ASSESSMENT OF PHYSICOCHEMICAL PARAMETERS AND HEAVY METALS IN EFFLUENTS FROM ODOGUNYAN INDUSTRIAL ESTATE, LAGOS, NIGERIA

Owalude, S.O.*, Odebunmi, E.O. and Babalola, K.O.

Department of Chemistry, University of Ilorin, P.M.B. 1515, Ilorin, Nigeria
Corresponding author's email: owalude@unilorin.edu.ng

ABSTRACT

The impact of the industrial effluents from Odogunyan industrial estate on the environment was assessed by determination of some physicochemical characteristics of the effluent samples and comparison with discharged standards to ascertain the efficiency of industries' wastewater treatment process. The physicochemical parameters were evaluated by conventional methods while heavy metals in the effluent samples were analyzed using Atomic Absorption Spectrophotometer (AAS). The results for all the effluents revealed that temperature was in the range of 32- 35 $^\circ C$; Biochemical Oxygen Demand (BOD) 43-86.7 mg/L; Chemical Oxygen Demand (COD) 492-888 mg/L; Total Dissolved Solids (TDS) 1667-13333 mg/L; Total Suspended Solids (TSS) 350-1000 mg/L; Total Solids (TS) 3350-14333 mg/L; Electrical Conductivity 166.5-12390 μS/cm; Total Acidity (TA) 12-60 mg/L and total hardness 24-56 mg/L. The AAS analysis results showed the average metal levels in mg/L as 0.7-1.15, 0.05-0.44, 2.0-45.0, 0.01 and 0.03-0.17 for Zn, Cu, Fe, Cd and Cr respectively. Thus, there is a need for proper remedial measures of the effluents before their discharge into the water bodies.

Keywords: Water, pollution, textile industries, industrial wastes, Atomic Absorption Spectroscopy.

INTRODUCTION

Water is one of the most important and abundant components of the ecosystem. All living organisms on the earth require water for their survival and growth. Water is essential to all forms of life and it constitutes a large percentage of the weight of plants, animals and humans (Driskell, 2000). Water is also very important in agriculture, manufacturing, transportation and many other human activities. Despite its importance, water is the most poorly managed resource in the world; about 1.1 billion people of the world's population do not have access to safe water (Praveen *et al*., 2016). This is as a result of the increase in human population, industrialization, use of fertilizers in agriculture and other man-made activities which make water to be highly polluted with different harmful contaminants.

Industrialization is highly important for the social and economic development of a nation. A good percentage of the population in both developed and developing countries are gainfully employed by these industries (Baer and Hervé, 1966). This has led to poverty reduction and improved standard of living among the citizens of these countries, however environmental considerations should be given a high priority when citing industries. In developing countries such as

Nigeria, very little attention is given to possible damage to the environment by the discharged effluents when industries are being cited (Amuda *et al*., 2006). This environmentally unacceptable practice poses a serious threat to public health and even industrial estates are indiscriminately located in residential areas in most state capitals and large urban centers in Nigeria (Adebisi and Fayemiwo, 2010). The industrial wastes from these industries are generally discharged into the environment resulting in adverse effects on humans and the environment at large. Aquatic lives, soil and crops are also polluted in the process causing harmful damages to the health of humans, animals and the entire environment (Hossain *et al*., 2010). Industrial effluents having high heavy metals content are of special concern because their intake leads to poisoning and chronic health problems in aquatic animals and by extension to humans (Owa, 2013). The discharge of organic wastes into river water causes leads to an increase in BOD, COD and TDS. Also, total suspended solids and faecal coliform in the water body, making the water unsuitable for domestic and industrial uses. It is therefore desirable to determine the level of these pollutants in water before being applied for both domestic and industrial uses; this will provide data on the degree of contamination in these water bodies and

also help the concerned authorities in the formulation of desirable future environmental policies (Moldovan, 2005; Said and Hamed, 2006). The selection of parameters for testing of water solely depends upon what the water is used for and the level of quality and purity required (Patil *et al*., 2012). This research is therefore focused on the evaluation of physicochemical parameters and determination of heavy metals content of the industrial effluents from an industrial estate located at Odogunyan area in Ikorodu, Lagos, Nigeria. The area under study is a large expanse area of land housing several manufacturing industries producing textile, detergents as well as iron and steel. The level of the toxic pollutants discharged by these industries into the environment and the environmental impact were assessed and reported in this study.

