Speciation of Potentially Toxic Metals from the Mine Impacted soils: A Case Study of Riruwai Mining Area, Kano State, Northern Nigeria

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Received: 5 May 2023; Accepted: 20 June 2023; Published: 25 July 2023

Abstract

Mining and smelting sectors contribute significantly to global growth on a regional and local scale. However, the operations are causing significant environmental damage, particularly pollution of the soil with potentially toxic metals (PTMs). In this study, the speciation of PTMs (arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc) from the mine impacted soils of Riruwai mining area was investigated using a modified Tessier sequential extraction method. The findings of the study revealed that all the PTMs examined are considerably associated with non-residual fractions in all the sampling locations. This indicates that all the PTMs investigated in the study area may be highly mobile in the soils and hence potentially toxic. The concentrations of PTMs in the soil’s geochemical fractions were found to vary with the sampling locations, with active mining sites significantly (p ≤ 0.05) recording the highest value, followed by farmland, while the lowest value was reported at the control site. Therefore, to protect the environment and the health of the local populace, PTMs in the soils of the study area should be regularly monitored and a treatment program should be put in place.

Keywords: Mining; toxic metals; fractions; Riruwai; Speciation

Introduction

Mining is contributing substantially to the world economic growth. The activities however, have resulted in environmental deterioration, notably soil contamination with potentially toxic metals, PTMs (Aliu et al., 2016; Jiménez-Oyola et al., 2021). Arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc are examples of PTMs that are known to be hazardous and harmful to humans and animals as well as the environment (Fazekašová & Fazekaš, 2020; Focus et al., 2021). These PTMs are frequently found in industrial waste, including mining waste, electronic waste, and other hazardous waste. When these wastes are not properly handled, PTMs can leach into the soil, groundwater and subsequent accumulation in the biota, causing serious threats to both the environment and public health (Focus et al., 2021; Wang et al., 2022). The transfer of PTMs from soils to other media such as air and water, and then to biological systems, as well as their potential
hazards, is dependent on their speciation patterns in the soils (Liu et al., 2013).

A common technique of determining the speciation of PTMs in environmental samples is sequential method. This method involves the sequential separation of metal species based on their chemical association with different soil fractions (Li et al., 2022). By analyzing the metal concentrations in each of the fractions, it is possible to determine the speciation of the PTMs and their potential mobility and bioavailability in the environment (Meng et al., 2018; Li et al., 2022). The different fractions that PTMs exist in soil include organic matter-bound, exchangeable, reducible, and residual fractions (N’goran et al., 2022).

The distribution of these fractions determines the potential movement and availability of the metals in the soil and, hence their toxicity. If they are predominantly in non-residual fractions (organic matter-bound, exchangeable, reducible), they may be leached down to the soil profile and reach groundwater or they may be taken up and accumulated by plants, invertebrates, humans and other animals (Sinduja et al., 2022). PTMs that exist primarily in residual forms pose little to no risk to living organisms or the environment (Nevidomskaya et al., 2021). Therefore, measuring total PTMs contents in soils cannot provide an accurate assessment of the negative effects of mining activities (Jahromi et al., 2020). It is therefore necessary to identify the geochemical fractions of metals in soils in order to evaluate the risks of metal contamination and propose viable remediation approaches. For this reason, this study was aimed at studying the speciation of PTMs from the mine-impacted soils of Riruwai Zn-Sn Mine in Kano State, Northern Nigeria. To the best of our knowledge, no similar studies were carried out in the study area.

Methods

Description of the Study Area

Riruwai mining area is located between latitudes 10°43’97” and 10°45’01” N and longitudes 8°43’3” and 8°47’39” E (Fig. 1). The area has a total area of 129 km² (Alhaji et al., 2017; Lambu, 2019). The climate was classified as tropical savanna by the Köppen climatic classification system. The mean annual rainfall varied between 400 and 1,200 mm each year, with a minimum temperature of 14.02 °C and a maximum temperature of 32.03 °C (Yakubu, 2016). In terms of geology, Riruwsai is one of the new granite complexes in Nigeria. The complex is an excellent example of decayed alkaline volcano roots that formed in the early Jurassic as part of a sequence of anorogenic centers that formed sequentially over time. It is surrounded by a collection of calc-alkaline meta-igneous and metamorphic rocks that have undergone metamorphism and a Cambrian age modification (Olasehinde et al., 2012).

