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HEAVY METAL CONCENTRATIONS AND ASSOCIATED RISK BY SOME PHAEMACEUTICAL PRODUCTS AROUND SAMARU-ZARIA NIGERIA BASED ON XRF AND ASS METHODS.

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Abstract

In this study, we evaluate the level of heavy metals presence by some pharmaceutical products in Samau-Zaria - Nigeria. Samples were collected and suggested to Energy dispersive X-ray fluorescent spectrometry (EDXRF) where eighteen (18) elements were detected and Varian AA240 Atomic absorption spectrophotometer (AAS) test methods which indicate that the test average of Pb, Cd, Cr, Ni and Fe are 0.02916 mg/kg, 1.283933 mg/kg, 0.00942 mg/kg, 0.422673 mg/kg and 0.31278 mg/kg respectively. Cr had the lowest concentration, whereas Cd had the greatest. Suspension medicines have higher Fe levels than tablets. Cd was ranked higher than Cr, Pb, Ni, and Fe in terms of chronic daily intake (CDI). Lead varied from 1.174 mg/kg to 9.687 mg/kg (average: 4.822 mg/kg), Cd from 7.21 mg/kg to 7.94 mg/kg (average: 7.738 mg/kg), Cr from 1.03 mg/kg to 8.45 mg/kg (average: 4.29 mg/kg), Ni from 1.06 mg/kg to 9.09 mg/kg (average: 3.158 mg/kg), and Fe from 1.07 mg/kg to 8.59 mg/kg (average: 2.352 mg/kg). Pb > Cr > Ni > Fe > Cd was the order of the total hazard quotient (THQ). The following ranges applied to Pb, Cd, Ni, and Fe: Pb increased from 2.029 mg/kg to 9.661 mg/kg (average: 5.104 mg/kg), Cd decreased from 0.001441 mg/kg to 0.001587 mg/kg (average: 0.0015 mg/kg), Cr decreased from 1.174 mg/kg to 8.454 mg/kg (average: 3.699 mg/kg), Ni increased from 1.10577 mg/kg to 6.9187 mg/kg (average: 2.922 mg/kg), and Fe decreased from 1.227 mg/kg to 4.511 mg/kg (2.523 mg/kg). Pb, Cr, and Fe levels did not meet WHO/FAO guidelines it is therefore recommended that precautionary action should be applied.

Keywords: Heavy-metals, health-hazards, Samaru-Zaria, EDXRF and AAS

1. INTRODUCTION

Heavy metals in some pharmaceutical products affect human lives is so many ways. For instance, result in to soil pollution, affect air quality (if locally manufactured) and destroy body cell and tissues etc. This is due to the inherent

toxicity of certain contaminants (Jaishankar et al., 2014, Nagajyoti et al., 2010). Heavy metals most often considered toxic to humans are Lead, Mercury, Arsenic, Cadmium, Tin, Cobalt, Nickel and so on. Some heavy metals such as Zinc, Copper, Chromium, Iron and Manganese are required in small amounts but these same elements can be toxic in higher quantities (Lewen et al., 2004). Heavy metals are also referred to as metals having atomic weight greater than Sodium, and possess some level of toxicity (Adepoju & Adekoya, 2014). These metals could be toxic and essential and may cause damage to vital organs of the body like heart, liver, kidney and brain at high concentration (Chervona et al., 2012). Heavy metals such as Cr, Mn, Co, Cu, Fe and Zn play important biochemical roles in the life processes of many organisms, and their presence in trace amounts are essential (Akoto et al., 2014).

Heavy Metals constitute an important class of toxic substances which are ingested into the human body in numerous ways (Duffus, 2002). In control amount, they constitute an integral part of the herbal, traditional and orthodox medicine, however toxic in large quantity. The impact of these toxic agents on human health is currently an area of intense interest due to the ubiquity of exposure. high concentrations Fe and Mn can cause pathological events such as the iron oxides deposition in Parkinson's disease. Excess Cu had been associated with liver damage and Zn may produce adverse nutrient interactions with Cu. Also, Zn reduces immune function and the levels of high-density lipoproteins. Ni is highly toxic at high concentration. It can cause gastrointestinal distress, increase red blood cells and reduce lung functions. Nduka et al., (2020), conduct an assessment on risk of heavy metals from consumption of locally manufactured pain-killer drugs in Nigeria. Thirty different locally manufactured painkiller drugs were randomly sampled from pharmaceutical shops within Awka in October 2016. The drugs were pulverized, sieved and ashed before digestion using conc aqua regia HCl: HNO3 (3:1), carcinogenic heavy metals (arsenic, cadmium, chromium, mercury, nicked and lead) were analyzed using Varian AA240 atomic absorption spectrophotometer (AAS). Risk assessment was carried out using US EPA model.

