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Physicochemical and mineralogical characterization of Brazzaville landfill soils: Assessment of mercury (Hg), nickel (Ni) and zinc (Zn) ion contamination

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Abstract

The aim of this study was to characterize in depth the soils from six urban landfill sites in Brazzaville (Diata, Glacière, Kinsoundi, Moukondo, Tsiémé and Mikalou) by assessing their physicochemical properties, mineralogy and metal ion contamination (Hg, Ni, Zn). Using samples taken according to a rigorous methodology, we analysed water content, pH, organic matter, mineralogy via X-ray diffraction (XRD), and metal ion concentration using Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES). The results highlight significant variability in parameters between sites, with concentrations of Hg^{2+}, Ni^{2+} and Zn^{2+} systematically exceeding regulatory thresholds, underlining the potential environmental and health impact. Statistical analyses reveal strong correlations between certain physicochemical parameters and the presence of heavy metals, indicating a potential relationship between soil composition and metal contamination. Furthermore, the distribution of parameters and heavy metals is predominantly asymmetrical, except for organic matter in the Tsiémé landfill, where a symmetrical distribution is observed. These results contribute to a better understanding of the dynamics of urban soil contamination, and underline the urgent need for sustainable and appropriate management of these landfill sites.

Keywords: Characterization; Physicochemical properties; Mineralogy (XRD); Soil contamination; Heavy metals; Urban landfills

1. Introduction

Urban landfill sites are a major component of waste management in many developing cities, particularly Brazzaville. Their accumulation leads to environmental and health risks, mainly associated with the leaching of toxic substances, particularly heavy metals and other toxic elements, which are responsible for transferring pollutants to groundwater and into the food chain [1]. Soil contamination by heavy metals is due to an increase in the total concentrations of these elements in the soil following major anthropogenic inputs [2].

The dumping of solid waste in the ground, without any prior treatment or protection of the subsoil at landfill sites, can be a risk factor for environmental contamination by heavy metals [3,4,5,6]. In soil pollution by heavy metals, soil-metal cation interactions play a key role in regulating the behaviour of these heavy metals, particularly their distribution in the solid phase. Released pollutants can be transported by infiltration water to the underlying natural soil and groundwater. The accumulation of heavy metals in soils is a concern in agricultural production because of their adverse effects on crop growth, food quality and environmental health [7].

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Among these contaminants, mercury (Hg), nickel (Ni) and zinc (Zn) are of particular concern because of their toxicity, bioaccumulation and persistence in the environment.

Mercury, often associated with electronic or medical waste, poses a high risk due to its conversion into methylmercury, a powerful neurotoxicant. Nickel and zinc, partly from industrial activities and batteries, can contaminate soil and groundwater, affecting biodiversity and human health [8].

The degree of metallic contamination in soils is often assessed on the basis of the total content of trace metallic elements determined in the surface horizon [9]. Many researchers have carried out research into soil contamination by heavy metals from landfill sites [10,11,12]. These studies have shown that landfill sites are among the sources of metal contamination in soil.

The objective of this study is to characterize the soils of Brazzaville's six landfill sites in terms of their physicochemical and mineralogical properties, and to quantify their levels of Hg^{2+}, Ni^{2+} and Zn^{2+} contamination, in order to provide data for informed environmental management.

2. Materials and methods

2.1. Location and description of the study area

Located between latitudes 4°6' and 4°23' South and longitudes 15°5' and 15°25' East. Brazzaville is the capital and most populous city of the Republic of Congo. It is located in the south of the Republic of Congo, on the banks of the River Congo, on the north bank of the Pool Malebo opposite Kinshasa. It has a very extensive peri-urban area in both its northern and south-western parts. Brazzaville has a population of around 1,838,348, covers an area of over 309 km2 and has 9 arrondissements. Figure 1 shows a map of the city of Brazzaville with the selected landfill sites.

