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Physico-Chemical Properties and Heavy Metals Assessment of Wastewater Discharged from Selected Cosmetic Industries in Nigeria

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In this study effluents discharge from three cosmetic industries within Lagos and Ogun State of Nigeria were analyzed for heavy metal concentration using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) technique and measurement of selected physicochemical properties was also carried out. The heavy metals analyzed include: Cadmium (1.173±0.737 mg/L), Lead (1.173±0.749 mg/L), Zinc (1.154±0.691 mg/L), Nickel (1.134±0.718 mg/L), Iron (1.186±0.746 mg/L) and Mercury (0.294±0.227 mg/L). Mercury shows the lowest concentration among the determined heavy metals while Iron (Fe) has the highest concentration in the effluents. The effluents from two industries located in Lagos State (sample C and B) contain higher concentrations of all heavy metals under study compared to the effluents from the cosmetic industry located in Ogun State (sample A) with total mean concentrations of 1.72 mg/L, 0.88 mg/L and 0.46 mg/L respectively. One way ANOVA analysis of the heavy metal distribution in the effluents of the three cosmetic industries shows no statistical significant difference indicating that difference observed in the concentrations of the heavy metals is not dependent on the location but rather due to the process of production. The physicochemical properties: Electrical conductivity (EC), Biological oxygen demand (BOD), Chemical oxygen demand (COD), Total solid (TS) and Total dissolved solid (TDS) shows average values of 3034.9±14.27 µS/cm, 92.90±3.57 mg/L, 206.67±1.42 mg/L, 8791.78±23.46 mg/l and 1350.35±6.97 mg/L respectively which are above the regulatory bounds of World Health organization and Standard organization of Nigeria.

Keywords: Heavy metals, cosmetics effluent, ICP-OES, water quality, Nigeria.

1. Introduction

In present times, water which is one of man's greatest resources is facing huge pollution threats due to the continuous discharge of pharmaceuticals, textile and personal care products which are being released consciously or unconsciously into water sources due to poor regulatory frameworks especially in the developing countries such as Nigeria (Aniyikaye *et al*., 2019). This act of indiscriminate discharge of industrial wastewater has led to the continuous decline in freshwater quality, with adverse environmental and health implications (Bhat *et al.,* 2018). In addition, the effects of these contaminants are poorly known as they are scarcely biodegradable and as such have become an environmental nuisance and public health issue (Bautista *et al.,* 2010). This development has attracted attention from both governmental and nongovernmental organizations worldwide (Drozdova *et al.,* 2019).

The potential adverse effects on humans and the entire ecosystem due to the direct disposal of cosmetics effluent into the aquatic environment without proper treatment have been widely reported to lead to compromise in the quality of the receiving water bodies (Olaniyi and Nwadiogbu, 2012).

Substances found in cosmetic wastewater belong to the group called pharmaceuticals and personal care products (PPCPs). These include drug and cosmetic ingredients, dietary supplements and products of their metabolism. The main sources of PPCPs emission into the environment are through industrial and household discharge. As a result of low PPCP concentration in the water environment, questions were raised regarding their microbial resistance, chemical persistence, and the synergistic effects of various compounds (Tijani *et al.,* 2013).

The existence of high concentration toxic heavy metals in generated wastewater could be sourced from the numerous chemicals used for cosmetics formulation, especially pigments of mineral origin as well as machinery during production. Some components of these wastes contain potentially toxic chemical elements which when released into the environment may penetrate and leach into the subsurface environment and subsequently settle in the soil and sediment of water bodies (Olaoye and Oladeji, 2015).

Cosmetic wastewater has been reported to pose very high Chemical Oxygen Demand (COD) (>100000 mg/l), Biological Oxygen Demand (BOD) and Total Organic Carbon (TOC) levels, high concentrations of organic nitrogen and organic phosphorus (Bogacki *et al.,* 2011). The direct discharge of cosmetic wastewater can cause eutrophication and reduce the available dissolved oxygen level. Such adverse conditions make aquatic life difficult and tend to accelerate genotoxicity and microtoxicity in aquatic organisms (Verma *et al.,* 2012). A similar study by de Melo *et al.* (2013) showed wastewater from cosmetic company contains high concentration of COD and BOD that is above the acceptable values lay down by international regulatory body. This was revealed by conducting an aquatic toxicity bioassay for eco-toxicological characterization using *C similis*, *C. dubia*, and *P. subcapitata* as an organism at different conditions. The findings of this study also proved that the effluents can be a potential public and environmental health hazard.

