ASSESSMENT OF HEAVY METALS IN EDIBLE CLAYS SOLD IN ONITSHA METROPOLIS OF ANAMBRA STATE, NIGERIA

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Abstract: This study was undertaken to determine and compare the level of heavy metals- zinc, lead, cadmium, copper, mercury and cobalt in two species of edible clay (Nzu and Ulo) sold in four major markets namely; Ochanja, Ose, Relief and Okpoko, in Onitsha metropolis of Anambra state, Nigeria. Heavy metals in the edible clay samples were analysed by atomic absorption spectrophotometry. Mean concentrations were computed from three replicate measurements. Data from the markets were compared by One-Way ANOVA while data between the clay types were analyzed by independent t-test. The result of the study indicate that mean concentrations (ppm) of Zn, Pb, Cd, Cu, and Co showed no significant difference [(P =.981, .479, .335, .333 and .613) for Zn, Pb, Cd, Cu, and Co respectively] among markets. The mean concentration (ppm) of Hg (2.335±0.276) in Ochanja market however, differ significantly (P =.045) from those of the other markets, 0.621±0.244, 0.796±0.280 and 0.251±0.190 for Ose, Relief and Okpoko, respectively. Statistically reliable differences were found for Zn (P =.000) and Co (P =.003) type 1(nzu) and 11 (ulo) mean concentrations. However, mean concentrations of Pb, Cd, Cu and Hg in type 1 and type 11 clays did not differ significantly [(P =.391, .288, .243, and .613) for Pb, Cd, Cu and Hg, respectively]. There were high concentrations of Hg in virtually all samples and Cd in few samples. The high levels of Cd and Hg in the analyzed edible clays make them unhealthy for human consumption. However, further research is required to validate the reliability of these findings.

Keywords: Cadmium, edible clay, heavy metals, mercury, permissible limit

Introduction

Geophagia is defined as deliberate consumption of earth, soil or clay (Alexander *et al.*, 2002; Abraham *et al.*, 2013). It is related to pica a classified eating disorder characterized by abnormal cravings for non-food substances (Alexander *et al.*, 2002; Abraham *et al.*, 2013). It exists in humans, most often in rural or pre industrial societies among children and pregnant woman (Alexander *et al.,* 2002). Geophagia is not limited to any particular age group, race, sex, geographic region or time period (Abraham *et al.*, 2013). It is a traditional, cultural or religious activity (Vermeer and Frate, 1979), which has been observed during pregnancy (Wodwodt and Kiss, 1999), or as a remedy for disease (Vermeer and Ferrell, 1985; Dominy *et al.*, 2004). Culturally speaking, the practice amongst many of the kaolin (clay) eaters emanates from having doubtlessly watched their mothers or close relatives eat the clay (Bisi-Johnson *et al.*, 2010). Many of the studies on geophagia have advanced many more other reasons for this phenomenon around the world. In southern parts of U.S.A, pregnant women who traditionally ate substances like clay, corn starch and baking soda believed that such substances helped to prevent vomiting, helped babies to thrive, cured swollen legs and ensured beautiful children (Mcloughlin, 1987). In parts of Africa, rural areas of the United State and villages in India, clay consumption is correlated with pregnancy (Geophagy, 2017). In Australia, some Aborignes eat white clay for medicinal purposes (Bisi-Johnson *et al.*, 2010). In Haiti, South Africa, Malawi, Zambia, Zimbabwe and Swaziland, geophagy is widespread (Bisi-Johnson *et al.,* 2010). In South Africa, the eating of clay is mostly observed among pregnant women. In urban South Africa, young women believe that earth eating will give them a lighter colour (making them supposedly more attractive) and soften their skin (Alexander *et al.*, 2002). Studies have also established that geophagia is rife among the Tanzanians and Kenyans in the eastern part of Africa, as well as, Senegal, Mali and Nigeria in West Africa (Bisi-Johnson *et al.*, 2010), and South Asia (Abraham *et al.*, 2013).