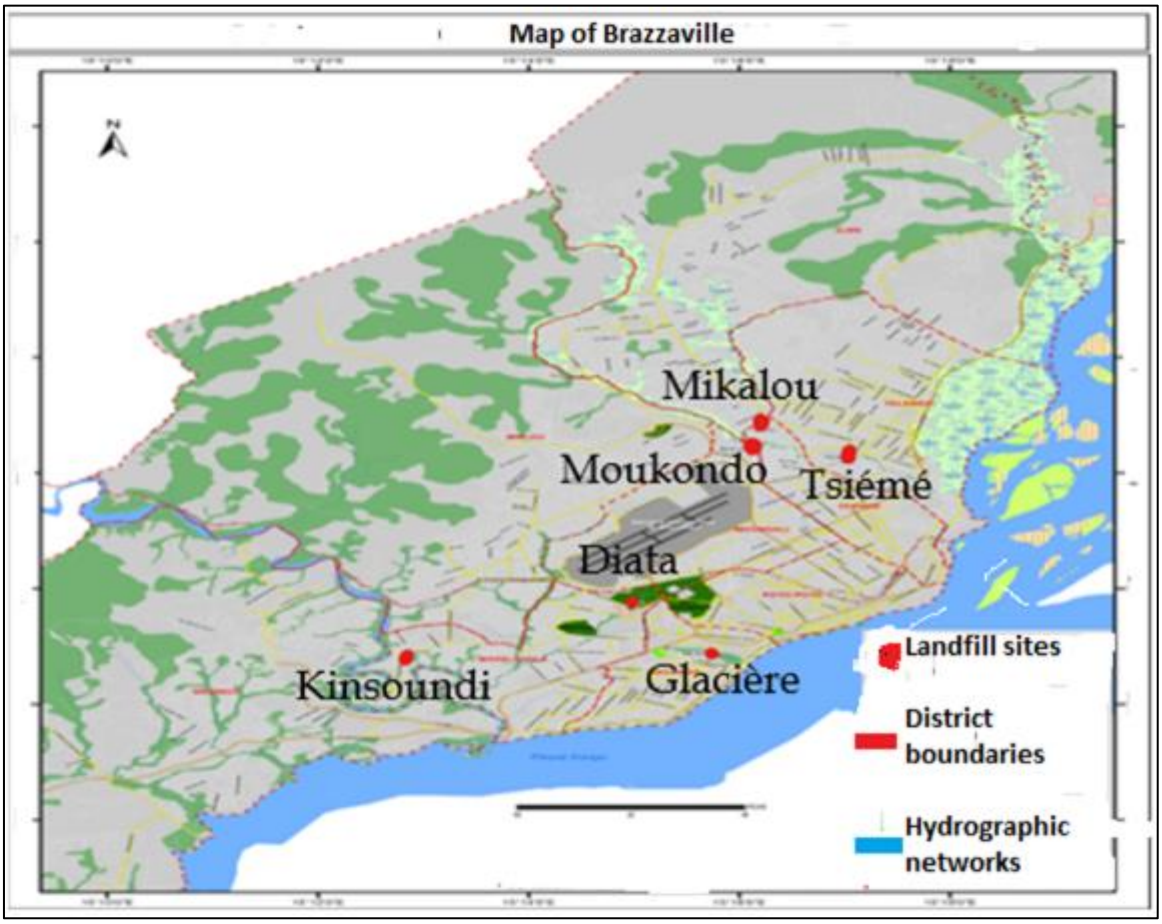

Figure 1 Map of Brazzaville (red dots represent landfill sites)

Table 1 Geographical coordinates of the landfills studied

Contact details	Kinsoundi	Glacière	Diata	Moukondo	Tsiémé	Mikalou
Latitude (°)	04.28635	04.28619	04.27211	04.22756	04.22994	04.21942
Longitude (°)	015.21398	015.26225	015.24971	015.26872	015.28378	015.26983

2.2. Sample collection and processing

Soil samples were collected from a depth of 0-25 cm, using a stainless-steel auger, in accordance with ISO standard 18400-102 [13]. Three sampling points per site were selected according to a systematic plan to represent spatial variability. The samples were stored in clean plastic bags and transported to the laboratory. In order to obtain a composite sample for each landfill, 2 kg of each sub-sample was mixed together, air-dried for ten days, ground using an agate mortar, sieved to 2 mm and stored under stable conditions.

2.3. Physicochemical analysis

- Measurement of water content:

Water content was determined by weight lost after heating at 105°C to constant mass (ISO 11465) [14].

- Measurement of pH:

The pH was measured in the supernatant of a 1:2.5 (soil/water) soil suspension using a HANNA-type pH meter in accordance with the standard (AFNOR, 1953) [15].

- Measurement of organic matter

The Walkley and Black method was used to determine the organic matter content of soil samples. This method is based on the oxidation of organic carbon by an excess of potassium dichromate ($K_2Cr_2O_7$) in an acid medium, followed by an assay of this excess using Mohr's salt [16].

2.4. Mineralogical analysis

Mineralogical analyses using X-ray diffraction on samples of these soils were carried out at the scientific and innovation centre of the Felix Houphouët-Boigny University (Abidjan, Republic of the Ivory Coast). Powder X-ray diffraction (XRD) patterns were recorded at room temperature on Bruker's Siemens D5005 diffractometer over the 2θ angular range from 5° to 90° with a scan speed of 2° per minute (increment: 0.05). The radiation used was CuKα (λ=1.5406 Å). The collection voltage and current are 40 KV and 35 mA respectively.