2. MATERIALS AND METHODS

2.1 Materials

The materials employed to carry out this research work include: Energy dispersive X-ray fluorescent spectrometer (EDXRF), Atomic absorption spectrometry AAS Machine (AA-6800-Shimadzu Japan), Whatman Filter Paper, Agate Mortar and Pestle, Hand gloves, Distilled Water**,** Tissue Paper**,** Towels**,** White Plane Sheets**,** Volumetric Flask, Masking Tape and Mesh.

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2.2 The Study Area

Samaru is a town and ward in Sabon Gari Local Government of Kaduna state, Nigeria. The town is a semi-urban area in which Ahmadu Bello University main campus is located. Samaru is one of the most popular towns in Zaria with different ethnic groups living together in peace and harmony. Samaru is home for all tribes (Aboh *et al.*, 2015). Samaru is located on latitude 110' 25'N and Longitude 40' 26'E with two basic seasons, which are dry season and the rainy season. The seasons of Samaru enable farmers to produce good farm products at the end of every season. Figure 1, presents the digital map of the study area.

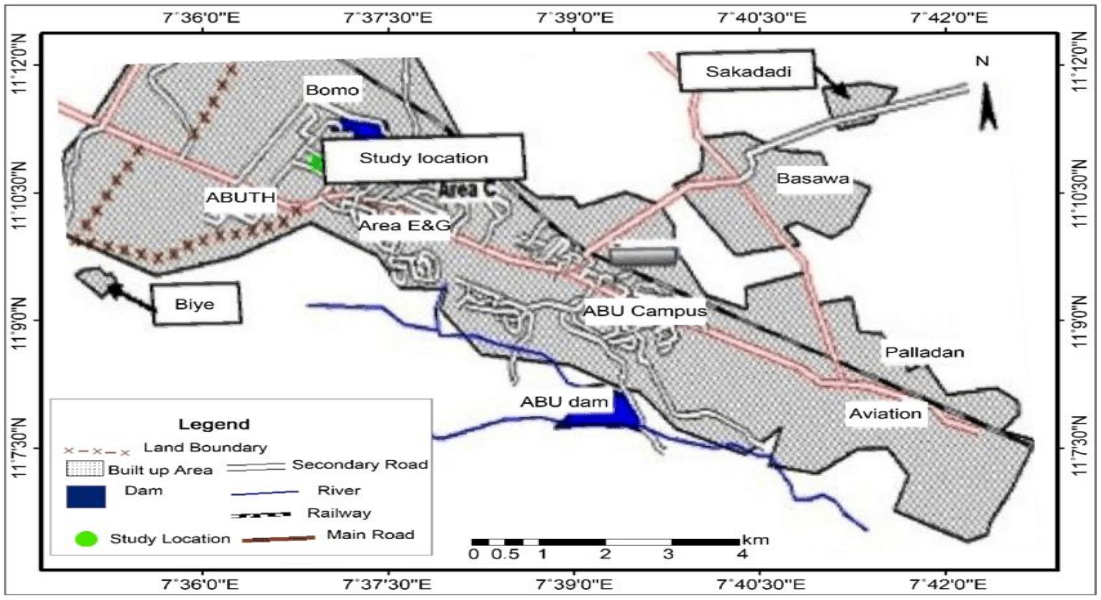

Figure 1: Map of the study area (Chidowe *et al.*, 2019)

2.3 Method of sampling

A. Sample Collection

Fifteen brands of pharmaceutical dosage forms that include 10 tablets, 2 syrups and 3 suspensions were purchased from different pharmaceutical stores in Zaria. Amongst the samples, 3 products were manufactured locally in Nigeria and 12 products were imported from Saudi Arabia, Greece, Egypt, Malaysia, Germany, USA and India. All the samples collected were coded for easy identification and then placed in plastic containers. The samples were then taken to the laboratory for preparation and analysis.