Clay for consumption in Nigeria is known as calabash chalk. Calabash chalk - also known according to language/ locality as Argile, Calabar stone, Calabash clay, Ebumba, Lacraie, Mabele, Ndom, Nzu, Poto and Ulo – is a generic term used for naming these Nigeria, geophagical materials (Abraham *et al.*, 2013). Most of the calabash chalk samples are clay rich soil materials that have been dried and/or baked into blocky or spherical unit. Geophagia in Nigeria is noted to be especially associated with pregnant women who consume earth materials to alleviate the symptoms of morning sickness (Abraham *et al.*, 2013). Pregnant women from the Igbo tribe – a large ethnic group of eastern Nigeria were recorded as the main eaters of calabash chalk (clay) (Abraham *et al.*, 2013). Some samples of Nzu and Ulo are shown in Figs. 1 and 2, respectively.


Fig. 1: Some sample of Nzu


Fig. 2: Some samples of Ulo

Clays contain large amounts of trace minerals. It is common to see as many as 75 different trace minerals in Montmorillonite clays as shown in Table 1 (US Patent, 2017). There are obvious risks in the consumption of clay that is contaminated by animal or human faeces, in particular, parasite eggs, such as round worm, that can stay dormant for years, can present a problem. Other dangers associated with geophagia include ingestion of a variety of bacteria, various forms of soil contamination and intestinal obstruction (Alexander *et al.*, 2003; Bisi-Johnson *et al.*, 2010). In the traditional societies, there is a widespread practice to heat-treat (bake) the earth before consumption, and this tends to mitigate the risks to some extent (Alexander *et al.*, 2003).

Table 1: Montmorillonite components (Average nutrient content per 1000 kg)

Montmorillonite components	Average nutrient content per 1000 kg
Silicon	6933
Aluminium Silica	2505
Sodium Chloride	1320
Potassium	1293
Protein	1116
Calcium	1104
Sulphur	431
Iron	431
Magnesium	224
Chlorine	164
Titanium	61.9
Carbon	48.2
Sodium	37.2
Barium	10.5
Phosphate	8.62
Strontium	6.46
Cesium	4.93
Manganese	4.04
Thorium	2.69
Uranium	2.69
Arsenic	1.97
Chromium	1.89
Molybdenum	1.64
Nickel	1.62
Iodine	1.28
Lead	1.17
Cerium	1.08
Rubidium	0.983
Antimony	0.781
Gallium	0.673
Germanium	0.673
Neodymium	0.539
Zinc	0.539
Lanthanum	0.486
Bismuth	0.385
Zirconium	0.269
Rhenium	0.269
Thallium	0.269
Tungsten	0.218
Vanadium	0.215
Ruthenium	0.210
Baron	0.189
Bromine	0.140
Cobalt	0.129
Selenium	0.110
Symposium	0.107
Fluorine	0.102
Scandium	0.0997
Samarium	0.0943
Nobelium	0.0754
Copper	0.0593
Praseodymium	0.0539
Erbium	0.0539
Hafnium	0.0539
Ytterbium	0.0377
Lithium	0.0377
Yttrium	0.0323
Holmium	0.0296
Cadmium	0.0296
Palladium	0.0189
Terbium	0.0161
Thulium	0.0161
Gold	0.0161
Tantalum	0.0135
Iridium	0.0135
Lutetium	0.0108
Europium	0.0108
Rhodium	0.0108
Tin	0.0108
Silver	0.00808
Indium	0.00808
Oxygen	0.00539
Mercury	0.00269
Tellurium	0.00269
Beryllium	0.00269

Source: ^US Patent 6962718 Table available at http://www.freepatentsonline.com/6962718.html

Apart from mineral elements in air and water, soil or clay serves as reservoir of chemical and biological agents. Among the chemical agents are heavy metals, radioactive gases and organic chemical (Bisi-Johnson *et al.,* 2010). Heavy metals (Fe, Mn, N, Cu, Zn, Co, Cr, V, Ti, Cd, Hg, Mo) and trace metals as well as Se and F occur naturally in soil (Wodwodt and Kiss, 1999). However, concentrations are frequently elevated because of contamination (Christoph *et al.*, 2001). Minerals such as iron, copper, zinc and manganese are essential and play important roles in biological systems. Meanwhile, mercury, lead and cadmium are toxic, even in trace amounts. However, essential minerals can also produce toxic effects at high concentrations (Nurnadi *et al.*, 2013).

