Assessment of Heavy Metals and Physico-Chemical Parameters of Stream Associated With Vegetable-Oil-Industrial Effluent in Zawan Community, Jos South- Plateau State, Nigeria

[1]Gonap, B.J., [2]Onekutu, A., [3]Ebah, E.E

[1]Department of Biological Science, Faculty of Science,University of Agriculture Makurdi, Benue State
[2]Department of Biological Science, Faculty of Science, University of Agriculture Makurdi, Benue State
[3]Department of Biological science, Faculty of Science, University of Agriculture Makurdi, Benue State

Abstract: The development of industries and extensive urbanization has enhanced water consumption and pollution resulting from problems of industrial waste disposal. This Industrial development results in the generation of industrial effluents and if untreated it results in water and soil pollution. This study was carried out to check the impact of the effluent discharged by the vegetable-oil-producing industry and its possible environmental pollution level of the surrounding habitat. Of the collected and analyzed water and soil samples, it was found that all the isolated metals have their concentration in the water and soil ranging above that that was found in the control sample. A total of eight (8) heavy metals: Copper (Cu), Nikel (Ni), Manganese (Mn), Chromium (Cr), Lead (Pb), Iron (Fe), Zinc (Zn), and Cadmium (Cd) were identified from the water and soil samples collected. Of the eight isolated metals: Ni, Cr, Pb, Fe, Zn, and Cd were noted to occur above their respective permissible limits as approved by the Federal and State Environmental Protection Agencies. Pb was absent in soil sample A while Mn was not detected in water samples C,D and F and soil sample D, E and F. In reference to the control samples, all the water samples analyzed recorded Zn(27.39±0.02)ppm at water point B with a remarkably high concentration while in the soil samples, Fe was remarkably high in concentration followed by Pb.The concentration of Cu was found to have slightly exceeded that of the control which was put at (0.21±0.01)ppm only at water sample point B and E. Compared to the control (0.32±0.01ppm), Pb has shown all concentrations above that of the control sample, with a remarkable high concentration (13.94±0.02)ppm and (17.36±0.02)ppm in water samples of point A (effluent discharge point) and point D (mining point) respectively. The Physicochemical parameters measured and recorded for the different sampling points analyzed were: Total dissolved solids (TDS), turbidity, chlorine (Cl$^-$) content, dissolved oxygen (DO), biochemical oxygen demand (BOD), total suspended solute (TSS), alkalinity, oil and grease. The samples were compared to a control sample and of all the parameters taken, three (TDS, turbidity and alkalinity) showed significant values above that of the control sample at most of the points with their values peaking at points B and C. One-way ANOVA was used to analyze results of the different sample points and mean differences were sorted out based on Tukey-Kramer's Multiple Comparisons Test using InStat3 Software, 2013 (significant at $P<0.05$) while paired sample t-test carried out to test for difference in means of concentrations (ppm) for both soil and water samples using SPSS software. Creating awareness of this finding to the public is recommended.

Keywords: Heavy Metals, Effluent, Pollution, Industry, Awareness, Physicochemical.

1. INTRODUCTION

Water is one of the most important and abundant compounds of the ecosystem. All living organisms on the earth need water for their survival and growth. As of now, only earth is the planet having about 70% of water [22]. For water to be usable, it must be free from pathogens, dissolved toxins/ 'heavy' metals and disagreeable turbidity, odour, colour and taste. Such usable water, in most communities, is obtained from surface sources such as streams, river, lakes and hand dug wells. Good health is dependent upon a clean potable (drinkable) water supply [22]. However, due to increasing human population, industrialization, use of fertilizers in the agriculture and man-made activity makes surface water to be highly polluted with different harmful contaminants [21,17]. The oil refinery industry is one of the important sources of pollutants to the environment being a significant water consumer and consequently a large waste water producer [1]. In the last 50 years In particular, human exposure to heavy metals has risen dramatically as a result of an exponential increase in the use of heavy metals in industrial processes and products. In natural organisms, some trace metals are essential at low levels but they can be toxic at higher concentrations. They enter into the living organisms' body through the food chain causing different diseases and damages in them (even humans) [13]. Humans are exposed to metals via the following pathways: diffusion into the bloodstream through the lungs and skin, drinking contaminated water, and eating contaminated food, such as fruits and vegetables [23]. Having excessive amounts of heavy metals such as Pb, Cr and Fe, as well as heavy metals from industrial processes are of special concern because they lead to chronic poisoning in living organisms especially aquatic animals [8,1,16]. High levels of pollutants mainly organic matter in river water causes an increase in biological oxygen demand, chemical oxygen demand, total dissolved solids, total suspended solids and fecal coli form [7]. They make water unsuitable for drinking, irrigation or any other use [10]. Water pollution has many sources. The most important sources of heavy metals in the environment are the anthropogenic activities such as mining, smelting procedures, steel and iron industry, chemical industry, traffic and agriculture as well as domestic activities which are discharged into rivers [2]. Trace amounts of heavy metals are always present in fresh waters from the weathering of rocks and soils [4,6,12]. Even though natural water may contains some types of impurities whose nature and amount vary with source of water. They may be contaminated with heavy metals thereby making the water toxic and dangerous for biological systems [16,18]

