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Assessment of Heavy Metal Speciation and Mobility in By-Products from Wastewater Treatment Plant in Ouagadougou

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Abstract

The wastewater treatment plant (WWTP) generates by-products such as treated wastewater (TWW) and dried sewage sludge (DSS). This study assesses the speciation and mobility of metals in dry sewage sludge collected in 2022 and 2024 using a three-step sequential extraction based on the BCR method. The highest total metal concentration was Mg, followed by Zn > Cu > Mn > Cr > Ni for both years, indicating a relatively stable distribution over time. It suggests that the sampling year does not significantly affect the chemical stability or binding forms of metals in sewage sludge. Mg and Mn were predominantly associated with the exchangeable and acid-soluble fraction (F1) in both years. Cu and Cr were mainly concentrated in the oxidizable fraction (**F**3). Ni showed a relatively uniform distribution across fractions and years, with a consistent proportion in the oxidizable fraction. The undetectable Cd, Co, Hg, and Pb in raw and treated wastewater indicate a low industrial contribution of wastewater to the waste treatment plant. The sewage sludge analysis showed high levels of total organic matter, phosphorus, and nitrogen, suggesting its potential use as an agricultural soil amendment. The results provide important insights into metal behavior, informing safe sludge management and its potential use in agriculture.

Keywords

Heavy Metals, Mobility, Sequential Extraction, Sewage Sludge, Wastewater

1. Introduction

The reuse of raw wastewater as well as treatment by-products is a common practice, particularly in countries with arid or semi-arid climates, and in urban vegetable farming and horticulture sectors. This trend is mainly driven by drought, insufficient irrigation water, and declining soil fertility, thereby promoting the use of wastewater and treatment by-products in urban agriculture [1]. In Ouagadougou, the capital of Burkina Faso, vegetables and cultivated trees are largely irrigated using water from dams, wells, and boreholes, and particularly with raw wastewater and by-products from the Kossodo wastewater treatment plant [2]. The water entering the wastewater treatment plant (WWTP) originates from various sources, including industry, commerce, and the hospitality sector, and carries a range of pollutants, among which heavy metals are generally not efficiently removed by conventional treatment processes. This process is to achieve stabilization, dehydration, and reduction of pathogens in the product from the treatment of sewage sludge. Thus, the reuse of this treated wastewater necessitates to enhance the treatment process to prevent any environmental and human risk [3] [4]. In many countries, wastewater treatment produces two main by-products: treated wastewater (TWW) and dried sewage sludge (DSS), which have shown great interest in agriculture because of their important content in organic matter, phosphorus and nitrogen and promoted as an important organic substitute for agricultural chemicals [1] [5]-[7]. The reuse of treated wastewater and sewage products has become a widespread practice to address agricultural challenges, especially in regions with arid or semi-arid climates facing severe water deficiency [3] [8]. Study has shown that in Ouagadougou, especially in the horticultural sector, 75% of producers use treated wastewater [2]. The most important concern is based on the presence of some heavy metals, which were not taken up during the wastewater treatment and can provoke phytotoxic problems and pose a risk to human health [3] [7]. The risk of heavy metals is evaluated due to their non-biodegradable nature, their toxicity, and their chemical form (speciation), which characterizes their mobility. A given metal can be essential and toxic, depending on the dosage, exposure levels, organism, and recipient population [9] [10]. Due to their non-biodegradable nature, environmental persistence, toxicity, and chemical forms (speciation) that determine their mobility, heavy metals can be absorbed by cultivated plants [11]. This accumulation in plant tissues may contaminate the food chain and pose a risk to human health. Numerous studies have focused on evaluating the quality of products derived from wastewater treatment and sewage sludge [1] [2] [12]. These works focused mainly on determining organic pollutant levels, physicochemical parameters (pH, temperature, conductivity, turbidity), pollution parameters (BOD, COD, TSS), and the presence of pathogenic microorganisms (bacteria, viruses, parasites). These analyses have made it possible to better characterize the health aspects related to the use of these sewage products in agriculture. However, a significant limitation is that most of these studies neglect to analyze the speciation of heavy metals present in these products. For a comprehensive assessment of environmental impacts, it is crucial not to limit oneself to measuring total metal concentrations, but also to consider their chemical speciation and mobility in the soil. In this context, this study aims to evaluate the fractional distribution of heavy metals in the sewage product of Kossodo wastewater treatment plant in the city of Ouagadougou, Burkina Faso, with a view to their reuse in agriculture.

