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# HEAVY METAL CONTAMINATION OF PHARMACEUTICAL PRODUCTS COMMONLY SOLD IN NIGERIA

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## Abstract

The study evaluates the contamination by heavy metals of pharmaceuticals commonly used in Nigeria. Pharmaceutical samples in sixteen pharmacological groups in different dosage forms were purchased from registered pharmacies and patent medicine shops from Abraka and Obiaruku in Delta State, Nigeria. The samples were digested using a bi acid mixture of $HNO_3$ and $HClO_4$ in the ratio of 6:1 and analyzed using the Atomic Absorption spectrometer (AAS) for the presence of lead, chromium, cadmium, zinc, nickel, manganese, iron, copper and cobalt. The result reveals significant contamination by all the metals investigated with the exception of copper and cobalt. The contamination may be attributed to wear and tear of manufacturing machinery, contamination from water, packaging and raw materials. It is recommended that machinery used for the manufacture of pharmaceuticals should be well maintained and replaced were necessary and strict adherence to good manufacturing practice (GMP) as required by regulatory agencies.

Keywords: Pharmaceutical, Heavy metals, Contamination, Product

## Introduction

Twenty four percent of global disease burden for adults and children under the age of five is caused by environmental exposures (WHO, 2015). In Nigeria, forty-four percent of deaths is traceable to poor environment (Alo, 2008). A potent but often ignored environmental factor that affects all quality of life is the inevitable use of pharmaceutical products. Pharmaceuticals are products of medical and industrial advances used for the diagnosis, treatment and prevention of

diseases and ailments, which have replaced to a large extent our reliance on crude herbal preparations.

Most pharmaceutical products are made by the synthesis of chemicals both organic and inorganic. Relatively few are derived from plants and animal sources. As a result, many of the most effective pharmaceuticals and diagnostic agents that have been introduced into medicine over the last few decades are metal based. Today drugs employing heavy metals have been extremely effective for cancer treatment and other life threatening diseases (CHMR, 2008).

A majority of the population invariably use pharmaceutical products almost daily either as food supplements, analgesic, antihypentensive etc. Sustained exposure of our body to heavy metals will lead ultimately to the accumulation of toxins in our tissues and organs causing nutritional deficiencies, hormonal imbalances, neurological disorders, autoimmune reactions, cancer, and other chronic pathological conditions (Pham-Huy *et al.,* 2008). Infact heavy metal toxicity maybe the root of many health disorders today. Heavy metal ion produces large quantities of free radical compounds, which destroys lipids, proteins and DNA in the cellular system.

Free radicals damage our cells, prevent cell stabilization and create an overly acidic terrain in the body (Choi *et al,* 1999). Also heavy metals cause the body to produce cholesterol and prevent the absorption of calcium. And, the more free heavy metal ions in the body, the more buildup of acidic waste and hence the more calcium is deposited in the arteries (Alabaster and Lloyd, 1980). Bryan (1976) reported that the threat heavy metal toxins pose to our health is beginning to gain attention. However, heavy metal toxicity is a condition that often goes overlooked in traditional medical diagnosis. While it is rare for an individual to experience a disease or health condition solely from a heavy metal toxin, it is reasonable to conclude that these toxins exert a dramatic effect on the health of an individual and contribute to the progression of many different debilitating conditions.

Moreover, taking ill and our ability to recover from one is almost always traceable to biochemical reactions in the body with heavy metals playing a significant role. Moreover, heavy metals can interact with pharmaceuticals in the body to alter their pharmacological effects (Stargart *et* al., 1989). The interaction of aluminum ions with tetracycline to reduce the absorption of the latter is well known. Hence physical and pharmacological interactions between drugs and heavy metals may lead to altered biological actions, prolonged side effects through delayed tubular excretion and biotransformation. Such interactions may also results in anaphylactic reactions and other untoward forms of drug effects leading to cumbersome management of diseases and hospital induced illness (Woods *et al.,* 2007).

The term “heavy metal contaminants in pharmaceuticals” may seem a misnomer in some cases considering the fact that heavy metals are currently used in pharmaceuticals for therapeutic purposes. For example, aluminum as the hydroxide is used as an antacid for relieving the symptoms of peptic and other forms of hyperacidity. Selenium and copper are employed as nutritional supplements because of the belief that they have a functional role in haemoglobin formation. However, heavy metals found in pharmaceutical products where they are not expected to play any therapeutic role and in excess of the desired quantity when used therapeutically may be regarded is a contaminant.

In tablets, contamination may occur from raw materials, active ingredients, excipients e.g. corn starch used as binders, magnesium stearate used as lubricants, disintegrants and lactose used as fillers. Contamination may also occur during granulation including sieving, granular flow from hopper to the die for compression. The sieve, hopper, die and punch in a tableting machine are all made of stainless steel and therefore potential sources of contamination. Liquid preparations, creams, ointments, liniments e.t.c may be contaminated by the raw materials,, active ingredients and excipients (sugar, suspending agents, ointment bases usually petroleum jelly, emulsifiers, water, \preservatives, oil, surfactants etc).