Study Site:

Zawan community which has this Geographical Coordinates as 9° 45' 0" North, 8° 52' 0" East, is situated in Du district of Jos South [11]. It has an approximate population of 76,000 persons, and it is basically bare ground characterized by few ponds, streams and degrading forest patches that are rapidly diminishing due to anthropogenic activities like farming, urbanization and deforestation activities in the area. It habitat the grand cereal and oil Mills Company.

Plate 1: Map of Zawan Community Showing Industry, Water sources and Residential Areas

2. MATARIALS AND METHODS

Introduction:

Rapid increase in the pollution of our environment due to increase in industrialization, developmental processes, anthropogenic activities are major factors of pollution in the world. If not control, the problem of pollution of the environment continues to be on the increase. To keep this environmental risk in check, some safety measure must be applied. However, from the variety of analytical methods available, no standard method of the analysis has been selected for particular types of environmental samples (soil, water, plant, air, and sediment).The analytical procedures applied to a particular pollutant depend on the facilities and the experience existing in the laboratory [16].

Sampling or Sample collection:

Water and soil samples were collected from the streams (at the stream inlet, center and outlet) along a 1.5km transect, eastward of the company, along the streams' direction of flow. The samples were collected at intervals of 300m away from each other at the sampling locations in Zawan Community, Plateau state, Nigeria. The important parameters considered in these studies include Heavy metals, Total suspended solids, Total dissolve solids, Dissolve oxygen, Biochemical oxygen demand, Alkalinity, Chloride, Oil and grease. Water samples were collected in sampling bottles avoiding floating materials. The stoppers of the sample containers were closed properly to prevent outside contamination. The containers were labeled describing the name of the sampling point, date and conditions under which each was sampled. The collected samples were transported by standard methods as illustrated in [5,16]

Soil samples were collected at depths of 0 to 5 cm and 5 to 10 cm from six sites, stored in plastic bags and transported to the laboratory for heavy metal extraction and analysis. The soil samples were oven dried at 105°C for 24 h, followed by grinding and sieving using 0.18 mm sieve. Dry soil sample (0.5g) was poured into a graduated test tube and mixed with 2 ml of aqua regia 1:3 (1 conc. HCl: 3 conc. HNO_3). The mixture was digested on a hot plate at 95°C for 1 h and allowed to cool to room temperature.

The sample was then diluted to 10 ml using distilled water and left to settle overnight. The supernatant was filtered prior to analysis using AAS as specified in Adams (1991).

Reagents and Materials:

Some reagents used were Sodium Thiosulphate, Manganase (ii) sulphate, alkali-azide reagent, Anhydrous calcium carbonate, calcium chloride, Sodium sulphate, Potassium dichromate, Hydrochloric acid, Nitric acid etc.

Materials used include: Measuring cylinder, Weighing balance, Dessicator, Seperatory funnel, Beakers, Volumetric flask. etc

Quality Assurance Protoco:l

All reagent used were of analytical grade. Contamination was checked by running blanks of all determination. Distilled water was used. All glass apparatus used were first soaked in hot soap solution, later in H2SO4/HNO3 acid(1:1)mixture, and then rinsed with copious amount of hot water followed by several portions of distilled water. All plastic containers were equally given the same treatment

Analytical Methods:

Heavy Metals:

Analysis of heavy metals in soil and water samples [such as Lead (Pb), Copper (Cu), and Zinc (Zn)] was done using Perking Elmer Analyst 100 Atomic Absorption Spectrophotometer equipped with Perking Elmer HGA 850 Graphite Furnace and Perking Elmer AS 800 Autosampler with a computer interface for operation and readings display, Varian Spectra AAS with SpectrAA55.

