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Lead (Pb) Concentrations in Soil, Air and Fruits of Sweet Orange (*Citrus sinensis* L. Osbeck) in Selected Landuse in Port Harcourt Metropolis, Rivers State, Nigeria

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Abstract

The study investigated the concentrations of Pb in soil, air and fruit of *C. sinensis* in selected landuse types in Port Harcourt Metropolis, Rivers State, Nigeria. Five fruits of *C. sinensis* were collected randomly in residential, commercial, industrial and natural forest (control). The weight, length and width of the *C. sinensis* fruits were measured. A total of three soil samples were collected around the *C. sinensis* trees where the fruits were collected into well labeled polythene bags and thereafter taken to laboratory for analysis. Pb concentrations in *C. sinensis* and soil samples were analysed using atomic absorption spectrophotometer (AAS). The concentration of Pb in the air was measured for 14 days using Aeroquel 500. The significant relationship between Pb concentration in the *C. sinensis* fruits, soil and air was determined using Spearman's rank correlation. Findings showed that the mean weight and width of *C. sinensis* fruits were highest in the residential landuse while the mean length of *C. sinensis* fruits was highest in the industrial landuse. However, the mean Pb concentration in the *C. sinensis* fruit was highest in the industrial landuse (0.46±0.15 mg kg^{-1}), commercial landuse (0.33±0.09 mg kg^{-1}) and the least was found in the natural forest (0.25±0.06 mg kg^{-1}). The mean Pb concentration in the soil was highest in the industrial landuse (0.177±0.16 mg kg^{-1}) and commercial landuse (0.121±0.10 mg kg^{-1}). However, the mean Pb concentration in the air was highest in the industrial landuse (0.85±0.09 mg kg^{-1}) and followed by commercial landuse (0.30±0.17 mg kg^{-1}). The correlations between the Pb concentration in the *C. sinensis* fruit and soil ($r=0.768$, $p<0.05$) and air ($r=0.642$, $p<0.05$) were significant. The study concluded that the Pb concentration in the *C. sinensis* fruits was higher than the WHO standard; hence people should be discouraged to consume them, especially those from the industrial and commercial areas.

Key Words: Pb concentration, soil, air, *C. sinensis*, landuse

Introduction

Heavy metals in which Pb is inclusive are contaminants in the environment which are influenced with various human activities (Taghau et al. 2011). Pb and its compounds are very harmful to the human body and animals whenever they gain access to food chain through drinking water, air, soil, plant tissues and contaminated food (Sharma and Dubey 2005; Hung et al. 2014). However, Pb concentrations in the cities in recent time can affect the products from urban gardening which continues to boom worldwide to enhance food security particularly in developing coun-

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tries (Food and Agriculture Organization of the United Nations [FAO], International Fund for Agricultural Development [IFAD] and World Food Programme [WFP] 2013; von Hoffen and Saumel 2014).

Generally, environmental pollutants in the urban areas are from different sources in different landuse with varying degrees of concentrations. For instance, automobile exhaust constitutes 75-80% of the gross air pollution in Nigeria while the remaining comes from the dust arising from untarred roads, smoke and gases from industries and the burning of bush and refuse (Taghau et al. 2011). The Pb level in Nigeria's super petrol is in range of 210-520 mg/litre (Ademoroti 1986). Pb and leaded compounds have been used in a wide variety of products found in and around our homes including paint, ceramics, pipes and plumbing materials, solders, gasoline, batteries, ammunition and cosmetics. Pb may travel long distance before settling to the ground where it usually sticks to soil particles. Since Pb is not biodegradable, once soil has become contaminated, it remains a long-term source of Pb exposure. Metal pollution has a harmful effect on biological systems and does not undergo biodegradation (Pehlivan et al. 2007). Meanwhile, Pb is element with atomic number 82, atomic weight 207.19, and a specific gravity of 11.34 and it is a bluish or silvery-grey metal with a melting point of 327.5°C and a boiling point at atmospheric pressure of 1,740°C (Tangahu et al. 2011).