B. Sample Preparation

Sample preparation is recognized as the major source of errors and if not done properly, may affect the final results. Therefore, close attention was paid to every sample to avoid cross-contamination. This section presented the procedure that

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was followed in preparing the samples for heavy metals analyses using Flame AAS and XRF techniques. All the collected drug samples were prepared by dry ashing procedure followed by acid digestion method (Soylak *et al.*, 2004). The mean product weight of 5 to 10 sample units (Tablets) or 10 ml solutions (liquid) for each sample was determined. The weighed product unit was grounded into a fine powder using mortar and pestle before wet digestion. 2.0g powder or 15ml liquid of each sample was weighed using an electronic weighing balance and put in an empty clean beaker. 10ml of Nitric acid (HNO_3), 2ml of 60% Per chloric acid ($HClO_4$) and 5ml of concentrated Sulphuric acid (H_2SO_4) were added to each sample in the beaker and mixed up with the sample using a glass rod. The sample was then heated on a hot plate for about 1hour and allowed to be digested up to dryness at 100^{o}C, allowed to cool at room temperature and filtered in to a standard 60mL sample bottle and were made up to the mark with distilled water. The filtrate was then kept ready for analysis.

C. Sample Analysis

The heavy metals analysis was carried out using atomic absorption spectrophotometer with model (AA-6800 Shimadzu Japan) situated at National Research Institute for Chemical Technology located at km 4, Old Kano Road Basawa, Zaria, Kaduna state. Different concentrations of standard solutions were run on the instrument to obtain the calibration curves for each metal using measured absorbance and the corresponding concentration. The instrument was set to zero by reading a reagent blank. Each of the prepared samples were aspirated in to the instrument and read at least three (3) times. The average value of the concentration was taken for each metal in each sample. The results of the analyses were validated by digesting and analyzing standard reference materials (Lichens coded IAEA-336) following the same procedure. Distilled and Deionized water were used throughout the experiment and all reagents were of analytical grades. The thermos-fisher EDXRF Instrument situated at the central laboratory of Umaru Musa University, Katsina, is a compact energy dispersive x-ray spectrometer designed for the elemental analysis of a wide range of sample. The system is controlled by a PC running the dedicated analytical software.

3. RESULTS AND DISCUSSIONS

From the EDXRF results, eighteen elements were found, which are: Fe, Cu, N, Zn, Al, Mg, S, P, Ca, K, Mn, Rb, Sr, Br, Cl, V, Cr and Na respectively. Tables 1 and 2, present the WHO/FAO Permissible limit and the heavy metal concentrations.

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Table 1: WHO/FAO Permissible limit of the analyzed Heavy metals

S/N	Heavy metal	WHO/FAO Permissible limit (mg/kg)
1	Lead (Pb)	0.3
2	Cadmium (Cd)	0.2
3	Chromium (Cr)	0.1
4	Nickel (Ni)	0.1
5	Iron (Fe)	0.3

Table 2: Heavy metal concentrations of the studied drug samples

S/N	Product Code	NAFDAC Number	Batch Number	Concentration (Mg/Kg)				
				Pb	Cd	Cr	Ni	Fe
1	A1	04-2303	170	0.0146	1.3031	0.0068	0.8897	0.5379
2	A2	04-0266	L141a	0.0215	1.2811	0.0001	0.3859	0.5014
3	B1	A4-3483	Aos186	0.0576	1.3045	0.004	0.7071	0.4114
4	B2	04-2580	811077	0.0135	1.2742	0.0021	0.1802	0.4000
5	B3	04-2744	90409	0.0165	1.2893	0.0068	1.1138	0.4057
6	C1	04-4054	So939sz	0.0002	1.2591	0.0067	0.2357	0.2884
7	C2	04-0633	A2o1869	0.0278	1.2275	0.012	0.4645	0.2884
8	C3	A11-0015	4537z	0.0528	1.2282	0.001	0.6239	0.2427
9	C4	B4-7698	201128	0.0207	1.279	0.0129	0.3767	0.267
10	C5	A4-2069	04/01	0.0326	1.2976	0.0001	0.0208	0.1463
11	C6	04-0694	Aa42559	0.0449	1.3106	0.0144	0.6678	0.2404
12	C7	04-3221	E911	0.0212	1.3099	0.0175	0.1548	0.2568
13	C8	B4-7823	Gt20305	0.0561	1.3519	0.0313	0.0069	0.3194
14	C9	04-1984	A529514	0.0121	1.2646	0.0072	0.5084	0.2044
15	C10	04-0716	0020617	0.0453	1.2784	0.0184	0.0039	0.1815
Minimum	--	--	--	0.0002	1.2275	0.0001	0.0039	0.1463
Maximum	--	--	--	0.0576	1.3519	0.0313	1.1138	0.5379
Average	--	--	--	0.02916	1.283933	0.00942	0.422673	0.31278