The sources of contamination in soils are multifarious and include agricultural and industrial pollution (Tuchschmid *et al.*, 1995). Heavy metals are toxic and poisonous in relatively high concentrations. Two factors contribute to the deleterious effects of heavy metals as environmental pollutants. Firstly, they cannot be destroyed through biological degradation as in the case of most organic pollutants. Secondly, they are easily assimilated and can bioaccumulate. Bioaccumulation is the gradual build up over time of a chemical in a living organism. This occurs either because the chemical is taken up faster than it can be used or because the chemical cannot be broken down for use by the organism (Nurnadi *et al.*, 2013; Beer, 1999).

The clays (Nzu and Ulo) are obtained from subsurface area in a local community (Nteje) in Anambra State and sold by its merchants to traders who retail them in the markets. In Nteje locality, there is no proper/organized waste disposal method, hence, the residents engage in indiscriminate dumping of refuse on the land. For instance, lead is used to produce batteries, ammunition, metal products like devices, etc. The local residents use batteries a lot to power their torch light and radios and many a times you could see used batteries discarded on the land. Mineral fertilizers contain some cadmium. The farmers make use of fertilizers in farming. Cadmium from the fertilizers can leach into the soil. Mercury occurs naturally in air which eventually settles onto land. It is also emitted into the environment through combustion of coal. According to Abraham *et al*. (2013), any ingested geophagical material such as Nzu and Ulo has the potential to release mineral nutrients and potential hazardous elements (PHE) when they come in contact with digestive fluids. The edible clays sold in Onitsha metropolis may be poisonous and therefore harmful to the health of consumers because of possible contamination with heavy metals. Moreover, there is insufficient data on the quality of edible clays sold in Onitsha.

The aim of this study is to determine heavy metal concentration of edible clays sold commercially in Onitsha markets.

Materials and Methods

Analar grade HNO_3 and HCl were used to digest the clays which were subsequently subjected to heavy metal analysis using Varian FS240 Atomic Absorption Spectrophotometer manufactured by Varian Inc., Australia.

Sample collection

Two types/varieties of clay identified by a soil scientist from the Department of Crop and Soil Science, Faculty of Agricultural Sciences, National Open University of Nigeria, popularly known as Nzu and Ulo amongst the Igbo tribe of the Eastern part of Nigeria and sold commercially in an unpackaged manner were purchased from four spatially located markets in Onitsha, Anambra State of Eastern part of Nigeria. These markets were Ochanja, Ose, Relief and Okpoko. The edible clays sold in these markets are usually obtained from Nteje located at 6º 16’ 0””North 6º 55’ 0’’East. In each market, three replicate samples of each clay type were purchased and used for the study.

Sample preparation and analysis

The samples for metal analysis were digested according to the method described by Heu *et al*, 2002. Each variety of clay was ground using a porcelain mortar and pestle and sieved with 5 mm sieve and oven dried at 100ºC to constant weight. 1g of the dried sample was accurately weighed into a pre-cleaned 250 mL glass beaker and digested with aqua regia (a solution of freshly prepared HNO_3 and HCl in the ratio 1:3) and heated to 100ºC in a water bath until the solids dissolved completely. The solution was left to cool and 20 mL of deionized water was added to the cooled solution to dilute it. The diluted solution was then filtered through Whatman 110 mm filter paper into a previously pre - cleaned beaker. The filtrate was transferred into a sterile sample bottle previously washed with 10% nitric acid, washed severally with deionized water, and analyzed for heavy metals using Varian FS240 AA Spectrophotometer with detection limit of 0.001 ppm. The AAS machine was operated under the following conditions: measurement mode (integrate), slit width (0.5 nm), gain (78%), lamp current (4.0 mA), flame type (air/acetylene), air flow (13.50 L/min), acetylene flow (2.00 L/min), and burner height (0.0 mm). Standards used were 1000 ppm stock solutions of spectrascan SS (1202, 1217, 1214, 1201, 1132, and 1108) for Zn, Pb, Cd, Cu, Hg, and Co respectively. The machine runs in triplicate and the average absorbance of the sample was calculated by referring to the appropriate calibration curve based on commercial standards.

