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PHYSICOCHEMICAL COMPOSITION AND HEAVY METAL DETERMINATION OF SELECTED INDUSTRIAL EFFLUENTS OF IBADAN CITY, NIGERIA

[1]Okoyomon, O. O., [*2]Kadir, H. A., [3]Zango, Z. U., [4]Saidu, U., & [3]Nura, S. A.

[1]Department of Chemistry, University of Ibadan, Oyo State, Nigeria
*[*2]Department of Quality Assurance & Control, Dangote Cement Plc, Kogi State, Nigeria*
[3]Department of Chemistry, Al-Qalam University Katsina, P.M.B 2137, Katsina, Nigeria
[4]Department of Chemistry, Sule Lamido University Kafin Hausa, P.M.B 048, Jigawa State, Nigeria

***Corresponding Author's E-mail:** hkadir2012@hotmail.com* ***Phone**: +2348038793929*

ABSTRACT

The rise of heavy metal presence in environmental waters has made it necessary to continuously examine industrial effluents to maintain the quality of the environment. The focus of this study is centered on determining the heavy metal concentrations and some physicochemical parameters in twelve industrial effluents samples collected from various locations across Ibadan city. A composite sampling method was utilized to obtain representative effluent samples of the 12 Industries (categorized into food, beverage, tobacco, plastic, Pharmaceutical, chemical, and allied industries) and borehole samples from around the city were used as control. The effluent samples were digested by nitric acid (HNO_3) and analyzed for cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), and lead (Pb) using the atomic absorption spectrophotometric method (AAS). Some physicochemical parameters such as pH (Jenway 3510 pH meter), total dissolved solids (Hanna TDS meter), total suspended solids, and phosphate were determined. The heavy metal mean values were compared with Federal Environment Protection Agency (FEPA) and the United States Environmental Protection Agency (USEPA) standard values shown in table 1. The mean concentrations of heavy metal in the industrial effluent samples were Cu (0.32 mg/L), Pb (0.037 mg/L), Ni (0.50 mg/L), Co (0.037 mg/L), Cd (0.016 mg/L), Fe (54.0 mg/L) and Cr (0.44 mg/L). It was found that Chemical and allied industries have the highest concentration for metals such as Fe (128 mg/L), Ni (1.1 mg/L), and Cu (0.27 mg/L) while Cr (0.0067 mg/L) and Co (0.08 mg/L) were obtained in the Food/Beverage and pharmaceutical industries respectively. Conclusively, the industries around the Ibadan city stand as potential contributors to pollution, hence a periodical and continuous assessment effort are recommended.

Keywords: *heavy metals, industrial effluents, Ibadan, pollution, spectroscopy*.

INTRODUCTION

Wastewater discharged from industries is one of the major sources of water pollution, and many industrial wastes were discharged in form of effluents into rivers, lakes, and coastal areas. As a result, it constitutes serious problems in the environment that cause negative effects to the ecosystem and human life (Awoyemi *et al.*, 2014). Organic wastes include pesticide residue, solvent and cleaning fluids, dissolved residue from fruits and vegetables, and lignin from pulp and paper processing industries. Effluents can also contain inorganic wastes such as brine salts and metals. The clean Water Act has standards for the permitted release of a limited number of contaminants into waterways. Thus, industries are required to pre-treat their water by neutralizing the chemically active components, recycling, dilution, or extraction and collection for proper disposal (Audu and Idowu, 2015).

In Nigeria, economic development is premised on industrial productions. There are four major industrial estates in Ibadan, namely, Oluyole, Olubadan, Old Lagos Road, and New Lagos scheme (Lagos-Ibadan expressway) industrial estates (Adebisi and Fayemiwo, 2011). The Oluyole industrial estate is the most developed and is situated in the western part of the city; it also has the largest concentration of industrial plants. By contrast, the other estates are occupied by only a few plants. Major industries in Ibadan include Tobacco, manufacturing industry, brewery industry, pharmaceutical, food, furniture making industry (Adebisi and Fayemiwo, 2011). Others include agricultural products and processing industries like cocoa, timber, palm oil, and cassava (Adelekan and Alawode, 2011).

