



# FRACTIONATION AND MOBILITY OF CADMIUM, LEAD, CHROMIUM AND COPPER IN THE VICINITY OF DYE PITS IN ZARIA CITY, NORTHERN NIGERIA



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**Abstract:** The sequential extraction provides information on mobility and stability of various metal species which can be used to evaluate the movement of such metals in soils. The effect of dye effluent on the fractions, spatial distribution, and mobility of cadmium (Cd), lead (Pb), chromium (Cr) and copper (Cu) was investigated in five vicinity of dye pits in Zaria city, northern Nigeria. Total concentrations of Pb, Cr and Cd recorded were at maximum values of 181.57, 228.57 and 40.55 mg kg<sup>-1</sup>, respectively. However, the concentration of Cu was within acceptable limit of 200 mg kg<sup>-1</sup> while concentrations of Pb, Cd, and Cr were above the threshold levels. The sequential extraction procedure was used to fractionate the soil into six operationally defined species. Speciation of the heavy metals indicated that 50-70% of the metals were associated with the resistant soil fraction; this represents heavy metals held in the crystal lattices of minerals with strong bindings and consequently are not easily released into the environment. However, the effect of chemicals in the dye effluent, under the influence of pH, could release these metals into the soil-exchange site thereby increasing the concentrations of labile Cd, Cr and Pb in soils which may consequently aggravate the potential hazard for the redistribution and translocation of these metals into the food chain and underground water.

**Keywords:** Heavy metals, metal mobility, metal speciation, soil contamination

## Introduction

The discharge of toxic effluents from various industries adversely affects water resources, soil fertility, aquatic organisms and ecosystem integrity. Contaminants in soil can easily be taken up by plants along with nutrients contained in the soil which when consumed by man may pose a risk hazard (Abdu *et al.*, 2011b). In Africa, over 50 percent of the population is rural and directly reliant on locally grown food (Brown, 2001). This farming can be sustainable if farmers invest in fertilizer, and in soil and water conservation, but these pressures further expose this valuable resource to degradation (Brown, 2001).

In a developing country like Nigeria where there is virtually little or no waste treatment and disposal regulations, waste are disposed indiscriminately which increase the pollution level (Ogungbemi, 2002). The textile and dyeing industries discharge large volume of waste water after the dyeing process, this have been reported to alter the colour and qualities of soil and water bodies posing serious environmental problems (Khan *et al.*, 1995). Industrial effluents consist of organic compounds along with inorganic complexes and other non-biodegradable substances such as heavy metals.

Heavy metals particularly, Pb, Cr, Cd and Cu are widely used for the production of colour pigments in textile dyes. These heavy metals which are transferred to the environment are potentially toxic and can bio-accumulate in the human body, aquatic life, natural water bodies and also possibly trapped in the soil (Mathur *et al.*, 2005).

The total content of heavy metals is made up by their different forms (fractions), which can be related to chemical species, as well as to their different solubility and mobility in soil environment. Sequential extraction of elements is used to identify these fractions which enable us determine the geochemical species of metals in soil.

Zaria is a major city in Kaduna state which is one of the most developed states in northern Nigeria and dyeing activities are some of its dominating activities. It is on record that the first textile industry in Nigeria was established in Kaduna

(Jibrin, 2004). Since dyeing activities is a very important economic activity among Zaria city inhabitants, their method of improper disposal is likely to have adverse effect on the ecosystem, thereby affecting the health status of the populace. This is as a result of heavy metal and other chemical contaminants deposition from these wastes into the soil. Activity of heavy metals in a complex soil matrix is dependent on the chemical species of the metals present. Thus, knowledge about the chemical speciation of heavy metals provides information on the bioavailability and toxicity of such metals (Abdu, 2010).

## Material and Methods

### Study area

Zaria lies between latitude 11° 4' 0" N and longitude 7° 42' 0" E and located on a plateau at a height of about 2200 feet above sea level in center of northern Nigeria and more than 400 miles (634.71 km) away from the sea (Yahya *et al.*, 2011). It possesses a tropical continental climate with the tropical climate more pronounced during the dry season, especially in December and January. The city lies within a region which has a tropical savanna climate with distinct wet and dry seasons (Maiwada and Renne, 2007). The mean annual rainfall is 1060 mm (Owonubi *et al.*, 1991).

