Analysis of Mercury Levels in Agricultural Soils and Citrus Varieties from Benue State, Nigeria

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Abstract: Mercury levels in agricultural soils and three citrus varieties (Washington, Ibadan Sweet and Valencia) in seven (7) local government areas of Benue State were determined using standard techniques. The mean physico-chemical parameters of the soils ranges from: pH (5.20 – 6.10); CEC (10.5 – 12.9 cmolkg\(^{-1}\)); organic carbon (2.35 – 3.39%) and organic matter (6.17 – 7.37%). The mean Hg levels in the soils ranges from 20.5 – 21.2 µgkg\(^{-1}\). The mean proximate parameters are: fat in Valencia (0.330%); fibre in Valencia (0.490%); moisture in Valencia (87.4%); ash in Valencia (1.80%) and carbohydrate in Ibadan sweet (17.7%). The mean Hg concentration in the citrus juice ranges from: Washington (0.770 – 6.35 µgkg\(^{-1}\)); Ibadan sweet (0.770 – 6.53 µgkg\(^{-1}\)) and in Valencia (0.770 – 5.22 µgkg\(^{-1}\)). The order of accumulation of Hg in the citrus varieties is Ibadan sweet > Washington > Valencia. The concentrations of Hg in the three citrus fruit juice and soil do not exceed the WHO/FAO permissible limits of 100 and 1000 µgkg\(^{-1}\) for foods and agricultural soils respectively. However, continues monitoring is hereby recommended.

Keywords: Agricultural Soil, Washington, Ibadan Sweet, Valencia, CV-AAS

1. Introduction

The need for Nigeria to key into minamata convention on mercury in line with United Nations Environmental Programme precipitated the motive of this research. Orange (Citrus sinensis) fruits play an essential role in human nutrition as they are good sources of antioxidants such as ascorbic acid, carotenoids; tocopherol and phenolic compounds [1]. It also contains some other nutrients such as proteins, carbohydrate, fibre and minerals. Vitamin C (ascorbic acid) is the most dominant nutrient in orange fruits, it is essential for the synthesis of collagen and its deficiency leads to scurvy which causes loss of teeth [2]. Globally, Nigeria ranks 9th position among the world top 10 producers of citrus sinensis with Benue state giving the highest annual production of the commodity [3]. Oranges are very important protective foods and useful for the maintenance of good health, prevention and treatment of various diseases [4]. Frequent consumption of fruit is associated with reduced risk of cancer, cardiovascular diseases, stroke, Alzheimer disease, cataracts, and some of the functional declines associated with aging [5]. The large input of mercury (Hg) into the arable lands has resulted in the widespread occurrence of mercury contamination in the entire food chain. Hg is a unique metal due to its existence in different forms e.g., HgS, Hg\(^{2+}\), Hg\(^0\) and methyl-Hg. However, in agricultural soil, ionic form (Hg\(^{2+}\)) is predominant [6]. The persistence nature of heavy metals in the environment and subsequent accumulation in fruits is discouraging the intake of this commodity [7]. Dietary exposure to mercury has been identified as a risk to human health through the consumption of vegetable crops [8]. Food safety is important for the survival of all living organisms involved in the food chain. In most developing countries like Nigeria, the quest for rapid economic growth through industrialization and modern agriculture have resulted in a concomitant inflow of several contaminants like heavy metals into the environment [9]. Despite the benefits of fruit consumption on human health, heavy metal contents in fruits can be toxic when they exceed the recommended health levels or when they bio-accumulate in the body over a long period [10]. This study aims at determining the level of mercury in three orange fruits juice (Washington, Ibadan
sweet and Valencia), soil and some physico-chemical properties of the soil.