Cosmetic wastewater contains various compounds, both organic and inorganic. The organic compounds includes, proteins, ethers, esters, aldehydes and ketones, alcohols, carboxylic acids, and their complex derivatives (Bogacki *et al.,* 2011). Inorganic compounds include: acids, hydroxides, salts, oxides and heavy metal compounds. Every year there is increasing amounts of heavy metals being used and incorporated into cosmetics products. These heavy metals are toxic to human health and the environment at trace level concentrations, which is a major cause of concern (Drozdova *et al.,* 2019). Heavy metal ions gets absorbed when in contact with human body by forming complex species with the binding sites of proteins e.g. carboxylate, $-NH_2$ and -SH resulting in cell death of the cells and lead to different ailments such as cancer genetic mutation, deformation, cancer, kidney problems and kidney diseases (Zainya, 2017).

As a result of inadequate supply of drinkable water in developing countries such as Nigeria, water from neighbouring rivers is used for domestic purposes in cities like Ogun. The vast communities depends on the water bodies for various activities such as fish farming, irrigation, recreation, transportation, cooling, etc. In order to protect the lives of humans, plant and aquatic animals relying on these water bodies for survival, therefore it is of utmost importance to ascertain and monitor the quality and compliance level of the released industrial wastewater with the relevant regulatory standards such as World Health Organization (WHO, 2011) and Standard organization of Nigeria (SON). Hence, this study assesses the treated wastewater qualities from cosmetic industries in Ogun and Lagos state, Nigeria.

2. Materials and Methods

2.1 Sampling Sites

Samples of wastewater were collected at the outlet of Wastewater treatment plants of three different cosmetic industries in Mowe, Ogun state, Oke-Afa, Lagos state and Fatahi-Atere road, Mushin Lagos. The first industry is a hair care product company located in Mowe, Ogun State. The second sample site is a body and hair product manufacturing plant located in Oke-Afa Okota, Lagos. The third sample site is a body care product company that produces body cream and lotion, fragrance and face powder located at Fatahi-Atere road, Mushin Lagos

2.2 Collection of Wastewater Samples

Wastewater samples were obtained from Wastewater treatment plants of three selected cosmetic companies in Lagos and Ogun state, Nigeria during January, 2020. The samples were collected in clean polystyrene bottles of 1L storage capacity. Prior to wastewater collection, the bottles were washed properly and rinsed thoroughly several times with distilled water. The samples for each industry were collected thrice (i.e morning, afternoon and evening) and pooled together to form a composite sample. Samples were labeled as;

Sample A: Composite wastewater from Beauty Field Multi Concept, Mowe, Ogun State

Sample B: Composite wastewater from Cybele Cosmetics limited, Oke-Afa, Lagos State

Sample C: Composite wastewater from Mega M limited, Fatahi-Atere road, Mushin Lagos.

After collection, the pH and conductivity of the wastewater samples were measured onsite using a pH meter (Metrohm, USA) and portable conductivity meter (Thermo Orion, model 162A,) respectively.

2.3 Sample Pretreatment and Preservation

Two bottles were filled with each sample. One was acidified with 1mL of 65% concentrated HNO_3 acid which was used for trace elements preservation, and another non-acidified sample was used for other parameters screening. Collected samples were labeled, transported to the laboratory and stored in the refrigerator at 4ºC prior to further analysis.

2.4 Parameters Analysis

The samples collected were screened for parameters differentiated into three categories namely physical parameters which included pH, conductivity, temperature, alkalinity, total dissolved solids, salinity and turbidity; chemical/ biological parameters which included biological oxygen demand (BOD), chemical oxygen demand (COD), Total organic carbon (TOC) and heavy metals analysis of Hg, Pb, Fe, Cd, Zn, & Ni. All parameters were analyzed using standard methods.