A total of five locations were selected amongst the dyeing centers with one location selected from a non-dyeing centre which serves as a control. The locations selected are KofarKuyanbana (KK), KofarJatau (KJ), Kusfa, Mabuga, AngwanIya (AI) and KofarGayan (KG) (the control). These locations represent the major dyeing centers in Zaria city.

### Soil sampling and preparation

Soil samples were collected in the immediate vicinity of dye pits. Composite samples consisting of 15 auger points were collected at a depth of 0-15 cm. The samples were collected at < 1 m radius from the dye pit. The soil samples were air dried, thoroughly homogenized, crushed with porcelain pestle and mortar and sieved through 2 mm mesh size for physicochemical analysis (Table 1).

Table 1: Concentration of total heavy metals in soils receiving dye effluents in Zaria city (Mean±SD)

Location	Cu (mg/kg)	Cr (mg/kg)	Pb (mg/kg)	Cd (mg/kg)
KG	27.29±1.18 <sup>b</sup>	21.10±7.00 <sup>d</sup>	120.60±23.00 <sup>c</sup>	40.55±5.17 <sup>a</sup>
Kusfa	29.04±5.17 <sup>b</sup>	47.22±9.23 <sup>c</sup>	127.37±23.01 <sup>c</sup>	13.07±2.45 <sup>f</sup>
KJ	32.20±4.27 <sup>ab</sup>	181.43±42.5 <sup>ab</sup>	181.52±32.21 <sup>a</sup>	19.45±5.67 <sup>d</sup>
Mabuga	31.24±7.27 <sup>ab</sup>	228.57±18.4 <sup>a</sup>	158.54±35.23 <sup>b</sup>	23.56±6.34 <sup>c</sup>
AI	42.47±9.10 <sup>a</sup>	154.65±47.2 <sup>ab</sup>	155.83±32.11 <sup>b</sup>	31.24±7.23 <sup>bc</sup>
KK	34.25±8.20 <sup>ab</sup>	99.95±23.4 <sup>b</sup>	181.57±20.23 <sup>a</sup>	35.48±9.34 <sup>b</sup>

Mean with the same alphabets along the column are not different at P≥0.05

**Analytical procedures**

Soil pH was determined using a glass electrode pH meter, Particle size analysis was determined by the hydrometer method as described by Gee and Bauder (1986). Organic carbon (OC) was determined by the wet oxidation method as described by Nelson and Sommers (1982). Total Nitrogen was determined by the method of Bremner and Mulvaney (1982). Available phosphorus was determined following the procedure described by IITA (1979) using the Bray- 1 extraction method (Bray and Kurtz, 1945).

**Total Pb, Cd, Cr and Cu**

Total concentration of Cd, Pb, Cu and Cr in the soil was determined in Atomic Absorption Spectrophotometer (AAS) (Model AA 6680, Shimadzu, Kyoto, Japan) following digestion of a air- dried and finely ground soil samples with a mixture of HNO<sub>3</sub>, HF and HCl (Lim and Jackson, 1986). The digested soil sample was set aside to cool and then filtered through Whatman no. 42 filter paper. Total concentrations of Pb, Cd, Cr and Cu were determined in AAS.

**Sequential extraction of metals**

Thesequential extraction procedure modified from Tessier *et al.*, (1979) was used to fractionate the soil into six operationally defined species as follows:

- (i) **Water soluble (WS):**Two grams of air-dried soil was weighed into plastic bottle, 25 ml of distilled water was added and extracted at room temperature for 2 h. The mixture was centrifuged, decanted and filtered to get the supernatant into a clean plastic vial and the concentration of Cu, Pb, Cr and Cd was determined in AAS.
- (ii) **Exchangeable (EXCH):** The residue from (i) above was extracted at room temperature for 30 min with 20 ml of 1M NH<sub>4</sub>OAc solution (pH 7.0) with continuous agitation. The filtrate was decanted into clean plastic vial and the concentration of Cu, Pb, Cr and Cd was determined in AAS.
- (iii) **Bound to carbonates (CARB):**The residue from (ii) above was extracted by adding 20 ml 1M NaOAc at room temperature adjusted to pH 5.0 with acetic acid (HOAc). Continuous agitation was maintained for 5 h. The mixture was centrifuged, decanted and filtered to get the supernatant in to a clean plastic vial and the concentration of Cu, Pb, Cr and Cd was determined in AAS.
- (iv) **Bound to Fe and Mn Oxide (Fe-MnO):**The residue from (iii) above was extracted with 20 ml of 0.04 M NH<sub>2</sub>OH-HCl in acetic acid. The experiment was performed at 96°C by heating in water bath for 6 h with occasional agitation. The mixture was centrifuged, decanted and filtered to get the supernatant into a clean plastic vial and the concentration of Cu, Pb, Cr and Cd was determined in AAS.
- (v) **Bound to organic matter (OM):** Five milliliters of 0.1N HNO<sub>3</sub> and 10 ml of 30% H<sub>2</sub>O<sub>2</sub> was added to the residue from (iv) above, and the mixture was heated to 85 °C for 5 hours with occasional agitation. After cooling, 15ml of 3.2M NH<sub>4</sub>OAc in 20% (v/v) HNO<sub>3</sub> was added and agitated continuously for 30 min. The addition of NH<sub>4</sub>OAc was designed to prevent adsorption of extracted

metals onto the oxidized sediment. The mixture was centrifuged, decanted and filtered to get the supernatant into a clean plastic vial and the concentration of Cu, Pb, Cr and Cd was determined in AAS.

- (vi) **Residual fraction (RES):** The residual fraction was calculated by subtracting the sum of the fractions (i-v) from the total determined above.

$$RES = MT - (WS + EXCH + CARB + Fe - MnO + OM)$$

$$MT = TOTAL METAL CONCENTRATION$$

**Statistical analysis**

Cadmium, Pb, Cr and Cu mobility were assessed based on the weakly bound (mobile: F1 + F2) and the moderately mobile (F3) fractions (Abdu *et al.*, 2012; Kabala and Singh, 2001). The relative mobility index was calculated as a mobility factor (MF) Kashem *et al.*, 2007) using the following equation:

$$MF = (F1 + F2 + F3)/(F1 + F2 + F3 + F4 + F5 + F6) \times 100$$

The numerator represents the mobile to moderately mobile fractions which included the water or readily soluble, the exchangeable, and carbonate-bound fractions. Depending on the prevailing soil conditions, metals in exchangeable and carbonate fraction can be very active and bioavailable and can transform to the acid soluble fraction when the pH of the soil increases (Li *et al.*, 2010).

**Results and Discussion**

**Total Cu, Cr, Pb and Cd Concentrations in the Soil**

The total heavy metals concentration showed wide variations. The lowest concentration of Cu (27.29 mgkg<sup>-1</sup>) was recorded at KofarGayan while the highest concentration of 42.47 mgkg<sup>-1</sup> was recorded at AngwanIya (Table 2), this concentrations was below the threshold levels given by the regulatory bodies. This may be due to minimal use of copper in the dye chemical composition. Chromium concentration ranged from 21.10 mgkg<sup>-1</sup> to 228.57 mgkg<sup>-1</sup>. Highest concentration was recorded at Mabuga and the lowest at KofarGayan. These values are very much high when compared with standard thresholds for arable soils. This shows that more dye activities occurs at Mabuga. Most of the Cr values obtained were higher than that reported by Abdu *et al.* (2011a) in waste water irrigated garden soil in Kano, northern Nigeria. There is possibility of Cr contamination in the dyeing area, since Cr is one of the dye chemical constituent and 2-10% of the dye is loss in the soil (Welham, 2000). The non biodegradable nature of Cr is responsible for its persistence in the soil; it undergoes transformation in to various mobile forms before ending in environmental sink.