2. Materials and Methods

2.1. Sample Collection

Benue State lies within the lower River Benue trough in the middle belt region of Nigeria with Landmass of 34,059 square kilometers and a population of 4,253,641 in 2006 census. Agriculture is the mainstay of the economy engaging over 75% of the working population and its geographic coordinates are longitude 7° 47’ and 10° 0’ East, Latitude 6° 25’ and 8° 8’ North and it is characterized by an annual rainfall of 1250 - 1500 mm. Orange (citrus) and soil samples were collected from seven (7) Local Government Areas between January - April, 2018, because of the dominance in orange production. Orange farms were located from three (3) council wards of each Local Government where the citrus sinensis (sweet orange) fruits (Washington, Ibadan Sweet and Valencia) samples were plugged into a polyethylene container hanged on a stick and bulked based on varieties, placed into appropriately labeled sacks and transported to the Departments of Crop Production and Biology, Federal University of Agriculture Makurdi, where the plant parts (orange fruits) were taxonomically identified and authenticated by professionals, also, the soil samples were taken using auger at the depth of 0 – 20 cm after which the samples were conveyed to the laboratory of Chemistry Department Federal University of Agriculture Makurdi for storage and pretreatment.

2.2. Sample Preparation and Digestion

The orange fruits were washed with tap water and then with distilled water after which the juice was squeezed manually into separate beakers. About 200 mL of juice was obtained from each sample. The juice was filtered, mixed well and exactly 1.0 g of the homogenized orange sample was weighed into 100 mL volumetric digestion flask and a mixture of 2 mL H2O, 4 mL HNO3 - HClO4 (1:1) and 10 mL H2SO4 were added. The mixture was then heated at a temperature between 150 and 250°C until the solution was clear. The sample solution was then cooled and diluted to 100 mL with distilled water. Also, exactly 1.0 g of the homogenized soil sample was weighed into 100 mL volumetric digestion flask and the same digestion procedure applied. A blank and standard solution digests using Hg solutions were subjected to the same treatment and the contents analyzed for mercury using cold vapour atomic absorption spectrophotometer [11].

### Table 1. Physico-chemical Parameters of the Soil.

| Local Government Areas | pH in 0.01 M CaCl2 | pH in Distilled Water | Sand% | Silt% | Clay% |
|------------------------|-------------------|-----------------------|-------|-------|-------|
| Kastina-Ala             | 5.50 ± 0.3        | 7.10 ± 0.4            | 76.0 ± 3 | 8.23 ± 0.1 | 15.7 ± 3 |
| Konshisha              | 5.20 ± 0.4        | 6.10 ± 0.2            | 76.1 ± 5 | 7.70 ± 0.6 | 16.2 ± 5 |
| Kwande                 | 6.10 ± 0.2        | 6.70 ± 0.3            | 77.0 ± 3 | 7.18 ± 0.8 | 15.8 ± 3 |
| Logo                   | 5.70 ± 0.4        | 6.90 ± 0.3            | 73.7 ± 0.1 | 8.14 ± 0.1 | 18.1 ± 1 |
| Ukom                   | 5.70 ± 1          | 6.40 ± 0.3            | 81.2 ± 0.1 | 7.84 ± 0.03 | 11.0 ± 0.1 |
| Ushonjo                | 5.70 ± 0.4        | 6.40 ± 0.3            | 77.0 ± 5 | 8.52 ± 0.6 | 14.5 ± 5 |
| Vandeikya              | 6.10 ± 0.2        | 6.70 ± 0.5            | 72.2 ± 3 | 7.89 ± 1 | 19.9 ± 3 |

### Table 2. CEC, Organic Carbon and Organic Matter of the Soil.

| Local Government Areas | CEC (emolkg-1) | Organic Carbon% | Organic Matter% |
|------------------------|----------------|-----------------|-----------------|
| Kastina-Ala            | 11.48 ± 1      | 2.94 ± 1        | 7.35 ± 2        |
| Konshisha              | 10.53 ± 2      | 2.89 ± 0.3      | 7.37 ± 1        |
| Kwande                 | 12.86 ± 1      | 2.53 ± 1        | 6.42 ± 2        |
| Logo                   | 12.10 ± 0.2    | 3.21 ± 1        | 6.17 ± 2        |
| Ukom                   | 11.23 ± 1      | 2.35 ± 1        | 6.63 ± 0.3      |
| Ushonjo                | 12.45 ± 1      | 3.39 ± 1        | 6.90 ± 1        |
| Vandeikya              | 11.91 ± 1      | 3.30 ± 0.1      | 7.08 ± 1        |