## Materials and Methods

### Sampling

One Hundred (100) samples of various brands of pharmaceuticals from sixteen pharmacological classes in different dosage farms were purchased from pharmaceutical shops and patent medicine stores in Abraka and Obiaruku in Delta State, Nigeria. The sample were manufactured in Nigeria, India and China. None of the sample have expired and are all registered with the National Agency for Food and Drug Administration and Control (NAFDAC) in Nigeria. The pharmacological therapeutic classes of pharmaceutical products purchased include the following:

i. Analgesics
ii. Antimicrobials
iii. Antimalarials
iv. Antihypertensives
v. Antidiabetics
vi. Anthelmintics
vii. Antituberculosis
viii. Anticonvulsants
ix. Antidiarrhoeas
x. Antipsychosis
xi. Antiasthmatics
xii. Haematmics
xiii. Antiulcers
xiv. Antihistamines
xv. Corticorsteroids
xvi. Contraceptives

### Chemicals and Reagents

The perchloric acid and concentrated nitric acid used were of analytical grade. They were manufactured by Merch Kga A of Germany and BDH Limited Poole, England respectively. The distilled water used was double distilled (DD).

### Sample Preparation

Two grams (2g) of sample was accurately weighed on electronic balance into an Erlenmeyer Flask 2m1 of perchloric acid and 12m1 of concentrated nitric acid are added into the flask The bi-acid digested mixture was heated on a hot plate in a fume cupboard and the temperature maintained at 122°C until dense white fumes appear. The flask was then allowed to cool to room temperature.

50 ml of distilled water was then added and digestion continued until a clear solution was formed. The solution was cooled and made up to 50 ml with 1isti11ed water (Musa and Flomza, 2009).

### Chemical Analysis

The levels of metals (Cd, Pb, Ni, Cr, Cu, Co, Fe, Mn and Zn) contaminants in the sample solutions were analyzed using atomic absorption spectrometry (Analyst 200, Norwalk CA, USA).

### Quality Control/Assurance

The laboratory table and glass wares were thoroughly cleared to avoid cross contamination. Blank was used to correct instrument readings and spike recovery method was used to check the analytical procedure. The spike recovery achieved for the metals were greater than 89.4%.

### Statistical Analysis

Analysis of variance (ANOVA) was used to test for the difference within the same pharmacological groups studied and lukeep test was used to compare differences in the mean concentrations.

**Results**

The study of heavy metal contamination in pharmaceutical samples purchased are presented below in Tables 1 to 6.

Table 1: Levels of heavy metal contamination (µg/g) in some brands of analgesics

| S/N | . ANALGESICS (A) | Brand Code | Pb | Cd | Cr | Ni | Mn | Fe | Zn | Cu | Co |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Tcb, Ibuprofen 200mg/Tab Paracetamol 500mg Caffeine 50mg | A1 | Nd | Nd | 1.9 | 0.5 | 1 | 224.03 | 10.53 | 1.55 | 3.45 |
| 2 | Cap Indomethacin 25 mg | A2 | 11.00 | 0.2 | 2.7 | 3.1 | 22.9 | 224.78 | 7.18 | 2.75 | 2.58 |
| 3 | Cap Piroxican 20mg | A3 | 3.00 | Nd | 8.8 | 1.7 | 3 | 170.11 | Nd | 0.125 | Nd |
| 4 | Cap Piroxicam 20mg | A4 | Nd | 0.4 | Nd | 1 | 2.6 | 19021 | 6.12 | 0.3 | Nd |
| 5 | Cap Piroxicam 20mg | A5 | Nd | Nd | 0.6 | Nd | Nd | 50.3 | 2.11 | 0.55 | 0.73 |
| 6 | Tab Aspirin 300 mg | A6 | 17.1 | Nd | 9.4 | 0.6 | 27.7 | 6.7 | 23.1 | 8.32 | 2.58 |
| 7 | Cap Tramadol 50mg | A7 | Nd | 0.2 | Nd | Nd | Nd | 24.81 | Nd | 0.1 | Nd |
| 8 | Inj Diclofenac 75mg | A8 | Nd | 2.4 | 0.2 | 5 | 1.7 | 27.73 | 38.42 | 1.4 | 8.93 |
| 9 | Pu/v Aspirin 760mg/Caffeine 60mg | A9 | Nd | Nd | Nd | 1.7 | 3 | 36.28 | 14.43 | 2.75 | Nd |
| 10 | Tab Diclofenac 100mg | A10 | 2.5 | Nd | 3.63 | 1.8 | 0.8 | 31.93 | 7.35 | 40 | 0.05 |
| 11 | Tab Ibuprofen 400mg | A11 | 3.6 | Nd | 3.4 | 2.1 | 4.23 | 162.05 | 7.35 | 1.18 | 2.93 |
| 12 | Tab Ibuprofen 400mg | A12 | 6.1 | Nd | Nd | 3.7 | 11.1 | 67.05 | 5.93 | 0.63 | 1.95 |
| 13 | Tab lbuprofen 400mg | A13 | 43.5 | 1.6 | 1.8 | 14.4 | 4.93 | 34.13 | 8.13 | 2.3 | 0.45 |
| 14 | Cap Ibuprofen 200mg/ Paracetamol 500mg/Caffeine 50mg | A14 | Nd | 1 | 24.4 | 4.4 | 28.7 | 29.3 | 2.85 | Nd | Nd |
| 15 | Tab Paracetamo1 500mg | A15 | Nd | Nd | 7.9 | 0.7 | 1.5 | 81.2 | 9.12 | 2.06 | Nd |
| T | Tab Paracetamol 500mg | A16 | 1.1 | Nd | 3.8 | 1 | 1.33 | 34.13 | 7.73 | 2.3 | 0.45 |
| 17 | Tab Paracetamol 500mg | A17 | Nd | Nd | Nd | Nd | 1.2 | 109.56 | 4.6 | 3.6 | Nd |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 18 | Tab Paracetamol 500mg | A18 | Nd | 1.1 | Nd | 1 | 2.1 | 19.56 | 0.83 | 0.18 | Nd |
| 19 | Tab Paracetamol 500mg/Aspirin 300mg | A19 | Nd | Nd | 1.4 | 0.5 | 2.0 | 52.83 | 3.21 | 13.3 | Nd |
| | | Mean | 4.62 | 0.78 | 3.68 | 2.27 | 6.3 | 82.98 | 8.37 | 4.39 | 1.27 |
| | | S.D | 9.84 | 0.67 | 5.84 | 3.6 | 9.08 | 72.57 | 9.08 | 9.25 | 2.02 |