Figure 1. Geographical map of the study area

Soil Sampling and Collection

Forty-eight (48) soil samples were collected at a depth of 0–20 cm from three (3) sampling locations, including 18 from active mine sites, 21 from farmlands, and 9 from control sites using a systematic sampling strategy with the help of a steel soil auger. A grid of equal space lines was constructed, sampling sites were partitioned into equal sub-areas, and an equal distance of 10.00 meters was measured at intervals between each sampling site. Top layers were collected because, when soils are contaminated with heavy metals, the highest levels of metals are usually found at the surface layer of the soil profile (Mazurek et al., 2017). Control soil
samples were collected 6.00 km distant from other sampling locations in the area with the same lithology, but with little or no human intervention as reported by Ngole-Jeme & Fantke (2017). At each sampling location, three sub-samples were collected within a distance of 5 m surrounding the location and mixed together to form a composite sample. A 1.00 kg from the mixed samples was selected by quartering method, stored in a polyethylene bag, labelled and transported to laboratory for analyses (Fan & Wang, 2017; Yang et al., 2017).

Samples of soil were air-dried in the laboratory for five (5) days at room temperature. The large fragments, stones and gravels were removed. The homogenized soil was pulverized, sieved through a 2 mm polyethylene sieve, stored in polyethene bags and labelled appropriately for further analyses.

**Determination of PTMs Speciation in Soil**

A modified Tessier sequential extraction technique adopted by Uba et al. (2008) and Zhang et al. (2020) was used for soil partitioning. As part of the modification, ammonium ethanoate was used instead of magnesium chloride to prevent the re-adsorption or precipitation of the leached metals on the surface of the soil substrate due to the metal complexing power of ethanoate (Sagagi, 2013).

**Determination of Exchangeable Phase (F1)**

A 1.00 g of air-dried soil was shaken in a 50 cm³ polypropylene centrifuge tube at room temperature with 10.00 cm³ of 3.5 mol/dm³ ammonium ethanoate at a pH of 7.00 for 1 hour, centrifuged and the supernatant was decanted and made up to 100.00 cm³ with deionized water. The residue was reserved for further analyses (Zhang et al. 2020).

**Determination of Oxidizable Phase/Bound to Organic Matter (F2)**

A 10.00 cm³ of 8.80 mol/dm³ hydrogen peroxide and 6.00 cm³ of 0.02 mol/dm³ trioxonitrate (V) acid were added to residue from F1 and agitated for 5 hours at 98 °C.

10.00 cm³ of 1.00 mol/dm³ ammonium ethanoate was added centrifuged and supernatant made up to 100.00 cm³ with deionized water. The residue was reserved for further analyses (Uba et al., 2008; Anju et al., 2010)

**Determination of Acid Soluble Phase /Bound to Carbonate (F3)**

A 25.00 cm³ 0.05 mol/dm³ of disodium ethylenediaminetetraethanoate solution was added to the residue from F2, agitated for 6 hours and centrifuged. The supernatant was decanted into a 100 cm³ volumetric flask and made up to 100.00 cm³ with deionized water. The residue was reserved for further analyses (Sut-Lohmann et al., 2022).

**Determination of Acid Reducible Phase /Bound to Fe-Mn Oxides (F4)**

A 17.50 cm³ of 0.10 mol/dm³ hydroxylamine hydrochloride and 17.50 cm³ of 3.50 mol/dm³ ammonium ethanoate were added to the residue from F3, agitated for 1 hour and centrifuged. The supernatant was into a 100 cm³ volumetric flask decanted and made up to 100.00 cm³ with deionized water. The residue was reserved for the determination of residual phase /bound to silicates (F5)

**Determination of Residual Phase /Bound to Silicates (F5)**

A 5.00 cm³ of concentrated hydrochloric acid, 15.00 cm³ of concentrated trioxonitrate (V) acid and 5.00 cm³ of concentrated hydrofluoric acid were added to the residue from F4 and digested in an acid Teflon cup. The mixture was heated using electric hot plate at 180 °C until the solution was dried. The beaker was removed and cooled to room temperature. The dried solution was diluted to 100.00 cm³ with deionized water (Vuong et al. 2022).

The extract from each extraction step was used for the determination of arsenic, cadmium, chromium, mercury, nickel, manganese, lead and zinc at their respective wavelengths using Microwave Plasma Atomic
Emission Spectrometer (Model Number 4200, Agilent, USA).