2.4.1 Determination of Physical Parameters

The pH of the samples was measured with a digital pH meter (Metrohm, USA). The pH electrode was calibrated using buffers of pH 4.0, 7.0 and 10.0. The electrode was immersed into the wastewater sample and readings were recorded. Temperature was determined using a thermometer. Electrical conductivity (EC) and total dissolved solids (TDS) were determined by a conductivity meter (Thermo Orion, model 162A, USA). Total solid (TS) are determined as a residue left after evaporation of unfiltered samples while the total suspended solids (TSS) were determined through gravimetric method as described by Sulieman *et al*., (2010)

2.4.2 Determination of Chemical /Biological Parameters

The biological oxygen demand (BOD), total organic carbon (TOC) and chemical oxygen demand (COD) were determined by titration method (APHA 2005). Dissolved oxygen was measured with Jenway Model 9070 waterproof meter. The wastewater samples collected were immediately processed after collection for the determination of initial oxygen and incubated at 20 °C for 5 days for the determination of BOD_5. The results are shown in Table (3).

2.5 Determination of Heavy Metals

Heavy metals (Cd, Ni, Pb, Zn, Fe, and Hg) were determined using Inductively Coupled Plasma-Emission Spectroscopy (ICP-OES, VARIAN720-ES). The wastewater samples were subjected to nitric acid digestion according to method described by Yayintas *et al.,* (2007).

2.5.1 Reagents and Solution Preparation

All reagents used were of analytical reagent grade (Merck, Germany). Deionized water was used through all experiments. Stock standard solution was prepared daily by appropriate dilution of AccuTrace, the reference standard (Item Number is ICP-07W-1, USA) in double-distilled deionized water with 1–5 drops concentrated nitric acid. This solution was appropriately diluted in double-distilled deionized water for prepared metals standard calibration solution concentrations. Also the blank was prepared with double-distilled deionized water containing 1–5 drops concentrated nitric acid.

2.5.2 Sample Digestion

100cm^3 aliquot from a well-mixed, HNO_3-preserved sample was transferred to a 250cm^3 beaker and 2.0 ml nitric acid and 1.0 ml of hydrochloric acid was added. The beaker was placed on a hot plate for solution evaporation. The hot plate was placed in a fume-cupboard and was previously adjusted to provide evaporation at a temperature of approximately but not higher than 85 °C. The beaker was covered with an elevated watch glass to avoid sample contamination from the fume hood surroundings. The volume of the sample aliquot was reduced to about 20 ml by gentle heating at 85 °C without boiling. Then the lip of the beaker was also covered with a watch glass to reduce additional evaporation and gently refluxed for 30 minutes and cooled. The sample solution was quantitatively transferred to a 50ml volumetric flask and made up to the mark with deionsed water. The sample solution was analyzed for Cd, Ni, Pb, Zn, Fe, and Hg using Inductively Coupled Plasma-Emission Spectroscopy(ICP-OES) PerkinElmer®Optima™ 7300 DV ICP-OES instrument (PerkinElmer, Inc. Shelton, CT, USA) equipped with WinLab32™ for ICP Version 4.0 software for simultaneous measurement of all analyte wavelengths of interest. Three different linear concentration standards were prepared, (0.5ppm, 1.00ppm and 2.00ppm) and aspirated into ICP-OES system before conducting sample analysis, and linear curve was prepared. All metals having good linear graph with correlation coefficient > 0.999 were observed in the preparation of standard curves. After the completion of standard curve preparation, one standard check and one QC check are analyzed. (Al-Musharafi *et al.,* 2013)

The instruments working conditions and parameters were presented in Table 1.

2.5.3 Spike Recovery

One Spiked sample were analyzed for every 10 sample study and recovery of spiked concentration has been studied. The spike recoveries were observed at minimum of 85% for all metals.