MATERIALS AND METHODS

Sample Collection: Samples of industrial effluents were collected from four different industries (iron and steel – IEI, textile waste – IET_1, textile waste – IET_2 and soap and detergent – IED). For collection, bottles were cleaned with tap water, rinsed under the drain water, uncapped and water was collected from beneath the surface. Air bubbles were removed and the bottles were capped immediately following a literature procedure (Besselievre and Schwartz, 1976). Each sample bottle was labeled with the necessary information like the name of the industry, location, date and time of collection.

Physical Parameters

The Temperature: This was measured at the time of sample collection. The temperature was measured with the aid of a mercury thermometer. The thermometric bulb containing the mercury was vertically immersed into the effluent and allowed to stand for some minutes till the temperature reading was steady before obtaining the readings.

Colour and Odour: Each effluent sample was transferred into a clean glass test tube; the colour was evaluated visually while the odour of the effluent samples was smelled and noted as described in a previous report (Kulandaivel *et al*., 2014).

pH: The pH of each industrial effluent was measured at the time of sample collection with a portable field digital pH meter (Hanna Instrument). The meter was calibrated with a buffer of 4.00 and 7.00, it was then inserted into the effluent sample and pH measurement was recorded after the reading is stable. The electrode will then be rinsed with deionized water before taking another measurement.

Turbidity: The turbidity of each effluent sample was also analyzed at the point of collection. The turbidities were measured using a digital turbidity meter (2100AN HARCH Model). The meter was standardized with deionized water and then introduced into each effluent sample. The turbidity reading of each sample in Nephelometric Turbidity Units (NTUs) were then recorded.

Total Suspended Solids (TSS): A filter paper was dried to a constant weight at 103 to 105 °C in an oven and was allowed to cool at room temperature after which it was weighed on a balance. 10 ml of the effluent sample was measured and filtered using filter paper placed in a funnel. The filter paper was removed gently and dried to constant weight within this temperature range in the oven. It was then allowed to cool. The weight of the filter paper and the residue was determined on a weighing balance and recorded.

$$Total\ Suspended\ Solids\ (mg/L) = \frac{(B - A) \times 1000}{Volume\ of\ Sample\ (mL)} \qquad (1)$$

Where: A = weight of filter paper only; B = weight of filter paper + dried residue; mL = volume of effluent.

2.2.6 Total Dissolved Solids: Each effluent sample was stirred with a magnetic stirrer and a portion of the effluent was filtered. 10 mL portion of the filtrate was measured into a pre-weighed evaporating dish and dried in an oven. The dish was placed in a desiccator and allowed to cool to room temperature and then weighed. The total dissolved solids content of the water was calculated using the relationship:

$$Total\ Dissolved\ Solids\ (mg/L) = \frac{(B - A) \times 1000}{Volume\ of\ sample\ (mL)} \qquad (2)$$

Where: A = weight of dish only, mg; B = weight of dried residue + dish, mg

2.2.7 Total Solids: Total solids were obtained by adding the total dissolved and suspended solids, using this relation:

$$TS = TDS + TSS \qquad (3)$$

Electrical Conductivity (EC): This was measured for each sample at the time of collection with a Handheld Conductivity Meter (Jenway Conductivity Meter 4510 model). The probe was dipped into the bottle containing the samples until a stable reading was obtained and recorded (Chinedu *et al*., 2011).

Chemical Parameters

Chemical Oxygen Demand (COD): The COD of each sample effluent was determined as follows: A blank solution was prepared by pipetting 100 ml of distilled water into 250 ml conical flask. A 10 ml portion of 25% H_2SO_4 and 20 ml of 0.01 M $KMnO_4$ was added. Then, 10 ml of the water sample was measured into another 250 ml conical flask and diluted with 90 ml of distilled water. A 10 ml portion of 25% H_2SO_4 and 20 ml of 0.01 M $KMnO_4$ were added to the solution and was heated in a water bath for 30 minutes. The solution was allowed to cool and 10 ml of 10% KI solution was added. The resulting solution was then titrated against 0.05 M $Na_2S_2O_3$ for some time to expel some iodine and 3 drops of the freshly prepared starch solution was added. The titration was continued to a colourless endpoint.

