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ORIGINAL ARTICLE

The assessment of heavy metal pollution in river sands of Jalingo, Nigeria using magnetic proxy parameters, pollution, and ecotoxicological indices

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Abstract The measurement of environmental magnetic properties and metal contents of sands from Nukkai (NKR), Mayo-Gwoi (RMYG) and Lamurde (LR) rivers located in Jalingo, Nigeria are reported. We seek to determine the extent of anthropogenic impact on the river sands using magnetic-proxy-parameters, pollution and ecotoxicological indices and multivariate statistics. Measurements of magnetic susceptibility χ, frequency-dependent susceptibility χfd%, anhysteric remanent magnetization, isothernal remanent magnetization and X-ray fluorescence were undertaken. The average values of the χ (in $\times 10^{-8}$ m^3 kg^{-1}) were 24.53, 12.76 and 39.27 for NKR, RMYG and LR sites respectively, implying that the magnetic minerals in the sands were mostly ferrimagnetic. The mean χfd% value of 2.64%, 4.85% and 3.53% for NKR, RMYG and LR respectively suggest that the study area was dominated by multi-domain magnetic grain sizes. The value of the S-ratio is $\sim$ 1 in all river samples, suggesting that low coercivity magnetic minerals (e.g., magnetite) dominated the samples. The mean concentrations of Ti, Zr, Sn, Ba and Pb were higher than the background values in the studied samples. All the estimated pollution indices puts the level of pollution of the river sands between low and moderate pollution with Sn, Ba and Pb as the elements of concern. Multiple sources of metal contents such as fertilizers, pesticides, waste dumps and vehicular sources etc. were found. Significant positive correlations between magnetic parameters (particularly, SIRM) and some heavy metals and pollution/ecotoxicological indices were obtained, showing that magnetic methods could be used as a geochemical proxy for pollution assessment.

Keywords Magnetic susceptibility · Ecotoxicological risk index · Pollution index · Heavy metals pollution · River

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1 Introduction

Environmental magnetic properties measurements have recently gained popularity as a proxy method of screening and assessing the level of pollution in environmental materials such as soils, sediments, dusts etc. Besides its use in pollution assessment, the method is also being applied in climatic studies (Warrier and Shankar 2009; Lyons et al. 2010), archaeology (Asanulla et al. 2017), sediment movement and provenance (Lepland and Stevens 1996).

Heavy metals and magnetic minerals of lithogenic, pedogenic or anthropogenic origin are usually deposited in coastal environments, thus acting as sinks to these deposits. According to Venkatachalapathy et al. (2013), magnetic particles in coastal sediments are usually derived primarily from fluvial and eolian transportation. For example, iron oxides that originate from anthropogenic sources such as emissions from factories, automobiles etc. may also contribute significantly to the magnetic properties in the aquatic environment. This occurs mostly in coastal areas located near highly populated urban cities and industrial sites. Usually, anthropogenic pollution has a strong magnetic signature resulting in a strong correlation between magnetic properties and metal contents. This relation has enabled the application of magnetic methods to discriminate between different pollution sources and to spot contaminated portions (Panaiotu et al. 2005).

One of the key magnetic properties that have strong affinity with heavy metals and have been used widely in pollution studies is magnetic susceptibility. It is the measure of the ease with which materials acquire magnetization in the presence of an applied field at room temperature. The value of the magnetic susceptibility is a function of the concentration and type of magnetic mineral in a material. It should also be noted that the magnetic susceptibility value of a sample is the summation of the diamagnetic, paramagnetic, canted antiferromagnetic, ferromagnetic, and ferrimagnetic minerals present in the sample. But the susceptibility of diamagnetic minerals is small and negative and that of paramagnetic minerals is small (but positive), they usually represent a small fraction of the sample's susceptibility. The susceptibility signal of a given material will thus be controlled by ferromagnetic and ferrimagnetic minerals. This is not always true in all situations. Sediments from lakes and oceans always have a low contribution from ferromagnetic and ferrimagnetic minerals (Geiss 1999) such that the susceptibility signal originates from diamagnetic and paramagnetic minerals. Again, it should be noted that where these lake/ocean sediments are polluted from industrial and other sources, the ferrimagnetic and ferromagnetic minerals dominate. The amount of superparamagnetic (SP) ferrimagnetic minerals in samples can be determined if magnetic susceptibility is measured at different frequencies. In this case, it is termed frequency-dependent -magnetic susceptibility (Dearing 1999; Evans and Heller 2003). The broad range of magnetic parameters can be divided into three classes: those that determine the content or concentration of magnetic minerals (e.g., magnetic susceptibility, anhysteric remanent magnetization and isothermal remanent magnetization), others that determine the sizes of grain in a material (e.g., frequency-dependent susceptibility, hysteresis parameters and ratios of susceptibility and remanence) and those that determine the magnetic minerals in the samples (e.g., acquisition curves for isothermal remanent magnetization, thermomagnetic curves and S-ratio) (Evans and Heller 2003; Basavaiah 2011). All these measurements are grouped into a sub-discipline of Geomagnetism termed Environmental magnetism. Environmental magnetic techniques adopt a multidisciplinary (Physics, Chemistry, Biology, Geology, Geophysics and Geography etc.) approach to characterizing magnetic compounds in rocks, soils, sediments, and dusts). The use of environmental magnetism methods has the advantage of being fast, cheap, nondestructive and sensitive. Measurements can be made on a large quantity of samples in a short time. It can be used as a quick reconnaissance method to select pollution hotspots for subsequent geochemical measurements which consumes time.

Magnetic methods have been used to assess inputs from anthropogenic substances in river sediments in many countries in Europe (Knab et al. 2006; Franke et al. 2009; Novakova et al. 2013) China (Zhang et al. 2011; Xue et al. 2014) and India (Chaparro et al. 2008; Venkatachalapathy et al. 2013). For instance, the magnetic susceptibility measured in Brantas River, Jawa Timur, Indonesia was found to exhibit a strong linkage with Fe and Co concentration (Mariyanto et al. 2019). Jordanova et al. (2003) identified the spherical magnetic particles that originated from anthropogenic processes in the sediment of Danube River, Bulgaria. In the aquatic environment, more studies have been made on sediment samples than beach sands. Surdarningsih et al. (2017) compared the magnetic properties of sand and boulder samples from Citarum River, West Java Province, Indonesia and found that the sand samples had higher magnetic susceptibility than the boulder samples. The magnetic properties of Iron sands from Tor River Estuary, Sarmi, Papua, Indonesia were found to originate from a high fraction of iron oxide Togibasa et al. (2018). In Nigeria, the environmental magnetic method has not been fully explored, especially in the aquatic environment. The present work is focused on the application of magnetic parameters as pollution indicators, especially from their relationship with metal contents. The applicability of magnetic methods as pollution proxy is further examined in this paper.

The assessment of soil pollution by heavy metals has been the traditional method of determining the level of contamination of soil, sediments, and water (Zhang et al. 2011; Jaishankar et al. 2014; Wang et al. 2019). Basically, the effect of heavy metal concentration on humans, plants and other organisms is determined by the level of toxicity on the environment. To this end, many assessment indices and criteria have been developed. According to Kowalska et al. (2018), these indices can be classified into six categories: (1) contamination factor, single pollution index, and geo-accumulation index. They give information on the levels of pollution by individual heavy metals analysed, (2) here in this group, information about the total pollution of a site by heavy metals analyzed is given, e.g., sum of pollution index, Nemerow pollution index, vector modulus of pollution index, contamination degree, modified contamination degree, background enrichment factor and contamination security index, (3) indices that tell the heavy metal sources, e.g., enrichment factor and multi-element contamination. (4) Others such as potential ecological risk, sediment quality guidelines and the probability of toxicity, explain the potential ecological risk (5) the exposure factor, which talks about the area that has the highest ability to accumulate heavy metals and (6) biogeochemical index, that gives information on the heavy metal potential of accumulation in the top horizon. It is the aim of this study to also investigate the degree of pollution in the study area using various pollution indices, determine the ecotoxicological risk of heavy metals on the studied water ecosystem, attempt an explanation of the sources of metal pollution using multivariate statistics and carry out a correlation analysis between pollution indices and magnetic parameters with a view to establishing the reliability of magnetic parameters to serve as a proxy for estimating these indices. Jalingo, the study area is an emerging urban town with no major industrial activities around the three rivers, Nukkai, Mayo-Gwoi and Lamurde. However, Jalingo has been relatively peaceful in the troubled and crisis ridden Northeast region of Nigeria and has thus witnessed the influx of persons from other parts. The economic activities in the area are suspected to receive a tremendous boost in near future. Efforts are been made by the government to site industries and factories in the region. It is hoped that the present study will serve as a future reference resource for researchers, policy makers, and urban development planners.

2 Materials and method

2.1 Study area and method of data collection

The study was conducted in Jalingo. Jalingo is the capital of Taraba State, Northeastern Nigeria which lies between latitudes 8° 50′ and 8° 55′ N and between longitudes 11° 17′ and 11° 26′ E (Fig. 1). Details about the study area can be found in Kanu et al. (2017). The geology of the study area is that of the basement complex which is undifferentiated and principally contains gneiss, migmatites and granites (Obaje 2009).

Surface samples were randomly collected from three rivers: Nukkai River (NKR), River Mayo-Gwoi (RMYG) and Lamurde River (LR) that flow across Jalingo Metropolis. Both RMYG and LR flow into NKR which empties into the River Benue. A lot of agricultural activities are undertaken around the river banks during the dry season (October to early April). Mostly cereals and vegetables are usually grown and harvested before or at the onset of the rainy season. The rainy season usually starts from late April to late September. Organic and inorganic fertilizers are usually applied on the soils to enhance crop yield. It is suspected that these chemical compounds from the river banks and surrounding agricultural fields may be washed into the river by erosion during the rainy season and may be retained in the sand/sediments. The river banks are mainly alluvium or sandy loam soils. A total of 104 samples were collected, with 34 samples from NKR, 33 from RMYG and 37 from LR. The samples were collected when the water dried up between January and March, 2012.

The collected samples were packaged in plastic containers and taken to the laboratory where the samples were dried in air for some days at laboratory temperature. Also, samples were subjected to crushing and sieving through a sieve mesh (2 mm). For the magnetic measurements, about 8–10 g of the sieved samples were filled in small polythene covers bags which were then tightly packed in 8 cm^3 nonmagnetic plastic containers.

2.2 Magnetic measurements

The magnetic measurements of the 104 samples were performed following Thompson and Oldfield (1986), Walden et al. (1999), Kanu et al. (2017). AGICO MFK1-FA Kappabridge was used to carry out the measurement of magnetic susceptibility (mass-specific) at 976 Hz (χlf) and 15,616 Hz (χhf). The two frequencies enabled the determination of the dependence of magnetic susceptibility on the frequency and this can give valuable information of the presence (or absence) of ferrimagnetic minerals in the super paramagnetic (SP) grain size range (< 0.03 μm) in a sample. The frequency-dependent susceptibility expressed in percentage (χfd %) was calculated using the relation:

$$\chi fd(\%) = \frac{\chi lf - \chi hf}{\chi lf} \times 100\% \tag{1}$$

where χlf (hereinafter referred to as χ) is the low field magnetic susceptibility.