2.5. Determination of metal ions

The concentrations of Hg, Ni and Zn were determined by ICP-OES after mineralization of 0.5 g of the composite soil sample with 9 ml of hydrochloric acid (HCl) and 3 ml of nitric acid (HNO3) (aqua regia). This step was carried out at a temperature of 95°C for a duration of 75 minutes using a heating block. The mineralisate obtained was reduced to 50 ml in accordance with the standard NF ISO 11466 [7].

2.5.1. Speciation of heavy metals

The sequential extraction scheme was used to study the speciation of heavy metals in soil samples collected in the following stages:

- **Stage 1**: exchangeable fraction: 1 g of soil sample was dissolved in 16 mL CaCl2 (1 M) and stirred for 2 h at room temperature.
- **Stage 2:** oxidizable fraction (organic fraction and sulphides): the pellet from stage 1 is dissolved in 16 mL of the mixture H2O2 8.8 M + HNO3 0.02 M (v/v) (5+3) and stirred for 5 h at room temperature and for 1 h at 98°C. The extraction was carried out in 10 ml of 3.5 M CH3 COONH4 and placed under agitation for 1 h at room temperature.
- **Stage 3:** acid-soluble fraction (carbonates): the pellet from stage 2 is dissolved in 35 ml of 1 M CH3COOH + 0.6 M CH3COONH4 (v/v) (1+1) and stirred for 5 h at room temperature.

- **Stage 4:** reducible fraction (Fe and Mn oxide): the pellet from stage 3 is dissolved in 35 mL NH2OH, 0.1 M HCl + 25% (v/v) CH3COOH (1+1) and stirred for 4 h at room temperature and 1 h at 98°C. Extraction is carried out in 10 mL 3.5 M CH3COONH4 and stirred for 1 h at room temperature.
- **Stage 5:** residual fraction: the pellet from stage 4 is calcined for 2 h at 550°C in a muffle furnace. The residue is solubilised in 20 mL of concentrated HCl (12 M) and HF 40% (v/v).

Sequential extractions were performed in polyethylene centrifuge tubes. Between two stages, the suspensions were centrifuged for 30 min at 5,300g. Heavy metals were measured in the supernatant of each phase [18].

3. Results and discussion

3.1. Physicochemical characteristics

3.1.1. Moisture content (MC)

The results (Table 2) show that moisture content varies from 2.7% (Glacière) to 38% (Mikalou), indicating differences in texture and water saturation per sampling campaign.

The highest water content values were consistently recorded during the third sampling campaign, while the lowest values were recorded during the first campaign for the Kinsoundi, Diata, Moukondo, and Tsiémé dumps, and during the second campaign for the Glacière and Mikalou dumps. The increase in water content during the third campaign can be explained by the fact that it was carried out during the rainy season. Soils are wetter in the rainy season than in the dry season.

The evolution of the landfill is guided by microbiological activity. This activity depends on the landfill's water content. Waste can remain intact for a very long time due to the low moisture content within the waste mass [19]. Generally, degradation and gas production rates increase as the water content of the waste increases [20].

3.1.2. pH values

The pH varies between 6.7 (Mikalou) and 7.2 (Diata), characteristic of moderately acidic to neutral soils (Table 2).

The variation in pH recorded in the landfill soils is shown in Figure 3. The pH recorded in these soils is around neutral, which could indicate that these soils are not rich in carbonate. These results are in agreement with those of the XRD, whose diffractogram shows very little carbonate at around 2θ=30°.

The pH values obtained in these landfill soils are similar to those found in the soil of the uncontrolled landfill of Tangier in Morocco [21], but they do not agree with those found in 2015 by El Baghdadi et al [22], in soil solutions from the landfill of the city of Béni-Mellal in Morocco, in fact, they had recorded basic pH values. The average pH values obtained for our study at the various sites are acceptable [23].

3.1.3. Rate of organic matter (OM)

Organic matter ranges from 0.13% (Moukondo, Kinsoundi and Diata) to 9.9% (Kinsoundi) (Table 2). The average organic matter content recorded in the landfills is higher than the guide value (1% to 2%) accepted for mineralised soils. These average organic matter values are even higher than those recorded in the soil of the Bè-Aveto landfill in Lomé, Togo [1]. The presence of organic matter in landfill soils can be justified by the fact that fermentable matter accounts for a large proportion of the physical composition of household waste, and the presence of limestone in a soil increases the total organic matter content. The presence of organic matter can also be linked to the strong mineralisation of dead leaves or cardboard waste caused by the action of specific enzymes [24].

