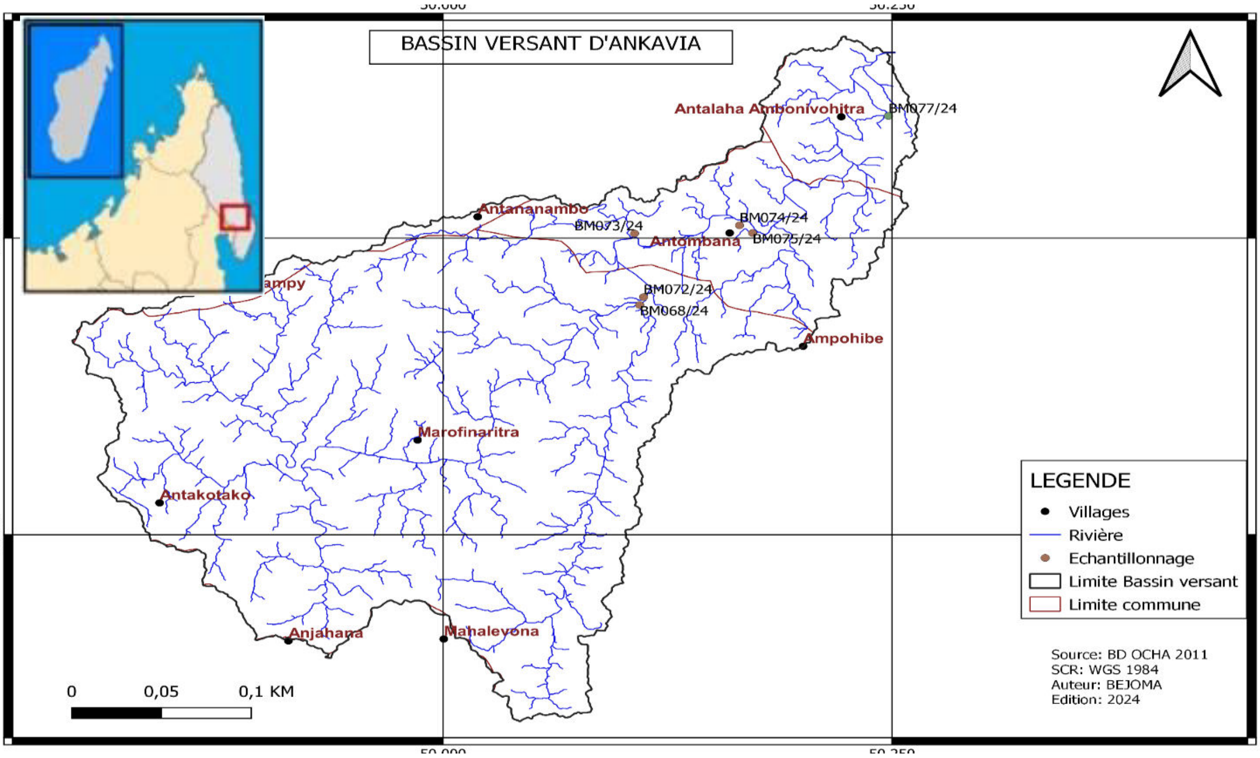

Fig. 1: Potential sampling Map

2. Study Framework

This study was conducted at the Nuclear and Environmental Physics Laboratory (LPNPE) at the University of Antananarivo, Madagascar. Elemental analyses were performed using energy-dispersive X-ray fluorescence spectrometry (ED-XRF), a technique that allows for the simultaneous identification and quantification of chemical elements present in samples. The figure below illustrates the overview of this spectrometer.

Fig 2: Amptek EXP-2 device used for sample analysis

3. Sample Preparation

Several samples were collected in the field for laboratory analysis. Sample preparation followed a standardized protocol aimed at obtaining homogeneous and representative specimens for ED-XRF analysis: drying, grinding, sieving, and conditioning.

Drying: The samples were calcined at 450°C for 30 minutes in a muffle furnace (Nabertherm, model L9/11) to remove organic matter, thus reducing matrix effects that could interfere with spectrometric analysis.