Nevertheless, waste production is also a function of every industrial process which is usually associated with the discharge in form of wastewater, gases, and solid wastes. Industrial wastewater effluents are recognized as contributors to the pollutions of soil and water (Zango *et al.*, 2020). The contamination from heavy metals is a serious environmental phenomenon due to their apparent toxicities to both flora and fauna. They possessed devastating poisonous risks to humans, resulting in various toxic effects (Armaya'u et al., 2020). Lead (Pb) has been associated with kidney dysfunction, cancer, anemia, and mental retardation in children (Adebola & Babalola, 2017*; Olagunju, et al.*, 2020). Chromium (Cr) and Cadmium (Cd) have been identified as hepatotoxic, neurotoxic, renal-toxic, and carcinogenic heavy metals associated with lung, kidney, and liver cancer (Adebola and Oyeleke, 2017). Zinc (Zn) is described as a respiratory disorder, while copper (Cu) was known to inhibit thyroid and adrenal hormones production. Nickel (Ni) and iron (Fe) caused pathogenesis and oxidative stress in the body cells and caused immune disorders (Nnaneme, 2021). Hence, environmental management is needed to curtail these pollutants from the environment, hence why it is of interest to environmental scientists, public health experts, environmental policymakers, and regulatory agencies (Chukwulobe and Saeed, 2014).

Previous studies have revealed the presence of heavy metals in environmental samples across the Ibadan city and the industries are significant contributors to environmental degradation. Oyeleke *et al.*, (2016), have identified heavy metals present in soil samples around the automobile battery factory in the city. Of all, Pb concentration is higher, up to 59.13 59.13±48.9 (range 5.00 - 182.00 mg/kg), which was four times the average normal level in the soil. Other heavy metals detected at Zn, Cr, Cd, Fe and Cu with concentration range of, 2.68±1.1 (range 0.4 - 5.2 mg/kg), 1.62±2.4 (range ND - 8.7 mg/kg), 0.08±0.09 (range ND - 0.24 mg/kg), 49.44±16.5 (range 12.5 - 70 mg/kg) and 4.94±2.6 mg/kg (range 0.5 - 10.5 mg/kg), respectively. The elevation of the level of Pb in the soils has made it unfit for agricultural activities (Peter et al., 2016). The heavy metals in selected roadside fast foods across the city were researched by Olajide *et al.,* (2019). The food samples analyzed include roasted fish, yam and plantain, "suya" meat, potato, corn, bean doughnut, and cake. Pb, Cu, Zn and Fe were reported in a concentration range of 0.032±0.01 – 0.077±0.05 mg/kg and 0.107±0.01 – 0.231±0.14 mg/kg, 0.023±0.01 – 0.039±0.04 mg/kg and 0.557±0.20 – 1.808±1.52 mg/kg respectively. Hg and Cd detection is below AAS sensitivity. The contamination was resulted due to environmental pollution from the industries and emissions from the automobiles engines (Olajide and Abiodun, 2019). Omenka and Adeyi (2016), have also investigated the presence of heavy metals in selected personal care products (PCPs) produced in the Ibadan city. The Estimated daily intake (EDI) and Health Risk Index (HRI) of the heavy metals in the PCPs were also calculated to assess the human health risks associated with the use of the products. Acid digestion and Atomic Absorption Spectrophotometry (AAS) analysis have revealed the concentrations of Zn (mg/kg) in the range of 3.75 - 19.3, 1.88 - 112,000, and 19.8 - 217 in creams, powders, and eyeliners, respectively. Thus, it was concluded the use of these products for a prolonged time may resulted in environmental and health risks of the heavy metals. Thus, authorities tasked to regulate the use of these toxic products particularly those imported into Nigeria (Omenka and Adeyi, 2016).