The chemical oxygen demand (COD) was calculated using this relation:

$$COD = \frac{(A - B) \times M \times 40000}{Volume \ (mL) \ of \ sample} \qquad (4)$$

Where: A = Titre value of sample; B = Titre value of the blank solution; M = Molarity of $KMnO_4$; V = Volume of sample

2.3.2 Dissolved Oxygen (DO): The amount of oxygen found in these determinations at the time of collection is the dissolved oxygen. The amount of DO was determined by following a reported modified Winkler's titration technique (Olaniyi *et al*., 2012).

2.3.3 Biochemical Oxygen Demand (BOD): Each effluent sample was divided into two portions. The DO level in ppm of the first portion was measured immediately using the method described above in 2.3.2 and recorded. The second portion was placed in complete darkness by wrapping the effluent sample bottle with aluminum foil at room temperature for 5 days. After 5 days, another dissolved oxygen reading was taken using the dissolved oxygen test kit. Then the Day 5 reading was subtracted from the Day 1 reading to determine the BOD level. The final BOD result was recorded in *ppm*.

2.3.4 Acidity: In a typical determination, a 50 ml portion of the effluent sample was measured into a clean 250 ml conical flask. Two drops of phenolphthalein indicator were added and the solution titrated against a standardized 0.01 M NaOH solution to a pink endpoint.

Acidity was calculated using equation

$$Acidity \ (mg/L) \frac{V \times M \times 100000}{Volume \ of \ sample \ used \ (mL)} \qquad (5)$$

Where V = volume of NaOH; M = molarity of NaOH; mL = volume of effluent

2.3.5 Alkalinity Level: The alkalinity level of each effluent sample was also determined. A 50 ml portion of the effluent sample was measured into a clean 250 ml conical flask, two drops of methyl red indicator was added and the solution was titrated against a standardized 0.01M HCl solution to a pink end-point. Alkalinity was calculated using the equation:

$$Alkalinity \ (mg/L) = \frac{V \times M \times 100000}{Volume \ of \ sample \ used \ (mL)} \qquad (6)$$

Where V = volume of acid used; M = Molarity of acid used; mL = volume of effluent

2.3.5 Total Hardness: A 25 mL of each effluent sample was measured into different clean 250 mL conical flasks. To this, 3 mL of ammonium chloride in concentrated ammonia buffer and 2 drops of Eriochrome Black T indicator was added. This was titrated against 0.01M EDTA solution until there is a colour change from violet to blue. The procedure was repeated two more times to obtain the average titrated value. Total Hardness was calculated using the equation:

$$Hardness \ in \ mg/L \ CaCO_3 = \frac{V \times M \times 1000}{Volume \ of \ sample \ used \ (mL)} \qquad (7)$$

Were M = Molarity of EDTA used; V = Volume of EDTA used; mL = volume of effluent

2.3.6 Heavy Metals

Iron (II), chromium (VI), zinc (II), lead (II), copper (II), manganese (II), nickel (II) and cadmium (II) ions in the aqueous solution were determined using Atomic Absorption Spectrophotometer.

RESULTS AND DISCUSSION

3.1 The temperature and pH of the samples: The results of the determined physicochemical analysis of the effluent samples collected from the four industries are presented in Tables 1 and 2. This is to assess the environmental impact of the waste effluents. The average temperature of the effluents ranges from 32 - 35$°C$. This value is within the Nigerian Environmental Standards and Regulations Enforcement Agency (NESREA) limit of 40°C (Adeolu *et al*., 2016). Portable water is generally tasteless and colourless with cool water being more palatable, any inorganic chemical contaminants that may affect taste and odour will therefore be affected by temperature. High water temperature aids the growth of microorganisms and may increase taste, odour, colour and corrosion problems. The temperature increase may become a barrier to fish migration and can affect their reproduction (Fenkes *et al*., 2016). Industrial dye effluents are generated at relatively high temperatures and their discharge into wastewaters may lead to an increase in temperature (Banat *et al*., 1996). The slight difference in the observed temperature of IET_2 with the WHO standard could therefore be attributed to heat exchange from the production process.