Grinding: The calcined samples were finely ground using an agate mortar and pestle to avoid metal contamination. Grinding continued until a homogeneous powder with a particle size less than 50 µm was obtained.

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Sieving: The resulting powder was sieved through a 50 µm mesh nylon sieve to ensure particle size uniformity of the final sample.

Packaging: A mass of 4.00 ± 0.01 g of each sieved sample was taken using an analytical balance (Sartorius, accuracy ± 0.1 mg) and placed in a high-density polyethylene sample holder fitted with a 4 µm-thick Ultralene support film.

III. EXPERIMENTAL RESULTS

Five (05) soil samples (BM 068/24, BM 072/24, BM 073/24, BM 073/24, and BM 077/24) were analyzed and taken in different locations, near the installed piezometers. The results of these analyses by the energy dispersive X-ray fluorescence method show that they contain several more important mineral elements such as: Lead (Pb), Cadmium (Cd), Iron (Fe), Copper (Cu), Zinc (Zn), Aluminum (Al), Manganese (Mn), Nickel (Ni), Calcium (Ca), Potassium (K), phosphorus (P) and Magnesium (Mg). These results show that the concentrations of mineral elements contained in the soil vary from one sample to another. The tables below represent the respective average contents of the mineral elements in the samples:

Table 1: Average content (n=5) in the soil at the bottom of the water from the wells coming from ANTALAHA

Mineral elements	BM 068/ 24	BM 072/ 24	BM 073/ 24	BM 074/ 24	BM 077/ 24
Mg [%]	0,065 ± 0,001	0,069 ± 0,001	0,644 ± 0,003	0,640 ± 0,003	0,415 ± 0,003
K [%]	0,49 ± 0,001	0,539 ± 0,001	< LD	< LD	3,043 ± 0,005
P [%]	0,059 ± 0,0002	0,062 ± 0,0001	0,078 ± 0,001	0,077 ± 0,001	0,163 ± 0,001
Ca [%]	0,056 ± 0,001	0,061 ± 0,001	0,241 ± 0,001	0,243 ± 0,001	0,87 ± 0,001
Al [%]	0,342 ± 0,001	0,359 ± 0,001	0,234 ± 0,001	0,232 ± 0,001	1,088 ± 0,01
Fe [%]	1,204 ± 0,001	1,308 ± 0,002	3,867 ± 0,02	3,866 ± 0,02	0,097 ± 0,001
Mn [mg/kg]	273,9 ± 0,8	303,4 ± 0,8	88,7 ± 1,3	81,8 ± 1,3	37,7 ± 0,7
Cu [mg/kg]	6,5 ± 0,2	7,2 ± 0,2	51 ± 1,0	52,5 ± 1,0	32 ± 0,6
Zn [mg/kg]	7,3 ± 0,1	8,1 ± 0,1	9,6 ± 0,5	9 ± 0,5	14,7 ± 0,4
Ni [mg/kg]	2,5 ± 0,2	2,6 ± 0,2	< LD	< LD	4,2 ± 0,4
Cd [mg/kg]	7,1 ± 0,3	7,9 ± 0,3	< LD	< LD	< LD
Pb [mg/kg]	1,1 ± 0,2	1,1 ± 0,2	< LD	< LD	24,4 ± 0,4
Si [%]	1,079 ± 0,001	1,144 ± 0,001	0,794 ± 0,001	0,791 ± 0,001	2,446 ± 0,02
S [%]	0,079 ± 0,0001	0,085 ± 0,0001	0,017 ± 0,0001	0,017 ± 0,0001	< LD
Ti [%]	0,025 ± 0,001	0,028 ± 0,001	0,031 ± 0,001	0,032 ± 0,002	0,784 ± 0,001
V [%]	< LD	< LD	< LD	< LD	0,011 ± 0,001
Cr [mg/kg]	10,8 ± 0,2	12,6 ± 0,2	< LD	2,3 ± 0,1	97,5 ± 1,1
Co [mg/kg]	< LD	< LD)	< LD	< LD	< LD
Se [mg/kg]	< LD	< LD	< LD	< LD	< LD
Rb [mg/kg]	7,7 ± 0,1	8,5 ± 0,1	3,9 ± 0,1	3,5 ± 0,1	154,7 ± 0,4
Sr [mg/kg]	51,3 ± 0,1	55,9 ± 0,1	220,3 ± 0,5	220,7 ± 0,5	268,7 ± 0,4
Y [mg/kg]	1,3 ± 0,3	1,4 ± 0,3	2,1 ± 0,2	1,7 ± 0,2	68,5 ± 0,3
Zr [mg/kg]	58,1 ± 0,7	58,8 ± 0,7	18 ± 1,6	16,5 ± 1,8	379,1 ± 2,9