**Quality Control and Statistical Analysis**

Appropriate quality control/assurance procedures and precautions were taken to ensure the reliability of the analytical results. To prevent cross-contamination, samples were handled with extreme caution. Throughout the study, analytical grade chemicals were used. All glass and plastic wares were immersed in 10.00% (v/v) trioxonitrate (V) acid overnight before being thoroughly washed three times with deionized water and dried in an oven. The wares were dried for 20 minutes in a desiccator before analysis (Asghraf et al., 2011). Reagent blanks were determined to minimize the errors caused by reagent contaminants. Each sample was analyzed in triplicate, and the results were expressed as the mean ± standard deviation. An analysis of variance (ANOVA) was used to establish whether there was a significant difference between the sampling locations. Tables and graphs were plotted using Microsoft Excel 2010.

**Result and Discussion**

As presented in Table 1, the speciation patterns of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc at the active mining site in exchangeable fraction (F1) are 7.49, 4.31, 3.90, 0.29, 90.61, 1.05, 21.23 and 18.23 mg/kg respectively. The values of all the PTMs are within the WHO/FAO (2001) limits of 20.00, 3.00, 100.00, 2.00, 2000.00, 50.00, 50.00, and 300.00 for agricultural soils. Manganese was found to be the highest in the exchangeable fraction, while mercury was found to be the lowest. In the oxidizable phase/bound to organic matter (F2), the concentrations of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc are 3.55, 1.09, 23.22, 0.17, 104.07, 5.44, 104.0 and 882.05 mg/kg respectively. In this fraction, zinc was recorded as the highest metal bound to oxidizable phase, while mercury was found to be the lowest.

This indicates that zinc is the highest heavy metal bound to organic matter among the studied PTMs. PTMs that are bonded to the organic matter may become available because some of them are water soluble (Elija et al., 2020; Ogunwale et al., 2021). The levels of PTMs in acid soluble fraction/bound to carbonates (F3) are: arsenic (6.38 mg/kg), cadmium (2.01 mg/kg), chromium (6.51 mg/kg), mercury (0.12 mg/kg), manganese (18.11 mg/kg), nickel (9.37 mg/kg), lead (34.31 mg/kg) and zinc (398.01 mg/kg). Zinc had the highest level, while mercury had the lowest. This suggests that in active mining site, considerable amount of zinc was bound to carbonates and very small fractions of mercury were bound to carbonates.

The levels of PTMs (mg/kg) in acid reducible fraction/bound to Fe-Mn oxides (F4) are as follows: arsenic (2.10), cadmium (11.28), mercury (0.07), manganese (47.91), nickel (3.07), lead (10.40) and zinc (58.43). In this fraction, zinc was the highest metal bound to Fe and its oxides, while mercury was least recorded. The levels of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc in residual fraction are 1.05, 0.07, 12.77, 0.05, 23.29, 2.06, 15.08 and 42.40 mg/kg respectively. Similar to other fractions, zinc is the highest heavy metal in the residual fraction, while mercury is the lowest. In this sampling site, very considerable amounts of heavy metals are found in non-residual fraction. The percentage contributions of PTMs to non-residual fraction as shown in Figure 2 are 1.04, 0.40, 2.40, 0.03, 13.95, 1.01, 8.54 and 72.61 % for arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc respectively.

The high percentage (%) association of PTMs with non-residual fraction is a reflection of their high mobility and hence bioavailability. The rate of mobility decreases in the following order: zinc > manganese > lead > chromium > arsenic > nickel > cadmium > mercury. Rodríguez et al. (2009) reported similar observation that PTMs are associated with non-residual fractions when they investigated PTMs distribution and
chemical speciation in tailings and soils around a Pb–Zn mine in Spain. Yiika et al. (2023) observed significant mobility of PTMs in artisanal gold mine tailings in eastern Cameroon.

In the farmland, the levels of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc in exchangeable fraction are 4.46, 0.36, 5.33, 0.13, 37.79, 1.34, 11.57 and 92.34 mg/kg respectively. Zinc is observed to be the highest contributor to this fraction while mercury is the lowest contributor. The concentrations of PTMs (mg/kg) in oxidizable fraction are: arsenic (3.12), cadmium (0.09), chromium (9.89), mercury (0.10), manganese (61.82), nickel (8.59), lead (25.10) and zinc (158.50). This fraction contains highest amount of zinc and lowest amount cadmium. The levels of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc in acid soluble fraction are 3.9, 0.10, 15.34, 0.07, 15.38, 0.71, 16.06 and 78.45 mg/kg respectively.