Table 2 Summary values of measurements of physicochemical parameters in landfill soil

Parameters	**TE (%) or Hu (%)**			**pH**			**M.O (%)**		
Compaigns	C1	C2	C3	C1	C2	C3	C1	C2	C3
Kinsoundi	12	14	18	7.14	7.01	7.17	9.9	0.13	0.15
Glacière	9	2.7	17	7.2	7.02	7.21	0.55	1.72	1.95

Diata	11.8	14	18	7.1	7.2	7.58	1.55	0.13	2.47
Moukondo	5.6	14	18	6.9	6.9	7.32	0.13	5.47	4.62
Tsiémé	15	16	23	6.9	6.9	6.95	3.69	7.24	5.43
Mikalou	15	14	38	6.9	6.7	8.02	2.27	8.15	0.16
Minimum	12	2.7	17	6.85	6.7	6.95	0.13	0.13	0.15
Maximum	15	16	38	7.14	7.2	8.02	9.9	8.15	5.43
Averages	1.4±0.63	12.45±3.25	20± 3.94	7.01± 0.14	6.96± 0.11	7.37± 0.34	2.92± 3.56	3.21± 2.56	1.2±2.02
WHO standards				6.5 – 8.5					

3.2. Mineralogical analysis of landfill soils

The results of X-ray diffraction (XRD) analyses of the soil samples studied, taken from the targeted public landfills, are shown in Figure 2 below. Applying Bragg's relation, the peaks located at 2ϴ (°) = 21; 26.5; 39; 42.5; 45.5; 50; 55; 60; 68 and 80 corresponding respectively to the following intereticular distances (dhkl in Å) 3.86; 3.065; 2.27; 2.105; 1.938; 1.816; 1.66; 1.52; 1.405; 1.256 and 1.082, are the characteristic peaks of quarters. The fact that the intensities of these peaks are the highest would reflect the abundance of quartz in these soils. The peaks located at 2ϴ = 29.5° in the diffractograms of the Tsiémé (PS01T XY) and Diata (PS01D (1) XY) landfill soils, corresponding to dhkl = 2.759 Å, could indicate the presence of calcite ($CaCO_3$). As the intensities of these peaks are low, this could indicate that calcite is not abundant in these landfill soils. This value of dhkl = 2.759 Å would also probably correspond to the (111) plane of sphalerite (ZnS) [25,26].

Given that quartz is a species characteristic of sand, the landfill soils studied are consequently sandy soils. We can deduce that these soils have a low water retention capacity, and may facilitate the migration of pollutants through the vertical movement of rainwater towards the water table.

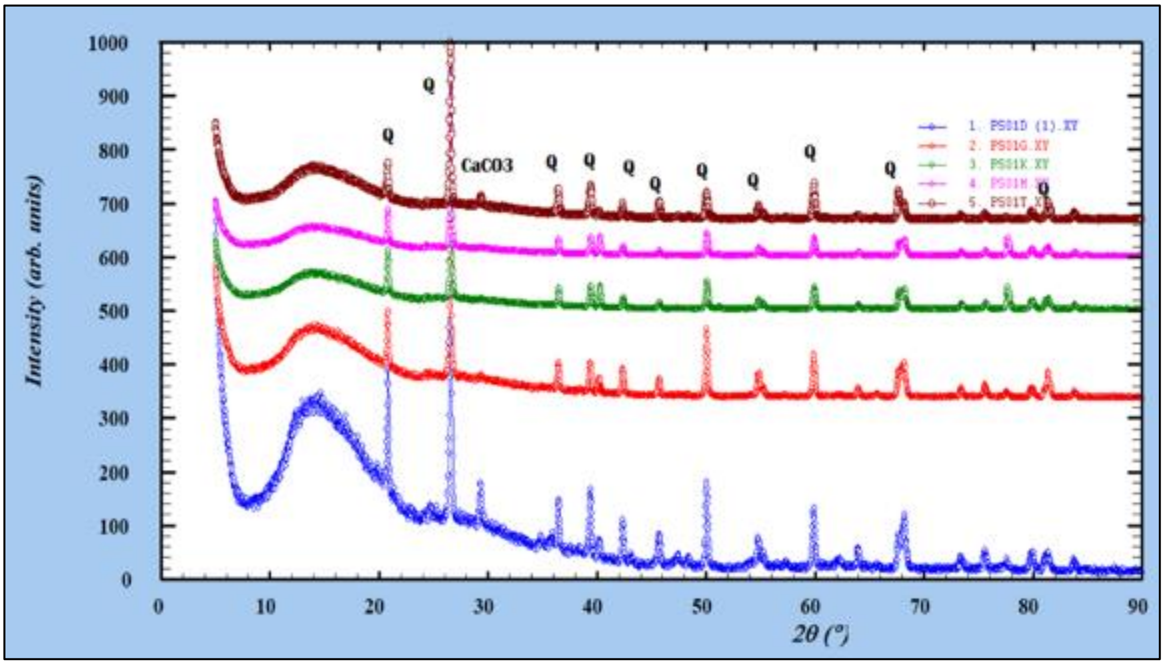

Figure 2 X-ray diffraction of soil from selected landfills

3.3. Concentrations of Hg^{2+}, Ni^{2+} and Zn^{2+}

Concentrations of mercury, nickel and zinc ions (Table 3) show significant variability.