The rapid detection of heavy metals around the city's water bodies resulted from the industrial effluents from the various factories and other domestic activities (Olagunju *et al.*, 2020). Recently Ganiyu *et al.,* (2021) studied heavy metals contaminations in drinking water samples across the wells from three residential areas in the Ibadan metropolis: The areas include the traditional core area (TCA), peri-urban area (PUA), and the urban area (UA)]. The heavy metals detected have been in the concentration of 3.930, 0.658, 0.0304, 1.698, and 0.501 mg/L of the for the Zn, Pb Mn, Fe, and Cd respectively. The amount of Zn, Fe, Pb, and Cd exceeded the standards in 60%, 86.7%, 100%, and 100% of ground-water samples, respectively. Ayoade and Nathaniel (2018), analyzed contamination of heavy metals in water and sediment in the tropical man-made lake of Dandaru reservoir located in the city. The heavy metals were reportedly present in the samples (except Ni which was not present in the sediment). The trend of the metals was Mn>Fe>Pb>Ni>Zn>Cu>Co>Cd>Cr in the water samples, Similarly, for the sediment, the order of the occurrence was Fe>Zn>Mn>Pb>Cu>Co>Cd>Cr. Pb, Cd, Ni, and Mn were higher compared to the recommendation of the National Environmental Standards and Regulations Enforcement Agency (NESREA), Nigeria, and World Health Organization (WHO) for domestic uses. Thus, discharges into the reservoir should be managed to safeguard the environment (Ayoade and Nathaniel, 2018).

Therefore, this present study is aimed at evaluating the physicochemical parameters (pH, phosphate, total suspended solids, and total dissolved solids) and heavy metal (lead, cadmium, cobalt, nickel, iron, copper, and cadmium) qualities of the industrial effluents collected from twelve industries in the city of Ibadan and to compare the results obtained with those of Federal Environmental Protection Agency (FEPA) effluent discharged standard for industrial effluents as well as checking the compliance of the industries concerning the FEPA regulations on permissible limits of effluents released.

MATERIAL AND METHODS

STUDY AREA

Samples were collected from 12 industries in Ibadan, Oyo State, Nigeria. These industries include the following: Energy foods, British American tobacco, Eagle flour mill, seven up bottling company, Zartech poultry, Yale foods limited, Vital foods, Procter and gamble West African, Black horse plastics limited, Dana Pharmaceutical, Bentos Pharmaceutical, and Balaji metal-works engineering limited. The industries are categorized (based on their products) into the following groups: food, beverage, tobacco, plastic, Pharmaceutical, chemical, and allied industries.

SAMPLING COLLECTION AND PROCESSING

Effluent samples were collected at the point of discharge by lowering the sample containers and allowing the effluents to flow in. The bottles were tightly covered after filling. In each case, the previously acid-cleaned containers were rinsed with effluent before sampling is done at each point. One liter sample container was used for sampling. Samples were collected at a week interval basis, the method utilized was "composite sampling" of the effluent channel for each industry. Samples for heavy metal determination were collected in containers and stabilized with nitric acid on site.

ANALYSIS OF SAMPLE

On-site, sample bottles filled with effluents were kept in a cooler containing ice to the laboratory. The effluent's pH was analyzed using a Jenway 3510 pH meter. It was calibrated using buffers 4 and 7 before use. Phosphate was analyzed by the colorimetric method and the concentration was measured at 880nm using a UV-Visible spectrophotometer. The total dissolved solids (TDS) in mg/L was determined using the Hanna TDS meter (APHA, AWWA, 2017). The total suspended solids (TSS) in mg/L was determined by the gravimetric method (Ademoroti, 1996). The water sample was filtered through a pre-weighed filter paper. The filter paper was then dried at 105 ^{0}C and re-weighed. The suspended solids were determined from the increase in weight of the filter paper. Wet digestion of samples was employed by the addition of 5 mL of 0.2 M HNO_3 to 50 mL of the effluent sample and an automated Q block wireless digester was utilized for 1 hour. Afterward, the determination of heavy metals (Cu, Pb, Ni, Co, Fe, Cd, and Cr) was carried out using atomic absorption spectrometry