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A. Discussion of Heavy Metal Concentration

Table 1 shows the concentration of the heavy metals investigated in the different types of pharmaceutical drug samples collected from patent medical stores in Samaru-Zaria area of Kaduna state, Nigeria. A total of fifteen (15) pharmaceutical drug samples were collected and analyzed. Analyzed values of Pb were found to range from 0.0002mgkg^{-1} to 0.0576 mgkg^{-1} with an average value of 0.02916 mgkg^{-1}. Values for Cd ranged from 1.2275 mgkg^{-1} to 1.3519 mgkg^{-1} with an average value of 1.283933 mgkg^{-1}. Values of Chromium ranged from 0.0001 mgkg^{-1} to 0.0313 with an average value of 0.00942 mgkg^{-1}. Results of Ni showed that, it ranged from 0.0039 mgkg^{-1} to 1.1138 mgkg^{-1} with an average value of 0.422673 mgkg^{-1}. And lastly, the results of Fe ranged from 0.1463 mgkg^{-1} to 0.5379 mgkg^{-1} with an average value of 0.31278 mgkg^{-1}. The heavy metal concentrations in the studied samples follows the order: Cd > Ni > Fe > Pb > Cr. The results indicated that Cd had the highest concentration in all the samples, while Cr recorded the lowest concentration. The levels of Fe were observed to be higher in suspension drugs and lowest in Tablet drugs.

B. Statistical Analysis

Statistical Bar charts were adopted to demonstrate a pictorial relationship between the calculated heavy metal concentration of different elements and permissible limits set by WHO and FAO as presented in Figures 2-6.

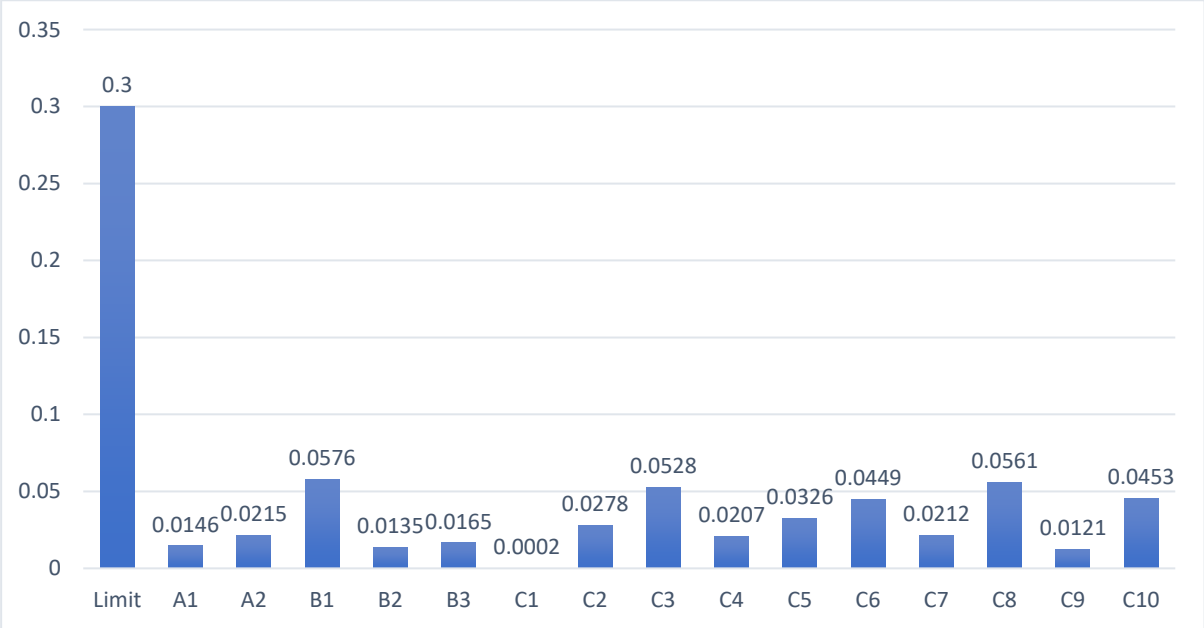

Figure 2: Chart for heavy metal concentration of Lead (Pb) from the collected 15 samples