However, the highest Pb concentration (Pb 181.57 mgkg<sup>-1</sup>) was recorded at KofarKuyanbana while the lowest concentration of 120.60 mgkg<sup>-1</sup> was recorded along KofarGayan. Lead concentration was also higher than that reported by Abdu (2010) and (Nwachuku *et al.*, 2010). Cadmium concentration was far above the limit, this was worrisome especially in the control. Highest concentrations of 40.55 mgkg<sup>-1</sup> at KofarGayan while the lowest concentration of 13.07 mgkg<sup>-1</sup> was recorded at Kusfa; these values are

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extremely higher than the 3.0 mgkg<sup>-1</sup> acceptable Cd concentrations in arable lands. The effluent released in the surrounding may be responsible for high Cd content; this corroborated the findings of Malarkodi *et al.* (2007) in contaminated soil with textile effluent in India. The observed Cd content may also be attributed to the various chemical

composition of the dye materials used in Zaria city. The very high Cd concentration observed in the control suggest that that dye effluent was not the primary source of Cd contamination, it might be as a result of other anthropogenic activities taking place or it may be associated with soil parent materials.

Table 2: Correlation coefficient matrix between the different Cu and Cr fractions

		Cr					
		WS	EXCH	CARB	OXIDE	OM	RESI
Cu	WS	1	-0.298	-0.342	-0.142	-0.229	0.163
	EXCH	0.938**	1	0.993**	0.799	0.987**	-0.980**
	CARB	0.988**	0.959**	1	0.792	0.990**	-0.976**
	OXIDE	0.790	0.638	0.745	1	0.848*	-0.868*
	OM	0.963**	0.983**	0.985**	0.0673	1	-0.997**
	RESI	-0.981**	-0.983**	-0.992**	-0.738	-0.994**	1

The lower left-hand section is correlation coefficient for Cu while the upper, right-hand part is correlation coefficient for Cr; \*, \*\* Significant at p $\geq$ 0.05 and 0.01, respectively.

Table 3: Correlation coefficient matrix between the different Cd and Pb fractions

		Pb					
		WS	EXCH	CARB	OXIDE	OM	RESI
Cd	WS	1	0.158	0.410	0.680	-0.359	-0.530
	EXCH	0.938**	1	-0.238	0.804	-0.233	0.203
	CARB	0.988**	0.959**	1	0.005	-0.406	-0.898*
	OXIDE	0.790	0.638	0.745	1	-0.455	0.059
	OM	0.963**	0.983**	0.985**	0.673	1	0.026
	RESI	-0.981*	-0.992**	-0.992**	-0.738	-0.994	1

The lower left-hand section is correlation coefficient for Cd while the upper, right-hand part is correlation coefficient for Pb; \*, \*\* Significant at p $\geq$ 0.05 and 0.01, respectively.

### Metal Fractionation in Soil

#### Chromium fractions

Chromium is mostly abundant in the residual fraction in all the locations. Up to 70% of Cr was in the residual fraction in KofarGayan (Fig. 1). The high percentage of a metal in the residual fractions in a soil indicates a lithogenic origin of the metal contaminants (McLean and Bledsoe, 1992). However, in our soil where high discharge of dye effluents containing high concentration of Cr, anthropogenic source of Cr could not be ruled out. Chromium in the water soluble fractions ranged from 2-4% across the locations with KofarGayan having the lowest value and Kusfa having the highest value.

The exchangeable fractions ranged from 2-10% with the lowest percentage observed at KofarGayan while KofarJatau and KofarKuyanbana both have the highest value. The relatively lower pH (5.4) in KG might be responsible for low solubility of Cr and hence its redistribution into the more resistant fraction. Carbonate-bound fraction has the highest percentage of 9% in Mabuga and AngwanIya, while the highest percentage of organic matter-bound fraction (19%) was found at Kusfa and the lowest fraction was found at Mabuga. The relatively high value of Cr in the exchangeable fractions at KofarKuyanbana and KofarJatau (10%) in both locations shows that there is possibility of Cr being mobile in this environment.