To measure the Anhysteric Remanent Magnetization (ARM), a 100 mT peak alternating magnetic field was imparted on the samples and a steady direct current of 0.05 mT field was simultaneously applied. The changing magnetic field takes away any form of hysteresis in the samples so that the resulting magnetization comes from the ferrimagnetic grains of Stable Single Domains (SSD) range that responds to the direct current–biased field. The Molspin Spinner Magnetometer was used to measure the remanence generated from demagnetizing the samples with an AF Demagntizer (D-2000). The ARM measured was changed to the susceptibility of ARM (χARM).

The Isothermal remanent magnetization in the samples was determined by magnetizing the samples with a molspin pulse magnetizer before measurement using the spinner magnetometer (Molspin). Magnetic fields were imparted to a maximum limit of 1 T and regarded as the saturation isothermal remanent magnetization (SIRM). The samples were subjected to 20 mT and 1000 mT forward field and then demagnetized step-by-step by 20, 30, 100, and 300 mT. Hard-IRM (or HIRM), soft-IRM and S-ratio were calculated from these measurements following Walden

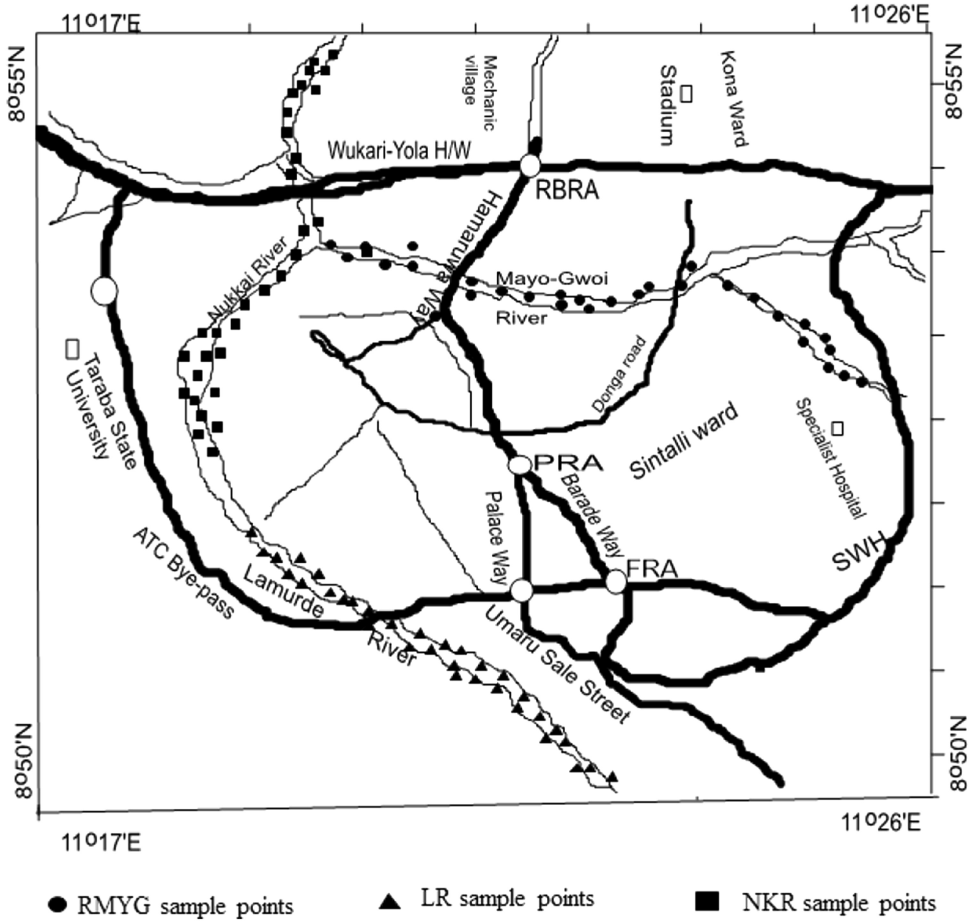


Fig. 1 Location map of Jalingo showing sampling points

et al. (1999) and Kanu et al. (2017) as displayed in Eqs. 2–6.

$$Soft\ IRM = \left(\frac{SIRM - IRM_{-30\,\mathrm{mT}}}{2}\right) \quad (2)$$

$$Soft\ IRM(\%) = \frac{SOFT}{SIRM} \times 100\% \quad (3)$$

$$HIRM = \left(\frac{SIRM - IRM_{-300\,\mathrm{mT}}}{2}\right) \quad (4)$$

$$HIRM(\%) = \frac{HIRM}{SIRM} \times 100\% \quad (5)$$

$$S_{ratio} = \frac{|IRM_{-300\,\mathrm{mT}}|}{SIRM}. \quad (6)$$

2.3 X-ray fluorescence (XRF) analysis

XRF analysis was carried out on 65 soil samples (17 from NKR, 21 from RMYG and 27 from LR). Measurement was made on powdered and homogenized samples using an X-ray fluorescence spectrometer (AMATEK SPECTRO XEPOS). Approximately three grammes of the sample were inserted into plastic cups supplied by the manufacturers and the Turboquant Powders method (www.amatek.com) was selected for the analysis which takes about 10 min. The standardization of the XRF measurements was achieved by the use of the certified reference materials (NIST SRM 2709), duplicate samples and programme blanks. The instrument is usually calibrated once a week and each year a global calibration is performed. In order to ensure that measurement could be reproduced, each soil sample was subjected to the measurement for three times. The calculated standard errors in all the analyzed metals were between 0.2% and 5.0% and a recovery rate of 94%–115% for the measured heavy metals was achieved. Furthermore, XRF measurements were performed thrice on selected 15 samples, constituting 22% of the samples, and compared with the initial measurement. A very strong positive correlation (r = 0.98–1.0) was achieved. Varying Limit of Detection (LoD) obtained for the various metals using the XRF instrument were Al (0.57 mg/kg), Ti, Ni (1.00 mg/kg), Mn (0.40 mg/kg), Fe (0.37 mg/kg), Cu, Zn, Ba (2.0 mg/kg), Sr (0.9 mg/kg), Zr (0.3 mg/kg), Sn (6.0 mg/kg) and Pb (2.8 mg/kg).

2.4 Assessment of level of contamination using pollution indices

Various indices including contamination factor, modified degree of contamination, Nemerow Pollution index, Pollution Load index, geo-accumulation index and enrichment factor were used to assess the level of pollution of the Jalingo river sands.

2.4.1 Contamination factor and modified degree of contamination

The contamination factor (*CF*) takes into consideration the measured heavy metal value with respect to the geochemical background and can be determined using Eq. 7 (Hakanson 1980; Kowalska et al. 2018).

$$CF = \frac{C_n}{C_b} \tag{7}$$

where C_n is the concentration of metal n, and C_b is the uncontaminated or background value. In this study, the background values of all analyzed heavy metals were taken from upper continental crust values given by Wedepohl (1995) and Rudnick and Gao (2014).

The modified degree of contamination (mC_d) was introduced by Abraham and Parker (2008) to assess the total metal contamination in the soil/sediment. It was calculated using Eq. 8.

$$mC_d = \frac{1}{n}\sum_{i=1}^{n} CF_i \tag{8}$$

where n indicates the number of metals analyzed and CF_i is the contamination factor of metal i.

2.4.2 Nemerow pollution index (PI_N)

According to Liu et al. (2015), the Nemerow pollution index can better expose the level of pollution in soils since it involves the evaluation of many heavy metals in soils. It was determined using the equation (Gao et al. 2016):

$$PI_N = \sqrt{\frac{\left(\frac{1}{n}\sum_{i=1}^{n} PI\right)^2 + PI_{max}^2}{2}} \tag{9}$$

$PI = \frac{concentration\ of\ heavy\ metal}{background\ value\ for\ the\ heavy\ metal}$ is the single pollution index and PI_{max} is the maximum value of the PI calculated for all metals. Five classes of the PI_N (Cheng et al. 2007) are as follows: safety domain ($PI_N < 0.7$), precaution domain ($0.7 \le PI_N < 1.0$), slightly polluted domain ($1.0 \le PI_N < 2.0$), moderately polluted domain ($2.0 \le PI_N < 3.0$) and seriously polluted domain ($PI_N > 3.0$).

2.4.3 Pollution load index (PLI)

The pollution load index is also used to check the overall assessment of the level of contamination in the soil. It is used to find out the extent of contamination caused by the continual addition of heavy metals to the soil (Varol 2011). It is calculated using Eq. 10 thus:

$$PLI = \sqrt[n]{CF_1 \times CF_2 \times CF_3 \times \cdots\cdots\cdots \times CF_n} \tag{10}$$

In Eq. (10), n is the total number of investigated metals (n = 11 in this study) and CF is the contamination factor. A *PLI* value ≤ 0 is an indication of clean and perfect environment without pollution and can serve as a control site, a *PLI* value > 0 and ≤ 1 expresses the baseline level of metal pollutants and a *PLI* value > 1 gives strong evidence of polluted soil (Pobi et al. 2019).

2.4.4 Geo-accumulation index (I_{geo})

The geo-accumulation index (I_{geo}) is an easy way of estimating the enhancement of heavy metals higher than background/baseline values. It was first introduced by Muller (1969) and is calculated thus:

$$I_{geo} = log_2\left(\frac{C_n}{1.5B_n}\right) \tag{11}$$

where C_n is the concentration of the analyzed metal and B_n is the geochemical background values. The value of 1.5 introduced in the formula is to account for eventual changes in the background values resulting from lithogenic processes in the soil. In this study, Bn is taken from the upper continental crust (UCC) given by Wedepohl (1995) and Rudnick and Gao (2014). Interpretation of I_{geo} to reveal the extent of contamination is carried out based on the following divisions: unpolluted ($I_{geo} \le 0$); unpolluted to moderately polluted ($0 < I_{geo} \le 1$); moderately polluted ($1 < I_{geo} \le 2$); moderately polluted to highly polluted ($2 < I_{geo} \le 3$); highly polluted ($3 < I_{geo} \le 4$); highly polluted to very highly polluted ($4 < I_{geo} \le 5$) and very highly polluted ($I_{geo} > 5$) (Muller 1981).

2.4.5 Enrichment factor (EF)

This pollution index is used to determine the likely effect of anthropogenic activities on the heavy metal contents in soils/sediment. In the *EF* method, the analysed metal is normalized using a reference element such as Al, Fe, Ti, Ca, Sc and Mn (Kowalska et al. 2018). EF is evaluated using the following expression:

$$EF = \frac{\left(\frac{C_n}{Al}\right)_{sample}}{\left(\frac{C_n}{Al}\right)_{background}} \tag{12}$$

where Cn is the concentration of measured heavy metal n in the samples and Al is the content of Aluminum used as a reference value. Aluminum was used for normalization because it is one of the most abundant elements in the Earth's crust and it is scarcely of anthropogenic origin. The *EF* is divided into the following categories: deficiency to minimal enrichment (< 1–2); moderate enrichment (2–5); significant enrichment (5–20); very high enrichment (20–40) and extremely high enrichment (> 40) (Loska and Wiechula 2003; Pobi et al. 2019).