The concentration of metals in ppm was calculated as:

$$X\ (ppm) = \frac{mass\ of\ solute\ (mg)}{mass\ of\ solution\ (mg)} \times 1000000 \quad (1)$$

The concentrations of the analyte in the digest were converted to concentration in ppm prior to AAS analysis using the above relationship. The concentrations of the various metals determined were in ppm from the computer print-out.

Statistical analysis

Results are presented as mean ± standard deviation; n equals 3, the number of triplicate measurements. Data obtained were subjected to One-Way ANOVA and t-test using Statistical Package for Social Sciences (SPSS), software for windows version 19.0 (IBM SPSS Statistics 19.0). Results from the different markets were compared using One-Way ANOVA whereas the clay types from the four markets were compared using t-statistics at the 95% confidence level. $P \leq .05$ was considered to indicate significant difference. Where the means differ significantly in the One-way ANOVA, Least Significant Difference (LSD) was used to determine which means differ.

Results and Discussion

Table 2 shows the mean concentrations of zinc in ppm contained in the calabash chalk obtained from Ochanja, Ose, Relief and Okpoko markets all in Onitsha, Anambra State of Eastern Nigeria and the permissible limit stipulated by FAO /WHO, 2001. Mean concentration of zinc (ppm) ranged from 2.540 in Relief to 3.210 in Ose in type 1 clay and 7.911 in Relief to 11.719 in Okpoko in type 11. Relief market had the lowest mean concentration of 5.226 ppm while Okpoko had the highest mean concentration of 7.172 ppm. One-way ANOVA of the mean concentrations of samples from the four markets did not show significant difference (P =.981). The mean concentrations in ppm of zinc in both clay types (Nzu and Ulo) were less than the permissible limit of 100 ug/g i.e 100 ppm of zinc in food substances (FAO/WHO, 2001). There were higher concentrations of zinc in type11 (Ulo) clay than type 1 (Nzu) clay in all the markets sampled. A t-test revealed a statistically reliable difference (P =.000) between the mean concentration of type 1 (2.737±0.181) and type 11 (10.332±0.067) edible clays. This may be attributed to the chemical nature/structure of the clays. Nzu is kaolin clay while Ulo fits into the description of bentonite clay. Bentonite clays have extremely flat crystals, they are shaped like credit cards or playing cards, and they stack or layer, held together with weak electric bonds (Clay minerals, 2017). Bentonite clays are good at adsorbing heavy metals and other positively charged substances because bentonite is negatively charged, hence, bentonite nanoparticles stick to heavy metals by adsorption. This property is not manifested in other clays like kaolin. The fact that zinc was detected in the two clay types and the concentrations were below the permissible limit for zinc makes them nutritive as zinc is an essential element and non-toxic (Clay minerals, 2017). In terms of zinc, type 11 edible clay used in this study has a higher nutritive value compared to type 1 clay.

Table 2: Mean concentration (ppm) of zinc in edible clay samples

Type/Market	Ochanja	Ose	Relief	Okpoko	Mean
Type 1 ·	2.568±0.146	3.210±0.046	2.540±0.527	2.629±0.007	2.737±0.181^a
Type 11	11.582±0.074	10.120±0.086	7.911±0.048	11.716±0.062	10.332±0.067^b
Mean	7.075±0.110	6.665±0.066	5.226±0.288	7.172±0.035	
PL					100

Type 1= Nzu, Type 11= Ulo Means with different superscripts differ significantly at α =.05, PL = FAO/WHO permissible limit

The permissible limit for lead in food substances by FAO /WHO, 1999 and FAO /WHO, 2001 is 0.2 ug/g (0.2 ppm) and 0.3 ug/g (0.3 ppm), respectively. The mean concentration of lead in clay obtained from Ochanja was higher than the permissible limit for lead in food substances. As shown in Table 3, there was no lead found in type 1 clay obtained from the four different markets used in this study, and type 11 clay obtained from Ose, Relief and Okpoko markets. The mean concentration of type 1 clay did not differ significantly (P =.391) from that of type 11. There was also no significant difference (P =.479) among the mean concentrations of the samples from the sampled markets. The high level of lead above the permissible limit observed in type 11 clay obtained from Ochanja market makes it unfit for consumption because lead is toxic even in trace amounts.