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Figure 2: shows the chart for heavy metal concentration of Lead (Pb) from the collected 15 samples, on the vertical axis of the chart, a scale of 0.05 to represent 1unit while on the horizontal axis of the charts, all the samples were shown with their respective values. The permissible limit of Lead (Pb) concentration set by WHO and FAO is 0.3mg/Kg as shown in table 1. it can be seen from Figure 2, none of the calculated results is above the permissible limit. So, it is safe to say that none of the analyzed drug samples has Lead content higher than the permissible limit.

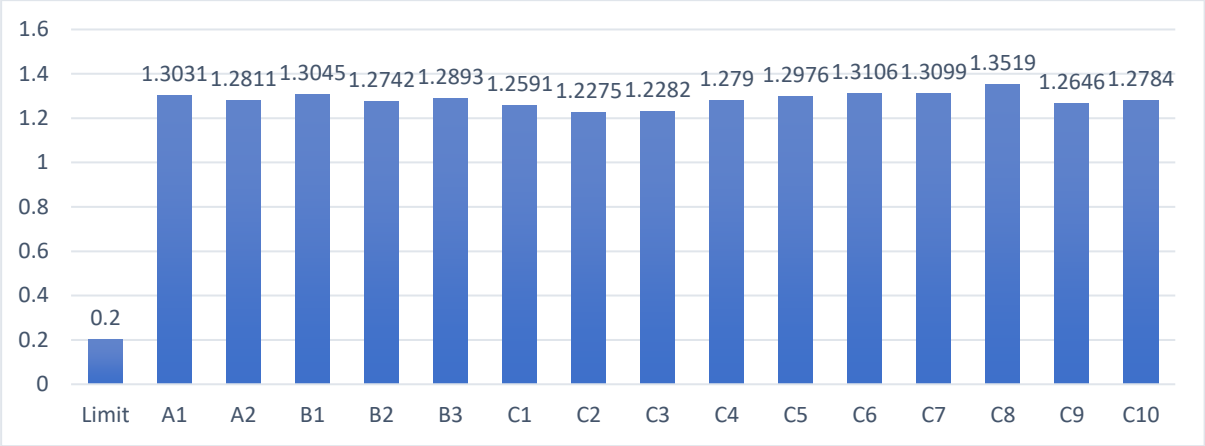

Figure 3: Chart for heavy metal concentration of Cadmium (Cd) from the collected 15 samples

Figure 3: Chart for heavy metal concentration of Cadmium (Cd) from the collected 15 samples. On the vertical axis of the chart, a scale of 0.2 to represent 1unit while on the horizontal axis of the charts, all the samples were shown with their respective values. The permissible limit of Cadmium (Cd) concentration set by WHO and FAO is 0.2mg/Kg as shown in table 1. it can be seen from Figure 3; all of the calculated results are above the permissible limit. So, it is safe to say that all of the analyzed drug samples have Cadmium content higher than the permissible limit. In this case, recommendation will be provided in the last chapter of this research.

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Figure 4: Chart for heavy metal concentration of Chromium (Cr) from the collected 15 samples

Figure 4: Chart for heavy metal concentration of Chromium (Cr) from the collected 15 samples. On the vertical axis of the chart, a scale of 0.02 to represent 1unit while on the horizontal axis of the charts, all the samples were shown with their respective values. The permissible limit of Chromium (Cr) concentration set by WHO and FAO is 0.1mg/Kg as shown in table 1. it can be seen from Figure 4; none of the calculated results are above the permissible limit. So, it is safe to say that none of the analyzed drug samples have Chromium content higher than the permissible limit.