2.5 Ecotoxicological determination of contamination by heavy metal

There are several sediment quality guidelines (SQG) that are recently used to characterize the level of soil contamination by individual heavy metals. This assessment criterion is achieved by comparing the metal concentration with the reference criteria. Two levels of comparison are usually adopted: (a) those below which hostile biological effects scarcely occur [e.g., LEL (Lowest Effect Level), TEL (Threshold Effect Level), ERL (Effect Range—Low) and MET (Minimal Effect Threshold)] collectively referred to as Threshold Effect Concentration (TEC) sediment quality guidelines and (b) those above which hostile biological effects are likely to take place (or expected to take place frequently) [e.g., SEL (Severe Effect Level), PEL (Probable Effect Level), ERM (Effect Range—Median) and TET (Toxic Effect Threshold)] collectively known as Probable Effect Concentration (PEC) Sediment quality guidelines (MacDonald et al. 2000; Benson et al. 2018). In this study, TEL, ERL, SEL, PEL and ERM were used to assess the quality of the river sands affected by the concentration of Ni, Cu, Zn and Pb. We further used the SQG values to calculate the mean probable effect level quotients ($mPEL_Q$), mean Effective Range -Median Quotients ($mERM_Q$), hazard quotients (HQ) and modified hazard quotient (mHQ).

2.5.1 Mean probable effects level quotients ($mPEL_Q$)

The mean probable effects level quotient was employed to investigate the probable biological effect of the combined heavy metals in the samples in the ecosystem. The $mPEL_Q$ was evaluated following Kumar et al. (2016) and Benson et al. (2018) as follows

$$mPEL_Q = \frac{\sum_{i=1}^{n} \frac{C_i}{PEL_i}}{n} \tag{13}$$

where C_i is the value of the metal (i) concentration, PEL_i is the corresponding Probable Effect Level value of the metal (i) and n the total number of determined metals. Different levels of $mPEL_Q$ and toxicity probability have been distinguished as follows: For $mPEL_Q \leq 0.1$ implies a low degree of contamination and 8% probability of toxicity; $mPEL_Q$ from 0.11 to 1.50 implies a medium degree of contamination and 21% probability of toxicity; $mPEL_Q$ from 1.51 to 2.30 implies medium–high degree of contamination and 49% toxic probability and $mPEL_Q > 2.3$ implies high degree of contamination and 73% probability of toxicity (Long et al. 2006; Kumar et al. 2016).

2.5.2 Mean effect range-median quotient ($mERM_Q$)

The adverse effect of heavy metals on the soil can also be evaluated using $mERM_Q$. It was calculated using Eq. 14 thus:

$$mERM_Q = \frac{\sum_{i=1}^{n} \frac{C_i}{ERM_i}}{n} \tag{14}$$

where C_i is the value of the metal (i) concentration, ERM_i is the corresponding Effect Range -Median value of the metal (i) and n the total number of determined metals. Different levels of $mERM_Q$ and toxicity probability has been distinguished as follows: $mERM_Q \leq 0.1$ implies low priority site and 9% probability of toxicity; $mERM_Q$ from 0.1 to 0.5 implies medium–low priority site and 21% probability of toxicity; $mERM_Q$ from 0.5 to 1.5 implies high-medium priority site and 49% toxic probability and $mERM_Q > 1.5$ implies high priority site and 76% probability of toxicity (Long et al. 2006; El-Alfy et al. 2020).

2.5.3 Hazard quotient (HQ)

The rate of heavy metal toxicity on both the organisms and the environment can be determined by evaluating the HQ. This was achieved using the equation:

$$HQ = \frac{C_i}{SQG} \tag{15}$$

where C_i is the measured content of heavy metal in the sample and SQG is the sediment quality guidelines and the threshold effect level (TEL) is the SQG used for the calculation of HQ in this work (MacDonald et al. 2000; Benson et al. 2018). Interpretation of HQ is based on the following classification proposed by Feng et al. (2011): $HQ < 0.1$ implies that there are no adverse effects; $0.1 < HQ < 1$ implies potential hazard; $1 < HQ < 10$ implies moderate hazard and $HQ > 10$ implies high hazards.

2.5.4 Modified hazard quotient (mHQ)

The mHQ is a new index established by Benson et al. (2018) to evaluate pollution in sediments by comparing

Table 1 Descriptive statistics of magnetic properties of sands from Jalingo rivers

Magnetic parameter	Unit	Nukkai river (n = 34) Mean ± SD	River Mayo-Gwoi (n = 33) Mean ± SD	Larmurde river (n = 37) Mean ± SD
χfd	$\times 10^{-8}$ m^3 kg^{-1}	0.51 ± 0.30	0.42 ± 0.15	0.92 ± 1.92
χfd %	%	2.64 ± 1.77	4.85 ± 2.38	3.53 ± 1.05
χ	$\times 10^{-8}$ m^3 kg^{-1}	24.53 ± 23.37	12.76 ± 8.59	39.27 ± 41.39
ARM	$\times 10^{-5}$ A m^2 kg^{-1}	2.33 ± 0.27	0.86 ± 0.10	2.19 ± 1.57
χARM	$\times 10^{-5}$ m^3 kg^{-1}	0.06 ± 0.01	0.02 ± 0.00	0.06 ± 0.04
ARM/χ	kA/m	3.51 ± 1.81	2.51 ± 0.93	4.21 ± 5.07
χARM/χfd	Dimensionless	320.49 ± 446.54	59.98 ± 28.51	140.63 ± 225.14
SIRM	$\times 10^{-5}$ A m^2 kg^{-1}	89.73 ± 49.11	80.14 ± 4.47	123.64 ± 10.54
ARM/SIRM	Dimensionless	0.06 ± 0.04	0.03 ± 0.01	0.03 ± 0.04
SIRM/χ	kA/m	1254.14 ± 2165.43	960.46 ± 477.63	2940.27 ± 3994.04
S-100	Dimensionless	0.64 ± 0.06	0.53 ± 0.05	0.60 ± 0.24
S-ratio	Dimensionless	0.93 ± 0.05	0.86 ± 0.03	0.94 ± 0.05
Soft IRM	Dimensionless	41.73 ± 24.79	23.43 ± 9.90	62.90 ± 111.41
Soft %	%	45.77 ± 2.35	45.38 ± 2.28	45.84 ± 2.95
HIRM	Dimensionless	3.39 ± 2.84	3.72 ± 1.89	3.66 ± 6.73
HIRM%	%	3.70 ± 2.56	7.17 ± 1.70	3.17 ± 2.32

SD standard deviation

single heavy metal contamination degree with different threshold levels provided by MacDonald et al. (2000). The *mHQ* was obtained using Eq. 16.

$$mHQ = \left[C_i\left(\frac{1}{TEL_i} + \frac{1}{PEL_i} + \frac{1}{SEL_i}\right)\right]^{1/2} \quad (16)$$

Accordingly, Benson et al. (2018) gave the following guides for the interpretation of *mHQ*: Extreme severity of contamination ($mHQ > 3.5$); very high severity of contamination ($3.0 < mHQ < 3.5$); High severity of contamination ($2.5 < mHQ < 3.0$); considerable severity of contamination ($2.0 < mHQ < 2.5$); moderate severity of contamination ($1.5 < \mathrm{mHQ} < 2.0$); low severity of contamination ($1.0 < mHQ < 1.5$); very low severity of contamination ($0.5 < mHQ < 1.0$) and nil to very low severity of contamination ($mHQ < 0.5$).

2.6 Multivariate statistical analysis

In this study, we made use of two multivariate statistical tools: Principal Component Analysis (PCA) and Path diagram to assist in the identification of likely source(s) of heavy metals and magnetic variables. In the PCA, the varimax rotation with Kaiser Criterion was used. In each principal component, the eigenvalue > 1 was considered while others are rejected because of the insignificant variance explained by them. The linkage between each group was identified using the path diagram. The path diagram is a pictorial representation of the relationship between the variables. The direction and strength of the correlation is indicated by the wideness of arrows that moves from the variables to the PCs. Both PCA and path diagrams were evaluated using JASP (0.16.1) statistical software. ORIGIN 5.0 was used to make boxplots.

3 Results and discussion

3.1 Environmental magnetic parameters

The environmental magnetic properties of beach sands and sediments from Nukkai River (NKR), River Mayo-Gwoi (RMYG) and Lamurde River (LR) are presented in Table 1. The result showed wide variability in the χ value of the LR which ranged from (2.36 to 161.57) $\times 10^{-8}$ m^3 kg^{-1} with an average value of 39.27 $\times 10^{-8}$ m^3 kg^{-1}. In NKR, χ ranged from (8.73–120.46) $\times 10^{-8}$ m^3 kg^{-1} with an average value of 24.53 $\times 10^{-8}$ m^3 kg^{-1} while in RMYG less variable value of χ (4.66–40.34) $\times 10^{-8}$ m^3 kg^{-1} was obtained with the least mean value of χ (12.76 ± 8.59) $\times 10^{-8}$ m^3 kg^{-1}. Generally, the results showed low to moderate enhancement in χ of the surface sediments from the river bodies. The highly variable χ values between samples may be attributed to the variable pollution sources in the river sand such as emissions from traffic, fertilizers, refuse dumps, atmospheric input from long-distance pollution sources etc. Usually, high magnetic susceptibility in marine sediments could result from different mechanisms such as diagenetic changes in magnetic mineralogy (Karlim et al. 1987), fine-grained iron oxides derived from anthropogenic sources (Chan et al. 2001), an increased input of detrital magnetite from soil erosion (Maher and Taylor 1988) and high content of magnetotactic bacteria in marine sediments (Lovely et al. 1987). The results of other concentration parameters (χARM, SIRM) were generally low compared to top soil samples from road-deposited sediments within Jalingo metropolis (Kanu et al. 2017). The obtained results of magnetic concentration-dependent parameters (χ, SIRM and χARM) indicates the presence of ferrimagnetic minerals in the beach sands.