< LD: below the detection limit

Analysis of the soil mineral profiles (BM 068/24, BM 072/24, BM 073/24, BM 073/24, and BM 077/24) reveals considerable variability in mineral element concentrations between samples. It reveals a diverse mineral composition characterized by significant levels of microelements and trace elements. Among the microelements, iron (Fe) dominates with a concentration of 2.446 ± 0.02% (BM 077/24), followed by silicon (Si) at 3.867 ± 0.02% (BM 073/24) and aluminum (Al) at 1.088 ± 0.01% (BM 077/24). These three elements therefore constitute the main matrix of the sediment, suggesting a high presence of aluminosilicates and iron-bearing minerals.

Alkaline and alkaline earth elements are present in moderate concentrations, with calcium (Ca) at 0.87 ± 0.001% (BM 077/24), zirconium (Zr) at 379.1 ± 2.9 mg/kg (BM 077/24), sulfur (S) at 0.085 ± 0.0001% (BM 072/24), iron (Fe) at 3.867 ± 0.02% (BM 073/24), magnesium (Mg) at 0.644 ± 0.003% (BM 073/24) and potassium (K) at 3.043 ± 0.005%

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(BM 077/24). These values indicate the probable influence of marine and terrigenous inputs in the formation of these soils.

Concerning heavy metals, several elements present notable concentrations: manganese (Mn) at 303.4 ± 0.8 mg/kg (BM 072/24), chromium (Cr) at 97.5 ± 1.1 mg/kg (BM 077/24) and zinc (Zn) at 14.7 ± 0.4 mg/kg (BM 077/24). Significant concentrations are also observed for nickel (Ni) at 4.2 ± 0.4 mg/kg (BM 077/24), copper (Cu) at 52.5 ± 1.0 mg/kg (BM 074/24) and lead (Pb) at 24.4 ± 0.4 mg/kg (BM 077/24). These high heavy metal levels could reflect anthropogenic contamination of the site, potentially linked to human or industrial activities. Trace element levels (Pb and Cd) vary from 1.1 ± 0.2 mg/kg (BM 072/24) to the detection limit, with an average of 8.32 mg/kg and an overall CV of 31.9%. The max/min ratio is 1.86.

Other elements such as zirconium (Zr) at 379.1 ± 2.9 mg/kg (BM 077/24), yttrium (Y) at 68.5 ± 0.3 mg/kg (BM 077/24), rubidium (Rb) at 154.7 ± 0.4 mg/kg (BM 077/24) and strontium (Sr) at 268.7 ± 0.4 mg/kg (BM 077/24) are also present in appreciable concentrations. It should be noted that Cobalt (Co), Vanadium (BM 068/24, BM 072/24, BM 073/24, BM 074/24) and chromium (Cr) are below the detection limit (DL) of the analytical method used.
This complex mineralogical composition, characterized by high levels of iron, chromium, manganese, zinc and nickel, suggests that the soils of the ANTALAHA district were probably influenced by natural geochemical processes superimposed on significant anthropogenic inputs.

IV. DISCUSSIONS

The heavy metal concentrations of chromium, nickel, copper, zinc, and lead were compared to the TEC (Threshold Effect Concentration) and PEC (Probable Effect Concentration) values to analyze the state of the sediment sampled for each study site **[1]**. The TEC corresponds to a concentration below which no effects are expected. The PEC corresponds to a concentration from which a high probability of effects is expected **[4]**.