Table 3: Mean concentration (ppm) of lead in edible clay samples

Type/Market	Ochanja	Ose	Relief	Okpoko	Mean
Type 1	ND	ND	ND	ND	
Type 11	3.171±0.064	ND	ND	ND	0.793±0.018
Mean	1.598±0.032				
PL					0.2 /0.3

Type 1= Nzu, Type 11= Ulo, ND= Not Detected; PL = FAO/WHO permissible limit

Table 4: Mean concentration (ppm) of cadmium in edible clay samples

Type/ Market	Ochanja	Ose	Relief	Okpoko	Mean
Type 1	1.027±0.442	0.282±0.080	ND	ND	0.327±0.130
Type 11	0.163±0.057	ND	ND	ND	0.041±0.130
Mean	0.595±0.249	0.141±0.040			
PL					0.1

Type 1= Nzu, type 11= Ulo, ND= Not Detected; PL = FAO/WHO permissible limit

The mean concentration in ppm of cadmium in the analyzed samples is shown in Table 4. The mean concentration Cd in type 1 clay ranged from 0.000 in Relief and Okpoko to 1.027 in Ochanja market. For type 11 clay, cadmium was detected only in the sample obtained from Ochanja market. Relief and Okpoko markets had 0.000 ppm mean concentration while Ochanja had the highest mean concentration of 1.027 ppm. There was no detection of cadmium in both clay types obtained from Relief and Okpoko markets as well as in type 11 clay obtained from Ose. A t-test revealed no significant difference (*P* =.288) between the mean concentrations of type 1 and 11 clays sampled. The same trend was observed for the One-way ANOVA of the various markets sampled (P =.335). The mean concentration of cadmium in both clay types from Ochanja and type 1clay from Ose were higher than the permissible limit of 0.1ppm set for cadmium in food substance by FAO /WHO (1999). The presence of cadmium in the clays from Ochanja market and type 1 clay from Ose above the permissible limit makes them unfit for consumption because of the adverse effect of cadmium. Cadmium is not an essential element for humans, animals and plants. Toxicity symptoms induced by cadmium include gastrointestinal disorders, kidney failure, hypertension, reduced pregnancy length and new born weight and disorders of the endocrine and/ or immune system in children.

Table 5 shows the mean concentrations in ppm of copper in the sampled clays and the permissible limit of copper stipulated by FAO /WHO, 2001. For type 1, the mean concentrations ranged from 0.116 in Relief to 0.220 in Ose while for type 11 the mean concentration ranged from 0.098 in Relief to 0.334 in Ochanja. There was no significant difference (*P* =.243) between the mean concentrations of type 1 and 11 clays used in the study. The same trend was observed among the markets (P =.333). The mean concentration in all the samples were lower than the permissible limit of 73 ppm set for copper in food substances by FAO/WHO, 2001.

As shown in Table 6, the mean concentrations in ppm of mercury were in the range 0.145 to 3.004 for type 1 clay and 0.357 to 1.666 for type 11 clay. Ochanja market had the highest mean concentration 2.335 ppm while Okpoko had the least (0.251 ppm). The mean concentration of edible clay samples obtained from Ochanja market (2.335±0.276) differs significantly (*P* =.045) from 0.621±0.244, 0.796±0.280, and 0.251±0.190 for Ose, Relief and Okpoko markets, respectively. The mean concentrations between the clay types (1 =1.185±0.282 and11 =0.817±0.213) however, did not differ significantly (*P* =.613). The mean concentrations of mercury for type I and type 11 clay samples obtained from Ochanja, Ose and Relief markets were higher than the permissible limit of 0.3 ppm set for mercury by FAO/WHO, 1999. For Okpoko market, the mean concentration of mercury in type I clay sample was less than the permissible limit while for type 11 clay sample, it was found to be slightly higher. Bonglaisin *et al.* (2011), reported a mean concentration of 368.4±376.9 µgg-1 of mercury in kaolin edible clay samples obtained from Nteje, Achala-Agu village. The high concentration of mercury in the analyzed samples could be attributed to both natural and anthropogenic sources. Naturally occurring mercury in the soil, atmospheric deposition, burning of coal, indiscriminate disposal and/or burning of mercury-containing waste as well as production of chlorine, metals and alkali are possible sources of mercury in the area where the samples were obtained. Nteje is located very close to Onitsha- a high industrial and commercial centre where various wastes are dumped indiscriminately on land. These wastes could be washed off to surrounding localities. Moreover, several coal mines were operated on commercial scale in Anambra where Nteje is found and other neighbouring states such as Enugu, Delta and Kogi between 1909 and 1999.The burning of coal over these years could have released a substantial amount of mercury into the environment. Mercury deposition in a given area depends on mercury emitted from local, regional, national, and international sources (USEPA, 2017). Once in the atmosphere, mercury is widely disseminated and can circulate for years, accounting for its wide-spread distribution (US Geological Survey, 2000. Some local mineral occurrences and thermal springs are naturally high in mercury. Mercury is a toxic metal even in trace amounts (Mercury and Health, 2017); therefore, its detection in food or edible substances makes such unfit for consumption.