RESULTS AND DISCUSSION

The data in Table 2 are the measured values of heavy metals concentrations of the analyzed industrial effluents. Cr was only detected at OLO5 (0.04 mg/L) and it was found to be within the acceptable range of FEPA and USEPA limits of <1 mg/L and 0.37 mg/L, respectively. The presence of Cr in the OLO5 effluent sample can be attributed to emanating from the poultry feedstock (Islam et al., 2017) Cr mean value (0.04 mg/L) was lower than the mean value previously obtained from rubber and brewery industrial effluent (0.079 mg/L) (Oguzie & Okhagbuzo, 2010). Cadmium was not found in OLO2, OLO1, TOG1, TOG2, POD1, POD2, POD3 industrial effluent samples, thou DUG-EF, OLO3, OLO5, OLO7, and ALK contained 0.01, 0.02, 0.01, 0.03, and 0.01 mg/L of cadmium respectively. These values conform with the FEPA limit of <1 mg/L, and USEPA's limit of 0.2 mg/L and there was no trace in the control sample, thereby indicating that the industries contributed to the presence of cadmium in the effluents. Oguzie and

Okhagbuzo (2010) reported cadmium value in effluents discharged which is transported to Ikpoba River in Benin City in the rainy season as 0.072 mg/L and, Siyanbola *et al.*, (2011) reported that increased cadmium in concentration inhibits bio-uptake of phosphorus and potassium by plants.

The Co concentration ranged from 0.15 mg/L at POD1 to 0.01 mg/L at TOG1 and TOG2, while there exists no trace of it at DUG and ALK. It is vital to highlight that, there is no recommended FEPA limit for Co but in comparison with USEPA standards, it was observed that POD1 values were slightly above the limit of 0.14 mg/L, while others were within range. The concentration for the control sample was lower than that of the effluent samples gotten from the industries.

Table 1: Mean Value for Heavy Metal Concentration in the Industrial Effluent Samples

Sample code	Cu	Pb	Co	Ni	Cd	Fe	Cr
DUG-EF	0.02	0.26	<0.053	0.01	0.01	0.40	<0.04
OLO1-PG	<0.025	0.06	0.01	0.02	<0.01	2.99	<0.04
OLO2-SBC	<0.025	0.03	0.02	0.04	<0.01	2.67	<0.04
OLO3-VFC	<0.025	0.02	0.03	0.01	0.02	<0.04	<0.04
OLO5-ZP	<0.025	0.07	0.03	0.01	0.01	97.54	0.04
OLO7-YF	<0.025	0.05	0.01	<0.06	0.03	79.38	<0.04
TOG1-BAT	<0.025	0.02	0.01	<0.06	<0.01	1.42	<0.04
TOG2-EFM	0.13	<0.18	0.01	0.09	<0.01	0.38	<0.04
POD1-BP	0.57	0.99	0.15	1.72	<0.01	0.44	<0.04
POD2-BMW	0.54	0.23	0.07	2.09	<0.01	254.70	<0.04
POD3-BH	<0.025	0.25	0.03	<0.06	<0.01	16.38	<0.04
ALK-DP	<0.025	<0.18	<0.053	<0.06	0.01	138.50	<0.04
CTRL DUG	<0.025	0.01	<0.053	0.01	<0.01	1.04	<0.04
CTRL OLO	<0.025	<0.18	0.02	<0.06	<0.01	2.60	<0.04
CTRL POD	<0.025	<0.18	0.01	0.02	<0.01	0.44	<0.04
CTRL ALK	<0.025	<0.18	0.01	<0.06	<0.01	3.23	<0.04
FEPA (1991)	<1	<1	ND	<1	<1	20	<1
USEPA	1.28	0.28	0.14	0.55	0.2	1.2	0.37