3.3.1. - Mercury ion

The results obtained for determining mercury levels in landfill soil are presented in table 3. These results indicate that the highest mercury levels were recorded during the second and third sampling campaigns. The mercury ion concentrations recorded in the soils of the Glacière, Diata, Tsiémé and Mikalou landfills are well in excess of the standard recommended by the WHO (2mg/kg) [27]. High concentrations of mercury ion in soils, compared with other elements, have also been reported by many authors [1,28]. This indicates significant contamination by this metal in the soils of these landfill sites. The high mercury ion levels could be explained by the fact that for a long time the landfill sites received waste containing mercury.

3.3.2. - Ion Nickel

Compared with the WHO standard, which sets a limit value for nickel at 35 mg/kg [27], it can be deduced that the soil at the Tsiémé, Diata and Moukondo landfills is contaminated and polluted by nickel. The high accumulation of nickel in certain landfills can be explained by the composition and nature of household waste. The main sources of nickel in household waste are metals, batteries, dyes used in textiles and glass [29].

Nickel levels in the Kinsoundi, Glacière and Mikalou landfills are low compared with those in the literature [20,30]. These low levels are thought to be due not only to the recovery of certain nickel-containing materials for possible recovery (recycling, reuse, etc.), but also to the fact that nickel is an element that is held very poorly by the soil, and often migrates to the depths [30].

3.3.3. - Zinc ion (Zn2+)

The average levels of zinc ion in landfill soils obtained in our study are well above the WHO standard (50 mg/kg) [27]. This is in agreement with the statements of Aurélie et al, [31], who estimated that the concentration of zinc ion is 5 to 127 times higher in landfill soils than in natural soils. The high concentrations of zinc ion in landfill soils may be linked to its use, given its importance worldwide. Zinc is the most abundant element in soils after iron. The presence of zinc in landfill soils is not homogeneous, which may be due to the heterogeneous nature of the waste or to non-uniform degradation of the waste [21]. Zinc in landfill soil may originate from kitchen utensil waste. High concentrations of zinc ion in soils, compared with other elements, have also been reported by many authors [20,21,32,23]. However, they are lower than those obtained in the soil from the Akouédo landfill [7].

Table 3 Summary of heavy metal concentrations in landfill soil (in red: concentrations above WHO standards; in bold: minimum and maximum concentrations)

Parameters	**Hg^{2+} (mg/kg)**			**Ni^{2+} (mg/kg)**			**Zn^{2+} (mg/kg)**		
Compaigns	**C_1**	**C_2**	**C_3**	**C_1**	**C_2**	**C_3**	**C_1**	**C_2**	**C_3**
Kinsoundi	0.2	0.25	1.9	18	21	34	690	5100	440
Glacière	2.4	2.7	3.8	57	12	15	850	540	580
Diata	0.44	1.2	1.8	7500	4300	27	12000	2800	1700
Moukondo	0.66	1.5	0.2	5400	57	11	710	1200	850
Tsiémé	0.38	1.9	2.3	70	12000	15	910	3600	1700
Mikalou	1.9	1.7	2..3	35	9	10	1000	450	380
Minimum	**0.2**	**0.25**	**0.19**	**18**	**9**	**10**	**670**	**450**	**380**
Maximum	**2.4**	**2.7**	**3.8**	**7500**	**12000**	**34**	**12000**	**5100**	**1700**
Averages	1±0.2	1.5±0.5	1.5±1.1	2180±3373.6	2733±611.2	16.33 ± 8.81	2693.3±4560.8	2281.67 ± 1551.7	762.9±568.9
WHO standards [27]	**1**			**35**			**50**		

The results of the physicochemical analyses highlight moderately acidic to neutral soils, with variable organic matter, which may influence metal retention capacity. The quartz-rich mineralogy indicates the significant presence of sand, which is responsible for the possible infiltration of metal ions at depth.

Concentrations of Hg2+, Ni2+ and Zn2+ ions vary considerably between sites, with Tsiémé showing exceptionally high levels, linked to the nature of the waste (industrial, electronic). The presence of Hg2+ ions in quantities close to or above regulatory thresholds indicates a potential risk to the environment and human health, particularly through bioaccumulation.

Factors influencing contamination include waste composition, water dynamics (permeability, saturation) and soil adsorption capacity. Leaching of metals into groundwater is likely, requiring increased monitoring.

3.4. Statistical test on the distribution of physicochemical parameters and heavy metals in landfill soils

3.4.1. - Physicochemical parameters

The results obtained from tests on the distribution of physicochemical parameters in landfill soils are presented in figures 3 to 5. These results show that these parameters are asymmetrically distributed, except for organic matter, which is symmetrically distributed in the Tsiémé landfill.