Table 2: Levels of heavy metal contamination (µg/g) in some brands of antimicrobials

| S/N | ANTIMICROBIALS (AM) | Brand code | Pb | Cd | Cr | Ni | Mn | Fe | Zn | Cu | Co |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 20 | Cap Ampicillin 250mg /Cloxacillin | AM1 | 1.4 | Nd | 5.5 | 0.2 | 18.6 | Nd | 11.7 | | Nd |
| 21 | Syl Ampicillin 125mg | AM2 | Nd | Nd | Nd | 1.2 | 2.9 | 60.78 | 5.28 | 2.58 | Nd |
| 22 | Syl Ampicillin 125mg /Cloxacillin | AM3 | Nd | Nd | 2.5 | 1.4 | 8.1 | 42.23 | 2.63 | 0.48 | Nd |
| 23 | Cap Amoxicillin 500mg | AM4 | Nd | 0.5 | Nd | 1.1 | 3.5 | 25.39 | 3.83 | 0.28 | Nd |
| 24 | Syl Ampicillin 125mg/5ml | AM5 | Nd | Nd | Nd | 2.3 | 4.4 | 57.85 | 2.23 | 0.23 | Nd |
| 25 | Cap Chloramphenicol 250mg | AM6 | 1.3 | Nd | 3 | 1.7 | 8.2 | 69.58 | 3.23 | 0.22 | Nd |
| 26 | Clotrimazole Pessaries 100mg | AM7 | 5.6 | 1 | Nd | 1.5 | 2.4 | 48.18 | 5.03 | 0.88 | 0.68 |
| 27 | ClotrimazolePessaries 100mg | AM8 | 4.3 | 0.5 | Nd | 1.9 | 16.1 | 13.9 | 2 | 2.1 | Nd |
| 28 | Tab Cotrimoxazole 480mg | AM9 | Nd | Nd | 16.1 | 1.7 | 4 | 36.06 | 2.23 | 0.08 | Nd |
| 29 | Cap Chloramphenicol 250mg | AM10 | Nd | Nd | Nd | 1.6 | 1.3 | 63.6 | 3.43 | 3.45 | Nd |
| 30 | Tab Cotrimoxazole 480mg | AM11 | Nd | Nd | Nd | 2.3 | 5.3 | 44.48 | 4.2 | 10 | 0.58 |
| 31 | Tab Erythromycia 250mg | AM12 | Nd | Nd | Nd | Nd | 1.3 | 50.13 | 6.12 | 3.11 | Nd |
| 32 | Inj Genticia 28omg | AM13 | 4.2 | Nd | 1.2 | 4.6 | 6.5 | 49.3 | 6.63 | 4.5 | Nd |
| 33 | Tab Griseofulvin 500mg | AM14 | 3.3 | 0.02 | 0.6 | 1.7 | 22.9 | 11.48 | 3.23 | 1.3 | 0.3 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 34 | Cap Lincomycin 500mg | AM15 | 0.5 | 0.05 | 1.7 | 2.4 | 2.7 | 30.1 | 7.5 | 1.58 | 0.48 |
| 35 | Tab Metronidazole 200mg | AM16 | 4.9 | 0.03 | 3.6 | 5.5 | 3.1 | 99.33 | 8.93 | 1 | 2.73 |
| 36 | Tab Metronidazole 400mg | AM17 | Nd | Nd | Nd | 1.8 | 8.3 | 48.05 | 1.63 | 0.05 | Nd |
| 37 | Tab Metronidazole 400mg | AM18 | 0.02 | Nd | 2.7 | 1.3 | 1.8 | 36.4 | 15.1 | 2.43 | Nd |
| 38 | Nystatin Pessaries 100,000iu | AM19 | 1.6 | Nd | Nd | 1.4 | 3.2 | 358 | 7.18 | 13.28 | Nd |
| 39 | Penicillin Skin Ointment 10,000iu | AM20 | 6.90 | 0.6 | Nd | 1.2 | 0.9 | 29.48 | 8.65 | 0.23 | Nd |
| 40 | Cap Tetracycline 250mg | AM21 | 4.9 | Nd | 3.5 | 5.5 | 7.8 | 48.2 | 3.88 | 1.08 | 0.88 |
| | | Mean | 1.85 | 0.13 | 1.92 | 2.01 | 5.93 | 59.21 | 5.46 | 2.33 | 0.27 |
| | | S.D | 2.34 | 0.26 | 3.45 | 1.21 | 31.93 | 72.06 | 3.49 | 3.39 | 0.62 |