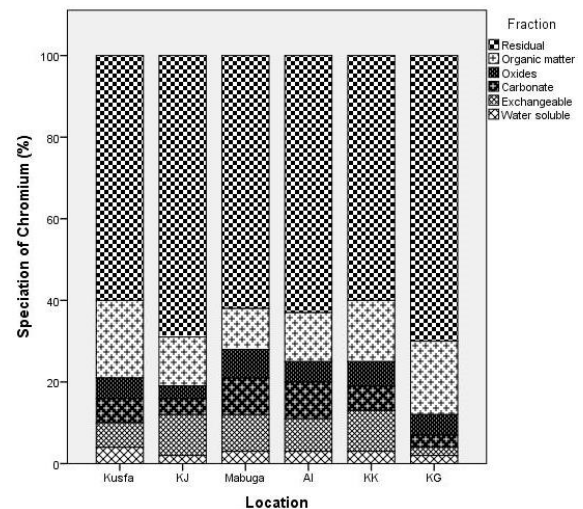


Fig. 1: Distribution of Cr in different geochemical fractions of soil in six locations in Zaria city

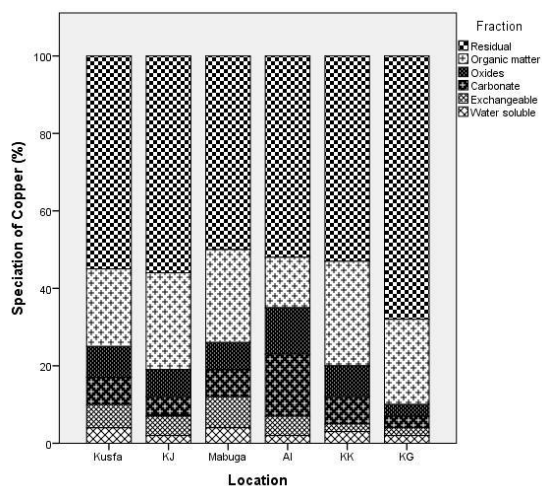


Fig. 2: Distribution of Cu in different geochemical fractions of soil in six locations in Zaria city

**Copper fractions**

Copper, likewise others were mainly found in the residual fraction (68%) in KofarGayan; relatively higher amounts of this metal were bound to organic matter (22%) and oxides of iron and manganese (3%). Insignificant concentrations were obtained in the other fractions. Similarly, Kusfa, KofarJatau, Kuyanbana and AngwanIya have a significantly high concentration of Cu fraction held unto the OM which ranged from 20-27% (Fig. 2).

At AngwanIya, carbonate-bound fraction was the next most important fraction after the residual. The carbonate bound fractions obtained was up to 16% of the total Cu concentration. Mabuga has 8% of the exchangeable bound fraction of the total Cu in the study area. Generally, heavy metals in the exchangeable and carbonate fractions are readily and potentially bioavailable (Filguerias *et al.*, 2002).

**Lead fractions**

In soil from Kusfa, Pb was found mostly in the residual fraction (77%), although a significant amount was also in the fraction bound to carbonate (12%), the other fractions were in smaller concentrations (Fig. 3). The relatively high percentage of carbonate-bound Pb fractions at this location is probably due to Pb being held by (exchangeable) electrostatic adsorption and that specifically adsorbed which may be available for biota uptake when it undergoes solubilization (Kabala and Singh, 2001; Kashem *et al.*, 2007).

In contrast, Mabuga and KofarJatau, have the organically bound fraction as the most important (12-14%), after residual, followed by that bound to carbonate (9-10%). The trend observed in these soils is an indication of it anthropogenic source. Similar findings were reported by Kabala and Singh (2001) and Kashem *et al.* (2007). Mabuga has the least value in the exchangeable fraction (1%) while that bound to oxide fraction (3%) was found in Kusfa and KofarKuyanbana. The exchangeable fraction has the least value of the total Pb from all the locations, this is in consonant with the work of Ashraf *et al.* (2012) that reported least Pb concentration in the water soluble fraction and also Ladipo *et al.* (2011) that reported Pb to be bound more to the exchangeable and carbonate fractions. A similar distribution of Pb in the exchangeable fraction was reported by Zerbe *et al.* (1999). The exchangeable metal ions measures those trace metals that are released most readily into the environment, this fraction corresponds to the form of metals most available for plant uptake and could be easily

released following any change in the ionic strength of the soil solution (Filguerias *et al.*, 2002).

