Determination of Heavy Metal levels in Soil and Vegetable Samples around Automobile Workshops in Iworoko-Ekiti, Nigeria

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Received: 06-FEB-2022; Reviewed: 13-MAR-2022; Accepted: 01-MAY-2022
https://doi.org/10.46792/fuoyejet.v7i2.795

ORIGINAL RESEARCH ARTICLE

Abstract- This study identified and determined levels of eighteen heavy metals in six soil and twelve vegetable samples (*Talinum triangulare* and *Amaranthus hybridus*) from selected automobile workshops at Iworoko Ekiti using Energy Dispersive X-ray Fluorescence Spectrometry (EDXRF). Identical samples were also collected outside this neighborhood as control and analyzed using the same analytical technique. The results indicated that the observed heavy metals in the samples from the sites and control were within the range (1.044-3180.224) mg/kg and (1.002-1845.744) mg/kg. The soil samples were slightly enriched in vanadium, manganese, nickel, copper ,zinc, arsenic, selenium and rubidium (Enrichment Factor (EF) = 1.033 – 2.179). Significant differences also exist between the levels of calcium, titanium, vanadium, chromium, manganese, iron, nickel, copper, arsenic, and yttrium (at $p < 0.05$, $t = 0.00002 – 0.03950$). The accumulation factors of the vegetable samples in *Amaranthus hybridus* were in the range 0.0006 – 824.1057 and those of *Talinum triangulare* were in the range 0.00019 – 338.76885. The relatively higher levels of chromium, manganese, iron, nickel, copper, zinc and arsenic in the vegetable samples from the sites compared with WHO/FEPA permissible limits was an indication of contamination: The consumption of vegetables in such an environment could be detrimental to health.

Keywords- Energy Dispersive X-ray Fluorescence analysis, Soil and vegetable samples, Automobile workshops

1 INTRODUCTION

Environmental pollution by heavy metals is a worldwide phenomenon that adversely affects human health and animals (Ingrid *et al.*, 2014; Mansour, 2016). Discharge of heavy metals from most of the vehicles on soil and vegetables causes a serious soil pollution which eventually affects the vegetables grown in that particular area (Mahfuza *et al.*, 2011). Soil contaminated with heavy metals through the repeated use of partially treated river water and unlimited use of fertilizers and pesticides is also one of the severe ecological problems (Ahmad and Goni, 2010).

Recent studies show that heavy metals are potentially toxic to humans and animals through soil and vegetables. Heavy metals absorption through the food chain by the living organism has been widely reported throughout the world (Muchuweti *et al.*, 2006). However, it has also been discovered that most of the human activities continuously release radiation or heavy metals from both terrestrial and extra-terrestrial sources.

The terrestrial radiation and heavy metals are released into the human body through the vegetables we feed on, and the amount depends on the types of soils, rocks, air, water, and food (Mahfuza *et al.*, 2011). Also, heavy metals can be discharged from most of the vehicles on vegetables and soil thereby causing pollution which eventually increase their levels in those media (Mahfuza *et al.*, 2011). Recent studies show that heavy metals are potentially toxic to humans and animals through soil and vegetables. When contaminated soil is used for crop production and the crops are consumed, they accumulate in vital organs (Sharma *et al.*, 2009). The accumulation of heavy metals in organs such as the kidney, liver and bones greatly cause health disorder in human body due to their non-biodegradable and persistent nature (Duruibe *et al.*, 2007).

However, plants react differently to the nutrients and metal concentrations through their changes in pigment concentration, water content, dry weight and growth. The summation of all the plant responses results in distinct light absorption and reflectance characteristics of the plants and invariably lead to soil contamination. Numerous studies have shown that metal and nutrient stress in plant give rise to changes in the spectra reflectance of the vegetation (Javid *et al.*, 2018) leading to several physiological and biochemical changes in plants which can significantly alter the availability of nutrients in the vegetables.