Physical Parameters:

Total Dissolved Solids (TDS)

Effluent sample was stirred with a magnetic stirrer and a measured volume was taken onto a glass fiber filter with applied vacuum. It was washed with three successive 10ml volumes of distilled water, allowing complete drainage between washings, and suction was continued for 3 minutes after filtration was completed. Total filtrate with washings was

transferred to weigh evaporating dish and evaporated to dryness on a steam bath. Evaporated sample was dried in the oven for 1 hour at 180±2°C, cooled in a desiccators and weighed [16]. The TDS is calculated by finding the difference in the weights of Total Solids (W1) and Total Suspended Solids (W2) expressed in the same units gives Total Dissolved Solids (TDS) [5].

Turbidity:

To record turbidity, the nephelometer was calibrated using distilled water (Zero NTU) and a standard turbidity suspension of 40NTU. The thoroughly shaken sample was taken in the nephelometric tube and the value is recorded. If the turbidity of the sample was more than 40 NTU, then the sample was diluted and the dilution factor was accounted in final calculations [5].

Oil and Grease:

The water sample was acidified previously at the sampling site (1:1) HCL to pH<2 or lower (generally, 5ml is sufficient for 1 liter sample). 200ml wastewater sample was transferred to a separatory funnel followed by the addition of 30ml n-hexane. It was shaken vigorously for 30 minutes for layers to separate. The n-hexane layer was drained through a funnel containing 10g Na_2SO_4 on a filter paper already rinsed with n-hexane. The extraction was repeated twice using 30ml n-hexane for each of the extraction. The funnel was rinsed with 10ml n-hexane after filtration in each case. All the hexane layers were mixed in a crucible already dried and weighed to a constant weight. The crucible was placed on a water bath until content evaporated completely. After which the crucible was placed in an oven for 5minutes. It was later removed and placed in the desiccator to cool and weighed. This was repeated until a constant weight was obtained. Blank was determined by taking 100ml n-hexane through the whole procedure [16]

Total Suspended Solute (TSS):

Glass fiber filter paper of diameter 5.5cm was dried to a constant weight at 103-1050C in an oven, cooled to room temperature in a dessicator and the weight was noted. Gooch funnel and rubber adapter were fixed to a filtering flask, the glass fiber was placed into the Gooch funnel carefully with the aid of a pair of tongs. The wastewater sample was thoroughly mixed on a magnetic stirrer, after which 100ml was quickly measured into the filtering apparatus. After filtration elapsed, a pair of tongs was used to remove the glass fiber carefully from the Gooch funnel and then dried to a constant weight at 103-105°C. The weight was noted [5,16].

Chemical Parameters:

Dissolved Oxygen (DO):

The water sample was put in a250ml bottle, followed by the addition of 1ml $MnSO_4$ solution and 1ml alkali-iodide-azide reagent well below the surface of the liquid. It was stoppered with care to exclude air bubbles and mixed inverting. It was allowed to settle for 2 minutes, after which 1ml concentrated H_2SO_4 was added by allowing the acid to run down the neck of the bottle, restoppered, and mixed by gentle inversion until dissolution was completed. At this stage, the iodine must have been uniformly distributed through the solution. 201ml of the solution was titrated with 0.0125M $Na_2S_2O_3$. $5H_2O$ solution to a pale straw color. 1-2ml of starch solution was added, which turns the color blue; the titration was preceded by adding the thiosulphate solution drop wise until the blue color disappears. The reappearance of blue color was disregarded [5,16].

DO (mg/l) = 16000*M*V /V2/V1 (V1-2)

Where

M= molarity of the thiosulphate solution

V= volume of thiosulphate used for titration

V1= volume of the bottle with stopper in place

V2 = volume of aliquot taken for titration

Biochemical Oxygen Demand (BOD):

The method consists of filling the samples in airtight bottles of specified size and incubating them at specified temperature (20 ^{o}C) for 5 days. The difference in the dissolved oxygen measured initially and after incubation gives the BOD of the sample. The sample having a pH of 7 is determined for first day DO. Various dilutions (at least 3) are prepared to obtain about 50% depletion of DO. using sample and dilution water. The samples are incubated at 20 ^{o}C for 5 days and the 5th day D.O is noted using the Oximeter. A reagent blank is also prepared in a similar manner [5].

Calculation: (D1 - D2) - (B1 - B2) X f

BOD = -------------------------- (in mg/L) P

D1 - 1st day D.O of diluted sample

D2 - 5th day D.O of diluted sample

P - decimal volumetric fraction of sample used.