Table 3: Levels of heavy metal contamination (µg/g) is some brands of antimalarials and antihypertensives

| S/N | ANTIMALARIALS (AL) | Brand Code | Pb | Cd | Cr | Ni | Mn | Fe | Zn | Cu | Co |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 41 | lnj. Chloroquine 40mg/ml | AL1 | 1.1 | Nd | 0.8 | 250 | 2.6 | 80.83 | 14.58 | 3.13 | Nd |
| 42 | Cap Chloroquine Phosphate 400mg | AL2 | 1.1 | Nd | 0.8 | 4.1 | 4.1 | 8.83 | 4.34 | 8.83 | Nd |
| 43 | Syr Artemether/Lumefantrine | AL3 | Nd | Nd | 6.0 | Nd | 6.1 | 305.25 | 11.7 | 11.2 | Nd |
| 44 | Tab. Amodaquine 200mg | AL4 | 1.1 | 0.6 | 2.1 | 0.9 | 5.2 | 78.25 | 9.14 | 5.13 | Nd |
| 45 | Tab Artesunate 50mg | AL5 | 7.6 | 0.3 | Nd | 2.3 | 11.5 | 50.25 | 4.58 | 3.85 | 1.2 |
| 46 | Tab Quinine 300mg | AL6 | 9.6 | 0.8 | 4.3 | 6.1 | 3.5 | 31.17 | 262 | 5.23 | 5.3 |
| 47 | Tab. Sulphadoxine 500mg /Pyrimethamine 75mg | AL7 | Nd | Nd | 4.1 | 4.4 | 3.7 | 112.3 | 12.13 | 2.15 | Nd |
| 48 | Tab. Arthemether/lumenfantrine 20mg /Luemantrine 120mg | AL8 | 1.6 | 0.05 | Nd | 1.4 | 3.2 | 51.11 | 6.3 | 1.19 | 0.9 |
| | | Mean | 2.76 | 0.22 | 2.26 | 2.68 | 4.98 | 59.11 | 8.17 | 5.09 | 0.93 |
| | | S.D | 3.49 | 0.32 | 2.27 | 1.99 | 2.86 | 19.32 | 0.06 | 1.1 | 2.67 |

| | ANTIHYPERTENSIVE (AH) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 49 | Tab Apresoline 25mg | AH1 | Nd | Nd | Nd | 1.3 | 3.4 | 20.50 | 2.60 | 4.15 | Nd |
| 50 | Tab Captopril 25mg | AH2 | 0.6 | 0.8 | 0.5 | 3.7 | 3.8 | 60.93 | 3.9 | 5.13 | Nd |
| 51 | Tab 1-lydrochiorothiazide 50mg/ Ameloride 75mg | AH3 | 6.2 | 0.4 | 0.5 | 3.9 | 12.1 | 41.25 | 1.24 | 0.025 | 2.35 |
| 52 | Tab Hydrochlorothiazide 50mg/ Amoloridc 75mg | AH4 | 4.3 | Nd | 0.7 | 3.1 | 14.6 | 51.15 | 3.12 | 1.5 | Nd |
| 53 | Tab Methyldopa 250mg | AH5 | 1.3 | 0.1 | Nd | 0.8 | 1.1 | 6.2 | 6.53 | 543 | Nd |
| 54 | Tab Methyldopa 250mg | AH6 | 0.2 | Nd | 0.1 | 1.4 | 1.7 | 37.45 | - 6.88 | 2.45 | Nd |
| 55 | Tab Methyldopa 250mg | AH7 | 1.2 | Nd | T | 1,4 | 1 | 28.83 | 5.28 | ‘4.14 | Nd |
| 56 | Tab Methyldopa 250mg | AH8 | 15.3 | 0.4 | 5.8 | 6.8 | 37.5 | 252 | 7.08 | 6.96 | 4.9 |
| 57 | Tab Hydrochlorothiazide 50mg | AH9 | Nd | 0.4 | Nd | 0.6 | 0.7 | 8.8 | 4.1 | 5.15 | Nd |
| 58 | Tab Nifedipine 20mg | AH10 | 3.9 | Nd | 2,9.7 | 15.7 | 1.8 | 27.8 | 4.78 | 0.88 | 1.95 |
| 59 | Tab Nifedipine 20mg | AH11 | 1.7 | Nd | Nd | 1.7 | 12 | 37.45 | 17.4 | 15.78 | Nd |
| 60 | Tab Methyldopa 250mg | AH12 | 22.2 | Nd | 1.6 | 0.9 | 2.9 | 46.33 | 3.95 | 3.44 | Nd |
| 61 | Tab Captopril 25mg | AH13 | Nd | Nd | 2.8 | 2 | 20 | 144.13 | 6.45 | 0.53 | 0.68 |
| 62 | Tab Methyldopa 250mg | AH14 | 4.3 | 0.5 | Nd | 1.6 | 1.3 | 24.8 | 8.63 | 0.55 | Nd |
| | | Mean | 4.34 | 0.19 | 3.05 | 3.2 | 6.86 | 58.9 | 5.33 | 4.69 | 0.76 |
| | | S.D | 6.56 | 0.26 | 7.83 | 3.96 | 9.99 | 67.3 | 2.05 | 4.37 | 1.48 |