C. sinensis originated from South East Asia around 4000 BC and were cultivated in China by 2500 BC (Nicolosi et al. 2008; Atta et al. 2012; Etebu and Nwauzoma 2014); and through migration and trade, the cultivation of *C. sinensis* was extended to Northern Africa and then to Nigeria. *C. sinensis* is one of the types of fruits produced in both rural and urban landscapes in Nigeria. *C. sinensis* accounted for about 70% of the total fruits produced in the world. Brazil, Florida (USA) and China are the three largest sweet orange producers (United States Department of Agriculture [USDA] 2009). Citrus is widely grown in Nigeria and many other tropical and subtropical regions (Piccinelli et al. 2008). In terms of volume in production, Citrus ranks after banana as the world second fruit crop with more than 108 million tons (FAO 2006). In Nigeria, about 930,000 tons of citrus fruits are produced annually from an estimated hectare of 3 million hectares of land (FAO 2008). Economically, oranges are important fruit crops, with an estimated 60 million metric tonnes produced worldwide as at 2005 for a total value of 9 billion dollars. Of this total, half came from Brazil and the United States of America (Goudeau et al. 2008; Bernardi et al. 2010). The global citrus acreage was nine million hectares with production put at 122.3 million tons (Xu et al. 2013). *C. sinensis* is a major source of vitamin C and contains sufficient amount of natural antioxidant such as folacin, calcium, potassium, thiamine, niacin and magnesium that builds the body immune system (Angew 2007; Etebu and Nwauzoma 2014).

There is always possibility of heavy metals accumulation on food items, food crops, plants, soil and air in the urban environments. Contamination of soils by heavy metals as a result of human, agricultural and industrial activities can be a source of accumulation of Pb due to its ability to be easily absorbed and accumulated in different plant parts (Sharma and Dubey 2005). Public concern is thus growing over potential environmental hazards that may be caused by the accumulation of Pb which may be a material of legacy of a site's industrial past or simply a function of its proximity to a freeway or some other sources of airborne pollution (Runk 2011; Seltenrich 2011; McClintock 2012). Pb is regarded as an element required by plant in small quantity but easily accumulate in different parts of the plant (Sharma and Dubey 2005; Hung et al. 2014). Plants like Citrus trees accumulate minerals essential for their growth from the environment and also accumulate heavy metals such as Cadmium (Cd) and Pb which have no known direct benefit to the plants (Ademoroti 1986).

Over the years, studies have reported the uptake of Pb by plants (Lane and Martin 1977) and its occurrence in plants and soil can be considered as an important pathway to food chain as high concentration of Pb can have toxic effects on the plants and man (Sharma and Dubey 2005). Studies have reported the accumulation of Pb on tomato (*Lycoperscon esculentum*) and okra (*Abelmoschus esculentus*) (Fatoba et al. 2012; Hung et al. 2014); vegetables, pea, soybean and cluster bean (Singh et al. 2012); maize, sunflower, willow and poplar (Kacalkova et al. 2014) and olive tree (Aghabarati et al. 2008). Investigations have been carried out on the levels of toxic metals (Pb and Cd) in selected varieties of fruits (banana, grape, guava, mandarin and orange) and farmland soils in Ethiopia (Yami et al. 2016). Similar studies have been reported in Nigeria (Igwegbe et al. 2013; Kalagbor et al. 2014); Pakistan (Akhtar et al. 2010); Kenya (Mausi et al. 2014); and India (Basha et al. 2014). Studies of Okonkwo et al. (2014) and Weli and Obisesan (2014) have reported the existence of urban pollution measured through the air quality in Port Harcourt Metropolis, Nigeria; which in some ways might have influence on tree crop tissues. This might also have influenced the urban soils because of the concentrations of synthetic organic contaminants such as polychlorinated biphenyls (PCBs) and the dioxins, polycyclic aromatic hydrocarbons, volatile organic compounds and heavy metals such as arsenic (Ar), cadmium (Cd), chromium (Cr), Pb, mercury (Hg), nickel (Ni) and zinc (Zn) exhibited by urban soils (Alloway 2004; Aichner et al. 2007). Very few of the previous studies considered the accumulation of Pb in *C. sinensis* tissues despite the high rate of consuming its leaf, fruit and bark for ethno-medicinal uses. This study therefore investigated the concentrations of Pb in soil, air and *C. sinensis* fruits in selected landuse types in Port Harcourt Metropolis because it becomes one of the fruits produced and sold in Port Harcourt City and studying its Pb concentration becomes a major concern in the recent times.