The frequency-dependent susceptibility χfd%, SIRM/χ, χARM/χ and ARM/SIRM are magnetic grain size-dependent parameters (Basavaiah 2011). χfd% had mean values of $2.64 \pm 1.77\%$, $4.85 \pm 2.38\%$ and $3.53 \pm 1.05\%$ in the NKR, RMYG and LR respectively (Table 1). The low values of χfd% ($< 4\%$) in all the sites implied that the samples were dominated by non–Super Paramagnetc (SP) coarse-grained ferrimagnetics from anthropogenic sources (Maher and Taylor 1988; Dearing 1999). Coarse-grained particles such as multidomain (MD) and pseudo single domain (PSD) grains are frequency independent and show similar susceptibility values at low and high frequencies (Dearing 1999). Further, ferrimagnetic minerals such as magnetite having MD characteristics exhibits soft magnetic properties, implying that they possess a small remanence and coercivity and hence can be magnetized and demagnetized with ease (Robertson et al. 2003). This behavior may be attributed to the freedom of the domain walls to move easily when the magnetic fields change. The low value of χARM/χ ratio obtained is an indication of MD grains dominating the sample which is in agreement with the results of χfd%. A high SIRM/χ ratio was obtained suggesting the presence of coarser magnetic grain size (Hu et al. 2007). According to (Evans and Heller 2003; Basavaiah 2011), smaller grains yield higher values of SIRM/χ because they are more efficient in acquiring remanence. SIRM/χ can also be used to infer paramagnetic contribution to the magnetic properties of sediments (Oldfield et al. 1985). This is sometimes inferred from an extremely low value of SIRM/χ. The ratio SIRM/χ can also be used to monitor mineralogical changes, higher values are indicative of hematite while lower values indicate magnetite (Van Oorschot 2001). Hence, the high value of SIRM/χ in the river sands could also be due to the presence of hematite. This conclusion needs confirmation from other measurements such as S-ratio and HIRM. The ARM/SIRM ratio is related to magnetic grain size. The low values of ARM/SIRM indicate coarse ferrimagnetic grain assemblages. Higher values represent finer grain- size (Venkatachalapathy et al. 2011).

S-ratio, Soft IRM and HIRM are parameters that give notice of magnetic mineralogy in samples. The mean values of S-ratio for the three rivers were 0.926, 0.857 and 0.937 for NKR, RNYG and LR respectively (Table 1). These values which are close to 1 (or approximately equal to one) showed a characteristic of the sample with low—coercivity and soft magnetite-like ferrimagnetic minerals (Venkatachalapathy et al. 2011; Kanu et al. 2017). The ratio of the mean values of soft IRM to HIRM is 92: 8% for NKR, 86: 14% for RMYG and 93: 7% for LR. This implied that the samples were mainly composed of ferrimagnets, though antiferromagnets may also be present. This is in agreement with the findings of Yang et al. (2019) who stated that the low magnetic signal of the hard coercive minerals can be overshadowed by the soft coercive fractions.

3.2 Relationship between magnetic parameters from Jalingo River sand

The relationship between environmental magnetic parameters is assessed using the Pearson correlation matrix (Table S1). The results show that χ is significantly correlated with other concentration dependent parameters: χARM (r = 0.610, $P < 0.001$) and SIRM (r = 0.881, $P < 0.001$), implying that the magnetic enhancement in the samples is mainly controlled by ferrimagnetic minerals. Since χARM is sensitive to SSD and SIRM is sensitive to MD grains, the significant correlation between χ and these parameters also suggested the dominance of SSD and MD grains in the samples. χ is also significantly correlated with mineralogic indicative parameters as follows: S-ratio (r = 0.192, $P < 0.05$), Soft IRM (r = 0.880, $P < 0.001$) and HIRM (r = 0.578, $P < 0.001$), indicating the presence of low coercivity ferrimagnetic minerals, however, the low correlation coefficient between S-ratio and χ (r = 0.192, $P < 0.05$) and the high correlation coefficient obtained between HIRM and χ (r = 0.578, $P < 0.001$) gives a strong indication of the presence of canted antiferromagnets at least to a reasonable concentration in the samples, notwithstanding the strong evidence of high ferrimanetic minerals in the river sands as evident in the high correlation between the soft IRM and χ. The inverse covariation existing between χ and χARM/χ (r = −0.217, $P < 0.05$), χ and χfd% (r = −0.191, $P < 0.05$) and χ and ARM/SIRM (r = −0.275, $P < 0.01$) suggests a relatively high input of coarser (MD and PSD) magnetite of anthropogenic source at sites with higher χ.

The plot of χfd% against χARM/SIRM could semi-quantitatively interpret the magnetic grain size (Dearing 1999). The analysis of the domain state of the surface river samples in Jalingo using Dearing's plot (Fig. 2) showed that the domain sizes were mainly MD, PSD and SSD. Most of the samples contained ≤ 50% SP grains.

3.3 Results of heavy metal concentration of Jalingo river sediments

The concentrations of elements in the three rivers sand under consideration in Jalingo town are presented in Table 2. The mean concentrations of the heavy metals varied in the following order: for NKR, Fe > Ti > Ba > Sr > Mn > Zr > Pb > Zn > Sn > Ni > Cu for RMYG, Fe > Ti > Ba > Mn > Zr > Sr > Pb > Sn > Zn > Ni > Cu and for LR, Fe > Ti > Ba > Sr > Zr > Mn > Pb > Zn > Sn > Ni > Cu. However, in all three river samples, the mean concentrations of Ti, Zr, Sn, Ba, and Pb were more than the geochemical background values, that is, the Upper Continental Crust (UCC) values taken from Wedepohl (1995) and Rudnick and Gao (2014). The level at which these metals exceeds the UCC values in the three rivers are as follows: For Ti, NKR (31.55%) > RMYG (25.78%) > LR (18.67%); for Zr, RMYG (36.62%) > LR (32.04%) > NKR (28.59%); for Sn, RMYG (86.95%) > NKR (86.91%) > LR (86.83%); for Ba, RMYG (63.14%) > NKR (63.07%) > LR (62.32%) and for Pb, RMYG (54.55%) > NKR (45.65%) > LR (43.78%). Also, Al exceeded the UCC values by 2.62% in NKR and 3.39% in RMYG but less than UCC by 1.28% in LR. The increase of the concentration of these metals above the UCC values implied that, their concentration has been influenced by anthropogenic activities. And the level of activities is indicated by the percentage increase above the UCC values. The presence of Sn in the soil according to (Senesi et al. 1999) are usually caused by manure addition to the soil, corrosion of metallic objects and metallic ores dispersal during movement. Also, the use of municipal refuse and sludge for amendment of soil can also contribute to tin accumulation in soils. When tin and its compounds are directly or indirectly ingested into the human body, it can be detrimental to health. According to the Agency for Toxic Substances and Disease Registry (ATSDR) (ATSDR 2005), ingestion of a high quantity of inorganic Tin may cause stomach aches, anaemic conditions and problems in the kidney and liver.

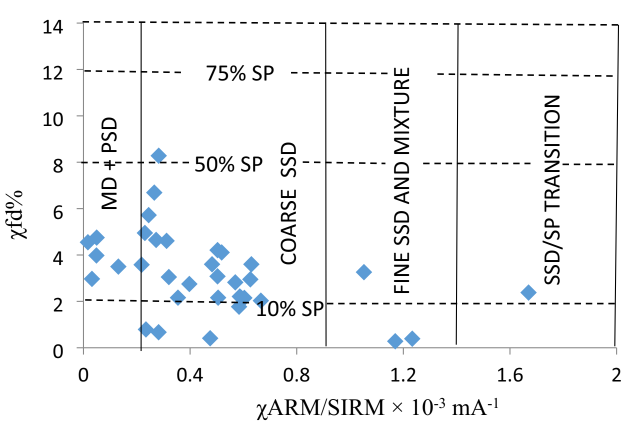


Fig. 2 Dearing plot for estimating the proportions of SP and non-SP grain sizes

Lead (Pb) in the soil can enter the human system if the contaminated soil is ingested directly. The concentration of lead in the river sands might be through deposition from automobile exhaust, fertilizers from nearby farming activities and leaded paints. Pb (and Cu) exposures have been found to cause damage to the kidney (Masri et al. 2021). As one of the known carcinogens, Pb can also cause lung, kidney and bladder cancers (Cempel and Nikel 2006). Zirconium (Zr) can be found in nature and through anthropogenic inputs but the sources from natural rocks are more abundant (Shahid et al. 2013). The anthropogenic inputs in the studied river sands may be from the indiscriminate dumping of wastes, atmospheric fallouts, phosphate fertilizers and animal manures. Titanium is one of the most available elements in the Earth's crust. TiO_2 finds applications in industries as the white colour in paints and plastics and a coloring agent in food and dyeing industries.

Notched box plots were used to compare the concentration of some elements within the different study sites (Fig. S1). It can be observed from the boxplots that there were no pronounced observable differences in the concentration of Ti and Sn within the three sites. Cu and Zn showed a similar trend (NKR > LR > RMYG) while Mn and Pb varied in the following sequence LR < NKR < RMYG. It was also observed that the highest concentration of the analysed metals are found in mostly in RMYG and NKR. For NKR, this result is expected since both LR and RMYG flow into the NKR. However, RMYG is located within the city with a lot of activities such as car wash, mechanic workshops, building renovation and construction activities including painting which could cause an increase in the contents of lead and even manganese in the RMYG over the others.

NKR showed the widest variability in the elemental concentrations. The concentration of Fe is highest in NKR and nearly equal in RMYG and LR. The results showed that there is no clear pattern in the values of the assessed metals in the three study sites. This implied that there are different levels of activities that resulted in the different elemental concentrations obtained in the river sands. Contributions may arise from both crustal and anthropogenic. Emissions from vehicular activities across the rivers, agricultural practices such as fertilizer and pesticide application, household waste disposals and erosion from factory sites might be some of the anthropogenic contributions to the elemental concentrations of the river samples.

Table 2 Descriptive statistics of elemental concentration of Nukkai (n = 17), Mayo-Gwoi (n = 21) and Lamurde (n = 27) rivers sand in mg/kg

Element	NKR Mean ± SD	RMYG Mean ± SD	LR Mean ± SD	UCC	TEL	ERL	PEL	SEL	ERM
Al	79,467.06 ± 4305.34	80,062.86 ± 4027.13	76,448.15 ± 4537.65	77,440	–	–	–	–	–
Ti	4553.47 ± 1809.44	4199.76 ± 1351.07	3832.33 ± 1177.59	3117	–	–	–	–	–
Mn	278.92 ± 141.72	328.55 ± 96.63	183.83 ± 59.16	527	–	–	–	–	–
Fe	9035.00 ± 4137.83	5908.19 ± 707.85	6552.74 ± 2100.46	30,890	–	–	–	–	–
Ni	6.83 ± 6.18	3.66 ± 0.60	4.67 ± 1.01	47	18	30	36	75	50
Cu	3.65 ± 0.97	2.08 ± 0.98	4.03 ± 2.61	28	35.7	70	197	110	390
Zn	17.15 ± 4.70	11.88 ± 1.87	25.82 ± 38.17	67	123	120	315	820	270
Sr	317.59 ± 11.97	275.66 ± 14.69	307.04 ± 19.19	320	–	–	–	–	–
Zr	270.26 ± 86.33	304.50 ± 130.45	283.99 ± 156.36	193	–	–	–	–	–
Sn	16.04 ± 2.68	16.09 ± 2.97	15.94 ± 3.25	2.1	–	–	–	–	–
Ba	1700.53 ± 66.35	1703.71 ± 83.11	1666.81 ± 110.63	628	–	–	–	–	–
Pb	31.28 ± 1.72	37.40 ± 1.83	30.24 ± 3.45	17	35	35	91.3	250	110

UCC upper continental crust (Wedepohl 1995; Rudnick and Gao 2014)

TEL threshold effect range (MacDonald et al. 2000; Benson et al. 2018)

ERL effect range low (MacDonald et al. 2000; Benson et al. 2018)

PEL probable effect range (MacDonald et al. 2000; Benson et al. 2018)

SEL severe effect level (MacDonald et al. 2000; Benson et al. 2018)

ERM effect range median (MacDonald et al. 2000; Benson et al. 2018)

3.4 Linkage between magnetic properties and geochemistry of the river sands

The magnetic and geochemical variables' relationship verification was carried out by Pearson's correlation method using JASP (version 0.16.1) statistical software. The outcome which is presented in Table 3 showed various degrees of correlation between the magnetic and the elemental variables. In general, two categories of metals affinity with magnetic parameters were observed: Group I elements (Ni and Sn) that clearly showed no significant affinity with any of the magnetic parameters and group II (Al, Ti, Fe, Cu, Zn, Sr, Zr, Ba and Pb) which displays a varying degree of affinity with some magnetic parameters.