Table 4: Levels of heavy metal contamination (µg/g) in some brands of antidiabetes, anthelmentics, antituberculosis and anticonvulsants

| S/N | ANTIDIABETES (AD) | Brand code | Pb | Cd | Cr | Nl | Mn | Fe | Zn | Cu | Co |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 63 | Tab Chlorpropamide 250mg | AD1 | 3.9 | Nd | N d | 1.1 | 8.5 | 30.13 | 8.23 | 6.23 | 0.55 |
| 64 | Tab Glipizide 5mg | AD2 | Nd | 0.1 | Nd | 1.3 | 4.6 | 31.48 | 3.63 | 0.53 | Nd |
| 65 | Tab Glucophapo 250mg | AD3 | 11 | 0.8 | 0.9 | 1.5 | 1.3 | 24.78 | 3.43 | 7.63 | Nd |
| 66 | Tab Glipizide 5mg | AD4 | 30.2 | 0.2 | 9.5 | 8.2 | 8.0 | 329.8 | 25.23 | 1.73 | 2.8 |
| 67 | Metformin 250mg | AD5 | Nd | 9.3 | Nd | 1.5 | 1.0 | 49.9 | 4.83 | 2.4 | Nd |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Mean | 9.02 | 0.28 | 2.08 | 2.72 | 4.68 | 43.22 | 9.07 | 3.7 | 0.67 |
| | | S.D | 11.83 | 0.31 | 4.03 | 3.07 | 3.56 | 134.10 | 2.04 | 0.65 | 2.17 |
| | ANTHELMINTICS (AT) | | | | | | | | | | |
| 68 | Tab. Levamisole 40mg | AT1 | 0.03 | Nd | 1.6 | Nd | 4.2 | 60.9 | 16.2 | 23.73 | Nd |
| 69 | Tab Mebendazole 100mg | AT2 | 9.8 | 0.2 | 5.6 | 4.9 | 39.8 | 408.25 | 0.48 | 0.18 | 4.55 |
| 70 | Tab Pyrantel Pamoate 100mg | AT3 | 15.7 | 1.2 | 12.6 | 37.4 | 6.4 | 53.93 | 8.11 | 1.53 | 2.75 |
| | | Mean | 10.51 | 0.47 | 6.4 | 14.1 | 16.8 | 174.36 | 8.26 | 8.48 | 2.43 |
| | | S.D | 4.87 | 0.64 | 5.63 | 20.32 | 19.95 | 121.17 | 3.72 | 3.87 | 0.4 |
| | ANTITUBERCULOSIS (AC) | | | | | | | | | | |
| 71 | Tab Ethambutol 500mg Pyrazinamide 150mg Isonizide 150mg/Rifampicin 300mg | AC1 | Nd | 0.2 | 1.9 | 1.9 | 2.6 | 114.3 | 2.28 | Nd | Nd |
| 72 | Tab Isoniazide 150mg | AC2 | 8.3 | Nd | 2.4 | 3.9 | *15.9* | 1042 | 1.19 | 0.03 | 1.1 |
| 73 | Tab Comb Dot | AC3 | Nd | Nd | 1..4 | 0.4 | 2.0 | 52.83 | 0.36 | 41.2 | Nd |
| 74 | Cap Rifampicin 300mg | AC4 | 0.09 | Nd | 0.1 | 1.5 | 2.6 | 4120 | 1.66 | l.10 | Nd |
| | | Mean | 2.11 | 0.05 | 1.45 | 1.9 | 5.78 | 90.44 | 1.28 | 13.74 | 0.37 |
| | | S.D | 4.16 | 0.1 | 0.18 | 1.4 | 6.76 | 32.96 | 0.96 | 23.78 | 0.64 |
| | ANTICONVULSANTS | | | | | | | | | | |
| 75 | Tab Carbamazepine 200mg | | 1.8 | 0.7 | 6.5 | 2.7 | 6.1 | 98.16 | 17.82 | 1.7 | Nd |
| 76 | Tab Phenobarbitone 30mg | | 9.9 | 0.3 | 1.8 | 7.7 | 4.4 | 133.77 | 33.25 | 1.38 | 3.85 |
| 77 | Cap Phenytoin 100mg | | 3.8. | Nd | 1.8 | 2.7 | 23.2 | 204.85 | 16.63 | 1.55 | 0.83 |
| | | Mean | 5.17 | 0.33 | 434 | 4.2 | 11.23 | 145.59 | 22.57 | 1.54 | 1.56 |
| | | S.D | 4.22 | 0.35 | 2.37 | 2.6 | 10.4 | 54.33 | 9.32 | 0.17 | 2.03 |

Table 5: Levels of heavy metal contamination (µg/g) in some brands of antidiarrhoea, antipsychosis, antiastimatics, haematinics and antiulcers