The pH of the effluent samples is 8.3, 7.8 and 7.0 for IEI, IET_1 and IET_2 respectively. These values are within both the WHO permissible limit of 6.5 – 8.5 and the NESREA limit of 6.0 – 9.0; therefore, there could be no danger to the receiving environment. The pH of IED is 10.7 which is well above the standard set by both the WHO and NESREA. Monitoring pH of marine waters is an important process in biological treatment. Aquatic organisms are very sensitive to pH changes

and most chemical reactions in the aquatic environment are controlled by a change in pH value, anything substance that could either make aquatic waters to be highly acidic or alkaline would therefore be a threat to marine life. The toxicity of heavy metals is pH-dependent; thus, the quality of wastewater effluent can also be improved by proper pH control (Banat *et al*., 1996). The pH value of 10.7 obtained for IED in this study suggests contamination by a strong base such as NaOH and Ca(OH)$_2$ which are important raw materials in the soap and detergent industry (Ogundiran *et al*., 2010).

3.2 Total dissolved solids, electrical conductivity and turbidity of the effluent samples: The TDS in the effluents ranges between 3000 - 13333 mg/L. This is far above the turbidity permissible level of 500 mg/L (WHO, 2003). A high content of dissolved solids affects the density of the water and reduces the solubility of useful gases like oxygen and consequently the utility of water for domestic, irrigational, and industrial applications (Lokhande *et al*., 2011). According to WHO, a TDS level of less than 600 mg/L is considered to be good quality water (WHO, 2003). TDS values determine the conductivity; therefore, the high electrical conductivity obtained for IED in this study indicates the utilization of electrolytes like common salts in the production processes (Sharma *et al*., 2014). Total dissolved solids also indicate the presence of inorganic salts and dissolved inorganic matter containing ions such as carbonates, sulphates, chlorides, calcium, sodium, potassium and magnesium (Emeka, 2015; Lokhande *et al*., 2011; Sharma *et al*., 2014). From the results presented in Table 1, electrical conductivity of 209.5, 194.9 and 166.5 μS cm^{-1} were obtained respectively for IET$_1$, IEI and IET$_2$. These values are within the approved limit of 1000 μS/cm by the WHO standard. The IED on the other hand has a conductivity value of 12390 μS cm^{-1} which is in much excess of the permissible limit. This indicates a high concentration of electrolytes in the samples and will therefore require treatment before being discharged into the water body. An increase in dissolved ions in water results in an increase in electrical conductivity and this is in line with the high total dissolved solids content obtained in the present study, this is also an indication of poor quality of the receiving water body (Hem, 1989).

Turbidity measures the cloudiness or haziness of fluids due to the presence of suspended particles that may not be visible to unaided eyes and it provides a very good test for water quality. When the turbidity of the water sample is high, the chances of contamination of water-borne diseases are very high since the suspended particles may shield the bacteria from disinfectants during water treatment. The estimated turbidity for IET1 and IET2 are within the permissible maximum limit of 10 and 25 NTU set by ISI while those of IED and IEI are well above the recommended level (Kumar and Puri, 2012). The higher turbidity in the industrial effluent samples could be attributed to high level of suspended materials, bacteria, plankton as well as dissolved organic and inorganic substances (Chinedu *et al*.,

2011). High turbidity can also impede the rays of light entering the river which eventually reduces the dissolved oxygen level in the water bodies (Akinloye and Olubanjo, 2014).

3.3 Total suspended solids, total solids and total hardness of the effluent samples: Any solid that is suspended in water and when on filtration cannot pass through a sieve of 2 μm pore size are considered total suspended solids while total solids are the combined content of all the ionized molecular or micro-granular organic and inorganic substances contained in a liquid. These solids can influence the level of dissolved oxygen in water thereby causing damages to the living organisms in water bodies (Chinedu *et al*., 2011). The permissible limit of 30 and 500 mg/L has been set for TSS and TS respectively by WHO and NESREA (Onuegbu *et al*., 2013). The values obtained for these parameters presented in Table 1 are well above the permissible limit, therefore these effluents need to be treated before being discharged into water bodies.