It was observed that χ, was strongly positively correlated with Ti (r = 0.406, $P < 0.001$) and weakly negatively correlated with Al (r = −0.356, $P < 0.01$) and Ba (r = −0.276, $P < 0.05$). χARM was seen to be significantly positively correlated with Fe, Cu, Sr and negatively correlated with Pb while SIRM correlated significantly

Table 3 Pearson correlation matrix between some magnetic and heavy metals from Jalingo River samples

Variable	χfd%	X	χARM	SIRM	S-ratio	soft IRM	HIRM
Al	0.091	**− 0.356**^{b}	− 0.191	**− 0.365**^{b}	− 0.018	**− 0.380**^{b}	− 0.098
Ti	− 0.245	**0.406**^{c}	0.142	**0.603**^{c}	0.013	**0.527**^{c}	**0.415**^{c}
Mn	**0.267**^{a}	− 0.143	− 0.248	− 0.093	− 0.148	− 0.129	0.095
Fe	0.018	0.105	**0.308**^{a}	0.151	0.145	0.129	− 0.056
Ni	− 0.039	0.012	0.180	0.052	0.077	0.040	− 0.016
Cu	− 0.025	0.053	**0.370**^{b}	0.069	**0.569**^{c}	0.096	**− 0.453**^{c}
Zn	− 0.056	0.218	0.186	**0.522**^{c}	**0.447**^{c}	**0.563**^{c}	**− 0.480**^{c}
Sr	− 0.185	0.088	**0.596**^{c}	0.042	**0.480**^{c}	0.041	**− 0.345**^{b}
Zr	**− 0.335**^{b}	0.230	− 0.031	**0.542**^{c}	− 0.063	**0.479**^{c}	**0.389**^{b}
Sn	− 0.085	0.128	0.064	0.119	− 0.022	0.129	0.049
Ba	− 0.027	**− 0.276**^{a}	− 0.063	**− 0.332**^{b}	− 0.074	**− 0.356**^{b}	0.037
Pb	0.172	− 0.240	**− 0.533**^{c}	− 0.102	**− 0.385**^{b}	− 0.123	0.221

$^{a}p < 0.05$, $^{b}p < 0.01$, $^{c}p < 0.001$

Bold values indicate significant correlation positively with Ti, Zn and Zr and significantly negatively correlated with Al and Ba. This implies that environmental magnetic parameters (in this case, χ, χARM and SIRM) can be used as a substitute for detecting the concentration of these metals, however, χ appears to be a weak proxy parameter as it showed low correlation strength with the heavy metals. A similar result was reported by Zhang et al. (2011) who also found SIRM to correlate with heavy metals better than χ in urban river sediments in China. It is therefore possible to provide a semi-quantification of pollution status in a soil at local or regional scale by the use of magnetic parameters. The correlation results also suggested that the sources of these metals were dominated by anthropogenic activities. The significant negative correlation obtained between χ and SIRM and Al and Ba signifies that the metals (Al and Ba) are of crustal or geogenic origin. In the river samples, χfd% exhibited weak correlation with only Mn (r = 0.267, $P < 0.05$) and Zr (r = -0.335, $P < 0.01$). The χfd%—Zr relation suggests that Zr concentration has been influenced by anthropogenic inputs. This was also supported by the strong positive covariation between Zr and SIRM. The increase in χfd% as Mn concentration increases in the river samples indicates that Mn originates from either crustal or weathering activities. S-ratio exhibited significant relationship with Cu (r = 0.569, $P < 0.001$), Zn (r = 0.447, $P < 0.001$), Sr (r = 0.480, $P = 0.001$) and Pb (r = -0.385, $P < 0.01$). Soft IRM correlates positively with Ti, Zn, Zr and negatively with Al and Ba. HIRM was strongly significantly positively correlated with the concentration of Ti and Zr and negatively correlated with Cu, Zn and Sr. It can be inferred from this result that increasing the concentration of Ti and Zr increased the concentrations of both magnetically soft magnetite and magnetically hard hematite in the samples while Cu, Zn, Sr and Zr increases as the soft magnetic fraction increased. The pollution of soils usually causes the elevation of the percentage of ferrimagnetic minerals leading to an increase in the S-ratio and soft IRM values (Yang et al. 2019). The negative correlation of Pb and χARM and S-ratio may indicate that the source (s) of Pb is/are different from other metals such as Ti, Cu, and Zn etc.

As a general guide, high magnetic susceptibility and elemental concentration give a high indication of an anthropogenic source. Alternatively, a high magnetic susceptibility and low concentration of elements point to a natural source, which tells of the dominance of geogenic sources (Spiteri et al. 2005; Canbay 2010). The strength of the correlation between magnetic susceptibility and element concentration represents the level of affinity of the elements to the ferrimagnetic minerals. Scatter plots of some magnetic parameters and heavy metals are shown in Fig. S2.

3.5 Assessment of level of contamination using pollution indices

3.5.1 *Contamination factor and modified degree of contamination*

In NKR, the contamination factor (CF) varied in the order Sn > Ba > Pb > Ti > Zr > Sr > Fe > Mn > Zn > Ni > Cu. A similar pattern was observed in the LR and RMYG with slight differences. In LR the order of CF was Sn > Ba > Pb > Zr > Sr > Zn > Fe > Mn > Cu > Ni and in RMYG it was Sn > Ti > Ba > Pb > Zr > Sr > Fe > Mn > Zn > Ni > Cu (Tables 4, 5, 6). The interpretation of the CF values are as follows: low contamination (CF < 1), moderate contamination (1 < CF < 3), considerable contamination (3 < CF < 6) and very high contamination (CF > 6) (Hakanson 1980; Pobi et al. 2019). The results indicated that all the sites experienced low contamination of Fe, Mn, Ni, Cu and Sr which had average CF values of less than one. Moderate contamination was observed in Ti, Zr, Ba and Pb while Sn showed very high contamination having CF values greater than 6 (Pobi et al. 2019). These observations are more clearly illustrated using boxplots (Fig. S3).

The average values of the modified degree of contamination were 1.58, 1.53 and 1.59 (Tables 4, 5, 6) in NKR, LR and RMYG respectively. The values of the calculated mC_d can be interpreted based on the following classification: low ($1.5 < mC_d < 2$), moderate contamination ($2 < mC_d < 4$), high contamination ($4 < mC_d < 8$), very high contamination ($8 < mC_d < 16$) and extremely high contamination ($mC_d > 16$) (Abraham and Parker 2008). According to Abraham and Parker (2008) classification, the result obtained signified low contamination of the samples as values were in the range $1.5 \leq mCd < 2$. The mCd provides a means of quantifying the overall degree of contamination.

3.5.2 *Nemerow pollution index (PI_N) and pollution load index (PLI)*

The Nemerow pollution index results revealed that Ti, Zr, Sn, Ba and Pb were the elements of concern with a potential threat to the rivers sand studied (Tables 4, 5, 6). The PI_N values between 2 and 3 indicate moderate pollution and value greater than 3 indicate serious pollution (Cheng et al. 2007). In NKR, Ti, Zr and Ba have a moderate effect on the soils while Sn (PI_N = 9.47) has a serious effect on the soils. Mn, Ni, Cu and Zn are in the safety region with $PI_N < 0.7$ while Fe and Sr are in the precaution domain and Pb in the slightly polluted domain (Table 4). In LR samples, Zr (PI_N = 3.84) and Sn (PI_N = 9.18) have serious pollution effects on the soil. Zn, Ba and Pb having

Table 4 Summary statistics of the analysed pollution indices for NKR samples

Heavy metal concentration in mg/kg												
Index	Statistics	Ti	Fe	Mn	Ni	Cu	Zn	Sr	Zr	Sn	Ba	Pb
CF	Mean ± SD	1.46 ± 0.58	0.53 ± 0.27	0.29 ± 0.13	0.15 ± 0.13	0.13 ± 0.03	0.26 ± 0.07	0.99 ± 0.04	1.40 ± 0.44	7.64 ± 1.28	2.71 ± 0.11	1.84 ± 0.10
PI_N		2.32	0.91	0.57	0.47	0.16	0.33	1.03	2.24	9.47	2.85	1.93
PLI	Mean ± SD	0.73 ± 0.13										
mCd	Mean ± SD	1.58 ± 0.18										
I_{geo}	Mean ± SD	− 0.14 ± 0.54	− 1.67 ± 0.81	− 2.46 ± 0.52	− 3.60 ± 0.69	− 3.58 ± 0.43	− 2.61 ± 0.41	− 0.59 ± 0.05	− 0.15 ± 0.39	2.33 ± 0.24	0.85 ± 0.06	0.29 ± 0.08
EF	Mean ± SD	1.4 ± 0.54	0.51 ± 0.27	0.29 ± 0.15	0.14 ± 0.12	0.13 ± 0.03	0.25 ± 0.06	0.97 ± 0.04	1.37 ± 0.42	7.43 ± 1.08	2.64 ± 0.12	1.79 ± 0.08

Table 5 Summary statistics of the analysed pollution indices for LR samples

Heavy metal concentration in mg/kg												
Index	Statistics	Ti	Fe	Mn	Ni	Cu	Zn	Sr	Zr	Sn	Ba	Pb
CF	Mean ± SD	1.23 ± 0.38	0.35 ± 0.11	0.21 ± 0.07	0.09 ± 0.02	0.14 ± 0.09	0.39 ± 0.57	0.96 ± 0.06	1.47 ± 0.81	7.59 ± 1.55	2.65 ± 0.18	1.78 ± 0.20
PI_N		1.87	0.56	0.39	0.14	0.42	2.12	0.99	3.84	9.18	2.76	2.23
PLI	Mean ± SD	0.17 ± 0.17										
mCd	Mean ± SD	1.53 ± 0.24										
I_{geo}	Mean ± SD	− 0.35 ± 044	− 2.17 ± 0.42	− 2.87 ± 0.35	− 3.95 ± 0.28	− 3.53 ± 0.58	− 2.46 ± 0.93	− 0.65 ± 0.09	− 0.13 ± 0.46	2.31 ± 0.31	0.82 ± 0.10	0.24 ± 0.14
EF	Mean ± SD	1.26 ± 0.46	0.36 ± 0.13	0.22 ± 0.09	0.10 ± 0.02	0.15 ± 0.12	0.42 ± 0.69	0.97 ± 0.03	1.53 ± 1.04	7.73 ± 1.71	2.69 ± 0.09	1.81 ± 0.31