| S/N | ANTIDIARRHOEA (AR) | Brand name | Pb | Cd | Cr | Ni | Mn | Fe | Zn | Cu | Co |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 78 | Tab Loperamide 4mg | AR1 | 6.7 | Nd | 5.3 | 3.3 | 26,6 | 261.14 | 11.22 | 0.03 | 2.23 |
| 79 | Oral Rehydration Salt | AR2 | Nd | 1 | 4.4 | 2.4 | 28.7 | 224.03 | 10.53 | 1.56 | 3.45 |
| | | Mean | 3.35 | 0.5 | 4.85 | 3.85 | 27.65 | 242.59 | 10.88 | 0.78 | 2.84 |
| | | S.D | 4.73 | 071 | 0.63 | 0.77. | 1.48 | 26.21 | 0.68 | 1.08 | 0.86 |
| | ANTIPSYCHOSIS (AC) | | | | | | | | | | |
| 80 | Tab chlorpromazme 50mg | AC1 | 10.6 | 0.3 | 6.6 | 4.8 | 36.2 | 97.23 | 24.19 | 0.83 | 1.45 |
| 81 | Tab triflouroperazine 5mg | AC2 | 7.2 | 0.1 | 3.9 | 5.0 | 11.2 | 67..33 | 20.0 | 1.18 | 1.78 |
| | | Mean | 8.9 | 0..2 | 5.25 | 4.9 | 23.7 | 82.28 | 22.09 | 1.01 | 1.62 |
| | | S.D | 2.4 | 0.14 | 1.91 | 0.14 | 17.68 | 21.14 | 2.93 | 0.25 | 0.23 |
| | ANTIASTHMATICS (AA) | | | | | | | | | | |
| 82 | Tab Aminophylline 100mg | AA1 | 10.6 | 0.3 | 0.3 | 4.5 | 12 | 53.93 | 8.4 | 15.73 | 2.75 |
| 83 | Tab Salbutamol 4mg | AA2 | 6.8 | Nd | 0.4 | 3 | 53.6 | 200.73 | 7.83 | 13.21 | 2.43 |
| 84 | Tab Salbutatnol 2mg | AA3 | 1 | 2.3 | 1.1 | 10 | 1.5 | 39.78 | 3.5 | 1.53 | Nd |
| | | Mean | 6.13 | 0.87 | 0.6 | 2.87 | 22.37 | 98.15 | 10.16 | 10.16 | 1.73 |
| | | S.D | 4.83 | 1.25 | 0.44 | 1.7 | 27.55 | 89.12 | 2.68 | 7.58 | 1.5 |
| | HAEMATINICS (H) | | | | | | | | | | |
| 85 | Tab Ferrous Sulphate 300mg | H1 | 0.7 | 0.044 | 4.7 | 2.8 | 15.7 | 305.19 | 10.5 | 16.63 | 0.3 |
| 86 | Cap Iron/Muitivitamin/ Minerals | H2 | Nd | Nd | 1.7 | 2.6 | Nd | 224.9 | 3.48 | 1.63 | .1.16 |
| 7 | Cap Iron/Multivitamin/ Minerals | H3 | Nd | Nd | Nd | Nd | 30.5 | 154.08 | 5.53 | 2.81 | Nd |
| 88 | Tab Vitamin B complex | H4 | Nd | 2.1 | Nd | 1.5 | 12.2 | 160.12 | 7.11 | 3.2 | Nd |
| 89 | Tab Multivite | H3 | 0.5 | Nd | 3 | 2.2 | 9.1 | 89.62 | 1.1 | 2.06 | 1.01 |
| | | Mean | 0.24 | 0.43 | 1.88 | 1.82 | 13.5 | 186.78 | 5.54 | 5.27 | 0.49 |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | S.D | 0.34 | 0.93 | 2.02 | 1.14 | 11.15 | 82.6 | 3.57 | 6.39 | 0.56 |
| | ANTIULCERS (AV) | | | | | | | | | | |
| 90 | Tab Cimelidinc 400mg | AV1 | 2.0 | Nd | Nd | 2.0 | 7.6 | 109.55 | 4.60 | 3.60 | Nd |
| 91 | Cap Omeprazole 20m g | AV2 | Nd | 0.4 | Nd | 0.8 | 1.2 | 33.83 | 4.16 | 16.5 | Nd |
| 92 | Tab Ranitidine 150mg | AV | Nd | 0.1 | Nd | 0.5 | 3.8 | 178.6 | 9.68 | 11.23 | Nd |
| | | Mean | 0.67 | 0.17 | 0.0 | 1.1 | 4.2 | 107.3 | 6.15 | 10.44 | 0.0 |
| | | S.D | 1.15 | 0.21 | 0.0 | 0.79 | 3.22 | 72.4 | 3.07 | 6.49 | 0.0 |

Table 6: Levels of heavy metal contamination (µg/g) in some brands of anthistamines, corticosteroids and contraceptives

| S/N | Anthistamines (AM) | Brand name | Pb | Cd | Cr | Ni | Mn | Fe | Zn | Cu | Co |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 93 | Tab. Chlorpheniramine 4mg | AM1 | 6.6 | 6.4 | 14.1 | 1.7 | 3.9 | 77.63 | 18.23 | 2.22 | 3.89 |
| 94 | Tab. Chlorpheniramine 4mg | AM2 | Nd | Nd | Nd | 1.0 | 2.6 | 63.6 | 3.43 | 2.58 | Nd |
| 95 | Tab. Cyproheptadine 4mg | AM3 | 9.1 | 1.0 | 4.40 | 5.40 | 25.4 | 81.22 | 38.43 | 1.4 | 8.93 |
| 96 | Tab. Cyproheptadine 4mg | AM4 | 25.9 | 3.1 | 13.9 | 13.2 | 24.4 | 113.68 | 8.63 | 2.85 | 4.55 |
| | | Mean | 10.4 | 2.63 | 8.1 | 5.33 | 14.08 | 84.03 | 17.18 | 2.26 | 4.34 |
| | | S.D | 11.02 | 2.83 | 7.05 | 5.59 | 12.52 | 21.18 | 16.55 | 0.65 | 3.65 |
| | CORTICORSTEROIDS (C) | | | | | | | | | | |
| 97 | Tab. Prednisolone 5mg | C1 | Nd | Nd | Nd | nd | 1.1 | 39.9 | 6.88 | 3.95 | Nd |
| 98 | Tab. Dexamethazone 1mg | C2 | Nd | Nd | 6.0 | Nd | Nd | 56.3 | 7.11 | Nd | Nd |
| 99 | Tab. Bethametrazone 5mg | C3 | 1.2 | 0.91 | 5.2 | 3.1 | 7.1 | 43.89 | 6.83 | 5.82 | Nd |
| | | Mean | 0.4 | 0.3 | 3.73 | 1.03 | 2.73 | 46.64 | 6.94 | 3.26 | 0.0 |
| | | S.D | 0.69 | 0.52 | 3.26 | 1.79 | 3.82 | 8.46 | 0.15 | 2.97 | 0.0 |
| | CONTRACEPTIVES (CT) | | | | | | | | | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 100 | Tab. Ethinyestradiol 0.03/ Norgesterone 0.3mg Fenocs Fumerate 75mg | | 2.7 | Nd | 2.9 | 1.8 | 9.1 | 141.2 | 2.83 | 4.11 | 3.22 |