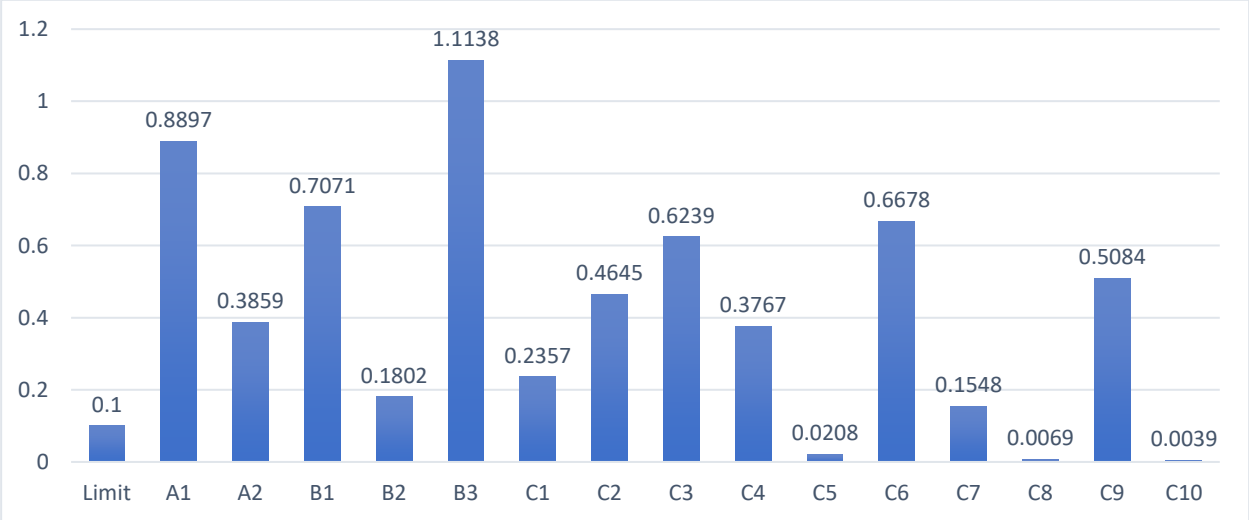

Figure 5: Chart for heavy metal concentration of Nickel (Ni) from the collected 15 samples

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Figure 5: Chart for heavy metal concentration of Nickel (Ni) from the collected 15 samples. On the vertical axis of the chart, a scale of 0.2 to represent 1unit while on the horizontal axis of the charts, all the samples were shown with their respective values. The permissible limit of Nickel (Ni) concentration set by WHO and FAO is 0.1mg/Kg as shown in table 1. it can be seen from Figure 5; Twelve out of Fifteen of the calculated results are above the permissible limit, only Three of the analyzed samples have results lower than the permissible limit. It was noted that, one of the samples (B3) has a value which is more than Ten times the permissible value (more than 1100%) which poses a serious concern.

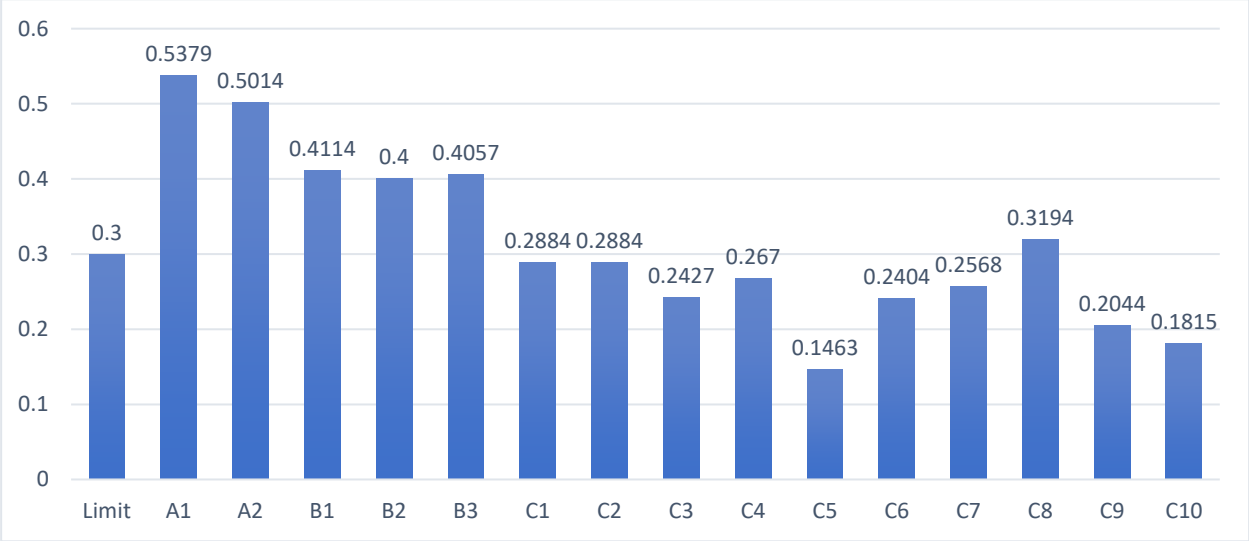

Figure 6: Chart for heavy metal concentration of Iron (Fe) from the collected 15 samples