Table 6 Summary statistics of the analysed pollution indices for the RMYG samples

Heavy metal concentration in mg/kg

Index	Statistics	Ti	Fe	Mn	Ni	Cu	Zn	Sr	Zr	Sn	Ba	Pb
CF	Mean ± SD	2.57 ± 5.76	0.62 ± 0.18	0.19 ± 0.02	0.08 ± 0.01	0.08 ± 0.03	0.18 ± 0.03	0.86 ± 0.05	1.58 ± 0.67	7.66 ± 1.42	2.71 ± 0.13	2.20 ± 0.11
PI_N		3.59	1.19	0.29	0.14	0.15	0.29	1.31	3.64	13.13	4.06	3.27
PLI	Mean ± SD	0.65 ± 0.07										
mCd	Mean ± SD	1.59 ± 0.15										
I_{geo}	Mean ± SD	− 0.22 ± 0.42	− 1.33 ± 0.42	− 2.98 ± 0.18	− 4.28 ± 0.23	− 4.48 ± 0.85	− 3.09 ± 0.24	− 0.80 ± 0.08	− 0.03 ± 0.54	2.33 ± 0.28	0.85 ± 0.07	0.55 ± 0.07
EF	Mean ± SD	1.29 ± 0.47	0.61 ± 0.18	0.18 ± 0.02	0.08 ± 0.01	0.07 ± 0.03	0.17 ± 0.02	0.83 ± 0.02	1.54 ± 0.69	7.44 ± 1.46	2.63 ± 0.18	2.13 ± 0.08

PI_N between 2 and 3 are in the moderately polluted region. Higher PI_N values were observed in the RMYG samples with Ti, Zr, Sn, Ba and Pb having $PI_N > 3$ (seriously polluted domain) (Table 6). Mn, Ni, Cu and Zn were in the safety domain with PI_N values less than 0.7 and Fe and Sr have a slight effect on the soil.

The average PLI values were 0.73 ± 0.13, 0.17 ± 0.17 and 0.65 ± 0.07 for NKR, LR and RMYG samples respectively (Tables 4, 5, 6). Only two samples, one each from NKR and LR had PLI values greater than one. All other samples had PLI values less than one (1), signifying that the soils were not polluted. This implied that effort should be made to maintain or even reduce activities that pollute the soil. Differences in the PLI values between the three rivers are shown in the boxplot of Fig. S4. NKR samples seem to show higher values compared to LR and RMYG.

3.5.3 Geo-accumulation index (I_{geo}) and enrichment factor (EF)

The I_{geo} values in all the analyzed metals fall in the unpolluted class ($I_{geo} < 0$), except Ba and Pb which were within the unpolluted to moderately polluted range ($0 \leq I_{geo} \leq 1$) and Sn which falls in the moderately polluted to highly polluted class with I_{geo} in the range $2 < I_{geo} \leq 3$ (Muller 1981). The I_{geo} results are presented in Fig. 3 and Tables 4, 5 and 6. The mean I_{geo} values of Sn are 2.33 ± 0.24 in NKR, 2.31 ± 0.31 in LR and 2.33 ± 0.28 in RMYG. For Ba and Pb, the average I_{geo} values are 0.85 ± 0.06 and 0.29 ± 0.08 in NKR, 0.82 ± 0.10 and 0.24 ± 0.14 in LR and 0.85 ± 0.07 and 0.55 ± 0.07 for RMYG respectively. The values of Sn, Ba and Pb are seen to be higher than other metals in all three rivers. The source of Sn in these river sands may be due to the accumulation of manure applied in nearby agricultural soils and municipal refuse wastes. Ba and Pb may be from automobile brake linings and exhaust (Kanu et al. 2017). Other sources of Pb may be from fertilizers applied on nearby farmlands and leaded paints.

The computed mean EF of all heavy metals in the three sites varied in the order Sn > Ba > Pb > Zr > Ti > Sr > Fe > Mn > Zn > Ni > Cu (Tables 4, 5, 6). Although slight variations were observed in LR, where the EF values of Zn was greater than Fe and that of Cu was greater than Ni. Sr, Fe, Mn, Ni and Cu displayed deficiency to minimal enrichment in all the samples, which indicates geogenic or crustal influence. This is equally the case for most samples of Ti, Zr and Zn. In LR, about 15% of samples had EF values of Ti > 1.5, 7% of samples had EF values of Zn > 1.5, 11% of samples had EF values of Zr > 1.5 and 100% of samples indicated EF of Pb > 1.5 (specifically, 1.63–3.23), which indicates the anthropogenic influence of these samples. All the samples in NKR showed that the EF values of Pb is greater than 1.5 (i.e., 1.66–1.90), 35% of samples show EF values for Ti > 1.5 and 18% of samples showed EF values of Zn > 1.5. Similarly, in RMYG, 19% of samples and 24% of samples have been enriched with Ti and Zr respectively. In all the rivers, Ba showed moderate enrichment while Sn was significantly enriched. Again, the EF has further exposed the high anthropogenic influence of Sn concentration in the river sands. Generally, EF > 1.5 depicts anthropogenic contribution to the metal contents while EF < 1.5 suggests the crustal or geogenic origin of metal (Zhang and Liu 2002; Kumar et al. 2016). Based on the criteria, Sn, Ba and Pb in NKR, Zr, Sn, Ba and Pb in LR and RMYG might have been influenced by anthropogenic sources.

3.5.4 *Ecotoxicological evaluation of contamination by heavy metal*

In order to further verify the quality of the river sands, the concentration of Ni, Cu, Zn and Pb were compared with the following sediment quality guidelines (SQGs): TEL, ERL, PEL, SEL and ERM (Table 2). This assessment was not carried out for Al, Ti, Mn, Fe, Sr, Zr, Sn and Ba since there were no SQGs specified for them. It was found that the contents of Ni, Cu, Zn and Pb in the investigated ecosystem were far lower than the reference SQGs values with few exceptions. In NKR, the concentration of Ni in one sample was 30.4 mg/kg which exceeded the TEL and ERL value of 18 and 30 respectively. Also, in LR, the concentration of Zn in a sample was 198.7 mg/kg which is higher than the TEL and ERL limit of 123 and 120 respectively. This implied that these samples must have been affected by anthropogenic activities and may pose ecotoxicological risk. In all the sampling sites, Pb seems to be the only element of concern with the likelihood of causing harm if unchecked. In RMYG, Pb (mean concentration of 37.40 mg/kg) is about 6.42% higher than TEL and ERL but very far below other SQGs (PEL, SEL and ERM). It should be noted that the concentration of Pb in about 95% of the samples was more than TEL and ERL. In NKR and LR, Pb is only 11.89% and 16.05% less than TEL and ERL respectively. It should be noted that Ni, Cu, Zn and Pb concentration in all the sites were far below the PEL, SEL and ERM values.

The estimated mean probable level effect quotient ($mPEL_Q$) in the studied river sands varied from 0.12 to 0.33 (mean, 0.15 ± 0.05), 0.11 to 0.35 (mean, 0.14 ± 0.05) and 0.12 to 0.17 (mean, 0.13 ± 0.01) for NKR, LR and RMYG samples respectively. The implication of this result is that the combined effect of Ni, Cu, Zn and Pb has 21% probability of being toxic to the aquatic ecosystem. The highest $mPEL_Q$ value was obtained from LR (Fig. 4). The $\mathrm{mERM_Q}$ varied in a similar manner as the $mPEL_Q$ (Fig. 4). The result estimated showed that $\mathrm{mERM_Q}$ varied from 0.10 to 0.25 (mean, 0.12 ± 0.04), 0.09 to 0.33 (mean, 0.12 ± 0.05) and 0.10 to 0.14 (mean, 0.12 ± 0.01) for NKR, LR and RMYG samples respectively. All the sites had an approximate average value of 0.12 which signifies medium to low priority sites and 21% probability of being toxic to biota.

The Hazard Quotients (HQ) estimated for Ni, Cu, Zn and Pb portrayed lead as the likely element to pose possible hazards to the biota. Ni, Cu and Zn had comparable values with few anomalies suspected to come from point sources (Fig. 5). The average values of Ni varied in the order NKR (0.38 ± 0.33) > LR (0.26 ± 0.06) > RMYG (0.20 ± 0.03); Cu: LR (0.11 ± 0.07) > NKR (0.10 ± 0.03) > RMYG (0.06 ± 0.03); Zn: LR