Zinc and mercury were the highest and lowest contributors respectively. The concentrations of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc in acid reducible fraction are 3.58, 0.25, 8.00, 0.04, 11.20, 1.47, 10.80, 48.47 mg/kg respectively. Zinc had the largest share of this fraction, while mercury had lowest share. The levels of PTMs (mg/kg) in the residual fraction are 0.41, 0.07, 7.70, 0.11, 9.01, 0.98, 9.45, 31.67 for arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc respectively. Zinc is the largest contributor to this fraction, while cadmium is the least. Non-residual fraction takes very significant part of this fraction with individual PTM contributions of: arsenic (2.37 %), cadmium (0.13 %), chromium (6.08 %), mercury (0.05 %), manganese (19.89 %), nickel (1.91 %), lead (10.02 %) and zinc (59.55 %) as presented in Figure 2. All PTMs levels fall within the ranges of limits set by WHO/FAO (2001).

In the control site, the concentrations of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc in the exchangeable fraction are 2.37, 0.18, 8.92, 0.003, 29.17, 2.80, 14.09 and 34.04 mg/kg respectively with highest contribution from zinc and lowest from mercury. The levels of arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc in the oxidizable fraction are 5.04, 0.15, 5.23, 0.002, 20.5, 3.46, 6.18 and 8.83 mg/kg respectively. Manganese is the largest contributor to this fraction while mercury is the lowest. The levels of PTMs (mg/kg) in the acid soluble fraction are: arsenic (2.24), cadmium (0.07), chromium (5.42), mercury (0.002), manganese (13.38), nickel (0.46) lead (1.57) and zinc (23.98). Zinc had the highest, while mercury had the lowest. In acid reducible fraction, the levels of PTMs (mg/kg) are 1.57, 0.05, 3.93, 0.001, 9.40, 0.29, 0.88 and 8.20 with highest contribution from zinc while the lowest from mercury. The levels of PTMs in residual fractions are: arsenic (0.34 mg/kg), cadmium (0.09 mg/kg), chromium (1.09 mg/kg), mercury (0.001 mg/kg), manganese (3.40 mg/kg), nickel (1.45 mg/kg), lead (0.31 mg/kg) and zinc (10.42 mg/kg). Zinc had the highest level in the residual fraction, while mercury was had lowest. Very significant amount of heavy metals are associated with non-residual fraction. The values of each PTM are within the WHO/FAO (2001) limits of 20.00, 3.00, 100.00, 2.00, 2000.00, 50.00, 50.00, and 300.00 for agricultural soils. The percentage contributions of each metal are 5.28, 0.21, 11.06, 0.004, 34.11, 3.30, 10.70 and 35.33 % for arsenic, cadmium, chromium, mercury, manganese, nickel, lead and zinc respectively (Figure 2).

Generally, the sequential extraction showed that all the PTMs examined are considerably associated with non-residual fractions in all the sampling locations. This implies that all the PTMs investigated in the study area are highly mobile in the soils. It has also been reported that PTMs released by anthropogenic sources, such as mining operations, are highly mobile in the soil environment and have a greater potential to harm the environment and humans than those released from geogenic sources (Kaasalainen & Yli-Halla, 2003; Kumar et al., 2003).
The levels of all PTMs in both residual and non-residual fractions were found to be significantly (p ≤ 0.05) higher than the rest of the locations. This is followed by farmland, while the lowest value was recorded at the control site. High values of PTMs in the active mining and farmland sites compared to the control site could be due to the fact that mining activities and farming involve the excavation of deep soil, which exposes previously undisturbed PTMs that were already buried beneath the soil’s surface, increasing their concentration level in the soil (Aliu et al., 2016; Ling et al., 2019; Adewumi & Laniyan, 2020; Hai et al., 2021).

Moreover, the variation of PTM concentrations across the sampling locations could be due to a number of factors, including changes in physico-chemical parameters, soil mineralization, and the application of fertilizer (Sungur et al., 2020; Jalali et al., 2023). Based on this findings, it is evident that all the PTMs investigated in the study area are highly mobile in the soil and can be bioavailable and hence toxic in the environment. Rodríguez et al. (2009), Liu et al. (2016) and Vuong et al. (2022) reported similar observations that PTMs from mining areas are associated with non-residual fractions. The speciation of PTMs in soil is regulated by soil pH, organic matter level, redox potential, and the presence of other elements. For example, in acidic soils such as those found in this study area (pH 6.05-6.26), as reported in our previous studies (Badamasi et al., 2022), heavy metals tend to be more soluble and mobile, which can increase their bioavailability and toxicity to plants and animals (Elijah et al., 2020; Ogunwale et al., 2021).