2. Materials and Methods

2.1. Description of the Study Sites

The Kossodo wastewater treatment plant (WWTP) (**Figure 1**), situated on the north-eastern outskirts of Ouagadougou, Burkina Faso, within the city's industrial zone (12.4561˚N, 1.4840˚W), processes both domestic and industrial effluents using a conventional activated sludge treatment system, followed by sedimentation and sludge stabilization. Its principal by-products—primary and secondary sludge—are periodically dewatered and stored in drying beds before their final disposal or further management.

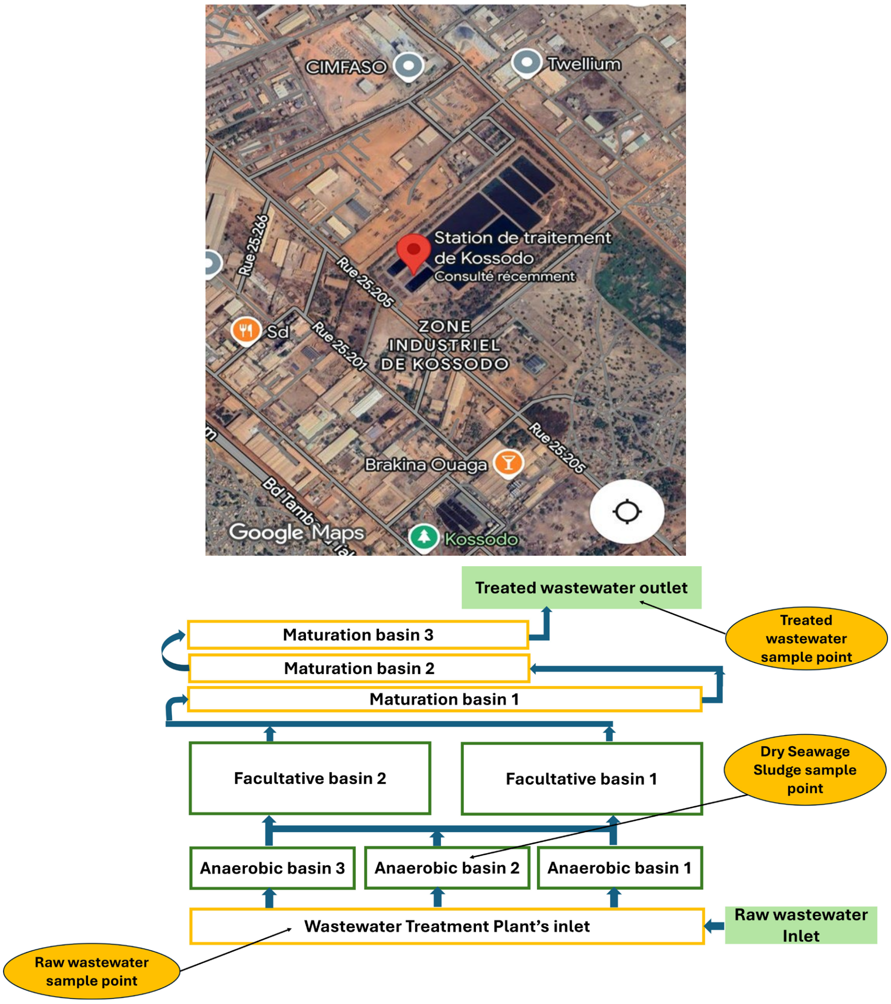

Figure 1. Localization of study site.

2.2. Experimental Design and Sample Collection

Three types of samples were collected: raw wastewater (RWW), treated wastewater (TWW), and dried sewage sludge (DSS) (**Figure 1**). Raw wastewater samples were collected at the plant's inlet, immediately after the homogenization manhole in the distribution channel, while treated wastewater samples were collected at the outlet, downstream of the final maturation basin. The dried sewage sludge was collected in polyethylene bags from anaerobic basin 2—which was undergoing desludging at the time—using polyethylene bags (**Figure 1**).