Another important and identified anthropogenic activity that can cause heavy metal pollution is the automobile workshops that deals with the auto-body works such as

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Section D- MATERIALS/ CHEMICAL ENGINEERING & RELATED SCIENCES
Can be cited as:
Oketayo O.O., Oke A.O., Adeyemi F.O., Akinnubi R.T., Ajao E.O., and Ayanda O.S. (2022): "Determination of Heavy Metal levels in Soil and Vegetable Samples around Automobile Workshops in Iworoko-Ekiti, Nigeria", *FUOYE Journal of Engineering and Technology* (FUOYEJET), 7(2), 222-228. http://doi.org/10.46792/fuoyejet.v7i2.795

car painting, panel beating, spray painting and mechanic or car repairs (John *et al*., 2019). In a similar study by Utang *et al* (2013), findings revealed that, at 0.05 level of confidence, significant variations exist in the levels of heavy metals such as mercury, lead and cadmium between the experimental sites and the control in soil. Same trend was obtained by Abidemi (2011) for cobalt, iron, lead and cadmium.

In this study, the enrichment and accumulation factors were calculated after determining the levels/concentrations of heavy metals in soil and vegetable samples from selected automobile workshops via a multi-elemental analytical technique.

2 MATERIALS AND METHODS

Six (6) soil samples were collected using a hand soil auger from automobile workshops around Iworoko/Ado Ekiti area at the exact point where the vegetables were uprooted. Iworoko-Ekiti is a well-known place in the city of Ado-Akiti. It is situated in Ekiti-East, Nigeria with geographical coordinates 7° 44' 0'' North, 5° 16' 0'' East. It has a main road that leads to Ado-Ekiti and other well-known places. The soil was dug at a depth of 0.15 cm (top-surface) and 15-30 cm (sub-surface depths). The samples were well-labelled in clean polythene bags and taken to the laboratory for analysis. Three identical soil samples from an unpopulated area in Iworoko, Ekiti state were also collected into different polythene bags as control. Six vegetable samples were collected, three of which were water leaf (*Talinum triangulare*) and the remaining three were African spinach (*Amaranthus hybridus*). All the samples were collected at different locations with separate hand gloves worn to prevent contamination. Also, similar vegetables were taken from a site where no anthropogenic activity occurs as control.

2.1 SAMPLE PRE-TREATMENT

The debris content of the soil was firstly removed. This was followed by air-drying it in a clean, well ventilated laboratory under ambient temperature. The dried samples were homogenized by grinding and filtered by passing through a 2 mm mesh size sieve to remove gravels larger than 2 mm in diameter. The samples were then put in polythene bags and well labelled for analysis. Stalks of each vegetable were removed with nylon gloves worn and cleansed properly. Each vegetable sample was freeze dried, weighed with XY2000 C model electronic balance, ground to obtain a homogenous mixture, labeled and kept in different clean labeled polyethylene bags to avoid contamination.

2.2 INSTRUMENTATION AND DATA ANALYSIS

The analysis of X-ray spectroscopy is given by the equation (1) (Fowler and Chieh, 2021).

$$E_x = RhC(Z-\sigma)^2\left(\frac{1}{n_1^2}-\frac{1}{n_2^2}\right) \qquad (1)$$

where E_x is the characteristic X-ray energy, R is Rydberg constant (R= 1.09737 x 10^7 m^{-1}), h is Planck's constant (6.6262 x 10^{-34} Js) and C is the speed of photons, Z is the atomic number, σ is Shielding constant, n_1 and n_2 are the energy series. For the spectrum Kα_1, shielding constant σ = 1, n_1 = 1 (K-shell), and n_2 = 2 (L-shell). Thus, (1) can be rewritten as follows

$$E_{K\alpha_1} = \frac{3RhC(Z-1)^2}{4} \qquad (2)$$

This law reveals the relationship between the x-ray energy and atomic number. This law is the theoretical basis for the qualitative analysis of material composition using XRF. A positive relationship exists between the count rate of the characteristic x-rays and the content of an element of the tested sample as shown in equation (3) (Asam *et al*., 2012).