Therefore, understanding the speciation of PTMs is important because it affects their mobility, bioavailability and toxicity in the environment (Chu et al., 2010; Vuong et al., 2022). By identifying the mineral components in which each metal has the greatest affinity, speciation assists in understanding the behavior of the metals in relation to their environment (Monterroso et al., 2014; Ogunwale et al., 2021). Mobility and bioavailability are regulated by dynamic equilibrium between the concentrations of these fractions instead of total metal concentrations. Therefore, measuring total PTMs levels in soil samples is inadequate for evaluating probable mobility, bioavailability and consequently, the toxicity of PTMs in the soil (Ahn et al., 2020; Kaninga et al., 2020).
Figure 2. Distribution (%) of PTMs in residual and non-residual fractions in the soils of Riruwai mining area

Table 1. Speciation Pattern of Some Heavy Metals (mg/kg) in Soil

<table>
<thead>
<tr>
<th>Metals fractions</th>
<th>As</th>
<th>Cd</th>
<th>Cr</th>
<th>Hg</th>
<th>Mn</th>
<th>Ni</th>
<th>Pb</th>
<th>Zn</th>
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<tbody>
<tr>
<td><strong>Active Mining site</strong></td>
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<tr>
<td>F1</td>
<td>7.49 ± 1.55</td>
<td>4.31 ± 0.23</td>
<td>3.90 ± 0.09</td>
<td>0.29 ± 0.15</td>
<td>90.61 ± 9.55</td>
<td>1.05 ± 0.02</td>
<td>21.23 ± 4.23</td>
<td>18.23 ± 5.66</td>
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<tr>
<td>F2</td>
<td>3.55 ± 0.23</td>
<td>1.09 ± 0.31</td>
<td>23.22 ± 7.11</td>
<td>0.17 ± 0.05</td>
<td>104.07 ± 10.00</td>
<td>5.44 ± 1.23</td>
<td>104.01 ± 17.04</td>
<td>882.05 ± 21.10</td>
</tr>
<tr>
<td>F3</td>
<td>6.38 ± 2.01</td>
<td>0.01</td>
<td>12.51 ± 5.12</td>
<td>0.07 ± 0.08</td>
<td>90.61 ± 9.55</td>
<td>1.05 ± 0.02</td>
<td>21.23 ± 4.23</td>
<td>18.23 ± 5.66</td>
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<tr>
<td>F4</td>
<td>2.10 ± 0.09</td>
<td>0.04</td>
<td>11.28 ± 5.15</td>
<td>0.07 ± 0.05</td>
<td>104.07 ± 10.00</td>
<td>5.44 ± 1.23</td>
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<td>882.05 ± 21.10</td>
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<tr>
<td>F5</td>
<td>1.05 ± 0.07</td>
<td>0.02</td>
<td>12.77 ± 6.31</td>
<td>0.05 ± 0.08</td>
<td>90.61 ± 9.55</td>
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<td>18.23 ± 5.66</td>
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<tr>
<td><strong>Farmland</strong></td>
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Values are mean ± standard deviation (n = 3). F1 = Exchangeable fraction, F2 = Oxidizable fraction/ bound to organic matter, F3 = Acid soluble fraction/ bound to carbonate, F4 = Acid reducible fraction/ bound to Fe-Mn oxides, F5 = Residual fraction/ bound to silicates.

Conclusion

In this study, the speciation of PTMs from the mine-impacted soils of the Riruwai mining area of Kano State, Northern Nigeria, was investigated. The findings of the study revealed that all PTMs studied in all locations were found to be considerably associated with non-residual fractions and hence highly mobile and potentially toxic. The values of PTMs were found to vary with the sampling locations, with active mining sites significantly (p ≤ 0.05) recording the highest value, followed by farmland, while the lowest value was reported at the control site. Therefore, a human biomonitoring study should be carried out on the residents of the mining area in order to evaluate the degree of PTMs transfer to humans.
Acknowledgement
The authors would like to thank Tertiary Education Trust Fund (TETFund) Nigeria for providing funding for this study. The authors are grateful to Mal. Mansir Muhammad Riruwai, Mal. Yaro Jinjiri, and Musa Shu’ailbu for their invaluable assistance during sample collections and reconnaissance surveys.

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