Figure 6: Chart for heavy metal concentration of Iron (Fe) from the collected 15 samples. On the vertical axis of the chart, a scale of 0.1 to represent 1unit while on the horizontal axis of the charts, all the samples were shown with their respective values. The permissible limit of Iron (Fe) concentration set by WHO and FAO is 0.3mg/Kg as shown in table 1. It can be seen from Figure 6; Six out of Fifteen of the calculated results are above the permissible limit, while Nine of the analyzed samples have results lower than the permissible limit.

C. Chronic Daily Intake (CDI).

The calculated results for chronic daily intake investigated in the different types of drug samples collected from patent medical stores in Samaru are presented in Table 3.

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Table 3: Chronic Daily Intake (CDI)

S/N	PRODUCT CODE	CDI (mg/kg)				
		Pb	Cd	Cr	Ni	Fe
1	A1	8.57143E-09	7.65E-07	3.99E-09	5.22E-07	3.16E-07
2	A2	1.26223E-08	7.52E-07	5.87E-11	2.27E-07	2.94E-07
3	B1	3.3816E-08	7.66E-07	2.35E-09	4.15E-07	2.42E-07
4	B2	7.92564E-09	7.48E-07	1.23E-09	1.06E-07	2.35E-07
5	B3	9.68689E-09	7.57E-07	3.99E-09	6.54E-07	2.38E-07
6	C1	1.17417E-10	7.39E-07	3.93E-09	1.38E-07	1.69E-07
7	C2	1.63209E-08	7.21E-07	7.05E-09	2.73E-07	1.69E-07
8	C3	3.0998E-08	7.21E-07	5.87E-10	3.66E-07	1.42E-07
9	C4	1.21526E-08	7.51E-07	7.57E-09	2.21E-07	1.57E-07
10	C5	1.91389E-08	7.62E-07	5.87E-11	1.22E-08	8.59E-08
11	C6	2.63601E-08	7.69E-07	8.45E-09	3.92E-07	1.41E-07
12	C7	1.24462E-08	7.69E-07	1.03E-08	9.09E-08	1.51E-07
13	C8	3.29354E-08	7.94E-07	1.84E-08	4.05E-09	1.88E-07
`14	C9	7.10372E-09	7.42E-07	4.23E-09	2.98E-07	1.2E-07
15	C10	2.65949E-08	7.51E-07	1.08E-08	2.29E-09	1.07E-07
Minimum	--	1.17417	7.21	1.03	1.06	1.07
Maximum	--	9.68689	7.94	8.45	9.09	8.59
Range	--	8.51272	0.73	7.42	7.83	7.52
Average	--	4.821917778	7.538	4.29	3.518	2.352

D. Discussion of Chronic Daily Intake (CDI)

Table 4.4 shows the chronic daily intake (CDI) measured in (mg/kg) x $10^{\pm 9}$ investigated in the different types of pharmaceutical drug samples collected from patent medical stores in Samaru-Zaria area of Kaduna state, Nigeria. A total of fifteen (15) pharmaceutical drug samples were collected and analyzed. The average chronic daily intake in the studied samples follows the order: Cd > Cr > Pb > Ni > Fe. The lowest value of Lead was found to be 1.174 and the highest value was 9.687 with an average value of 4.822. Cadmium's highest value was 7.94 while the lowest value was found to be 7.21 with an average value of 7.738. The values of Chromium ranged from 1.03 to 8.45 with an average value of 4.29. Nickel's calculated lowest value was 1.06 and the highest value was 9.09 with an average

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value of 3.158. Iron's lowest value was 1.07 whose highest value was 8.59 with an average value of 2.352.

E. Total Hazard Quotient (THQ)

The Calculated results for Total Hazard intake investigated in the different types of drug samples collected from patent medical stores in Samaru are presented in Table 4.