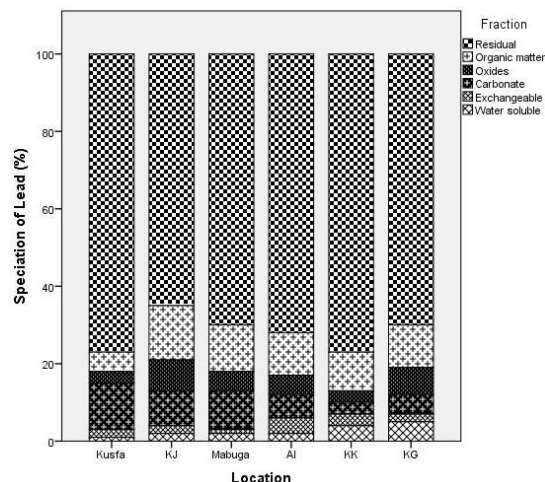


Fig. 3: Distribution of Pb in different geochemical fractions of soil in six locations in Zaria city

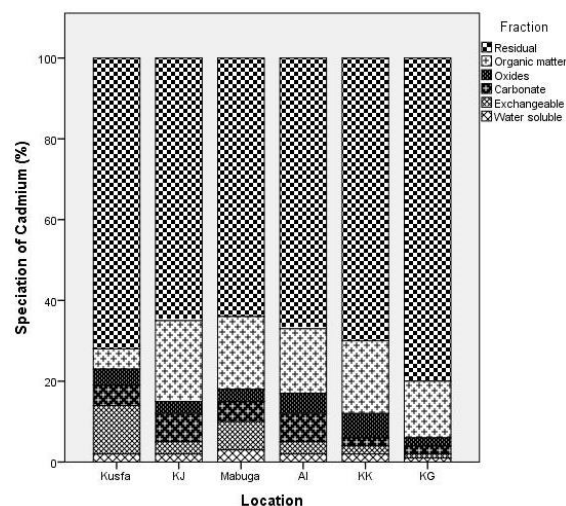


Fig. 4: Distribution of Cd in different geochemical fractions of soil in six locations in Zaria city

**Cadmium fractions**

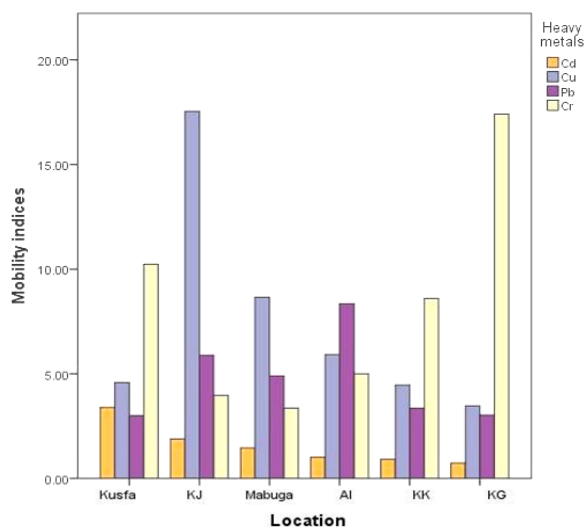
In KofarGayan, the bulk of the total Cd concentration was partitioned to the residual fraction (80 %) of the total Cd, followed by the organically bound fraction (20%). In Kusfa, the exchangeable fraction held as high as 12% of the total Cd (Fig 4). The relatively high levels of exchangeable Cd represent a higher risk of Cd mobility in the soil when it undergoes solubilization. Cd has a high potential to be released from the soil by simple ion exchange reactions (Rogan *et al.*, 2010), thereby representing potential phytotoxicity and human health hazards (Abdu *et al.*, 2012; USEPA, 2005).