Materials and Methods

Study Area

The study was carried out in Port Harcourt Metropolis which is located between latitudes 4°51' 30"N and 4° 57' 30"N and longitudes 6°50' 00"E and 7°00' 00"E. The study area is found in the sub-equatorial region and enjoys tropical climate. The mean annual temperature is 30°C and relative humidity is about 85% (Oyegun and Adeyemo 1999). The rainfall is seasonal, variable and heavy and mean annual rainfall is about 2,300 mm (Mmom and Fred-Nwagwu 2013). The prevailing wind is basically south-westerly and north-easterly and the wind speed is between 5 and 17 m/s (Utang et al. 2010). The topography of the study area ranges between 16 m and 40 m above the sea level. The vegetation is nourished with high rainfall which provides favourable condition for the growth of varieties of tall and big trees like *Swietenia macrophylla*, *Triplochiton scleroxylon*, *Terminalia superba*, *Elaeis guineensis* and *Raphia hookeri*. The soils of the area can be categorized as fresh-water brown loams and sandy loams (Umeuduji and Aiseobogun 1999).

Collection of C. sinensis Fruits Samples, Soil Samples and Measurement of Pb in the Air

The ripe fruits of *C. sinensis* from residential landuse (Mgbuoba), commercial landuse (Diobu), industrial landuse (Trans-Amadi) and natural forest (control) (Choba-Aluu axis). The size of residential landuse, commercial landuse, industrial landuse and natural forest was 1.29 sq km, 2.24 sq km, 3.45 sq km and 6.34 sq km respectively. The distance of residential landuse from commercial was 4.71 km, commercial landuse was 3.18 km from industrial landuse while industrial landuse was about 7.02 km from residential landuse. The minimum distance of the selected landuse types away from the natural forest was about 11 km. Majority of the industries was oil servicing companies. The road network in the commercial, residential and industrial landuse areas was relatively dense (Obafemi et al. 2011; Emenike and Ibezi 2017) but that of the natural forest was less dense. There were at least 10 tree stands of *C. sinensis* in each of the landuse area with age minimum of 12 years.

In each of the landuse; five (5) fruits were collected in different tree stand using random sampling technique. The selected fruits were of similar degree of maturity directly from their tree stand (Yami et al. 2016). Physical parameters such as weight, length and width of each selected *C. sinensis* fruit were measured. The weight was determined with weighing scale calibrated in kilograms whereas the length and width were measured with a calibrated tape rule in centimeters. The soils were well drained and three soil samples were collected around (maximum of 1 m radius) each *C. sinensis* tree stand whereby the *C. sinensis* fruit samples were obtained using soil auger at the depth of 0-15 cm. The soil samples were collected into well-labelled polythene bags. The soil samples were air-dried and sieved with a 2 mm mesh and prepared for laboratory analysis for the determination of Pb concentration only. Air quality device was used to record the level of Pb concentrations in the air in each landuse for two weeks (14 days). The measurement was done in the afternoon (12-2 pm) using Aeroquel 500.

Laboratory Analysis of Pb in C. sinensis Samples and Soil Samples

The fruit samples of *C. sinensis* were crushed and oven-dried at 70°C for 24 hours. After cooling at ambient temperature, the dried fruits were milled into fine powder and sieved by using a 2 mm mesh and thereafter kept in pre-cleaned screw capped polyethylene container for further analysis. The sample digests of the fruit was analysed in three replicates for Pb concentrations using Buck Scientific Atomic Absorption Spectrophotometry (AAS) (Buck Scientific Model 210/211 VGP, USA) of the Association of Official Agricultural Chemists (AOAC) standard. The soil samples were air-dried and carefully sieved with 2 mm diameter mesh in order to separate the soil from stones. Thereafter, the soil samples were taken to the laboratory for analysis to determine the concentration of Pb. 2 g of the air-dried soil samples were digested with 20 ml of 4 M HNO_3 using the Method 3050B (USEPA 1995; Chen et al. 2005). Extracts used for determining Pb was obtained by leaching soil samples using 0.1 N EDTA. The concentrations of extractable Pb in the solutions were determined using atomic absorption spectrophotometer (AAS) (Nazli and Hashim 2010; Rosemary et al. 2014). Accuracy of analysis was ensured through repeated analysis of samples against National Institute of Standards and Technology Standard Reference Material (SRM 1570) for Pb (Aghabarati et al. 2008). Satisfactory recoveries of 87.4% were obtained for Pb concentrations. To assure the accuracy of analysis, standard quality control practices were adopted during the analysis and blank samples were included in each batch of analysis (Rosemary et al. 2014). The elemental analyses of both *C. sinensis* fruits and soils samples were done in the Agronomy Laboratory of the University of Ibadan, Ibadan, Nigeria.