B1 - 1st day D.O of control

B2 - 5th day D.O of control

Chlorine (Cl⁻) Content:

Fifty grams of potassium chromate was dissolved in minimum amount of distilled water and silver nitrate was added drop wise till a red precipitate was formed. The mixture was allowed to stand for about 12 hours and diluted to 1000ml with distilled water. Then a known volume of filtered sample (50ml) was taken in a conical flask, to which about 0.5ml of Potassium chromate indicator was added and titrated against standard Silver nitrate till Silver dichromate ($AgCrO_4$) starts precipitating [5].

Calculation: (A-B) (N) (35.45)

Chlorides (Cl-) = --------------------   Sample taken in ml

Where,

A - Volume of silver nitrate consumed by the sample

B - Volume of silver nitrate consumed by the blank

N - Normality of silver nitrate

In principle, it is that, in alkaline or neutral solution, Potassium chromate indicates the endpoint of the silver nitrate titration of chlorides. Silver chloride was quantitatively precipitated before the red Silver chromate was formed [5].

Alkalinity:

Twenty five (25)ml sample was titrated with 0.01M H_2SO_4 acid solution using 3-5 drops of phenolphthalein indicator. The result was expressed as phenolphthalein alkalinity. Another 25ml sample was titrated with 0.01M H_2SO_4 with 3-5 drops of methyl orange indicator. Blank titration was carried out in both cases (APHA 1985; APHA 1992; Olaniyi *et al*., 2012).

Phenolphthalein alkalinity, mg/l CaCO3 = A*M*100,000/V

Where

A = volume of standard acid used

M= molarity of standard

Statistical analysis:

One-way ANOVA was used to analyze results of the different sample points and mean differences were sorted out based on Tukey-Kramer's Multiple Comparisons Test using InStat3 Software, 2013 (significant at $P<0.05$).Also,Paired sample t-test carried out to test for difference in means of concentrations (ppm) for both soil and water samples using SPSS software

3. RESULTS AND DISCUSSION

The result(s) and findings from the samples collected (from sample point **A**: effluent discharge point; **B**: pond that collect water from point A; **C**: stream flowing away from point B, with mining and farming activities around; **D**: stream flowing away from point C, with mining and farming activities around; **E:** stream flowing away from point D, with farming activities around; **F:** stream flowing away from point E, under the bridge of a main road towards less disturb place) are shown in the Tables below:

Table 1: Table Showing the Concentration (ppm) of Heavy Metals in Water and Soil Samples

	Copper (Cu)		Nickel (Ni)		Manganese (Mn)		Chromium (Cr)		Lead (Pb)		Iron (Fe)		Zinc (Zn)		Cadmium (Cd)	
Sample	Water	Soil	Water	Soil	Water	Soil	Water	Soil	Water	Soil	Water	Soil	Water	Soil	Water	Soil
A	0.20±0.03[b]	0.37±0.02[a]	0.98±0.02[b]	2.45±0.01[a]	2.06±0.01[a]	1.42±0.02[b]	1.66±0.01[a]	0.38±0.02[a]	13.94±0.02[a]	0.00±0.00[a]	2.88±0.02[b]	19.34±0.02[a]	6.84±0.02[a]	8.48±0.02[a]	4.27±0.01[a]	2.87±0.01[a]
B	0.54±0.02[a]	0.30±0.03[a]	2.77±0.03[a]	2.10±0.03[a]	0.79±0.02[a]	0.33±0.03[a]	3.45±0.02[a]	1.98±0.01[a]	1.88±0.02[a]	12.98±0.02[a]	2.28±0.01[a]	21.54±0.01[a]	27.39±0.02[a]	6.83±0.02[a]	6.68±0.02[a]	1.31±0.01[a]
C	0.24±0.03[a]	0.36±0.03[a]	1.96±0.03[a]	1.96±0.01[a]	0.00±0.00[a]	1.48±0.02[a]	1.09±0.01[a]	0.99±0.02[a]	6.17±0.01[a]	0.21±0.01[b]	6.67±0.01[a]	26.94±0.03[a]	8.94±0.01[a]	8.85±0.01[a]	3.48±0.02[a]	7.92±0.02[a]
D	0.14±0.01[a]	0.21±0.03[b]	1.32±0.01[a]	0.24±0.02[a]	0.00±0.00[a]	0.00±0.00[a]	3.11±0.01[a]	0.73±0.01[a]	17.36±0.02[a]	0.86±0.01[a]	5.49±0.02[a]	24.84±0.01[a]	0.45±0.03[a]	5.30±0.02[a]	2.78±0.03[a]	8.98±0.01[a]
E	0.48±0.02[a]	0.27±0.02[a]	2.10±0.01[a]	0.10±0.02[a]	0.20±0.02[a]	0.00±0.00[a]	2.28±0.02[b]	3.04±0.01[a]	8.17±0.02[a]	4.68±0.02[a]	3.96±0.01[a]	24.67±0.01[a]	3.96±0.02[a]	2.89±0.01[a]	4.78±0.01[a]	0.02±0.01[a]
F	0.19±0.04[b]	0.28±0.01[a]	2.55±0.02[a]	0.42±0.03[a]	0.00±0.00[a]	0.00±0.00[a]	1.54±0.02[a]	1.68±0.01[a]	9.70±0.01[a]	8.04±0.01[a]	3.51±0.02[a]	17.26±0.02[a]	3.69±0.02[a]	6.11±0.03[a]	0.24±0.02[a]	1.50±0.02[a]
Control	0.21±0.01	0.21±0.02	1.04±0.01	1.04±0.01	1.32±0.00	1.32±0.00	2.19±0.01	2.19±0.01	0.31±0.01	0.32±0.01	2.79±0.02	2.79±0.01	0.11±0.02	0.11±0.01	5.28±0.01	5.28±0.02