### Table 3. Fat and Fibre Contents of the Citrus Varieties.

| Local Government Areas | Fat%                  | Fibre%                  |
|------------------------|-----------------------|-------------------------|
|                        | Washington            | Valencia                | Washington            | Valencia                |
| Kastina-Ala            | 0.260 ± 0.1           | 0.330 ± 0.1             | 0.330 ± 0.1           | 0.230 ± 0.02           | 0.240 ± 0.04           | 0.220 ± 0.02           |
| Konshisha              | 0.220 ± 0.01          | 0.230 ± 0.02            | 0.220 ± 0.02          | 0.350 ± 0.1            | 0.300 ± 0.04           | 0.390 ± 0.02           |
| Kwande                 | 0.210 ± 0.01          | 0.260 ± 0.1             | 0.270 ± 0.01          | 0.410 ± 0.00           | 0.390 ± 0.01           | 0.420 ± 0.1            |
| Logo                   | 0.200 ± 0.01          | 0.230 ± 0.02            | 0.190 ± 0.03          | 0.220 ± 0.01           | 0.310 ± 0.1            | 0.220 ± 0.1            |
| Ukom                   | -                     | 0.210 ± 0.02            | 0.210 ± 0.03          | -                      | 0.250 ± 0.1            | 0.200 ± 0.02           |
| Ushonjo                | 0.310 ± 0.03          | 0.250 ± 0.02            | 0.310 ± 0.1           | 0.400 ± 0.01           | 0.370 ± 0.01           | 0.490 ± 0.1            |
| Vandeikya              | 0.270 ± 0.01          | 0.240 ± 0.04            | 0.250 ± 0.03          | 0.220 ± 0.02           | 0.230 ± 0.01           | 0.300 ± 0.04           |
4. Discussion

In this work, a total of 161 parameters (pH, sand, silt, clay, organic carbon, CEC, organic matter and mercury) were examined in the studied area.

4.1. Physico-Chemical Parameters of the Soil

4.1.1. pH

Table 1 shows the pH of the agricultural soils in 0.01 M CaCl₂ solution and distilled water in the range of 5.20 – 6.10 and 6.10 – 7.10, respectively. The lowest pH value was recorded in Konshisha and the highest in Kwande and Vandeikya in 0.01 M CaCl₂. This may be due to the ionization of more hydrogen ions in the soil solution of Konshisha than that of Kwande and Vandeikya. Similarly, the mean pH value in Ukum and Ushongo are low and the highest value is recorded in Katsina-Ala in distilled water, probably due to the same reason. The pH range of 4.56 – 7.71 in roadside soil from Wurukum, Gboko road, Federal lowcost and Federal medical centre was reported [12]. The pH of agricultural soils in selected farms in Abakra, Delta State revealed a pH range of 4.20 – 5.45 [13].

It may also be attributed to the high rainfall common in some of those areas which could leach out basic cations from the farmland soils [13].