Table 4: Total Hazard Quotient

S/N	PRODUCT CODE	Pb	Cd	Cr	Ni	Fe
1	A1	2.44898E-06	0.00153	7.98434E-07	2.61164E-05	4.51132E-05
2	A2	3.60637E-06	0.001504	1.17417E-08	1.13278E-05	4.2052E-05
3	B1	9.66173E-06	0.001532	4.69667E-07	2.07564E-05	3.45038E-05
4	B2	2.26447E-06	0.001496	2.46575E-07	5.28963E-06	3.35477E-05
5	B3	2.76768E-06	0.001514	7.98434E-07	3.26947E-05	3.40257E-05
6	C1	3.35477E-08	0.001478	7.86693E-07	6.91879E-06	2.41879E-05
7	C2	4.66313E-06	0.001441	1.409E-06	1.3635E-05	2.41879E-05
8	C3	8.85658E-06	0.001442	1.17417E-07	1.83141E-05	2.0355E-05
9	C4	3.47218E-06	0.001502	1.51468E-06	1.10577E-05	2.23931E-05
10	C5	5.46827E-06	0.001524	1.17417E-08	6.10568E-07	1.22701E-05
11	C6	7.53145E-06	0.001539	1.6908E-06	1.96027E-05	2.01621E-05
12	C7	3.55605E-06	0.001538	2.05479E-06	4.54403E-06	2.15376E-05
13	C8	9.41012E-06	0.001587	3.67515E-06	2.02544E-07	2.67878E-05
`14	C9	2.02963E-06	0001485	8.45401E-07	1.49237E-05	1.71429E-05
15	C10	7.59855E-06	0001501	2.16047E-06	1.14481E-07	1.52223E-05
Minimum	--	2.02963	0.001441	1.17417	1.10577	1.22701
Maximum	--	9.66173	0.001587	8.45401	6.91879	4.51132
Average	--	5.10382	0.001507533	3.698629333	2.922211429	2.623260667

ISSN: 3027-2335

F. Discussion of Total Hazard Quotient (THQ)

Table 4 shows the calculated results of Total Hazard Quotient (THQ) investigated in the different types of pharmaceutical drug samples collected from patent medical stores in Samaru-Zaria area of Kaduna state, Nigeria. A total of fifteen (15) pharmaceutical drug samples were collected and analyzed. The average total hazard quotient in the studied samples follows the order: Pb > Cr > Ni > Fe > Cd. The values for Pb ranged from 2.029 to 9.661 with an average value of 5.104. Cd has the lowest of 0.001441 and highest of 0.001587 with an average value of 0.0015. values for Cr ranged from 1.174 to 8.454 whose average value of 3.699. Ni values fall with 1.10577 and 6.9187 whose average value was 2.922. Values for Fe ranged from 1.227 to 4.511 with an average value of 2.623.

4. CONCLUSION

In conclusion, the concentrations of heavy metals such as; Mercury, Lead, Iron, Cadmium and Nickel that may be present in the sampled drugs using the Atomic Absorption Spectrometry (AAS) technique. Analyzed values of Pb were found to range from 0.0002mgkg^{-1} to 0.0576 mgkg^{-1} with an average value of 0.02916 mgkg^{-1}. Values for Cd ranged from 1.2275 mgkg^{-1} to 1.3519 mgkg^{-1} with an average value of 1.283933 mgkg^{-1}. Values of Chromium ranged from 0.0001 mgkg^{-1} to 0.0313 with an average value of 0.00942 mgkg^{-1}. Results of Ni showed that, it ranged from 0.0039 mgkg^{-1} to 1.1138 mgkg^{-1} with an average value of 0.422673 mgkg^{-1}. And lastly, the results of Fe ranged from 0.1463 mgkg^{-1} to 0.5379 mgkg^{-1} with an average value of 0.31278 mgkg^{-1} as shown in table 1. The heavy metal concentrations in the studied samples follows the order: Cd > Ni > Fe > Pb > Cr. The chronic daily intake (CDI) was measured in (mg/kg) x $10^{\pm 9}$. The average chronic daily intake in the studied samples follows the order: Cd > Cr > Pb > Ni > Fe. The calculated results of Total Hazard Quotient (THQ) were investigated. The average total hazard quotient in the studied samples follows the order: Pb > Cr > Ni > Fe > Cd. Statistical parameters such as minimum, maximum, range and average were calculated. Also, statistical chart (bar chart) was employed to make comparison of the findings with WHO/FAO permissible limit of heavy metal concentration.

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