Method of Data Analysis

The study made use of descriptive statistics to explain the levels of concentrations of Pb in *C. sinensis* fruits, soil and air. Spearman rank correlation statistics were used to determine the relationship between Pb concentration in *C. sinensis*, soil and air across landuse in Port Harcourt Metropolis at 0.05 significant levels. The concentrations of Pb in *C. sinensis*, and soil were compared with the permissible levels of World Health Organization (WHO) while United States Environmental Protection Agency (USEPA) permissible level was used for Pb concentrations in air. Also, Spearman's rank correlation statistics were used to determine Citrus fruit physical parameters (weight, length, width) and Pb concentration in *C. sinensis* at 0.05 significant levels. The analyses were carried out using SPSS version 20.0.

Results

Pb concentrations in C. sinensis Fruits, Soil and Air across the Landuse Types

The Pb concentrations in *C. sinensis* fruits, soil and air across the landuse types whereby the mean Pb concentrations in *C. sinensis* fruits in the natural forest was the least (0.25±0.06 mg kg^{-1}) while the highest was observed in the industrial landuse (0.46±0.15 mg kg^{-1}) (Table 1, Fig. 1). It was observed that the levels of Pb in *C. sinensis* fruits in commercial, residential and industrial landuse types than the permissible limits of WHO (0.3 mg kg^{-1}). Furthermore, the Pb concentration in soils surrounding the *C. sinensis*

Table 1. Pb concentrations (mg kg^{-1}) in *C. sinensis* fruits, soil and air across landuse

Landuse	*C. sinensis* Fruit		Soil		Air	
	Min-Max	Mean±SD	Min-Max	Mean±SD	Min-Max	Mean±SD
Natural Forest	0.21-0.32	0.25±0.06	0.00-0.17	0.065±0.09	0.02-0.35	0.19±0.16
Commercial	0.27-0.44	0.33±0.09	0.01-0.22	0.121±0.10	0.11-0.40	0.30±0.17
Residential	0.32-0.42	0.37±0.05	0.01-0.11	0.073±0.06	0.01-0.55	0.29±0.27
Industrial	0.37-0.63	0.46±0.15	0.01-0.32	0.177±0.06	0.77-0.95	0.85±0.09
WHO/USEPA	0.3*		0.85*		0.0015**	

F=3.04; p=0.098. *WHO Permissible Levels; **USEPA Permissible Level (USEPA 2016).

across the landuse was highest in the industrial landuse (0.177±0.16 mg kg^{-1}) and the least was observed in the natural forest (0.065±0.09 mg kg^{-1}). However, the mean Pb concentrations in soil in all the landuse investigated were lower than the permissible levels of WHO (85 mg kg^{-1}) while Pb concentrations in air were higher than the permissible level of USEPA (0.0015 mg kg^{-1}). The mean Pb concentration in air across the landuse types was highest in the industrial landuse (0.85±0.09 mg kg^{-1}) while the least was observed in the natural forest (0.19±0.16 mg kg^{-1}). Furthermore, the Pb concentrations in air in residential and commercial varied slightly. There was no significant variation in the Pb concentrations in *C. sinensis*, soil and air ($F=3.04$; $p=0.098$). Comparing the Pb concentrations between air and soil, the analysis showed that the mean Pb concentrations in air were higher than that of soil in all the landuse types (Fig. 1).