The RWW and TWW samples were performed in 2024, with triplicate samples collected at each sample. Wastewater samples were collected in 0.5 L polyethylene bottles, carefully labeled and stored in an ice-cooled container at 4˚C and transported to the laboratory, where they were stored at 4˚C until analysis. All bottles were washed in 1% (v/v) nitric acid, rinsed with ultrapure water, and oven dried. Dried sludge samples of 2024 and 2022 were collected in plastic bags of 30 × 50 cm. Physicochemical parameters of RWW and TWW, including pH, temperature, and conductivity, were measured *in situ*.

2.3. Sequential Extraction and Total Heavy Metals Concentration in DSS

A sequential extraction was performed following the Community Bureau of Reference (BCR) protocol to distinguish the different chemical forms of metals and to relate their distribution and mobility to environmental processes. In this study, three fractions were analyzed: the exchangeable and acid-soluble fraction (F1), the reducible fraction (F2), and the oxidizable fraction (F3). The residual fraction (F4) was subsequently derived by subtracting the sum of the three extracted fractions from the total metal concentration in the sample. The procedure applied in this study follows the protocol described in [13]. In summary, 1 g of dried sludge was sequentially treated with acetic acid (F1), hydroxylamine hydrochloride at pH 1.5 (F2), and hydrogen peroxide followed by ammonium acetate at pH 2.0 (F3). Each extraction step was carried out for 16 hours (overnight) at room temperature using a mechanical orbital shaker. The extract was separated from the solid residue by centrifugation (20 minutes at 3000 rpm), and the resulting supernatant was transferred into polypropylene centrifuge tubes and stored at 4˚C. The solid residue was then washed with 20 mL of ultrapure water, shaken for 15 minutes, and centrifuged under the same conditions.

The total heavy metal concentration in dried sewage sludge was analyzed by acid digestion in a microwave oven with a solution of 2.5 mL concentrated nitric acid HNO3, 7.5 mL concentrated chloridric acid HCl and 0.5 mL of hydrogen peroxide H_2O_2. The digested samples are filtered with a Whatman paper. The filtrate was diluted in a centrifuge tube with 50 mL of ultrapure water. The blank samples were prepared following the same procedure, with ultrapure water, 1% HNO_3 and 1% HCl.

The total heavy metal concentration and heavy metal concentration in each fraction were determined following standard methods from the US EPA 2007 using a microwave plasma atomic emission spectrometer (MP-AES), Agilent Model 4210. Three replicate measurements were made for each sample and the final concentration value for the sample was the average of the three measurements. The detection limits of Cr, Cu, Mg, Mn, Ni, and Zn were 0.005, 0.1, 5, 5, 10, and 1 mg/kg, respectively.

2.4. Physicochemical Analysis of RWW, TWW and DSS

In this study, key dissolved inorganic ions (NH_4^+ , NO_3^- , PO_4^{3-} , SO_4^{2-}), heavy metals and biological indicators (DCO, BOD_5, and TSS) were analyzed in both raw and treated wastewater. Sequential extraction of heavy metals in DSS was performed at the Physico-Chemistry and Electrochemistry Laboratory of Joseph KI-ZERBO University, while metals quantification in all samples was carried out at the SENEXEL laboratory. TSS was determined gravimetrically after filtration, and BOD_5 was measured using an OxiTop system following five days of incubation. DCO and dissolved inorganic ions (NH_4^+ , NO_3^- , PO_4^{3-} , SO_4^{2-}) were quantified by the molecular absorption spectrometry (DR3800 spectrophotometer).

The same procedure used for dissolved inorganic ions in raw and treated wastewater, was used to determine nitrate and phosphate in dried sludge, after been mineralized using Kjeldahl method for phosphate analysis, and treated with potassium chloride (KCl) solution followed by sulfuric acid (H_2SO_4)-salicylic acid ($C_7H_6O_3$) reagent, and finally sodium hydroxide (NaOH) for nitrate analysis. The organic matter in dried sludge was measured by the Walkley and Black method (1934). A 0.5 g subsample of dried sludge, sieved at 0.5 mm, was treated with 2.5 mL of potassium dichromate ($K_2Cr_2O_7$) and 5 mL concentrated sulfuric acid (H_2SO_4).

The heavy metals were measured with a microwave plasma atomic emission spectrometer (MP-AES), Agilent Model 4210. The analysis was performed as described by [14].