However, the distributions of Cd among the oxide, OM and carbonate fractions were in smaller concentrations. In the KofarJatau and Mabuga soils, the residual fraction was the most important; this was followed by the organically bound fraction which ranged from 18-20%. Water soluble fraction held the least with a percentage range of 2-3 of the total Cd concentration. Similar trend was observed at AngwanIya and KofarKuyanbana. Fractionation results partitioned the bulk of

the total concentration of Cd to the residual fractions (Fig. 4). The relatively high percentage of the residual fractions in this soil indicates a lithogenic origin of the metal contaminants (McLean and Bledsoe, 1992).

**Potential mobility of heavy metals in soil**

The mobility index of Liu *et al.* (2007) and Kashem *et al.* (2007) was used as a more accurate method of predicting the risk of contaminant transfer to the food chain or groundwater. The calculated mobility indices for the metals under study ranged from 0.76-3.39 for Cd; 3.00-8.35 for Pb; 3.34-17.41 for Cr and 3.46-17.59 for Cu (Fig. 5). These values showed that Cu was the most mobile in the soil followed by Cr while Cd was the least mobile of the four studied metals.



**Fig. 5:** Mobility indices for Cd, Cu, Pb and Cr across the study area in Zaria city

**Correlation between metal fractions**

According to Jones and Jarvis (1981), processes of metal mobilization-immobilization are affected by a variety of soil properties. To examine this influence, correlations between the six chemical phases of the sequential extraction have been established for all soils. For Cu, water soluble fraction correlate positively with the exchangeable fraction, carbonate fraction and organic matter fraction but correlates negatively with the residual fractions ( $P < 0.01$ (Table 2) of Cr indicating that the latter residual fraction may suppress the availability of these fractions thereby reducing the risk of plant uptake. No reliable correlation was observed between the fractions of oxides ( $P < 0.01$  (Table 2).

In contrast, water soluble fraction (WS) in Cr does not show any significant correlation with all the fractions. However, the exchangeable fraction correlates positively with carbonate fraction similar levels of correlation were also observed between the exchangeable, oxide and organic matter fractions ( $p < 0.01$  (Table 2). This suggests a strong buffering of the exchangeable Cr concentration by the oxide, carbonate and organic matter fractions, thereby increasing the risk of Cr release into the environment. The residual fraction correlates negatively with all the fractions, thus suppressing the availability of this metal for groundwater contamination or plant uptake.

Significant positive correlation was observed between the water soluble fractions, exchangeable fractions; carbonate bound fractions and organic matter fractions of Cd and Pb.

But the residual fraction of Cd correlates negatively with all the fractions of Pb (Table 3;  $p \leq 0.05$ ). This is an indication that the residual fraction may suppress the availability of these fractions thereby reducing the risk of plant uptake. However, for the Pb fractions only the residual fraction correlates negatively with the carbonate fraction of Cd (Table 3;  $p < 0.05$ ).

**Conclusions**

Toxic heavy metals such as Pb and Cd can be differentiated from other pollutants, since they cannot be biodegraded but can be accumulated in living organisms, thus causing various health disorders even at relatively lower concentrations. The contaminated soil samples showed the presence of high level of metal contamination as a result of contamination with textile effluent. Soil heavy metal concentrations trend in the locations followed the sequence Mabuga>KofarJatau >AngwanIya>KofarKuyanbana>Kusfa>KofarGayan. The total concentration of the metals was separated into operationally defined fractions by sequential extraction. The largest proportions for Pb, Cu, Cd and Cr were extracted in the residual phase, in which metals are strongly retained in the non-reactive soil minerals. The potential mobility of Cr, Cu, Pb, and Cd in the soil samples studied were quite high implying that under favorable conditions they can be released to pollute the environment. Correlation coefficients indicated that all metals were highly correlated with one another. Such strong inter element correlation is an indication that the elements originated from similar sources possibly subjected to the same pedogenic processes.

Though the volume of effluents from individual small-scale dyers might be small, the concentration of pollutants is generally high. The impact is significant where several producers are located at one place and discharge effluents into the soil. It is therefore concluded that the contaminated soil samples in Zaria city showed the presence of high level of metal contamination far from the prescribed limits, toxic in nature and require proper treatment before disposal on land as it is not safe for final release which is also harmful to man and other living organisms which consequently decrease soil fertility and damage food quality.

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