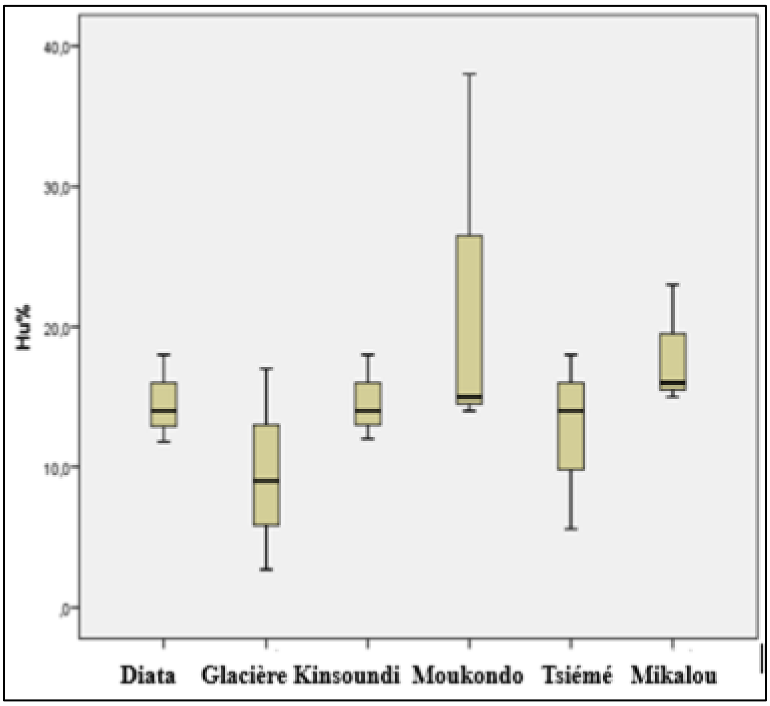

Figure 3 Distribution of moisture content in landfill soils

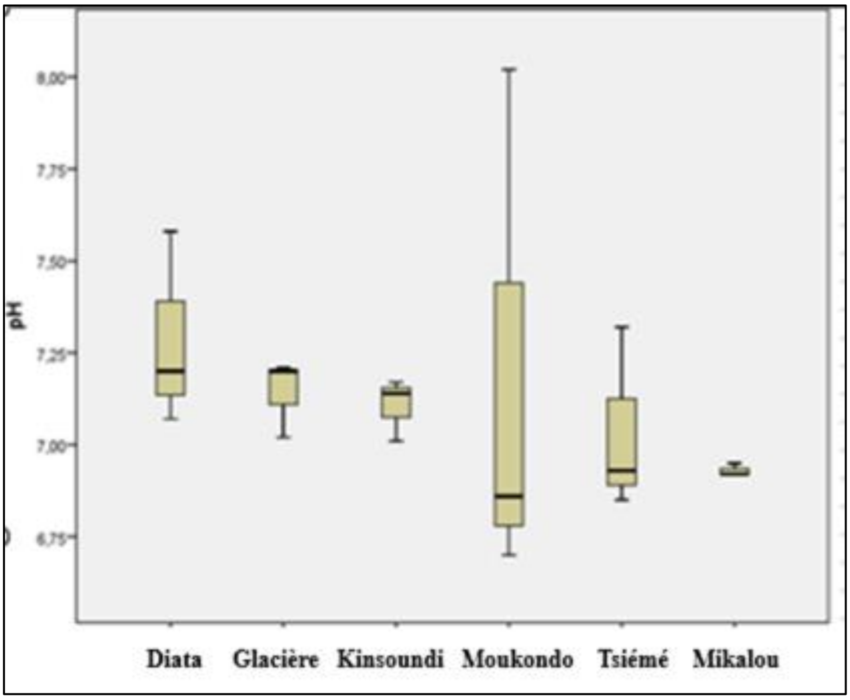

Figure 4 pH distribution in landfill soil

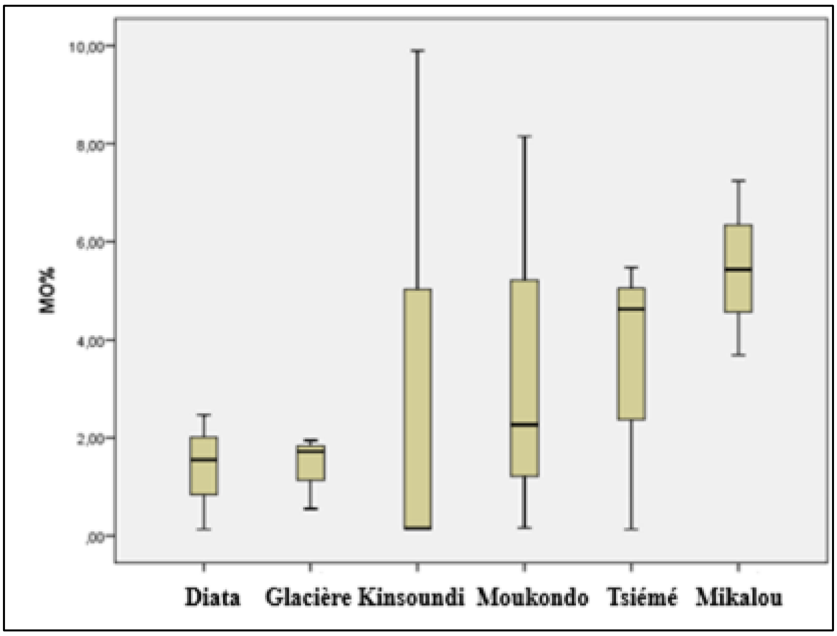

Figure 5 Distribution of organic matter in landfill soils

3.4.2. Concentration of Hg^{2+}, Zn^{2+} and Ni^{2+} ions

Figures 6 to 8 show that the dispersion of heavy metals (Hg, Zn and Ni) in landfill soils is asymmetrical. This indicates that these elements are not stable in these soils. This may be due to processes of migration, diffusion and differentiated contamination depending on proximity to sources. In fact, it is possible that a large proportion of the elements mobilised in the waste are dissolved through the complex chemical reactions that take place at the soil-waste interface and then transported by infiltration through the soil towards the groundwater. However, given that the analyses were carried out on composite samples, we can also suspect a dilution phenomenon by samples containing a majority of very low to moderate levels and a minority of very high levels of these elements, often associated with the proximity of specific pollution sources [34].