3. Results and Discussion

3.1. Physicochemical Parameters of RWW, TWW and DSS

The physicochemical parameters of dried sewage sludge (DSS) showed that the total organic matter, a key indicator of sludge composition and its potential for valorization, accounted for 5.97% of the dry matter (DM). The concentrations of nitrates and total phosphorus were 13 mg/kg and 161 mg/kg, respectively. These nutrients are essential for plant growth and contribute to the improvement of soil properties.

The physicochemical parameters of raw and treated wastewater are shown in **Figure 2**. pH was similar for raw wastewater (7.6) and for treated wastewater (8.1). The electrical conductivity (CE) was higher in the treated wastewater compared to raw wastewater (2118 µS/cm and 1929 µS/cm, respectively). These ions may include heavy metals released in more soluble (ionic) forms, which conduct electricity more effectively [15]. The turbidity values were 283.9 NTU in RWW and 228.9 NTU in TWW.

Ammonium concentrations were constant throughout the treatment process, with a value of 90 mg/L in RWW and 89 mg/L in TWW (**Figure 3**). This weak variation suggests limited nitrification efficiency within the biological treatment units. This is affected by a pH between 7 - 8.5 as determined in this study. In contrast, nitrate, and phosphate concentrations increased after the treatment process, reaching 176 mg/L of NO_3^-, and 39 mg/L of PO_4^{3-} in the TWW, compared to 100 mg/L NO_3^-, and 31 mg/L PO_4^{3-} in the RWW (**Figure 3**). This trend could be attributed to the inhibitory effect of certain metals on nitrate removal. Metal ions such as Cu^{2+} and Ni^{2+} are known to interfere with the activity of denitrifying bacteria, thereby disrupting the denitrification pathway and leading to the accumulation of NO_3^- in treated wastewater [16] [17]. The increase in nitrate concentrations, together with the stable ammonium levels, indicates that nitrification is occurring within the treatment process but is offset by the simultaneous production of ammonium resulting from the mineralization of organic nitrogen during treatment. Regarding organic pollution indicators, a decrease was observed in both Chemical Oxygen Demand (COD) and Biochemical Oxygen Demand (BOD_5), from 655 to 638 mg O_2/L and from 560 to 540 mg O_2/L, respectively. The BOD_5/COD ratio at the outlet (0.846) indicates that the raw wastewater remains highly biodegradable. This result also reflects the predominance of easily degradable organic matter, characteristic of domestic wastewater with limited industrial contribution.