Table 1. Instrument Operating Parameters

Parameters	Description
Nebulizer gas flow rate (L/min)	0.75
Sample Uptake Time (s)	20
Stabilization Time (s)	20
Replicates	3
Pump speed	15 rpm
Calibration correlation coefficient	0.995
Rf power	1.2kw
Wavelength	Table
Nebulizer pressure	200kPa
Sample Uptake delay	N/A
Sample introduction	Agilent SPS3
Rinse Time (s)	30

2.6. Data Analysis

Digestible samples were analyzed for concentrations of heavy metals: Cadmium (Cd), Lead (Pb), Zinc (Zn), Nickel (Ni), Iron (Fe) and Mercury (Hg) using ICP-OES. The results of the concentrations of the heavy metals were statistically analyzed for mean, standard deviation and one-way ANOVA using Origin Pro 8.5.

3. Results and Discussion

Table 2. Heavy Metals Concentration in Effluents from different Cosmetic Industries (mg/L)

Samples Site	Cd	Pb	Zn	Ni	Fe	Hg
A	0.524	0.512	0.567	0.505	0.532	0.095
B	1.021	1.020	0.980	0.980	1.026	0.245
C	1.974	1.986	1.915	1.917	1.999	0.541
Mean±SD	1.173±0.737	1.173±0.749	1.154±0.691	1.134±0.718	1.186±0.746	0.294±0.227
WHO Limit	0.003	0.01	-	0.07	-	0.006
SON	0.003	0.01	3.0	0.02	0.3	0.001

WHO= World Health organization (WHO, 2011); SON = Standard Organization of Nigeria (SON, 2007)

The use of ICP-OES in this study has been able to detect concentration of heavy metals to the lowest value of 0.095 mg/L. As noted earlier effluent sample from the cosmetic industry C (Lagos-state) show highest concentrations of heavy metals. Cadmium, lead and iron are predominant in the effluent of sample from cosmetic industry C with iron having the highest concentration of 1.999mg/L while mercury has the minimum concentration 0.541mg/L.

In a study conducted by Yayintas *et al.,* (2007), heavy metal concentrations were found between 0.00001-77.69610mg/L, comparatively result observed is higher than the concentration obtained in the present study.

Concentration of mercury remain the lowest from effluent of cosmetic industry B (Lagos-state) similar to the observation in industry C compare to the other heavy metal although the detected amount is lower than what was obtained in sample of cosmetic industry C for the same mercury. Lead, cadmium and iron are the major heavy metal constituent of the sample effluent from cosmetic industry B with Iron having the highest concentration of 1.026mg/L. Concentrations of heavy metals are detected at lowest amount in the effluent sample of cosmetic industry A (Mowe, Ogun state). Cadmium, lead and iron are also detected to be predominant in the effluent sample but mercury shows the lowest detected concentration of 0.095mg/L. The iron concentrations being highest (mean of 1.186mg/L) in the effluent samples can be attributed to the iron materials normally used in the construction of the machine employed at various stages and processes of production. The relatively high concentration of lead can be attributed to the fact that some of the selected industries are either using lead pipe or having lead as one of the major constituent of their cosmetic products. The health implications of mercury are disastrous ranges from negative effect on Central nervous system and tendency to effect brain tumours etc. The Food and Drug Administration (FDA) in the United States, prohibits the presence of any mercury Hg concentration in cosmetics and establishes maximum limit of 10µg/g for other heavy metals like lead, Pb for cosmetic to be applied on external region of the body. The use of ICP-OES has enable detection of mercury to level of 0.095mg/L. The discharge of these heavy metals into streams and other water bodies at the measured concentrations will posse danger to the environment. The acceptable concentrations of heavy metals from cosmetic industries as stipulated by World Health Organization (WHO, 2011) and Standard Organization of Nigeria (SON, 2007) are stated in table 2 (Izah *et al.,* 2016) but the measured values of concentrations of the heavy metals under study are beyond the acceptable value except zinc, this is an indication of threat to the environment against agitation for green environmental practices.

Table 3. One-way ANOVA for Significant Difference in Concentrations of Heavy Metals in Effluents of Cosmetic Industries

Source of variation	SS	df	MS	F-ratio	5% F-limit (F-table)
Between sample	4.986	2	2.493	16.10	$F_{(2,15)}$ = 19.43
Within sample	2.323	15	0.155		
Total	3.99	17			

SS= Sum of squares (variance) MS = Mean square df = Degree of freedom

P_{calc} = 0.000184 < 0.05 (P_{table})

Table 4. Results of Physicochemical Analysis of the Effluents.