Weight, Length and Width of C. sinensis Fruits across the landuse Types

The mean weight of the sampled *C. sinensis* fruits across

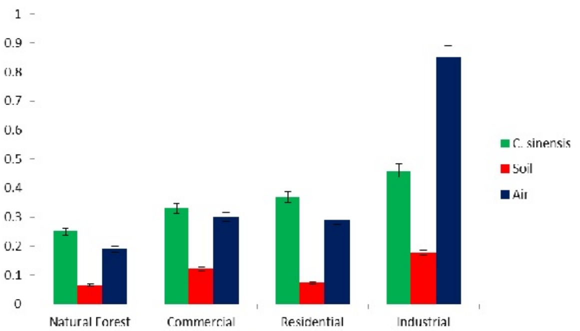

Fig. 1. Variations in Pb concentrations in *C. sinensis*, soil and air across the landuse types.

the landuse was 0.43 kg in natural forest and industrial landuse types, 0.40 kg in commercial landuse and 0.53 kg in the residential landuse. Thus, there was slight variation in the weight of *C. sinensis* fruits across the landuse Port Harcourt Metropolis; though the highest mean weight of *C. sinensis* fruits was observed in the residential landuse (Table 2). The mean length of *C. sinensis* fruit was 7.28 cm in the natural forest and 6.77 cm in the commercial landuse. In addition, the mean length of the *C. sinensis* fruit under residential landuse was 7.03 cm while it was 7.62 cm in the industrial landuse types (Table 2). Thus, the mean length of *C. sinensis* fruits was higher in the industrial and residential landuse types. The mean width of *C. sinensis* fruits was 6.86 cm in the natural forest, 5.76 cm in commercial landuse, and 7.37 cm in residential landuse while it was 6.69 cm in the industrial landuse. Slight variation in the mean width of *C. sinensis* fruits was observed across the landuse while the highest was recorded in the residential landuse.

Relationship between Pb concentrations in C. sinensis fruits, Soil, Air and Physical Parameters across landuse types

The correlation analysis existing between the concentration of Pb in *C. sinensis* fruits and soil was positive, relatively high and significant ($r=0.768$; $p<0.05$) (Table 3). Similarly, the correlation between Pb in *C. sinensis* fruits and Pb in air was positive and significantly high ($r=0.801$; $p<0.05$) (Table 3). However, the correlation between Pb concentrations in air and soil was positive and significantly high ($r=0.982$; $p<0.05$) (Table 3). The correlations between Pb concentrations in *C. sinensis* fruits and physical parameters (weight, length, width) of *C. sinensis* were generally low and not significant at 0.05 significant levels (Table 4).

Table 2. Physical parameters *C. sinensis* fruits across landuse

Landuse	Weight (kg)		Length (cm)		Width (cm)	
	Min-Max	Mean±SD	Min-Max	Mean±SD	Min-Max	Mean±SD
Natural Forest	0.40-0.50	0.43±0.06	6.86-7.62	7.28±0.39	6.85-6.87	6.86±0.01
Commercial	0.30-0.50	0.40±010	6.35-7.62	6.77±0.73	5.08-6.35	5.76±0.64
Residential	0.50-0.60	0.53±0.06	6.60-7.62	7.03±0.53	7.11-7.62	7.37±0.26
Industrial	0.40-0.50	0.43±0.6	7.11-8.13	7.62±0.51	6.60-6.86	6.69±0.15
F Value		0.75 (p=0.214)		0.86 (p=0.126)		1.15 (p=0.071)

Table 3. Spearman Rank Correlation Matrix of Pb Concentrations in *C. sinensis*, Soil and Air

	Pb in *C. sinensis* Fruits	Pb in Soil	Pb in Air
Pb in *C. sinensis* Fruits	1		
Pb in Soil	0.768* (p=0.031)	1	
Pb in Air	0.801* (p=0.025)	0.982* (p=0.003)	1

*Correlation is significant at the 0.05 level (2-tailed).

Table 4. Spearman Rank Correlation Matrix of Pb Concentrations between *C. sinensis* Fruits and Physical Parameters of *C. sinensis* Fruits

	C. sinensis Fruits	Weight	Length	Width
C. sinensis Fruits	1.000			
Weight	0.316 (p=0.115)	1.000		
Length	0.400 (p=0.07)	0.316 (p=0.116)	1.000	
Width	0.012 (p=1.124)	0.949 (p=0.001)	0.200 (p=0.173)	1.000

Discussion

The concentrations of Pb in soil, air, and *C. sinensis* in the industrial, residential and commercial landuse types of Port Harcourt Metropolis were higher than that of the natural forest. This is possible because Pb is generally attributed to atmospheric deposition downwind from industries and factories (Schulin et al. 2007) and exhaust emissions, traffic volume, vehicle type, topography and air-borne contamination from leaded fuels (Teichman et al. 1993; Sinegani 2007; Taghau et al. 2011; McClintock 2012; Weli and Obisesan 2014); and paints used for housing as interior and exterior paint always end up in the soil for plant uptake (Wu et al. 2010). No wonder, Jacobs et al. (2002) reported that 52% of houses built in the US before 1978 have yard soil Pb levels greater than 400 mg/kg. However, Saleh et al (2017) attributed the high concentration of Pb in the peel and pulp of citrus in Tehran to crude oil pollution while Aghabarati et al. (2008) attributed the high concentration of Pb in the root tips and proximal parts of the root of onions (*Allium cepa*) in the suburban areas of Tehran, Iran to irrigation using municipal effluent.