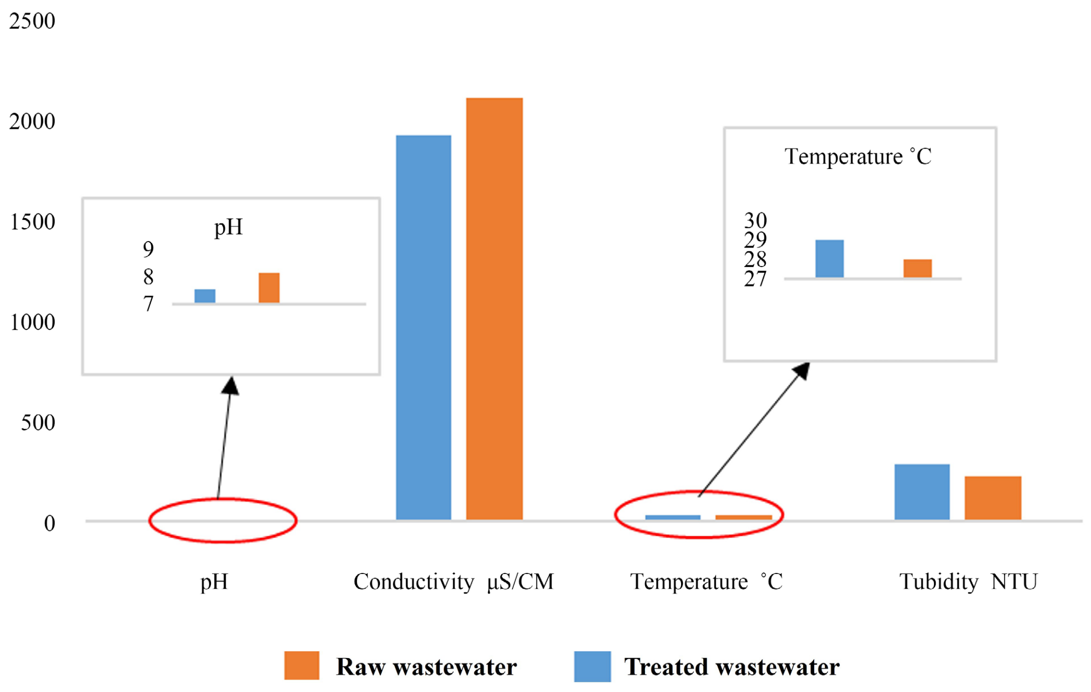

Figure 2. Physicochemical parameters (temperature, pH, electrical conductivity, and turbidity) of raw and treated wastewater.

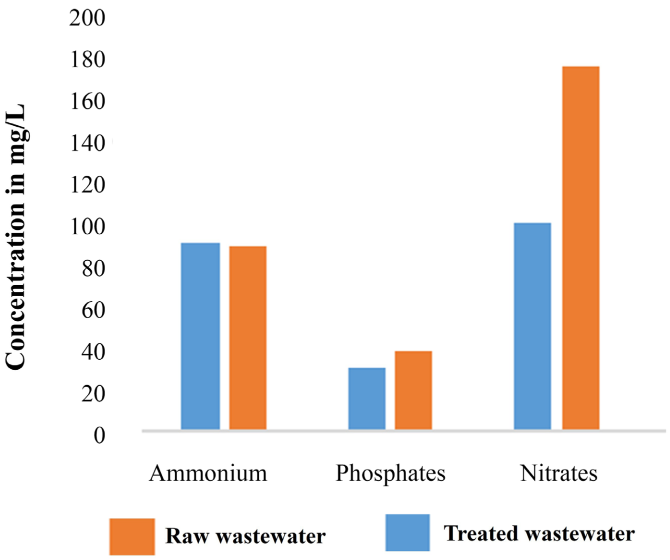

Figure 3. Concentration of inorganic ions in raw and treated wastewater in wastewater treatment plant.

3.2. Heavy Metal Concentration Raw and Treated Wastewater

The concentrations of heavy metals in both raw and treated wastewater are presented in Figure 4. The detection limits of Cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), Mg, manganese (Mn), nickel (Ni), lead (Pb), and zinc (Zn)were 0.005, 0.05, 0.005, 0.05, 0.1, 0.01, 0.02, 0.005, and 0.02 mg/L, respectively. The concentration of Cd, Co, Hg, and Pb was below detection limit in both samples. This absence indicates limited industrial contributions of these metals to WWTP. These findings are consistent with previous studies conducted in West African urban wastewater systems (Ouedraogo *et al.*, 2019) [2].