4.1.2. Sand

The result of the soil particles is presented in Table 1; the mean particle size is in the range of 72.2 – 81.2%. The lowest value was recorded in Vandeikya and the highest value in Ushongo. The analysis of irrigation water in river Katsina – Ala reported silt in the range of 72.2 – 81.2% [15]. The sand particles in soils around mechanic workshops in Makurdi were reported in the range of 73.4 – 83.3% [16]. The assessment of top soils in Wurukum abattoir reported silt content in the range of 10.70 – 17.70% in Konshisha than that of Kwande and Vandeikya. This may be due to the ionization of more hydrogen ions in the soil solution of Konshisha than that of Kwande and Vandeikya. Similarly, the mean pH value in Ukum and Ushongo are low and the highest value is recorded in Katsina-Ala in distilled water, probably due to the same reason. The pH range of 4.56 – 7.71 in roadside soil from Wurukum, Gboko road, Federal lowcost and Federal medical centre was reported [12]. The pH of agricultural soils in selected farms in Abakra, Delta State revealed a pH range of 4.20 – 5.45 [13]. The low pH of some orange farmland soils may be attributable to the presence of hydrogen cyanide from the perennial crops [14]. It may also be attributed to the high rainfall common in some of those areas which could leach out basic cations from the farmland soils [13].

4.1.3. Silt

The mean silt particles are in the range of 7.18 – 8.52% as shown in Table 1. The lowest value was recorded in Kwande and the highest value in Ushongo. The analysis of irrigation water in river Katsina – Ala reported silt in the range of 72.2 – 81.2% [15]. Silt content in the range of 10.70 – 17.70% in soils around Benue Cement Company revealed silt particle size in the range of 70.70 – 74.70% [17].

4.1.4. Clay

Table 1 shows the clay content in the range of 10.70 – 17.70% in soils around Benue Cement Company revealed clay particle size in the range of 70.70 – 74.70% [17].
which makes the soil compact.

4.1.4. Clay

The mean result ranges from 11.0 – 19.9% with the lowest value recorded in Ukum and the highest value in Vandeikya as presented in Table 1. The clay content of soil in the range of 10.2 – 16.6% was reported [17]. The clay content of top soil in Wurukum revealed a range of 21 – 57% [15] which is higher than the values recorded in this work. The clay content of soil was also reported in the range of 8.0 – 15% [18]. The clay contents of surrounding soils in ceramic and Pharmaceutical industrial areas gave a range of 7.6 -13.2% and 8.40 – 11.6% respectively [19].

4.1.5. Cation Exchange Capacity

The mean result of CEC ranges from 10.5 – 12.9 cmolkg\(^{-1}\) with the lowest CEC value recorded in Konshisha and the highest in Kwande as presented in Table 2. A study carried out on agricultural farmlands soils in Abakwa, Delta State, Southern, Nigeria reported Cation Exchange Capacity ranging from 1.04-7.96 cmolkg\(^{-1}\) [13]. A CEC of 13.0 and 91.1 cmolkg\(^{-1}\) in dumpsite waste in Yenagoa, Nigeria was reported [20].

4.1.6. Organic Carbon

The mean values of organic carbon ranges from 2.35 – 3.39% with the lowest value in Ukum and the highest value in Ushongo as shown in Table 2. The mean value of organic carbon in some selected site in Abeokuta were reported to be 0.02 - 8.48% [21] which is higher than the value recorded in work. The organic carbon in roadside soils in Makurdi metropolis in the range of 0.27 - 5.44% was reported [12] which is higher than the values recorded in this research. The organic carbon of some agricultural farms were reported in Delta State in the range of 0.95 – 2.02% [13] less than the value recorded in this work.

4.1.7. Organic Matter

The mean organic matter recorded is in the range of 6.17 – 7.37% with the lowest value reported in Logo and the highest in Konshisha as shown in Table 2. The organic matter content of soil in automobile mechanic village in Apir and North bank were investigated and the values reported are in the range of 1.46 – 2.39% [22] which is less than the values recorded in this research.