$$I_K = \frac{KI_0}{\mu_0+\mu_K} \times W_K \qquad (3)$$

where I_K and I_0 are the layer characteristics of the X-ray of the measured elements and the count rates of the incident X-ray, respectively. Moreover, μ_0 and μ_K are the absorption coefficients of the tested substance to the incident X-ray and the tested element to the layer characteristic X-ray, respectively. K is the constant related to the specific measurement device and should be determined by the calibration instrument $KI_0/(\mu_0+\mu_K)$. W_K is the measure of the content elements. The enrichment factor (EF) for each of the elements was determined by using crustal values and Fe as the reference element (Equation 4) (Oyebamiji *et al*., 2017). Thus, Equation 4 was used to determine how enriched the samples were in heavy metals.

$$E.F = \frac{X/(CONC\ of\ Fe\ (sample))}{Y/CONC\ of\ Fe\ (reference)} \qquad (4)$$

Where X is concentration of element in the sample and Y is the concentration of element in reference. The reference used in this research is the non-anthropogenic/contaminating site or control.

Since heavy metals translocate from soil to edible parts of the crops, the accumulation factors (AF) of each of the heavy metals was determined from the levels of these metals in the soil samples by the use of Equation (5) (Oyebamiji *et al*., 2017).

$$AF = \frac{Heavy\ metal\ conc.\ \text{in food crops edible part}}{Heavy\ metal\ conc.\ \text{in the}\ soil} \qquad (5)$$

In this study, 0.5 g of each dried sample was weighed and the elemental concentration was determined using the polarized EDXRF (SPECTRO LAB2000) Spectrometer.

2.3 QUALITY ASSURANCE AND STATISTICAL ANALYSIS

Standards and blanks were prepared for quality assurance and background correction as well as other sources of errors respectively. The equipment was properly calibrated with r ranging from 0.998 to 0.999 for the elements. This guaranteed the precision and the reliability of the results presented in this work. The data obtained were subjected to statistical analysis using Statistical Packages for Social Sciences (SPSS) software. The reference/control used in this research is the non-contaminating site.

http://doi.org/10.46792/fuoyejet.v7i2.795 http://journal.engineering.fuoye.edu.ng/

3 Results and Discussion

The comparison of the results observed with control for soil samples are shown in Figures 1a – b, while those of vegetables are depicted in Figures 2a – d. Tables 1 shows the comparison of the results obtained for vegetables in this study with FEPA/WHO limits. Tables 3 and 4 show the enrichment factors of the heavy metals in the soil and vegetables respectively. Table 5 gives the accumulation factors of heavy metals in the vegetable samples.