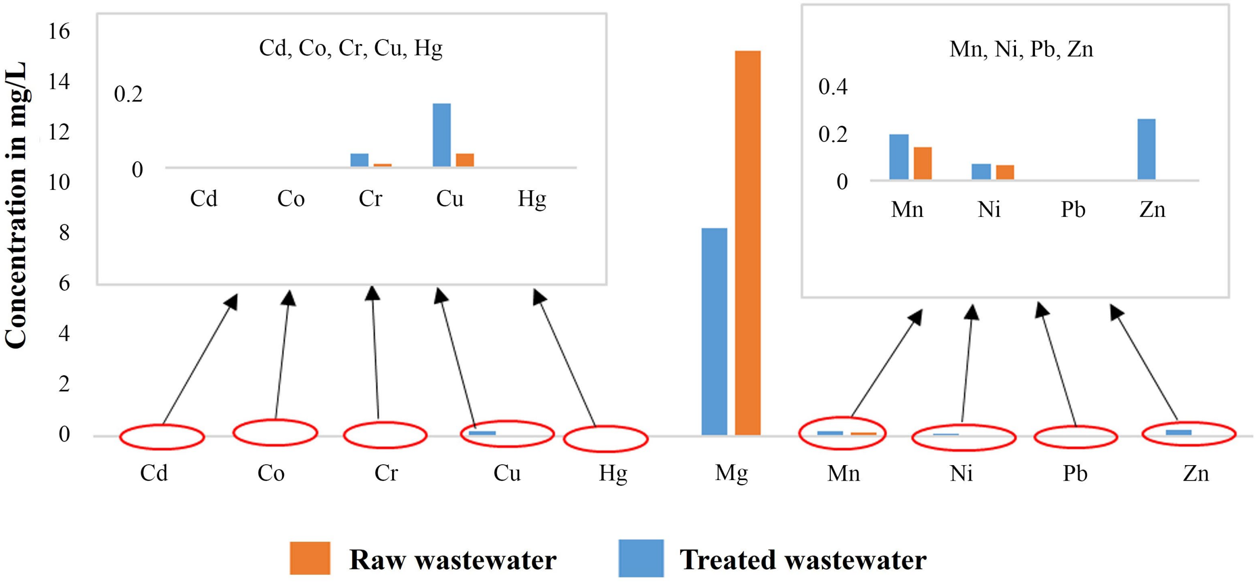

Figure 4. Concentrations of heavy metals in raw and treated wastewater from the Kossodo wastewater treatment plant.

Among all quantified elements, magnesium (Mg) exhibited the highest concentrations and was higher in treated wastewater (15.18 mg/L) than in raw wastewater (8.2 mg/L). In contrast, the concentrations of chromium (Cr) and manganese (Mn) decreased during treatment, from 0.04 mg/L to 0.01 mg/L for Cr and from 0.2 mg/L to 0.1 mg/L for Mn. Copper (Cu) and zinc (Zn), detected in raw wastewater at 0.2 mg/L and 0.3 mg/L, respectively, were not detected in the treated wastewater. This decrease in metal concentrations from the raw wastewater to the treated wastewater could be attributed either to the efficiency of the treatment process or to the precipitation of metals into insoluble forms (such as metal hydroxides), within the sludge. This precipitation is likely favored by basic pH (as it is found in this study), which promotes the formation of less soluble metal species that settle into the sludge phase.

3.3. Fractional Distribution of Heavy Metal in DSS

The trace metal (Cr, Cu, Mg, Mn, Ni, and Zn) concentration measured in dry sewage sludge collected in 2024 and 2022 is presented in **Table 1**. Regardless of the sampling year, Mg consistently exhibited the highest total concentration in the sludge, followed in descending order by Zn, Cu, Mn, Cr, and Ni. Total contents of Mg, Zn, Cu, and Mn were higher in DSS 2024 compared to DSS 2022, (Mg, 1882 mg/kg vs. 943 mg/kg; Zn, 855 mg/kg vs. 495 mg/kg; Cu, 374 mg/kg vs. 293 mg/kg, and Mn, 239 mg/kg vs. 180 mg/kg), while Cr, and Ni, were similar between the two years. The high phosphate levels observed in the DSS are likely to reduce the solubility of heavy metals in the sludge [18].

The sequential extraction performed on dry sewage sludge (DSS) collected in 2024 and 2022 (**Figure 5**) revealed consistent metal fractionation patterns, despite variations in total metal concentrations in both years. Mg and Mn were predominantly associated with the exchangeable and acid-soluble fraction (F1) in both years, indicating high mobility and potential bioavailability. In contrast, Cu and Cr were mainly concentrated in the oxidizable fraction (F3), reflecting their strong affinity for organic matter and sulfide phases. Zn exhibited contrasting behavior between the two years: while almost absent from the F1 fraction in DSS 2024, it was highly mobilizable in DSS 2022, with more than half of its total content occurring in F1. Nickel (Ni) showed a relatively uniform distribution across fractions and years, with a consistent proportion in the oxidizable fraction. Overall, the relative distribution of metals across the three fractions remained similar between the two sampling years, indicating that the year of sampling did not significantly influence the chemical stability or binding forms of the studied metals, suggesting that the sludge’s metal speciation is controlled primarily by its intrinsic physicochemical characteristics rather than temporal variations.