PARAMETER	Sample A	Sample B	Sample C	Mean±SD	WHO Limit	SON
pH	7.07	6.65	6.95	6.89± 0.12	6.00-8.00	6.5-8.5
Conductivity(µS/cm)	3025.0	3562.8	2517.0	3034.9±14.27	1000.0	1000
BOD(mg/l)	95.06	105.64	78.02	92.90±3.57	5-7	-
COD(mg/l)	214.84	221.84	183.35	206.67±1.42	40.00	-
TS(mg/l)	8435.07	9268.20	8672.09	8791.78±23.46	1500.00	-
TDS(mg/l)	1361.25	1532.00	1157.82	1350.35±6.97	500-600	500
TOC(%)	2.07	2.76	1.98	2.27±0.016	25.0	-
TA(mg/l)	95.6	70.0	77.5	81.0±0.81	600	100
Salinity(mg/l)	0.325	0.346	0.484	0.385±0.001	600	-

(WHO, 2011); (SON 2007)

The calculated F-ratio value (16.102) is less than the F-table value (19.43) indicating that there is no statistically difference in the concentrations of heavy metals in all the effluent samples based on the sources of the effluents, either Lagos state or Ogun state. The differences in detected concentrations of the heavy metals is not influenced by a particular industry and the location but can be attributed to processes involved in the production of cosmetics, the raw materials employed, the machines and other equipment used in production. Justifying this result is the report that metals such as Ag, Al, Au, Zn, Cd, Cu, Ni, Pb etc are either used to provide an iridescent metallic effect to the final cosmetics products or retain as impurities in the raw materials and most of the heavy metals are responsible for allergic dermatitis (Sainio *et al.,* 2000; Massadeh *et al.,* 2017).

The mean value of the pH for the effluents from the three companies is 6.89± 0.12. Although sample A from Ogun state is almost close to neutrality but all samples have pH within the acceptable range as recommended by WHO, (2012). None of the samples give values in acidic region. Low values pH is attributed to the use of materials such as benzoic acid, lactic acid and enzyme as raw materials while higher values are due to the use of caustic soda, NaOH as cleaning agent in the factories, (Walakira and Okot-Okumu, 2011). This implies minimal use of caustic soda as cleaning agents in the selected factories and the use of organic materials like ester with neutral. This observation is in agreement with observed total alkalinity value reportedly to be lower compared to the standard limit. The electrical conductivity values of the effluents are far beyond the acceptable limit and this shows a good correlation with the total dissolved solid (TDS). Pigment such as iron oxide, copper powder and bismuth oxychloride which are used during production of cosmetics can be attributed to the unavoidable higher electrical conductivities as reported in the result of (Mesko, *et al.,* 2017). Comparison of the BOD and COD with reference to the electrical conductivity shows that the predominance of the ionized product are not of organic origin but rather from inorganic raw materials employed and also the higher COD values can also be attributed to the different oxidizing agents used during production (Al-Dayel *et al.,* 2011). The higher COD values recorded for the effluents is in line with the report of (Bogacki *et al.,* 2011; de Melo et al., 2013). Total organic carbon percentage is below the acceptable value signifying the fact that raw materials employed in the selected industries for cosmetic production are inorganic in nature similar to the report of (Olaoye and Oladele, 2011).

4. Conclusion

In this present study, a number of toxic heavy metals (Pb, Cd, Ni and Hg) in all samples were found to be at concentrations exceeding acceptable limits determined by WHO and SON. Also, most of the results for the physicochemical parameters indicated non-tolerable quality. Therefore, it was established in the study that wastewater treatment plants under studied could not treat the inflow of wastewater received to acceptable quality, especially in lowering the concentration of Pb, Cd, Ni and Hg before discharging into the environment. It is therefore recommended that continuous monitoring by environmental agencies and maintenance of WWTPs infrastructure by respective industries should be done adequately.

Conflict of interest

The authors declare that there is no conflict of interests regarding the publication of this article.

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