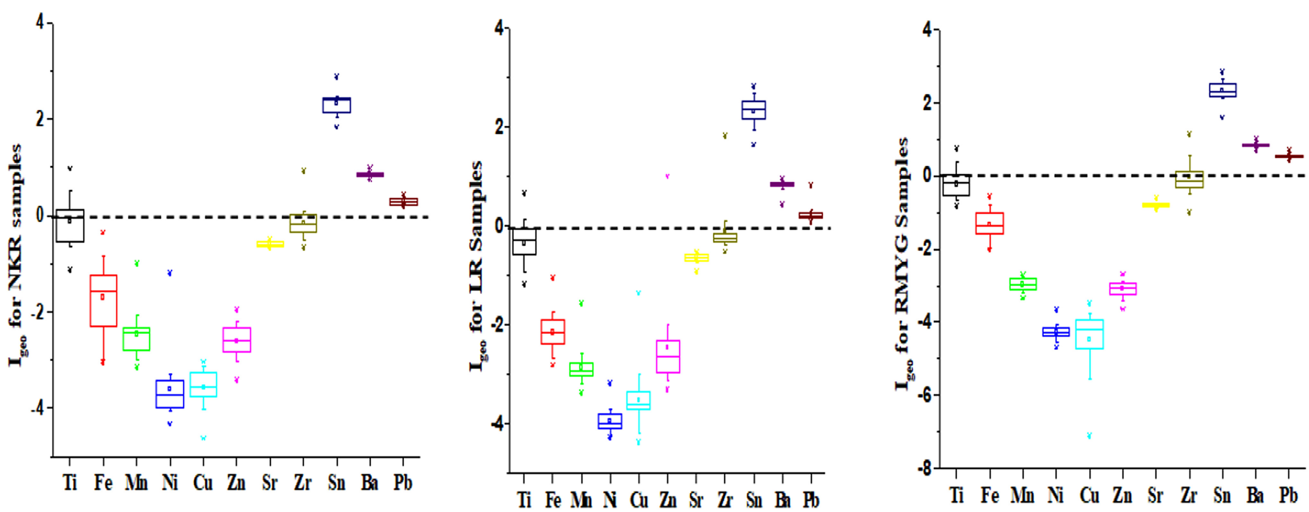


Fig. 3 Geo-accumulation index for the NKR, LR and RMYG samples

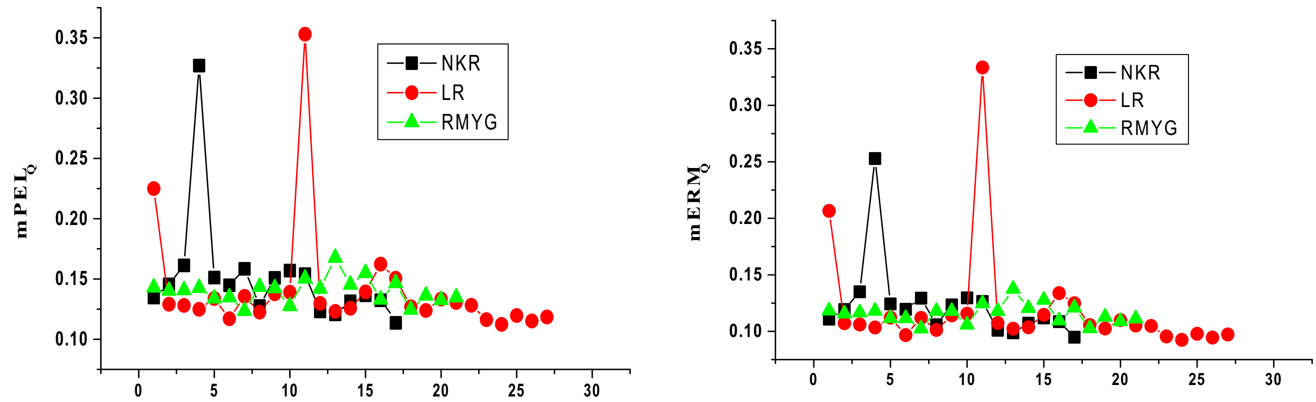


Fig. 4 Distribution of $mPEL_Q$ and $mERM_Q$ in the study areas

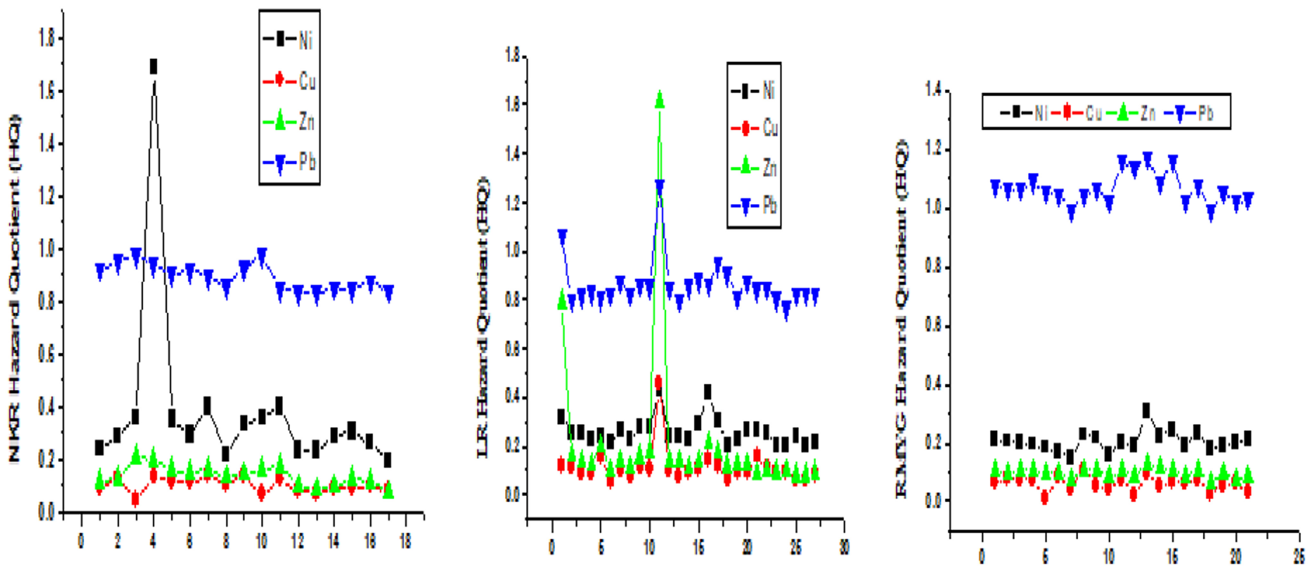


Fig. 5 Hazard quotients for NKR, LR and RMYG sand samples

(0.21 ± 0.31) > NKR (0.14 ± 0.04) > RMYG (0.09 ± 0.02) and Pb: RMYG (1.07 ± 0.05) > NKR (0.89 ± 0.05) > LR (0.86 ± 0.09). The mean values of the HQ in all the sites suggests evidence of potential hazards, being between 0.1 and 1 (Benson et al. 2018). Although some sampling points had values > 1 which indicate moderate hazard. The considerable high lead levels in all the sites should not be overlooked as its possibility of causing environmental and health problems is high. PEL$_Q$ and ERM$_Q$ results are in close corroboration with the HQ results on high Pb risks.

An attempt was made to investigate possible variation from the HQ results obtained if the modified hazard quotient (mHQ) formulated by Benson et al. (2018) is used instead. The results obtained further put Pb at the forefront of the element of concern (Fig. 6). Pb again dominated. In NKR, Ni varied from 0.58–1.71 (mean, 0.77 ± 0.25), Cu from 0.41–0.71 (mean, 0.59 ± 0.08), Zn from 0.95–1.57 (mean, 1.42 ± 0.71) and Pb from 1.68–1.82 (mean, 1.74 ± 0.05). In LR, the elements varied as follows: Ni, 0.59–0.86 (mean, 0.67 ± 0.07), Cu, 0.44–1.25 (mean, 0.61 ± 0.15), Zn, 0.98– 4.38 (mean, 1.42 ± 0.71) and Pb, 1.61–2.07 (mean, 1.71 ± 0.09) and in the RMYG, Ni: 0.51–0.73 (mean, 0.59 ± 0.05), Cu: 0.11–0.39 (mean, 0.29 ± 0.07), Zn: 0.32–0.44 (mean, 0.39 ± 0.03) and Pb: 1.23–1.34 (mean, 1.28 ± 0.31). It can be observed that from the average mHQ values that only Cu and Zn in RMYG indicates nil to very low severity of contamination (mHQ < 0.5). Pb in all the sites indicated moderate severity of contamination (1.5 < mHQ < 2.0) and other elements in all the sites indicated very low severity of contamination (0.5 < mHQ < 1.0). The findings from mHQ agreed reasonably well with other indices, thus justifying the use of this new index and confirming the statement of Benson et al. (2018) that the index is reliable

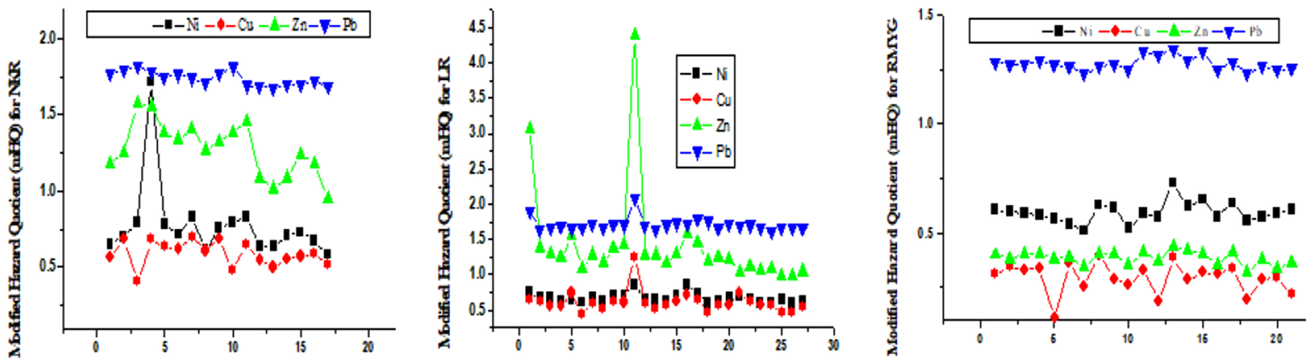
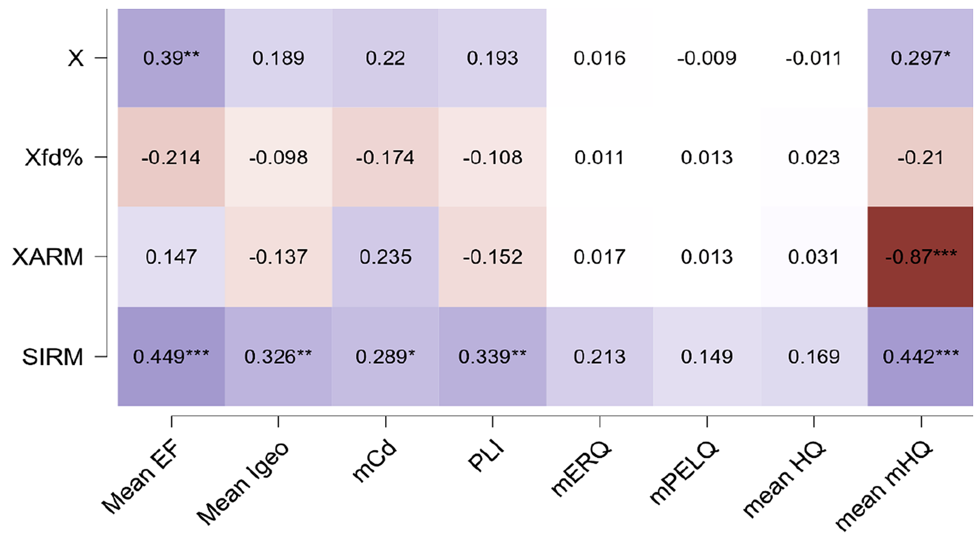


Fig. 6 Modified hazard quotients of Ni, Cu, Zn and Pb in NKR, LR and RMYG sand samples

Fig. 7 Heat map showing the covariation between magnetic properties and pollution and eco-toxicological indices. *$P < 0.05$; **$P < 0.01$ and ***$P < 0.001$



and useful tool for assessing the degree of pollution in marine ecosystems by heavy metals.