## Discussion

### Lead

From the study, 61% of all the samples studied shows significant contamination by lead. The level of lead contaminant ranges from 10.15mg/L among anthelmintic to a mean *value* pf 0.24mg/L in haematinics. The value is higher than the level of 0.01 mg/L to 1.08 mg/L obtained by Orisakwe and Nduka (2009) in some paediatrics syrups commonly, used in Nigeria. This may be due to the use of much lower quantity of pharmaceutical samples for analysis, in some cases, less than 500ng as against 2g used in the study. Moreover, tablets and capsules have higher contact with stainless steel materials in the course of their manufacture than the syrups. However, thirty-nine percent (39%) of the samples were below detectable limits (BDL). The contamination levels in 61% are significantly above the National Agency for Food and Drug Administration and Control (NAFDAC) tolerable limit of 0.001 mg/dL. Consequently, these observed values may increase the blood levels of patients with continuous use. Undoubtedly one of the o1dest occupational toxins as evidence of lead poisoning can be found dating back to Roman times. Some pharmaceuticals such as antidiabetics and some analgelsics used by arthritic patients are taken for life.

High levels of lead in the blood may lead to deleterious health consequences like autism; miscarriages and low birth weight of infants (Neda *et al,* 2017). Lead also accelerates ageing process, mental decline, infertility in couples, as it is associated with reduction in sperm count and motility. Other effects are anaemia and hypertension. (Kadzung, 2010).

### Cadmium

Cadmium contamination level observed in this study ranges from BDL in commonly used oral contraceptive taken daily to a mean value of 2.63 mg/L in anthistamines used for cold and skin allergic reactions. The level is similar to the highest level of 2.45 mg/L obtained by Orisakwe and Nduka (2009). However only a 46% of the samples shows Cd concentrations above the detectable limit. The level of cadmium contamination of pharmaceutical in this study is above the FAO/WHO tolerable daily intake of cadmium of 70 µg to 97 µg for an average body weight of 70kg man (WHO, 2004). Cadmium is absorbed through the - intestinal tract. Cadmium accumulation in the body may lead to clinical manifestation of kidney disorders like proximal tubular dysfunction (Hayes, 1997): Other target organs include bones (Osteomalacia) liver, lung, testis (oligospermea) and haemopoietic systems (anaemia) (Kokak and Akcil, 2006).

### Chromium

Chromium is considered to be one of the most environmental toxic pollutant in the world (Oliveir, 2012). Pharmaceutical contamination of chromium in this study ranges from a mean value of 5.10 mg/L in antihististamine to BDL among antiulcers. 68% of the samples

shows detectable limits with the highest value of 29.9 mg/L in nifedipine, a commonly prescribed antihypertensive and 24.4 mg/L in ibuprofen/paracetamol/coffeine combination an over — the — counter (OTC) medicine used routinely as an analgesics. Persistence exposure to chromium may result in skin rashes, stomach upset, respiratory disorders, weak immune system, kidney and liver damage, alteration of genetic material, lung and liver cancer and ultimately death. Tolerable limit for chromium is 0.005 mg/L (WHO, 2003).

**Nickel**

Nickel is considered a normal constituents of the diet and the dietary intake of nickel in most countries ranges from 100 -300 gig/day (Cempel and Nikel, 2006). In the pharmaceutical samples considered in this study, nickel level have been found to range from a mean value of 14.1 mg/L among anthelmintics to 1.03 mg/L in corticosteroids. Therefore, there is detectable level of nickel in all the classes of samples investigated. This may be expected as water generally contains nickel of at a concentration of about 100 mg/L (Nielsen *et al.*, 1999). Although, nickel has a bioavailability of 1 – 10% when ingested, high level of nickel is known to cause allergic dermatitis known as nickel itch (Yoshihisa and Shimizu, 2012). Other toxicological effects of nickel exposure include kidney damage, cardiovascular and immune systems and blood disorders (Cornell and Landis, 1984). These effects may occur when nickel is taken above the dietary limit' of 0.187—302 mg/day.