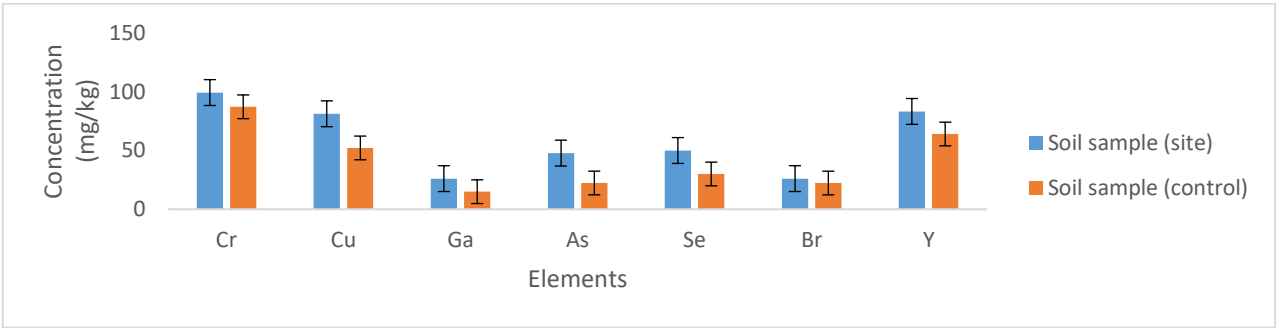

Fig. 1a: Levels of heavy metals in soil samples and control

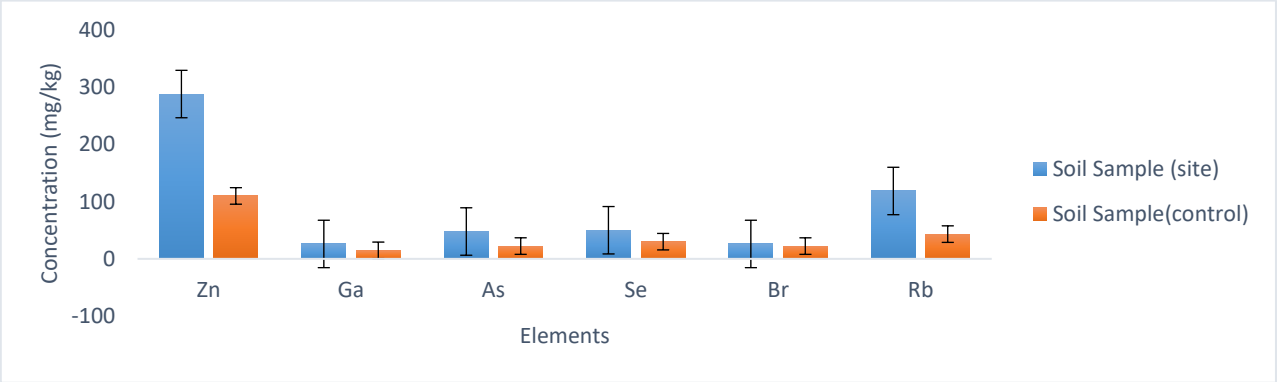

Fig. 1b: Levels of heavy metals in soil samples and control

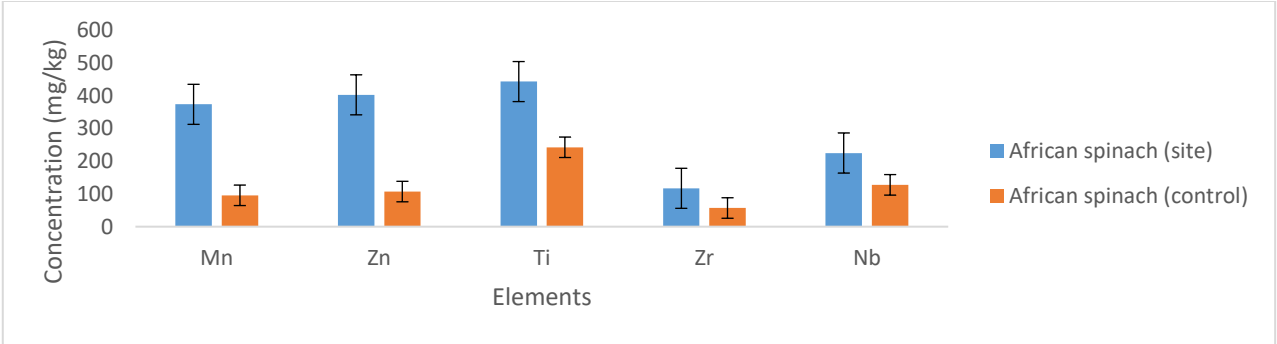

Fig. 2a: Levels of heavy metals in African spinach and control

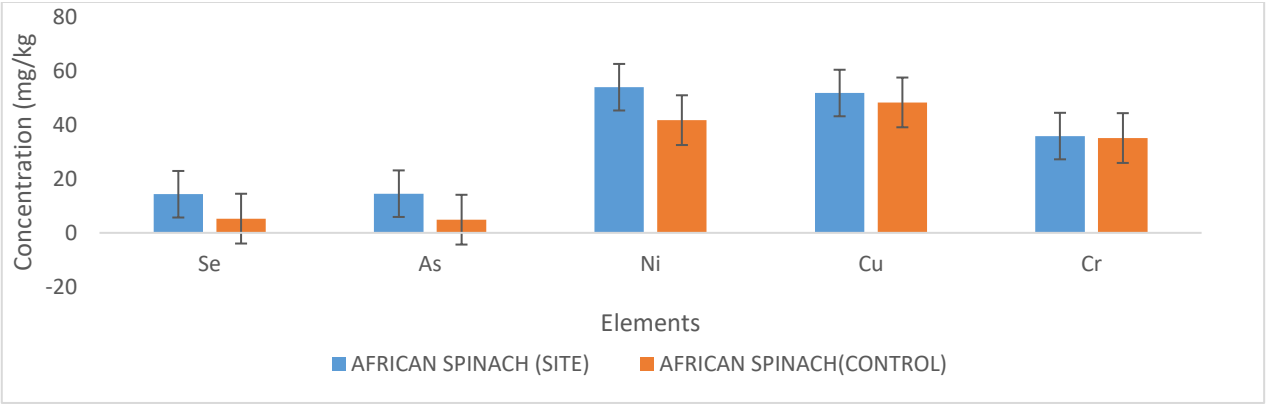

Fig. 2b: Levels of heavy metals in African spinach and control

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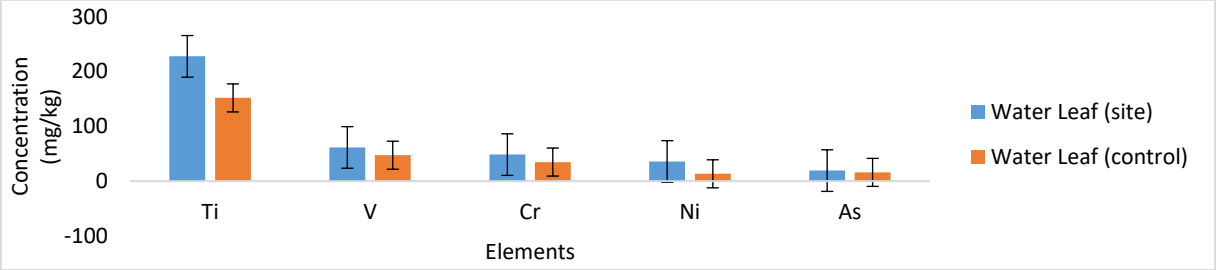

Fig. 2c: Levels of heavy metals in water leaf and control