CTRL: Control site; FEPA-Federal Environmental Protection Agency, Nigeria; USEPA-United State Environmental Protection Agency

Industrial Codes and Meaning

DUG-EF	=	Energy food
OLO1-PG	=	Procter and Gamble West African limited
OLO2-SBC	=	Effluent from Seven-up bottling company
OLO3-VFC	=	Effluent from Vital food company
OLO5-ZP	=	Effluent from Zartech Poultry
OLO7-YF	=	Effluent from Yale foods limited
TOG1-BAT	=	Effluent from British American Tobacco
TOG2-EFM	=	Effluent from Eagle flourmill
POD1-BP	=	Effluent from Bentos Pharmaceutical
POD2-BMW	=	Effluent from Balaji metal and Engineering Limited
POD3-BH	=	Effluent from Black horse
ALK-DP	=	Effluent from Dana Pharmaceutical company

The mean value for copper was 0.57 mg/L, 0.54 mg/L, 0.13 mg/L at POD1, POD2, and TOG2-EFM respectively, while the following samples: TOG1, OLO1, OLO2, OLO3, OLO5, and OLO7 shows no trace of copper. In comparison, it is noted that they are within the standard limit as stipulated and the non-detection of Cu in other samples could be attributed to sources of Cu not being an important raw material in their production processes. Otherwise, it might have been effectively removed.

It is an established understanding that iron is a vital element present in soil and water samples, and considering the above, Fe exhibited the highest concentration when compared to other heavy metals analyzed in the effluent samples. The mean Fe concentration varied from 0.40 mg/L at DUG-EF to 254 mg/L at POD2 and it was not detected at OLO3. Relatively to standard, the values obtained for the industrial samples conform to the standard limit (FEPA) with an exception of POD2 (254 mg/L), ALK (139 mg/L), and OLO5 (97 mg/L). Considering the USEPA limit of 1.2 mg/L, it is seen that most of the industries didn't conform except for DUG (0.40 mg/L) and POD1 (0.44 mg/L). An amount of 0.26 mg/L was detected in the control sample affirms the presence of iron in the environment. Hence, this can be due to the lack of effective treatment techniques exhibited by the industries. Fe in water imparts taste and promotes the growth of iron bacteria that accelerate the rusting process of ferrous metals that are exposed to water (Hamzah, Khohr, Abdolahi, and Ibrahim, 2013).

The mean Pb levels ranged from 0.02 mg/L at TOG1 to 0.26 mg/L at DUG samples and were not detected at TOG2 and ALK. The mean concentrations for Pb were within the FEPA (< 1 mg/L), and USEPA (0.28 mg/L) permissible limit. The negative effect of a high level of Pb present in the human body is damage to the kidney, central nervous system, brain, or even death, although the direct use of domestic water from these industrial vicinities may not pose a Pb threat (Hanaa et. al., 2000). The Ni concentration varied from 0.01 mg/L at DUG to 2.09 mg/L at POD2, which is not detected in TOG1, OLO7, POD3, and ALK. The mean concentration of Ni in almost all the effluent samples was within the FEPA (< 1mg/L) and USEPA (0.55 mg/L) except for POD2 (2.09 mg/L). The Ni concentration in the effluent samples was lower than the mean reported by Oguzie and Okhaghuzo (2010) in effluent from the brewery industry (0.122 mg/L), with exception of the effluent sample from POD2 (2.09 mg/L)

The heavy metal concentrations in the effluents based on the categories of industries are shown in Table 3. The mean heavy metal concentration (mg/L) in the effluents were in this order Fe >> Ni >> Cr >> Cu >> Pb >> Co >> Cd. The chemical and allied industry recorded the highest mean concentration for Fe (128 mg/L), Ni (1.1 mg/L), Cu (0.27 mg/L) than the other categories of industries. These metals were not within the FEPA limit except Cu (see Table 2). Pharmaceutical industry has the highest Co (0.08 mg/L) and Pb (0.50 mg/L) concentration. However, these values are still within the FEPA limit (< 1 mg/L). The food and beverage industry had the highest mean concentration for Cr (0.0067 mg/L) compared to other metals and it is based on the limit by Federal Environmental Protection Agency, NIGERIA (FEPA) (Agency, 1999).