The low concentrations of Pb in air, soil and *C. sinensis* in the natural forest landuse may be attributed to lower exposure to development and presence of tree that can absorb trace metals. This is in contrary to the findings of Rosemary et al. (2014) whereby Pb concentrations were higher in the uncultivated land indicating that non-agricultural sources, such as parent material and atmospheric deposition of Pb could have been responsible for the concentration. In addition, the dynamics of Pb concentrations in the air, soil and *C. sinensis* across the selected landuse types may be attributed to management practices (Rosemary et al. 2014).

The physical properties of the *C. sinensis* fruit especially the width was found within the normal range (6.5-9.5 cm) (Etebu and Nwanzoma 2014). All the physical properties insignificantly and positively correlated with Pb concentrations ($p > 0.05$) and thus the influence of Pb concentrations may not really have effects on physical properties of *C. sinensis* fruits. However, it is reported that heavy metals are known to have effect on plant growth and soil microflora (Roy et al. 2005; Taghau et al. 2014). Thus, pollutants deposited in the soil (through anthropogenic or human activities) remain there for a long time and acting as a source of further pollution in urban environments (Hjortenkrans et al. 2008; von Hoffen and Säumel 2014). The implication of the higher concentration of Pb in the urban soil than the natural forest may be imbalance of mineral nutrients in plants such as *C. sinensis*. It has been reported that Pb concentration in soil can block the entry of cations (e.g. K, Ca, Mg, Zn, Mn, Cu, Fe^{3+}) and anions in the root systems. Thus, anions like nitrate uptake declines in plants under exposure to Pb with an associated lowering of nitrate reductase activity and disturbed nitrogen metabolism (Sharma and Dubey 2005).

Higher concentration of Pb in *C. sinensis* fruits than the

WHO permissible level (0.3 mg kg^{-1}) could cause some disturbances in the human body systems. Pb being a serious cumulative body poison enters into the body system through air, water and food and cannot be removed by washing the fruits (Divrikli et al. 2006; Kalagbor et al. 2014). It has also been found to be toxic to the red blood cell, kidney, nervous and respiratory system (Taupeau et al. 2001; Kalagbor et al. 2014). Health implications of Pb in children may include behavioural disturbances, learning and concentration difficulties and people who may have been exposed to lead for a long time may suffer from memory deteriorations, prolonged reaction time and reduced ability to understand (WHO 1995; Sanyaolu et al. 2011). Furthermore, lead poisoning in children has been associated with hearing loss, learning difficulties and poor school performance, lower levels of tertiary educational attainment and a decline in lifetime earnings (WHO 2015).

The positive relationship of Pb concentration in *C. sinensis* and soil could be attributed soil particle size, pH, CEC, root surface area of plants, root exudates, mycorrhization, rate of transpiration, temperature, moisture and nutrient availability (Lee et al. 1998; Sharma and Dubey 2005; Taghau et al. 2011; Agharabati et al. 2012). Studies have shown that Pb concentrations in plants tissues are positively correlated with increasing total Pb content in soil (Kacálková et al. 2014). Also, Fe and Zn correlated positively between soil and fruits of banana, grape, guava, mandarin and orange (Yami et al. 2016).

Conclusion

The study has revealed the status of the concentration of Pb in *C. sinensis* fruits in the industrial, commercial and residential landuse to be higher than the WHO standards and as a result, it could be dangerous if they are consumed. It is therefore recommended that the octane rating of the petrol should be improved but not with lead additives and the use of leaded petrol should be discouraged. Consumption of *C. sinensis* fruits from industrial and commercial should be discouraged. The study can be extended to other landuse types to ensure absolutely good health for every individual residing and consuming *C. sinensis* in Rivers State, Nigeria.

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