4.2. Proximate Parameters of the Citrus Varieties

4.2.1. Fat Content

The result of the analysis revealed the levels of fat in the orange varieties in the range of: Washington (0.200 – 0.310%); Ibadan sweet (0.210 – 0.330%) and Valencia (0.190 – 0.330%). Low level of fat in Washington was recorded in Logo and highest level was observed in Ushongo, in Ibadan sweet, low level of fat was observed in Ukum and the highest in Katsina-Ala and in Valencia, the lowest levels was observed in Logo and the highest levels in Kastina-Ala as presented in Table 3. The fat content of oranges from Benue State and Rivers State were reported to be 0.65% and 2.0% [23] which are higher than the values recorded in this work probably due to time of collection of the samples. The fat content of *citrus sinensis* was reported to be 10.0% [24] which is by far higher than the values reported in this work. Proximate composition of *citrus sinensis* in Northern Nigeria reported the fat content of 0.06% [25] less than the values recorded in this work. The differences in the values of fat recorded in the citrus varieties could be due to species, the trend in the result showed that Valencia > Ibadan sweet > Washington.

4.2.2. Fibre Content

The result of the crude fibre of the three citrus varieties analyzed in this work are in the range of Washington (0.220 – 0.410%), Ibadan sweet (0.230 – 0.390%) and Valencia (0.200 – 0.490%). The lowest fibre content in Washington was recorded in Logo and Vandeikya and the highest in Kwande. In Ibadan sweet, the lowest value was observed in Vandeikya and the highest in Kwande and in Valencia, the lowest values was recorded in Ukum and the highest in Ushongo as shown in Table 3. The fibre content of oranges in Benue State and Rivers State revealed the mean values of 0.18 ± 0.06% and 0.41 ± 0.08% [23] this values are less compared to the ones recorded in this research and may be as a result of mineral composition and variety. The nutritional potential of *citrus sinensis* revealed the fibre content value of 13.5% [26] which is higher than the values obtained here may be as a result of time of sample collection and climate. The fibre content of *citrus sinensis* in Northern Nigeria was reported to be 2.82% [25]. The result of the analysis shows the trend in the fibre composition of the citrus varieties in order of Valencia > Washigton > Ibadan sweet.

4.2.3. Moisture Content

The moisture content reported in the three orange species ranges from: Washington (79.4 – 86.2%), Ibadan sweet (79.5 – 86.9%) and Valencia (80.4 – 87.4%). The lowest value of moisture content was observed in Konshisha and the highest in Ushongoin Washington, in Ibadan sweet, the lowest values were observed in Ukum and Vandeikya and the highest in Ushongo, in Valencia, the lowest value is recorded in Ukum and the highest in Ushongo as presented in Table 3. The moisture content of oranges in Benue State and Rivers State were reported to be 88.8 ± 0.07% and 88.9 ± 0.06% respectively [23], the values are higher than what is reported in this analysis. The moisture content of fresh juice from three citrus species were; sweet orange (80.5%), lemon (85.1%) and lime (75.6%) [27], which is within the range of the result of this analysis. The moisture content of orange *citrus sinensis* was reported to be 9.78% [26] which is far less than the values recorded in this work and other similar research works and this could be due to differences in species and season. The trend of the moisture content in the citrus varieties follows Valencia > Ibadan sweet > Washigton.

4.2.4. Ash Content

The result of this analysis revealed that the ash content of the three citrus varieties are in the range of Washington (1.20..
Northern Nigeria was reported to be 0.62%. The proximate composition of fresh juice from three citrus species recorded the ash contents in sweet orange (0.60%), lemon (0.50%) and lime (0.15%) [27], these values reported by these researchers are less than the values recorded in this work and may be due to species. The trend of the ash content in the citrus varieties are Valencia > Washington > Ibadan sweet.