Table 2: Mean value for heavy metal concentration in the industrial effluent samples based on the category of industries

Metals	Food beverage and Tobacco	Plastic	Chemical and allied	Pharmaceutical
Copper	0.021±0.049	<0.025	0.27±0.38	0.29±0.40
Chromium	0.007±0.016	<0.04	<0.04	<0.04
Lead	0.064±0.089	0.25±0.00	0.15±0.12	0.50±0.70
Cadmium	0.010±0.012	4.5±1.1	ND	0.0050±0.0070
Iron	26±43	0.18±0.15	128±170	69±98
Nickel	0.023±0.033	0.065±0.021	1.1±1.50	0.9±1.2
Cobalt	0.016±0.012	0.05±0.00	0.04±0.04	0.08±0.10

Table 3: Mean values for physicochemical parameters of the industrial effluents

Sample code	pH	TDS	TSS	Phosphate
DUG	6.72±0.45	677±410	298±33	1.19±0.31
OLO1	7.0±3.0	864±13	550±54	4.0±2.5
OLO2	9.80±2.0	674±550	165±150	0.90±1.20
OLO3	6.00±1.2	803±370	482±87	1.00±0.23
OLO5	6.1±1.3	503±370	482±87	1.22±0.70
OLO7	7.59±0.24	1210±120	345±97	7.05±0.55
TOG1	7.4±1.3	500±9.9	228±56	1.32±0.07
TOG2	8.06±8.12	292±280	240±260	1.4±0.7
POD1	8.090±0.042	289±3.5	250±14	0.874±0.048
POD2	6.37±0.37	1970±28	3620±12.0	5.91±1.20
POD3	7.06±0.16	330±4.2	141±80	2.61±0.28
ALK	6.53±0.37	99.5±0.70	107±46	0.694±0.059
FEPA limit	6-9	2,000	400	5

The pH value is a significant determinant of the quality of water that indicates the presence of alkali or acid in the samples of the water. It also affects chemical reactions, such as metal toxicity and solubility (Yusif, 2018). The mean pH was in the range of 6.13 to 9.77. Most of the effluent samples were within the acceptable FEPA pH limit of 6 – 9, except for OLO2 (9.77 mg/L) whose pH was the highest. Most of the sample of the collected industrial effluents pH also fall in Class II, otherwise called "Acceptable Quality". The mean values for pH in the samples were mostly lower when compared with the previous report for industrial effluents on the quality of water from Challawa River in Kano state which was 8.37 (Wakawa *et al.*, 2008). For TDS was 1970 mg/L at OLO1 and its lowest value of 99.5 mg/L at ALK. The entire effluent samples were within the FEPA standard (2,000 mg/L). The value for TSS was 3620 mg/L at OLO3 and its lowest value of 107 mg/L at ALK. A high value recorded for the TSS of the effluent samples indicates a high level of turbidity. The values of phosphate content were all below the allowable limit of FEPA.

CONCLUSION

Considering the above, this finding reveals the inadequate or lack of effluent treatment by chemical and allied industries before being discharged to the environment as the concentration of heavy metal was detected above other industries when compared. A high amount of Fe was recorded for some of the samples, this reveals that there exists a high level of contamination or pollution of the environs where these industries exist. On a positive note, generally, our findings reveal that the physicochemical properties and the levels of heavy metals in the effluent samples analyzed did not constitute any severe pollution threat presently, except for Fe. Relatively, it was observed that the parameters conform to acceptable standards which in turn connotes an acceptable and conducive range to both human and aquatic bodies. Hence, it is recommendable that, an evaluation of the surrounding ground and surface water be conducted.

CONFLICT OF INTEREST DISCLOSURE

No conflict of Interest

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