The estimated hardness values of the effluent samples reported in Table 1 are well below the WHO permissible limit of 500 mg/L (Kumar and Puri, 2012). The concentration of calcium and magnesium salts in water is a measure of its total hardness. When these salts are present in high concentration (> 200 mg/L) and combined with the interaction of other factors, such as pH and alkalinity, the water is considered to be hard. Such water samples will likely cause scale deposition in the treatment works, public water pipes and tanks. Hard water will require a high quantity of soap and detergent when used for washing and subsequent formation of scum is usually noticed. However, water with a hardness of less than 100 mg/L may have a low buffering capacity and so be more corrosive to water pipes since its capacity to resist pH changes becomes reduced (Hiscock, 2005). The hardness in water can be easily removed by simple boiling or addition of chemicals such as washing soda, sodium hydroxide and also through ion exchange technique (Ogundiran and Fawole, 2010; Sa'eed and Mahmoud, 2014).

3.4 Total alkalinity: The alkalinity levels were assessed in the effluent samples and the values 4800, 68, 83 and 72 mg/L were obtained respectively for IED, IEI, IET$_1$ and IET$_2$. These values are below the maximum permissible limit of 600 mg/L except that of IED which is well above the limit (Kale and Bandela, 2016). A high concentration of chemical species such as bicarbonates, hydroxides, phosphates and borates lead to high alkalinity in waters (Winter, 1998). The high alkalinity of IED can be a result of the hydroxides used in soap production which also accounts for the high pH of 10.7 (Adesoye *et al*., 2014).

3.5 Dissolved Oxygen and Biochemical Oxygen Demand: BOD values have been widely adopted as a measure of pollution effect in waters and it measures the amount of oxygen consumed by the organic matter (bacteria) and inorganic compounds while maintaining the organic matter at the similar conditions as those that occur in nature (Mohan *et al*., 2000). It measures how fast biological organisms use up oxygen in a

body of water over 5 days (BOD_5) and in combination with chemical oxygen demand, is one of the most common procedures for determining the level of putrefiable organic pollutants in water (Lokhande *et al*, 2011; Pujar et al., 2014). The data in Table 1 indicates that the BOD values of 52.4, 51.5, 86.7 and 43.8 mg/L were obtained respectively for the effluents IED, IET_1, IET_2 and IEI. These values were well above the NESREA permissible limit of 30 mg/L (Onuegbu *et al*., 2013). Low BOD value is an indication of good quality water and high BOD value indicates polluted water since the greater the BOD, the more rapidly oxygen will be depleted if such effluent is discharged into the water body. A high BOD value means the total dissolved oxygen becomes depleted and there is competition for available oxygen by aquatic organisms, they become stressed, suffocate, and eventually die (Lokhande *et al*., 2011; Sharma *et al*, 2014; Emeka, 2015).

3.6 Chemical Oxygen Demand: The average COD values obtained for the effluents in the present study lie between 492 and 888 mg/L, which is higher than the maximum allowed limit of 80 mg/L recommended by NASREA standard (Onuegbu *et al*., 2013). The Chemical Oxygen Demand (COD) is a measure of the oxygen equivalent of all organic matter in a sample that can be oxidized by a strong chemical oxidant at elevated temperatures (Mohan *et al*., 2000; Patil *et al*., 2012). The COD test takes considerably less time (2 h) than the BOD_5 test (5 days). The result obtained implies that further treatments are required to remove the chemical oxidants present in the effluents to prevent oxygen depletion when discharged into water bodies.

3.7 Metals content of the industrial effluents: The effluent samples were analyzed for metal contents and the results showed that the concentration of zinc ranges between 0.7 and 0.99 mg/L which are within the WHO and USA standard permissible level of 3.0 and 5.0 mg/L respectively ((Javed and Usmani, 2013)). The concentration of Zn (1.15 mg/L) in the IEI effluent was well above the FEPA standard of < 1 mg/L. Excessive concentration of Zn may result in necrosis, chlorosis and inhibited growth of plants (Lokhande *et al*., 2011). The concentration of copper in the effluent samples ranges from