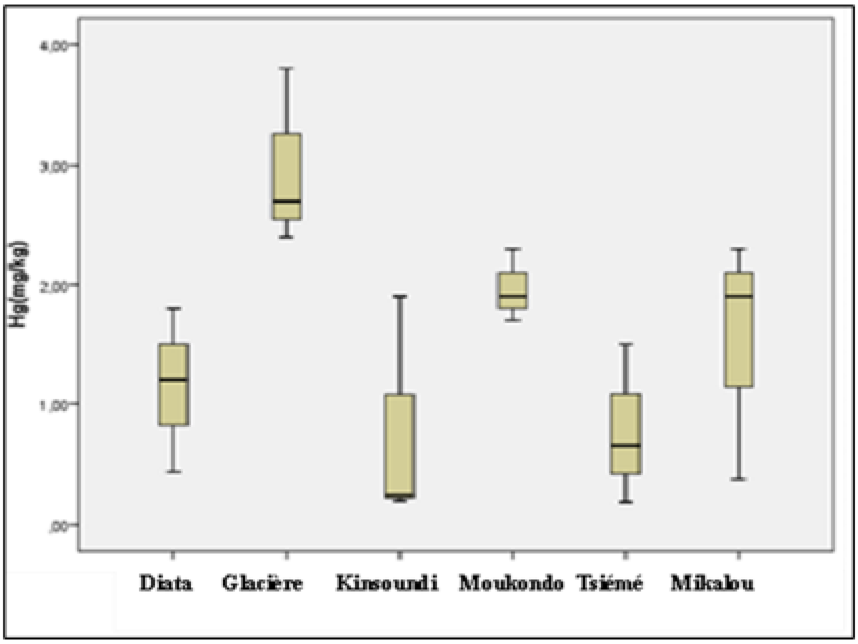

Figure 6 Distribution of mercury in landfill soil

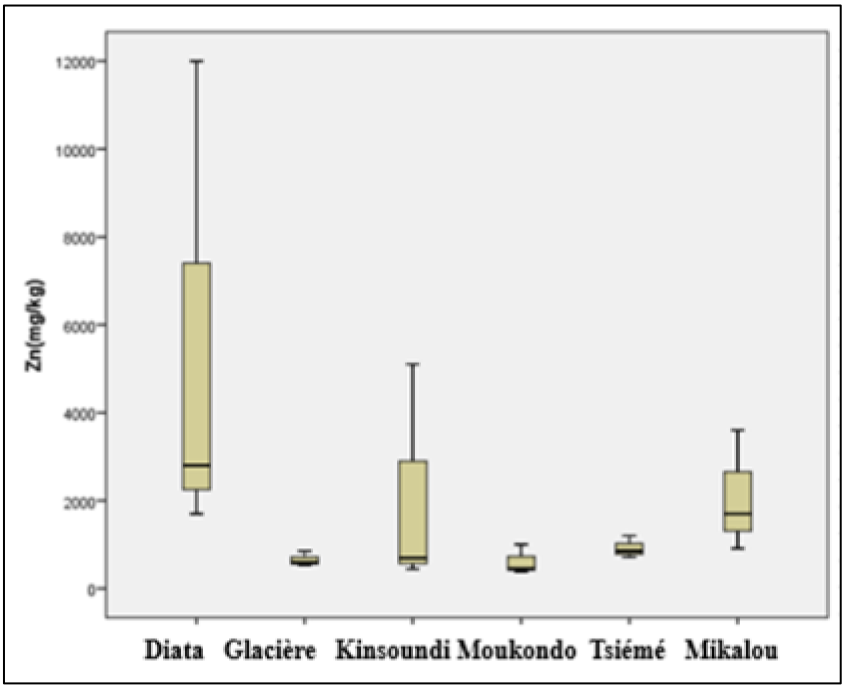

Figure 7 Zinc distribution in landfill soils

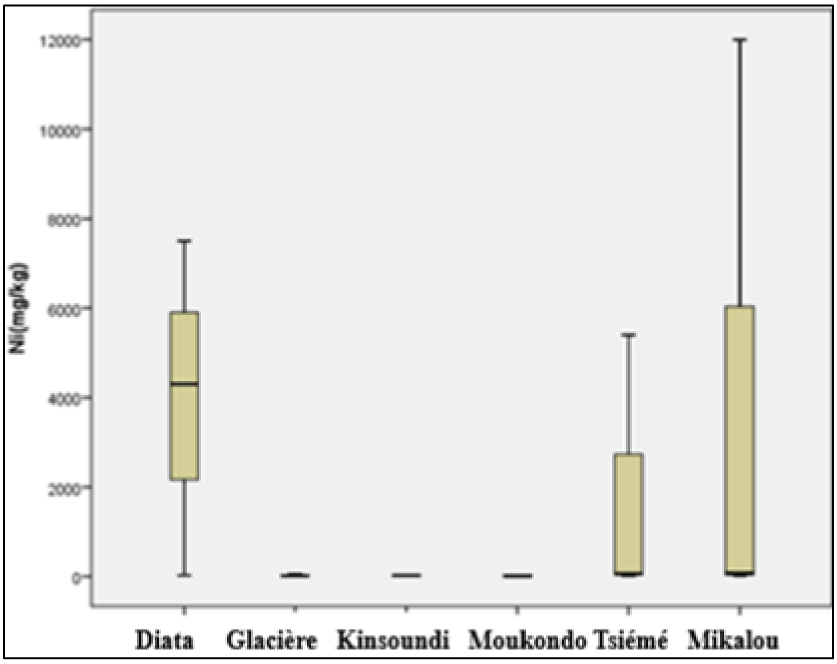

Figure 8 Nickel distribution in landfill soil

3.5. Analysis of the correlation between physicochemical parameters and heavy metals

Analysis of the correlation matrix between physicochemical parameters and metals reveals the presence of strong correlations between these parameters (Table 4); these include water content with organic matter (OM), pH with zinc (Zn) and nickel (Ni) with zinc (Zn).

Table 4 Correlation matrix between physicochemical parameters and metals in the soils of six (06) landfills (figures coloured red indicate highly correlated parameters)

	TE	pH	M.O	Hg	Ni	Zn
TE	1					
pH	0.030	1				
M.O	0.724	-0.659	1			
Hg	- 0.125	0.164	-0.232	1		
Ni	0.074	-0.192	0.103	-0.346	1	
Zn	-0.034	0.539	-0.386	-0.401	0.685	1

The strong associations between water content and organic matter, pH and zinc, and nickel and zinc show interdependence between these parameters. The strong correlation between water content and organic matter shows that the evolution of organic matter is guided by microbiological activity, which in turn depends on the water content in the landfill. The waste could remain intact for a very long time because of the low water content in the landfill. Furthermore, the association between nickel and zinc could indicate that these metals have the same origins, in particular anthropogenic activities [20].

4. Conclusion

Physicochemical and mineralogical analysis of the soil at selected landfill sites in Brazzaville reveals variable levels of mercury (Hg^{2+}), nickel (Ni^{2+}) and zinc (Zn^{2+}) ion contamination, with the highest concentrations being 3.8 mg/kg at the Glacière landfill site, 1,200 mg/kg at the Tsiémé landfill site and 1,200 mg/kg at the Diata landfill site. The Glacière, Tsiémé and Diata sites show worrying levels, which may be due to the presence of organic matter that favours the fixation of these metals.

Although the sandy nature of the soils studied could moderate the retention of metals by encouraging their infiltration into the water table, their accumulation remains an issue. Sustainable management of these sites must include measures for decontamination, containment and environmental monitoring.

Regular monitoring, the installation of containment barriers and raising local awareness of the risks associated with waste management should be put in place.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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