4.2.5. Protein Content
The result of proteins analyzed revealed the protein content of the three orange varieties to be in the range of (0.63 – 0.86%) Washington, (0.73 – 0.84%) Ibadan sweet and (0.62 – 0.89%) Valencia. The lowest value in Washington was observed in Kwande, the highest in Logo, in Ibadan sweet, the lowest value is in Ushongo, the highest in Konshisha and in Valencia, the lowest value is recorded in Ushongo and the highest in Konshisha as presented in Table 5. The protein contents of oranges from Benue and Rivers States were investigated, the mean values are; 0.44 ± 0.06% and 0.44 ± 0.07% [23]. The protein content of orange juice was also reported in the range of 3.80 – 3.65% [28], which is high compared to the value obtained in this analysis and this could be due to the fact that the leaves are the organs for photosynthesis in the plant and may store sufficient carbohydrates temporarily. The protein content of orange juice was also reported, the mean value is 0.74 ± 0.03% [23], which is less than the value reported in this analysis. The trend is Valencia > Washington > Ibadan sweet.

4.2.6. Carbohydrate Content
The carbohydrate content of three orange varieties gave a range of 11.1 – 17.3% in Washington, 10.2 – 17.7% in Ibadan sweet and 9.39 – 16.4% in Valencia. The lowest carbohydrate content was observed in Ushongo and the highest values were recorded in Konshisha in Washington. In Ibadan sweet, the lowest value was in Ushongo and the highest in Vandeikya also in Valencia, the lowest value was in Ushongo and the highest value in Vandeikya as shown in Table 5. The carbohydrate content of oranges from Benue State and Rivers State were reported to be 9.30% and 7.71% respectively [23] which are less than the values reported in this analysis. The carbohydrate content of citrus sinensis in Nigeria was reported to be 18.6% [29] which is in agreement with the values recorded in this research. The carbohydrate content of citrus sinensis was reported to be 42.9% [24] higher than the values recorded in this work. The low value of carbohydrate could be due to the unprocessed nature of the fruits. Processed fruits are expected to have higher values of carbohydrates, also species type. The trend of carbohydrate content in this analysis is Ibadan sweet > Washington > Valencia.

4.3. Hg in Orange Juice and Soil
The result as represented in Table 6 showed the range of Hg in the citrus varieties and soil as: Washington (0.770 – 6.35 µgkg\(^{-1}\)), Ibadan sweet (0.770 – 6.53 µgkg\(^{-1}\)), Valencia (0.770 – 5.22 µgkg\(^{-1}\)) and soil (20.5 – 21.2 µgkg\(^{-1}\)). The levels of Hg in Ushongo in Washington were below detection limits. High levels of Hg were observed in in Katsina-Ala in Washington (6.35 µgkg\(^{-1}\)), Ibadan sweet (6.53 µgkg\(^{-1}\)) and Valencia (5.22 µgkg\(^{-1}\)). Hg values reported in soils are 2410 µgkg\(^{-1}\) which is higher than the values reported in this work [30]. The levels of Hg in soils of Anka area were recorded in the range of 2153 – 12924 µgkg\(^{-1}\) which is higher than the values in this research [31]. The levels of Hg in industrial soils in Kaduna State were recorded to be 28800 µgkg\(^{-1}\) which is higher than the values reported in this research [32]. The levels of Hg distribution in polluted soils in the range of 220 – 1880 µgkg\(^{-1}\) were recorded which are higher than the values reported in this research [33]. The trend of Hg accumulation in the orange species is Ibadan sweet > Washington > Valencia. The accumulation may be due to orange type, solubility in soil, age, and volume of agrochemicals and the use of substances containing mercury (soaps, creams and batteries). The results of all the samples analyzed for Hg are below WHO/FAO (100µgkg\(^{-1}\)) limits for Hg in foods. Also, the level of Hg analyzed in this research in all the soils is below WHO/FAO (1000µgkg\(^{-1}\)) limits in agricultural soils.

5. Conclusion
The result of the analysis show that the level of Hg in the different orange fruits and soil analyzed do not exceed the prescribed WHO and FAO maximum permissible limit. Hg accumulation may lead to acute toxicity; metals enter into the food chain through polluted environment. The concentration of Hg in the orange varieties is in the order of Washington > Ibadan sweet > Valencia. Further studies should be carried out on other fruits since there is the probability of metal transfer from soil to fruits.

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