The sum of the three operationally defined fractions (F1 + F2 + F3) accounted for a large proportion of the total concentrations of Cu, Mg, and Mn, indicating that these metals are largely associated with labile, reducible, or oxidizable phases rather than being tightly bound within the mineral matrix. For Cu, the high proportion extracted (93% in DSS 2024 and 98% in DSS 2022) suggests that it is predominantly bound to organic matter and sulfides, consistent with its strong affinity for the oxidizable fraction (F3). Mg and Mn were mostly present in exchangeable, acid-soluble, and reducible fractions (F1 + F2), reflecting their association with more labile and potentially bioavailable forms. The slightly lower sum for Mg in DSS 2022 (58%) compared to DSS 2024 (93%) may be related to differences in sludge composition or organic matter content between the two years, but the overall pattern indicates that the majority of these metals are not present in the highly stable residual fraction (F4). The residual fraction (F4), representing metals not extracted by the first three steps, accounted for substantial proportions of Cr (62% in DSS 2024, 57% in DSS 2022), Ni (64% and 61%), and Zn (66% and 1%), highlighting that a large portion of these metals is tightly bound within the sludge matrix.

Table 1. Total concentration of heavy metal in dry sewage sludge (DSS) collected in 2024 and 2022.

Heavy metal	Cr	Cu	Mg	Mn	Ni	Zn
Limit of detection	0.005	0.1	5	5	10	1
			mg/kg			
DSS 2024	159	374	1882	239	67	855
DSS 2022	165	293	943	180	68	495

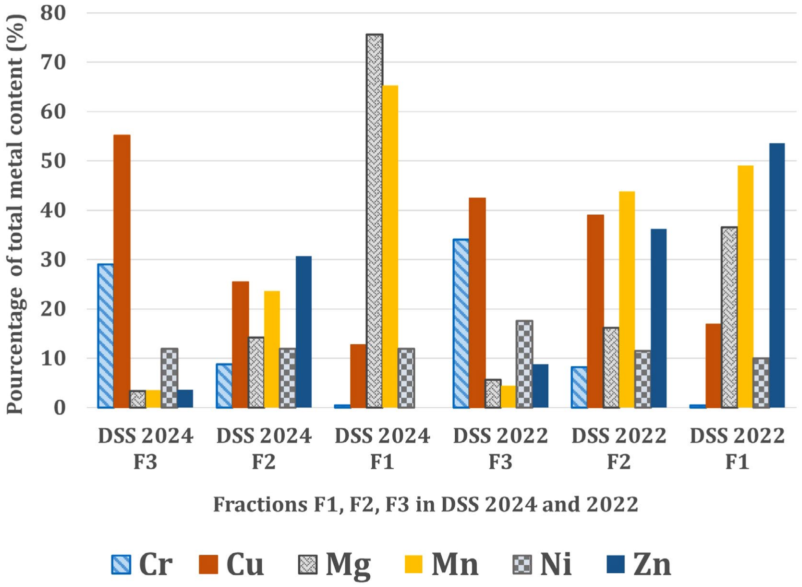

Figure 5. Distribution of metal fractions in dry sewage sludge (2024 and 2022).

4. Conclusion

The study aims to investigate the trace metal speciation and mobility in By-Products from Wastewater Treatment Plant in Ouagadougou. The present study highlights the fractionation of heavy metals in dry sewage sludge collected in 2022 and

2024. Despite minor differences in total metal concentrations, the distribution among fractions remained largely consistent over time. Magnesium, zinc, and copper were the most abundant metals, with Mg and Mn predominantly present in labile fractions (F1 + F2), whereas Cu and Cr were mainly concentrated in the oxidizable fraction (F3). Most metals, such as Cu, Mg, and Mn, are predominantly associated with the first three fractions (F1 + F2 + F3), accounting for 96%, 76%, and 95% of their total concentrations, respectively. The residual fractions (F4), calculated by difference, confirm strong immobilization of Cr and Ni. These. The relative distribution of metals across the three fractions remained similar between the two sampling years, indicating that the year of sampling did not significantly influence the chemical stability or binding forms of the studied metals, suggesting that the sludge's metal speciation is controlled primarily by its intrinsic physicochemical characteristics rather than temporal variations.

Authors' Contributions

YYMLD collected field samples, gathered general site information, and conducted literature reviews. SBMY conceived research ideas and wrote the first draft of the manuscript. IT supervised YYMLD to carry out the sample analysis. SBMY, SK and RT provided scientific supervision of study and coordinated the various analytical steps. All authors contributed to the writing and revision of the manuscript.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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