**Manganese**

Manganese as its sulphate is commonly used in pharmaceuticals as food supplements for bone development and is a constituent of enzymes (Siddiqui et al., 2014). This study shows high level of manganese with 96% of the sample having various level of manganese contamination. The highest mean value of 27.65 mg/L, was observed in antidiarrheoa. The least mean value of 2.73 mg/L was recorded among corticosteroids. Despite its human requirement however, high level of manganese may result in health problems. Mild manganese toxicity may lead to manganese psychosis with symptoms of asthenia, anolexia, insomnia, muscular pains, mental excitement hallucinations, unaccountable laughter impaired memory and compulsive actions. Moderate toxicity include speech disorder clumsy movement, abnormal gait, poor balance, hyperplexia in a the lower limbs, and fine tremor severe signs include rigidity, spasmodic laughter and Parkinson type syndrome (Driver et al., 2009; DeMaagd and Philip, 2015). With a daily requirement of 2.5 to 7.0 mg, the level of manganese contamination in this study is above the tolerable limit.

**Iron**

Iron has the highest level of contamination of all the metals considered in the study. The highest mean values of 242.58 mg/L was seen among the corticosteroids. All 100 % of the samples studied shows high level of iron contamination. This may be the result of wear and tear of the manufacturing equipment which are stainless steel composed predominantly of iron. The food and nutrition board (FNB) of the institute of medicine (IM) USA recommended' dietary allowance of 7-10 mg/day for children, 8 mg/day for adult and 27 mg/day during pregnancy for mothers (Cetin *et al.*, 2011). Iron is used for the treatment of iron deficiency anaemia and is stored in the spleen and bone marrow. Injection of large quality of iron salt may lead to several necrolizing gastritis with

vomiting and haemorrage (Baranwal and Singhi, 2003). Chronic iron toxicity also known as haemochromatoses results when excess iron is deposited in the heart, liver, pancreas and other organs, which may lead to organ failure and - death. The level of iron contamination among pharmaceuticals in this study' may be deleterious to health.

**Zinc**

Varying level of zinc was found in 98% of the samples studied. The highest mean concentration of 22.57 mg/L was found among anticulvusants with the least mean level of 1.28 mg/L among antituberculosis. A trace level of Zn is an essential element in man. The catalytic activities of about one hundred (100) enzymes are zinc dependent in the human body and also participate in cell signaling, release of hormones and apoptosis (Thomas, 2014). The recommended dietary allowances are 4 – 5 mg/day, 9 – 13 mg/ day and 13 – 19 mg/day for children, women and men respectively (Dufner-Beattie *et al.,* 2006). Although human body can tolerate high level of zinc, acute zinc toxicity (oral dose of 225 – 450 mg) can causes health problems like stomach cramps, skin irritation, vomiting, nausea and aneamia. Chronic zinc expose may lead to copper deficiency in man and very high level of zinc can damage the pancreas, distort protein metabolizing enzymes and artheroscierosis. It can be a danger to unborn foetus and breastfeeding mothers as the foetus and children can get exposed through blood and breast milk respectively. The National Research Council (NRC) recommended daily zinc intake between 10 to 20 mg /day (NRC, 1989). It is therefore unlikely that the use of pharmaceuticals considered in this study will result in zinc toxicity.

**Cobalt**

Among the metals investigated, cobalt have the least level of contamination of only 46%. Cobalt have BDL among antiulcers and corticosteroids and the highest mean value of 4.34 mg/L among antihistamines. At low concentration, cobalt plays a prominent role in the formation of cyanocobalamine viz B12. Exposure to high concentration of cobalt may lead to symptoms of cobalt poison characterized by visual impairment, hypothyrodism, peripheral neuropathy, rashes, cardiomyopathy, auditory and cognitive impairment (Leyssens *et al.*, 2017). Since *50%* of ingested cobalt is absolved through the intestine (Angelescu and Cristiana, 2003), long term use of pharmaceutical contaminated by cobalt may be hazardous However, cobalt contamination level among pharmaceuticals considered in this study is relatively safe.

**Copper**

Copper is present in 97% of the samples studied with the lowest mean value of 0.76 mg/L in antidiarrhoea. Antituberculosis has the highest level of contamination with a mean value of 13.74 mg/L. The blood level of copper is about 100 µg/100 ml in adult (Cesar and Fraga, 2005). In humans, copper is necessary for the development of connective tissue, nerve endings and bone. It also participates in iron metabolism. There is about 80 mg of copper in the adult body with the highest concentration in the head and brain. Medium intake of Cu ranges between 1.0 to 1.6 mg/day (Harris, 1997). Chronic copper toxicity is rare in humans and is mostly associated with liver damage. Acute Cu intoxication leads to gastrointestinal effect such as abdominal pain, nausea, diarrhea, vomiting. From the study, Cu contamination level is relatively safe as the risk of acute or chronic, toxicity is most improbable.

**Conclusion**

The study of one hundred (100) pharmaceutical samples in sixteen pharmacological and therapeutic classes commonly used in Nigeria shows a significant contamination by heavy metals with the exception of copper and cobalt. Iron has the highest contamination level of 100 % with cobalt of 46% as the least. Therefore, repeated use can lead to adverse health effects ranging from minor stomach upset to metal poisoning with lead and chromium being the most dangerous. In addition, untoward drug reactions and drug hypersensitivity reactions may occur. To protect consumers and patients from metal contamination that can undermine the safety and quality of pharmaceutical, drug manufacturing companies must follow strictly Good Manufacturing Practice (GMP) by abiding by the strict quality control (QC) standards required by the National Agency for Food and Drug Administration and Control (NAFDAC).

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