3.6 Relationship between magnetic susceptibility and some pollution indices

The use of magnetic susceptibility as a heavy metal pollution proxy has been affirmed by many authors (Yang et al. 2010; Kanu et al. 2017) and in this study. We assessed further the possibility of using magnetic properties as a proxy for pollution indices and ecotoxicological parameters. The Pearson correlation between magnetic parameters (χ, χfd%, χARM and SIRM) and pollution and ecotoxicological indices (PLI, mCd, mean I_{geo}, mean EF, $mPEL_Q$ and $mERM_Q$) was performed. The results which were displayed in a heat map (Fig. 7) showed great potential for magnetic concentration-dependent parameters acting as a proxy for the analysed pollution indices. It was observed that the χ, χARM and SIRM which depend on the concentration of magnetic minerals in a substance displayed varying degrees of covariation with PLI, mCd, mean I_{geo}, mean EF, $mPEL_Q$ and $mERM_Q$. This means that magnetic parameters (χ, χARM and SIRM) can effectively be used as proxy for the indices. χ showed moderate relationship with only mean EF ($r = 0.39$, $P < 0.01$) and mean mHQ ($r = 0.297$, $P < 0.05$). χARM showed poor relationship with other indices and a good relationship with only mean mHQ ($r = -0.870$, $P < 0.001$), implying that χARM might not be suitable for assessment of the pollution indices (except the mHQ) in the study area. SIRM correlated positively with mean EF ($r = 0.449$, $P < 0.001$), mean I_{geo} ($r = 0.326$, $P < 0.01$), mCd ($r = 0.289$, $P < 0.05$), PLI ($r = 0.339$, $P < 0.01$) and mean mHQ ($r = 0.442$, $P < 0.001$). The result showed that SIRM correlates well with all the pollution indices and among the ecotoxicological indices, it relates significantly with only mHQ. This implied that SIRM could be a suitable parameter for assessing the level of pollution in the study area. It is observed that SIRM exhibited the best relationship when compared to χ and χARM, confirming our earlier position that SIRM is a better magnetic proxy parameter for pollution assessment. The result of this study does not agree

Table 7 Principal component analysis results for the river sands. PC loadings greater than 0.5 are regarded as making significant contributions to each PC

Variables	Principal components					Uniqueness
	PC1	PC2	PC3	PC4	PC5	
Zr	0.871					0.149
Ti	0.808					0.192
SIRM	0.766					0.092
χ	0.573					0.329
χfd%	− 0.537					0.422
mCd					0.820	0.021
PLI		0.860				0.025
Fe		0.820				0.306
Zn		0.602				0.485
Cu		0.576	0.548			0.304
Ni		0.559				0.482
Mn		0.542	− 0.608			0.244
Pb			− 0.801			0.154
χARM			0.780			0.273
Sr			0.767			0.187
Ba				0.877		0.160
Al				0.856		0.187
Sn					0.977	0.028
Sum of square loadings	3.202	3.169	3.108	2.470	2.010	
Proportion variance	0.178	0.176	0.173	0.137	0.112	
Cumulative	0.178	0.354	0.527	0.664	0.775	

Applied rotation method is varimax

with that of Yang et al. (2010) who obtained good relation between χ, χARM and PLI. It was also noticed that all the magnetic concentration parameter relates significantly to the modified hazard quotient, making them a good proxy for the ecotoxicological index. Some of the relationships are displayed in a scatter plot (Fig. S5). To the best of our knowledge, no work has made attempt to investigate the relations that might exist between the magnetic properties and modified contamination degree, mean probable effect level quotient and mean effect-range median quotient. Therefore, more studies are required to authenticate the results of this study. However, since magnetic susceptibility is affected by geology and geographical location, the conclusions derived from similar results should be made in consideration of factors such as geology, geography, soil type etc.

3.7 Multivariate statistical analysis

The essence of the multivariate statistical analysis was for distinguishing and identification of heavy metal sources. The following parameters were subjected to the principal component analysis: χ, χfd%, χARM, SIRM, Al, Ti, Fe, Mn, Ni, Cu, Zn, Sr, Zr, Sn, Ba, Pb, PLI and mCd. The principal component analysis (PCA) indicated five (5) principal components (PCs) having a cumulative variance of 77.50% (Table 7). The uniqueness on Table 7 refers to the proportion of the variance of each parameter that was not explained by the components. The uniqueness indicated well representation of all the parameters ranging from Ni (51.8%) to mCd (97.9%) (Table 7). The first PC (PC1) comprised 17.8% of the explained variance with the following members: Zr, Ti, SIRM, χ and χfd%. This suggests a combination of lithogenic and anthropogenic sources. The magnetic concentration parameters present in this PC gives an indication of strong anthropogenic input. Zr and Ti are crustal elements but their average concentration in the river sands was higher than the UCC values (Table 2), and being in the same group with magnetic concentration parameters highlights strongly the addition of human activities to the river sands. Also, χfd% has a significant but negative value in PC 1. This further support the anthropogenic inputs to the environment. PC 2 has nearly the same number of parameters as PC 1 and contributes about 17.6% of the total explained variance. The members of this group are PLI, Fe, Zn, Cu, Ni, and Mn. This is clearly from pollution sources, resulting from man's activities. Zn may be obtained from pesticides applied on the nearby farms and waste (refuse) dumped into the water and surrounding lands that are washed or eroded into the

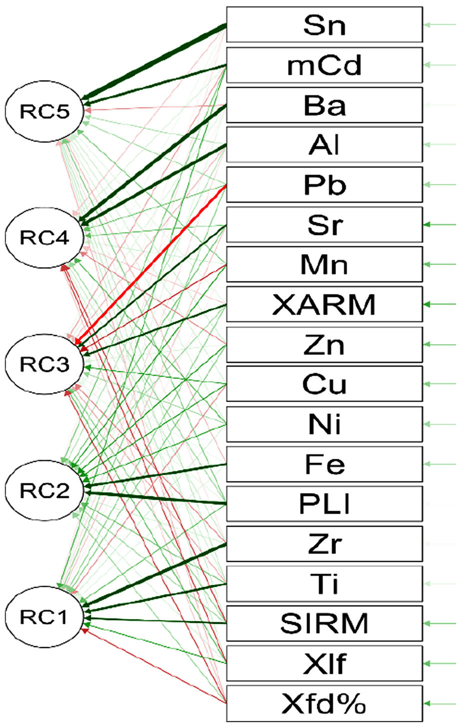


Fig. 8 Path diagram for the Jalingo river sands

river sands. Moreover, Zn and its compounds are utilized in the manufacture of fertilizers for agricultural purposes (Zhang et al. 2013). PC 3 contained 17.3% of the total explained variance and was composed of Cu, Mn, Pb, χARM and Sr. Cu contributes 54.8% to this PC and 57.6% to PC 2. This implied that Cu had dual sources (anthropogenic and lithogenic) in the river samples. Since copper is used as an essential ingredient in livestock feeds (Guan et al. 2018), it can be converted to animal manure and hence serve as a pointer to the application of animal manure (Zhang et al. 2012). Cupper accumulation in the soil can also result from the use of pesticides (Liang et al. 2017; Jiang et al. 2020). Furthermore, it should be noted that local farmers usually apply either fertilizer or pesticides without knowledge of the recommended quantity. That is to say, expert's advice is not sought in most instances. This could lead to high concentration of metals in the soil. Mn also exhibited dual sources in the studied area. It contributes 60.4% to PC 3 and 54.2% in PC 2. However, Mn and Pb were negatively correlated with χARM signifying that they may be from polluted sources while Sr may originate from geogenic sources. Pb enhancement in the river sands is traced to vehicular and traffic waste deposition and as an additive in paints (Kanu et al. 2017). The components of PC 4 which comprised 13.7% of the variance are Ba and Al. These are clearly from underlying rocks that cropped to the surface due to weathering activities. PC 5 contributes 11.2% of the total variance and consists of mCd and Sn. Sn in the river sands may originate from manure added to the nearby agricultural soils and metal scrapings from farm implements etc.

The path diagram (Fig. 8) is used to confirm the PC results. The path diagram represents the pictorial view of the relationship between the variables. The strength of the correlation is indicated by the wideness of the arrows which move from the parameters to each PC. The colour of the arrows is also instructive. Negative loadings are depicted in red while green depicts positive loadings. Also, greater loadings are indicated by wider arrows. The path diagram clearly grouped the parameters into five groups coincident with the results obtained from the PCA analysis.

4 Conclusion

The magnetic properties and metal contents of beach sands collected from three rivers in Jalingo Metropolis have been measured. The mean magnetic susceptibility values varied in the following order: LR (39.27×10^{-8} m^3 kg^{-1})->NKR (24.53×10^{-8} m^3 kg^{-1}) > RMYG (12.76×10^{-8} m^3 kg^{-1}). These results obtained indicated that there is an enhancement in the magnetic signal of the beach sand investigated and by extension increased contamination in the river samples. The χfd% values obtained from the three rivers were less than 4%, indicating that the samples were controlled by MD magnetic grain- sizes. This was also supported by Dearing's plot which established that MD and coarse SSD dominated the samples. MD + SSD grain sizes are characteristics of anthropogenic influence in the river sand samples. Furthermore, it was observed from S-ratio results that the river sands were controlled by low coercivity, and soft magnetic minerals. The significant correlation between magnetic susceptibility and other concentration-dependent magnetic parameters brings to light the dominance of ferrimagnetic magnetite-type minerals in the samples. This implied that the influence of diamagnetic and paramagnetic minerals in the river samples was subdued by the addition of different pollution sources containing ferrimagnetic minerals. There were significant differences in the concentrations of geochemical parameters in the three river samples; however, Ti, Zr, Sn, Ba and Pb have been enhanced above background value indicating anthropogenic influence. The high values of these metals may indicate a potential health threat to humans. The fact that higher values of heavy metals have been found in the samples from the river sands shows that there is a strong need for further studies to be carried out in the nearby coastal plains where a lot of agricultural activities is carried out. The heavy metals content of some of the vegetables grown needs to be measured also. A significant correlation existed between the magnetic parameters and some heavy metals, though at varying degrees. SIRM was found to exhibit the most significant correlation with the metals. This shows that there is great potential for the use of environmental magnetic methods, especially SIRM as a proxy for pollution assessment in the river samples. The magnetic method is fast and cost-effective and therefore can be used to complement the traditional geochemical methods of pollution assessment. Further attempt to properly investigate the pollution status and possible effect on human and the river ecosystems were made using several pollution (CF, Cd, mCd, PLI, PI_N, EF and I_{geo}) and ecotoxicological (SQG, HQ, mHQ, $mPEL_Q$ and $mERM_Q$) indices. Results showed low to moderate pollution and a low to moderate effect on biota was observed. However, Sn, Ba and Pb were the elements with a potential threat to humans and aquatic organisms. It must be noted that the study was carried out in a fast-growing city where industrial activities are low. The major contamination sources revealed by multivariate statistics are agricultural, waste/refuse dumps, metal scraps, and vehicular sources. Hence, government and other agencies must keep watch and maintain the current level. Another novel result deduced from this study is the discovery that a significant positive covariation exists between some magnetic properties (χ, χARM and SIRM) and pollution indices (mCd, PLI, mean EF and mean I_{geo}) and ecotoxicological index (mean mHQ). Again, SIRM showed the most significant interrelationship with these parameters. Thus, for the investigated Jalingo river sands, SIRM is the most suitable magnetic proxy parameter that can be used to infer the degree of pollution and risk level. However, more case studies from different pollution sources, geological backgrounds and geographical locations are required to authenticate the finding.

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Availability of data and materials Any additional data and material in respect of this manuscript will be made available on request.

Declarations

Conflict of interests The authors declare that they have no conflict of interest.

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