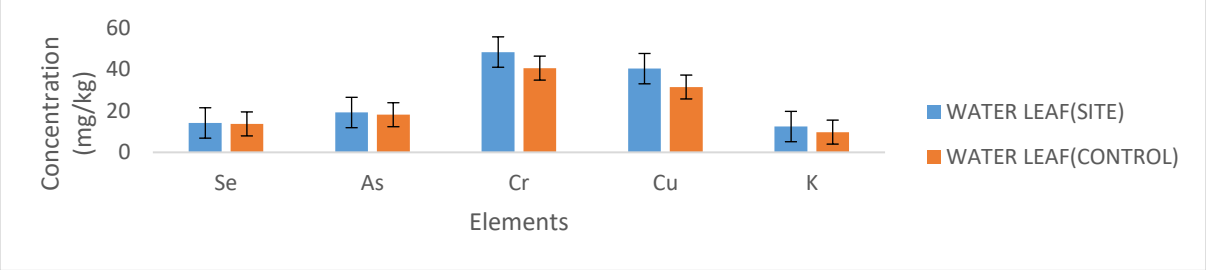

Fig. 2d: Levels of heavy metals in water leaf and control

Table 1. Comparison of the heavy metal content of the soil (mg/kg) with literature

Element	Soil samples (mg/kg) (In this study)	Soil samples (mg/kg) Aloysius *et al* (2013)	
		AP cluster	GBK cluster
Cu	81.217 ± 13.25	254.1 ± 236.0	1348.1 ± 1691
Zn	287.997 ± 16.35	295.5 ± 50.3	553.3 ± 284.8
Mn	1499.595 ± 86.04	58.8 ± 16.2	272.2 ± 106.2
Ni	195.495 ± 18.14	18.0 ± 4.2	12.7 ± 4.18
Cd	-	10.50 ± 24.59	12.7 ± 4.18

Note: AP = APIR and GBK = GBOKO (i.e the workshop places in Benue State).