Table 2: Threshold Effect Concentration (TEC) and Probable Effect Concentration (PEC) for some metals

Metals	Threshold Effect Concentration (TEC)	Probable Effect Concentration (PEC)
Cr (mg/kg)	43,40	111,00
Ni (mg/kg)	22,70	48,60
Cu (mg/kg)	31,60	149,00
Zn (mg/kg)	121,00	459,00
Pb (mg/kg)	35,80	128,00

Chromium
Chromium is present at five sites such as BM 068/24, BM 072/24, BM 073/24, BM 074/24, and BM 077/24, with levels of 10.8 ± 0.2 mg/kg, 12.6 ± 0.2 mg/kg, below the detection limit, 2.3 ± 0.1 mg/kg, and 97.5 ± 1.1 mg/kg, which do not exceed the PEC values. At BM 077/24, its concentration is twice the TEC value. These sites are likely at risk. Its health effects are carcinogenic **[5]**.

Nickel
Nickel is required at five sites, such as BM 068/24, BM 072/24, and BM 077/24, whose levels are 2.5 ± 0.2 mg/kg, 2.6 ± 0.2 mg/kg, and 4.2 ± 0.4 mg/kg, respectively, which do not significantly exceed the TEC and PEC values. Sites BM 073/24 and BM 074/24 have values below the detection limit. The nickel concentration does not exceed the PEC and TEC reference values. This means that these sites are not contaminated by nickel.

Copper
Copper is prevalent at sites such as BM 073/24, BM 074/24, and BM 077/24, with concentrations of 51 ± 1.0 mg/kg, 52.5 ± 1.0 mg/kg, and 32 ± 0.6 mg/kg, respectively, exceeding the TEC value. These three sites are at risk. At sites BM 068/24, BM 072/24, BM 077/24, BM 073/24, and BM 074/24, their concentrations are well below the PEC value. The presence of this element in the soil at the bottom of the wells of sites BM 073/24, BM 074/24 and BM 077/24 perhaps comes from discharges of metallurgical waste and also these sites are a southern outlet of the urban commune of ANTALAHA.

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Zinc

Zinc is required at sites BM 068/24, BM 072/24, BM 077/24, BM 073/24, and BM 074/24, with levels of 7.3 ± 0.1 mg/kg, 8.1 ± 0.1 mg/kg, 9.6 ± 0.5 mg/kg, 9 ± 0.5 mg/kg, and 14.7 ± 0.4 mg/kg, respectively, which do not exceed the TEC and PEC values. These sites do not pose environmental and ecological risks or hazards because their soils and well waters are not contaminated.

Lead

Lead is required at the four sites BM 068/24, BM 072/24, and BM 077/24, whose levels are 1.1 ± 0.2 mg/kg, 1.1 ± 0.2 mg/kg, and 24.4 ± 0.4 mg/kg, respectively. Sites BM 073/24 and BM 074/24 have values below the detection limit. The lead concentration does not exceed the PEC and TEC reference values. This means that the soil at these sites is not contaminated by the heavy metal lead. The sampling sites BM 068/24, BM 072/24, BM 077/24, BM 073/24 and BM 074/24 do not present any risk of environmental and ecological hazards, and cannot enter the human food chain through the consumption of groundwater that would have absorbed this toxic element.

V. CONCLUSION

The five soil samples collected from study sites BM 068/24, BM 072/24, BM 077/24, BM 073/24, and BM 074/24 were analyzed using the Energy Dispersive X-Ray Fluorescence (ED-XRF) method using XRS-FP2 software. The presence of trace elements such as magnesium, aluminum, silicon, phosphorus, sulfur, potassium, calcium, titanium, vanadium, manganese, iron, rubidium, strontium, yttrium, zirconium, ... which are essential but their excess in the body also causes diseases, and we also observe heavy metals such as chromium, nickel, copper, zinc, cadmium and lead. The most abundant elements in the soil samples are: aluminum, silicon, calcium and iron. Contaminants in soils from sites BM 068/24, BM 072/24, BM 073/24, BM 074/24, and BM 077/24 whose levels exceed the PEC reference values are chromium, nickel, copper, zinc and lead. However, the concentration of cadmium is below the detection limit. However, it should be noted that its effect on health is hypertension, liver damage and can lead to early death even at trace levels.

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