Table 5: Mean concentration (ppm) of copper in edible clay samples

Type/Market	Ochanja	Ose	Relief	Okpoko	Mean
Type 1	0.205±0.059	0.220±0.028	0.116±0.054	0.133±0.002	0.119±0.036
Type 11	0.270±0.044	0.270±0.044	0.098±0.078	0.315±0.020	0.245±0.032
Mean	0.270±0.044	0.270±0.044	0.107±0.066	0.224±0.011	
PL					73

Type 1= Nzu, type 11= Ulo; PL = FAO/WHO permissible limit

Table 6: Mean concentration (ppm) of mercury in edible clay samples

Type/Market	Ochanja	Ose	Relief	Okpoko	Mean
Type 1	3.004±0.490	0.674±0.222	0.917±0.253	0.145±0.163	1.185±0.282
Type 11	1.666±0.062	0.568±0.267	0.675±0.306	0.357±0.217	0.817±0.213
Mean	$2.335±0.276^a$	$0.621±0.244^b$	$0.796±0.280^b$	$0.251±0.190^b$	
PL					0.3

Type 1= Nzu, type 11= Ulo, Means with different superscripts differ significantly at α = .05, PL = FAO/WHO permissible limit

Table 7: Mean concentration (ppm) of cobalt in edible clay samples

Type/Market	Ochanja	Ose	Relief	Okpoko	Mean
Type 1	0.017±0.015	ND	ND	ND	0.004±0.004[a]
Type 11	0.354±0.039	0.299±0.076	0.142±0.030	0.437±0.025	0.308±0.042[b]
Mean	0.186±0.027	0.150±0.038	0.071±0.015	0.219±0.012	
PL					0.15-1.0

Type 1= Nzu, type 11= Ulo, ND= Not Detected, Means with different superscripts differ significantly at α .05, PL = FAO/WHO permissible limit

The mean concentrations in ppm of cobalt were in the range 0.142 to 0.437 for type 11 clay; whereas there was no detection of cobalt in type 1 clay except for the sample from Ochanja market as shown in Table 7 Okpoko had the highest mean concentration of 0.437 ppm while Relief had the lowest. There was no significant difference (*P* =.921) in the mean concentrations of samples from the four markets. Type 1 mean concentration differs significantly (*P* =.003) from that of type 11. However, the mean concentrations of all the samples were within the permissible limit of 0.15 – 1.0 ppm set for cobalt by FAO, 1985.

Conclusion

Based on the results of this study, the concentrations of the essential elements (zinc and copper) in the edible clays were very low compared to the permissible limits stipulated by FAO and WHO. Therefore, the nutritive values of the clays estimated from the concentrations of these essential elements were low. Concentrations of the toxic elements mercury and cadmium were relatively high. Heavy metals bioaccumulate, thus, continuous consumption of these clays contaminated with the toxic heavy metals observed in this study pose serious health threat to its consumers. The high concentrations of these toxic heavy metals might be due to indiscriminate waste disposal, burning of hazardous waste, agricultural activities, and combustion of fossil fuels. There is therefore great need to plan and implement proper waste disposal strategies in areas where these clays are obtained. There is also need to encourage the use of renewable fuels such as biogas and biodiesel.

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