Table 2. Levels of heavy metals (mg/kg) in vegetables and FEPA/WHO Limits

Element	Level in vegetable sample (mg/kg)				
	Amaranthus hybridus		*Talinum triangulare*		FEPA/WHO
	Site	Control	Site	Control	(2007) Standard
Ca	3.535	2.055	1.078	1.047	1000
Ti	475.582	266.837	227.897	173.635	1000
V	65.560	49.798	61.564	53.297	1000
Cr	42.139	41.181	48.456	40.712	2.300
Mn	373.799	95.822	228.214	85.175	6.640
Fe	3180.224	1845.744	1307.309	785.899	420.00
Ni	60.974	48.821	35.935	17.915	1000
Cu	58.174	54.261	40.435	31.565	40.000
Zn	402.802	107.232	144.566	48.921	60.000
As	15.803	5.775	19.251	18.182	1.400

Table 3. Enrichment factors of heavy metals in the soil samples

Element	Enrichment Factor	Degree of Enrichment
Ca	0.965	Not enriched
Ti	0.850	Not enriched
V	1.193	Slightly enriched
Cr	0.904	Not enriched
Mn	1.355	Slightly enriched
Fe	0.814	Not enriched
Ni	1.236	Slightly enriched
Cu	2.080	Slightly enriched
Zn	1.386	Slightly enriched
As	1.625	Slightly enriched
Se	1.323	Slightly enriched
Br	0.928	Not enriched
Rb	2.179	Slightly enriched
Y	1.033	Slightly enriched
Nb	0.884	Not enriched

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Table 4. Enrichment factors of the heavy metals in the vegetable samples

Element	*Amaranthus hybridus*		*Talinum triangulare*	
	EF	Degree of EF	EF	Degree of EF
Ca	0.998	Not enriched	0.619	Not enriched
Ti	1.034	Slightly enriched	0.789	Not enriched
V	0.764	Not enriched	0.694	Not enriched
Cr	0.594	Not enriched	0.715	Not enriched
Mn	2.264	Slightly enriched	1.611	Slightly enriched
Fe	1.000	Slightly enriched	1.000	Slightly enriched
Ni	0.725	Not enriched	1.206	Slightly enriched
Cu	0.622	Not enriched	0.770	Not enriched
Zn	2.180	Slightly enriched	1.776	Slightly enriched
As	1.588	Slightly enriched	0.637	Not enriched
Se	1.422	Slightly enriched	0.622	Not enriched
Br	0.638	Not enriched	0.633	Not enriched
Rb	2.328	Slightly enriched	0.853	Not enriched
Y	1.190	Slightly enriched	0.683	Not enriched
Nb	1.022	Slightly enriched	0.585	Not enriched

Note: EF = Enrichment Factor

Table 5. Accumulation factors of heavy metals in the vegetable samples

Element	Accumulation factors	
	Amaranthus hybridus	*Talinum triangulare*
Ca	0.0006	0.00019
Ti	0.0943	0.04517
V	0.2132	0.20016
Cr	0.4244	0.48806
Mn	0.2493	0.15218
Fe	824.1057	338.76885
Ni	0.3119	0.18381
Cu	0.7163	0.49786
Zn	1.3986	0.50197
As	0.3306	0.40279
Se	0.3067	0.28411
Br	1.2397	0.66292
Rb	0.5911	0.36450
Y	0.7885	1.68432
Nb	0.7442	0.88371

The levels of heavy metals in the soil samples were determined using the Energy Dispersive X-ray Fluorescence spectrometry (EDXRF). The results showed that the mean concentration of K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, As, Se, Br, Rb, Y, Zr and Nb in the soil samples at the site were within the range 3.859 – 9869.281 mg/Kg. The results in samples from the sites were found to be relatively higher than the control. Also, the soil samples from the sites were slightly enriched in V, Mn, Ni, Cu, Zn, As, Se, Rb and Y (with EF ranging from 1.033 to 2.179) but not enriched in Ca, Ti, Cr, Fe, Br and Nb (EF = 0.814 -0.965). Significant difference exists ($t < 0.05$) in the concentration of Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, As, Se and Rb between the sites and control (at $p < 0.005$, $t = 0.00001 – 0.01349$).

The values obtained for contamination pollution/accumulation factors to measure contamination pollution index were lower (for Cr, Cu, Ni and Zn) compared with the target (mg/kg) and the intervention values. In most cases, soil samples from the two sites were slightly enriched in heavy metals such as Ca, Ti, Mn and Zn for both vegetables but Ni only in *Talinum triangulare* but not enriched in other elements (EF < 1). The levels of heavy metals in the vegetable samples were also determined using the same analytical technique. The result indicated that the mean concentrations of Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, As, Se, Br, Rb, Y, Zr and Nb in the sites were within 1.078 – 3180.224 mg/kg. These results were also found to be relatively higher than the control for most of the heavy metals.