0.05 - 0.44 mg/L which falls within the approved maximum limit of 2.00 mg/L (Nazir *et al*., 2015). The effluents contain a high concentration of iron (2.0 - 4.5 mg/L) when compared with the approved USA standard of 0.3 mg/L (Javed and Usmani, 2013), and if discharged into the water body can be harmful to the human and aquatic organism. Most pathogenic organisms require Fe for rapid development and high concentration of Fe in water body will therefore increase its availability to these organisms (Tiwana *et al*., 2005). Cadmium concentration was found to be 0.01 mg/L for all the examined effluent samples. This is quite high when compared with the USEPA permissible limit of 0.005 mg/L but just within the maximum of 0.01 mg/L by WHO (Chauhan, 2013). Higher values of Cd in wastewaters indicate a high level of pollution which may results from dyes and paints pigments being utilized by manufacturing industries around the study area. Cadmium is reported to be toxic to both invertebrates and fishes and there have been a few recorded instances of poisoning in humans following consumption of contaminated fishes (Moore and Ramamoorthy, 1984). The permissible Cr concentration limit of 0.05 mg/L has been set for IED and IEI by WHO (Javed and Usmani, 2013), and effluents from both Spintex and Sunflag are within this limit showing an indication of good effluent treatment by these industries. For PZ and African Steel, the average Cr content is well above the permissible limit which indicates excessive pollution and poor effluent treatment. Acute toxicity of Cr to invertebrates is highly variable, depending upon species (Moore and Ramamoorthy, 1984). Acute toxicity in invertebrates and fishes has not been reported but Cr is generally more toxic at higher temperatures and its compounds are known to cause cancer in humans (Ember, 1975; Langard, 1990). The accumulation of Cr by plants results in the roots remaining small and narrow leaves displaying reddish-brown discoloration with small necrotic blotches (Lokhande *et al*., 2011). However, no detectable Nickel and Lead concentrations were found in all the effluents, this is probably due to the non-usage of these metals as raw materials by the industries under the study area.

Table 1: Results of the physicochemical analysis of the effluents

Parameters	IED	IET_1	IET_2	IEI	Standards
Colour	Colourless	Pink	blue	Clear with suspended black	3 NTU
Temperature (°C)	32	33	35	33	20-32[a], <40[b]
pH	10.7	7.8	7	8.3	6.5-8.5[a],6-9[b]
Turbidity (NTU)	97	10	3	37	5[a]
EC (μS/cm)	12390	209.5	166.5	194.9	1000[c]
DO_1 (mg/ L)	12.14	8.87	10.71	10.81	5.0[c]
BOD_5 (mg/ L)	52.4	51.5	86.7	43.8	30[b],50[c]

COD (mg/ L)	744	760	492	888	80[b],200[c]
Acidity (mg/ L)	ND	54	60	12	NA
Alkalinity (mg/ L)	4800	83	72	68	120-600[a]
Total hardness (mg/ L)	24	42	36	56	100[a]
TDS (mg/ L)	13333	1667	3500	3000	500[a]
TSS (mg/ L)	1000	400	350	350	30[a,b]
TS (mg/ L)	14333	2067	3850	3350	500[a,b]

[a] = WHO; [b] = NESREA; [c] = FEPA; ND = Not Detected; NA = Not Available

Table 2: Concentration of metals in the effluents

Heavy metals (mg/L)	Pz	Spintex	Sunflag	African Steel	Standards
Fe	2.0	3.23	2.0	45.0	0.3[a]
Cu	0.05	0.07	0.09	0.44	1[a,c]
Zn	0.99	0.96	0.7	1.15	3[a],<1[c]
Cr	0.22	0.05	0.03	0.17	0.05[a]
Cd	0.01	0.01	0.01	0.01	0.003[a]
Pb	ND	ND	ND	ND	0.01[a]
Ni	ND	ND	ND	ND	0.02[a]

[a] = WHO; [b] = NESREA; [c] = FEPA; ND = Not Detected; NA = Not Available

WHO: World Health Organization;   NESREA: Nigerian Environmental Standards and Regulations Enforcement Agency; FEPA: Federal Environmental Protection Agency

CONCLUSIONS

The results of the physicochemical analyses showed that most of the analyzed parameters did not show compliance with WHO, NESREA and FEPA standards. Although total hardness, total alkalinity and electrical conductivity obtained for IET$_1$, IET$_2$ and IEI were within the discharge permissible range but the results obtained for effluent of IED were still high. The results obtained from the AAS analysis confirmed the presence of different kinds of metal ions in the wastewater samples at levels generally higher than the permissible limits; hence contamination of the ecosystem is imminent. These results indicate high pollution of the environment under study and there is a need for the regulatory agencies to take necessary actions to forestall epidemics.

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