The results obtained from t-test between the sites and control showed that significant differences exist ($t < 0.05$) in Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn and As for *Talinum triangulare* (at $p < 0.05$, $t = 0.00002 – 0.03950$). Also, significant difference exists ($t < 0.05$) in concentration of Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, As, Se, Rb, Y, Nb in *Amaranthus hybridus* from the sites and the identical samples from the control ($p < 0.05$, $t = 0.00005 – 0.00860$). The mean concentration (mg/kg) of Cr, Mn, Fe, Ni, As, Se and Br in *Talinum triangulare* and *Amaranthus hybridus* were higher than the maximum permissible limits set by WHO/FEPA (Table 3). This was an indication that vegetables at this site were contaminated, whose consumption could be detrimental to health. Although, Mn is very essential for photosynthesis but continuous absorption could cause bio-accumulation in the body system which later becomes toxic and in turn renders the vegetables unsuitable for consumption. The vegetable

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samples from the site were slightly enriched in Ti, Mn, Ni, Zn, As, Se, Rb, (EF = 1.034 - 2.328) but not enriched in Ca, V, Cr, Cu and Br.

The results obtained for *Talinum triangulare* in the study were slightly higher in Cr, Ni and Cu but lower in Ca, Ti, V, Mn, Zn, As, Br and Se compared to *Amaranthus hybridus* in different automobile workshops. Moreover, the levels of Mn and Ni in the soil samples from the sites were higher in similar study by Aloysius (2013). This relatively high levels reported in soil samples at the sites could be attributed to the contribution by the associated allied industries such as painting, panel beating etc) in this study. The reverse was the case in Cu and Zn. In African spinach, the values reported for Cr, Mn, Fe, Ni, Cu, Zn, As, and Br were higher than those reported by Olukemi *et al* (2014) and Olajire *et al* (2018). The levels of heavy metals reported in this study were also relatively higher than those reported by Fayinminnu and Adekunle–Jimoh (2015) for Cr, Cu and Zn in Amaranthus hybridus vegetables grown in selected farms in Ibadan, Nigeria. This could also be an evidence of contamination and also indicated that the vegetables grown in the observed sites pose serious health risks if consumed.

4 Conclusions

The levels of eighteen heavy metals were determined in soil and vegetable samples from Iworoko-Ekiti using the Energy Dispersive X-ray Fluorescence Spectroscopy (EDXRF). The same technique was also used to analyze the identical samples from the control. The result gave the baseline levels of the metals in the plant and soil samples of the sites considered. The T-test results showed that significant differences exist between the levels of these heavy metals in the site and control ($p < 0.05$, $t = 0.00001 – 0.03950$). The soil samples were slightly enriched in V, Mn, Ni, Cu, Zn, As, Se Rb and Y with their enrichment factors ranging from 1.033 – 2.179.

Amaranthus hybridus samples were also slightly enriched in Ti, Mn, Zn, As and Se while *Talinum triangulare* samples were slightly enriched in Mn, Ni and Zn. However, toxic heavy metals such as lead (Pb) and cadmium (Cd) are not detected in the soil and vegetable samples. The mean concentration (mg/kg) of Cr, Mn, Fe, Ni, As, Se and Br in *Talinum triangulare* and *Amaranthus hybridus* were higher than the maximum permissible limit set by WHO/FEPA (Table 3). This was an indication that vegetables at these sites were contaminated as a result of the activities taking place in the study areas. The variation and the significant differences in the levels of the observed heavy metals in soil and vegetables between the sites and control may be due to dumping of metallic scraps, damaged/leftover corroding body parts of the vehicles, solvents, hydraulic fluid, spoilt/expired lubricants, tin cans at these workshops (Abidemi, 2011). It is therefore recommended that the locations of such workshops must be far away from the residents and residents of those areas should desist from vegetables grown at those sites.

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