Values are mean $\pm$ standard deviation (n = 3). [a]Values in the same column differ significantly (P<0.05) when compared with control, [b]Values in the same column are not significantly different (P>0.05) when compared with control. One-way ANOVA was used to analyze the results and mean differences were sorted out based on Tukey-Kramer's Multiple Comparisons Test using InStat3 Software, 2013.

Table 1: Table Showing the Mean Concentrations Physicochemical parameters in Water samples

water sample	TDS mg/l	Turbidity NTU	Cl2 mg/l	TSS mg/l	DO mg/l	BOD mg/l	Oil & grease mg/l	Alkalinity (pp caco3)
A1	1394±1.00[a]	52±1.00 [a]	0.1±0.02 [a]	1.00±0.22 [a]	3.69±0.54 [a]	9.67±0.60 [a]	10.9±0.60 [a]	429±0.04 [a]
A2	4020±2.00 [a]	1062±2.00 [a]	2.7±0.54 [a]	12.4±0.02 [a]	3.24±0.25 [a]	34.16±0.20 [a]	161.2±0.10 [a]	464±0.03 [a]
A3	1569±2.52 [a]	728±2.50[a]	1.8±0.03 [a]	4.9±0.10 [a]	2.52±0.50 [a]	6.64±0.51 [a]	0.4±0.21 [a]	472±0.02 [a]
A4	778±1.00 [a]	174±0.10 [a]	0.7±0.10 [a]	0.2±0.04 [b]	3.66±0.20 [a]	[a]6.90±0.30 [a]	0.3±0.08 [a]	455±0.01 [a]
A5	491±1.00 [a]	36±0.03 [a]	0.6±0.04 [a]	0.5±0.03 [a]	3.58±0.22 [a]	2.97±0.22 [a]	0.2±0.02 [b]	391±0.02 [a]
A6	650±0.25 [a]	93±0.02 [a]	0.8±0.03 [a]	0.2±0.01 [b]	4.47±0.30 [a]	3.06±0.81 [a]	0.4±0.01 [a]	418±0.10 [a]
control	245±0.01	4.42±0.01	11.21±0.02	0±0.00	126±0.30	8.18±0.56	0±0.00	0±0.00

The Physico-chemical parameters weremeasured and recorded for the different sampling points analyzed. The samples were compared to a control sample and of all the parameters taken, five (TDS,BOD, turbidity,Oil and Grease and alkalinity) showed high significant values (p<0.05) when compared to the control sample at most of the points with their values peaking at points B and C . It is most likely that these values are observed due to the accumulation of effluent discharged and anthropogenic disturbance at such sample points. It is also clearly found that the concentration of dissolve oxygen (DO) at all sample points where significantly low(p<0.05) in comparison to that of the control sampleand biochemical oxygen demand (BOD) concentration is high at point B indicating pollution. The other parameters taken were found to be below that of the control sample which suggests increased effect in physico-chemical parameters due to effluent discharge into the environment.Also, a total of eight (8) heavy metals: Copper(Cu), Nikel(Ni), Manganese(Mn),

Chromium(Cr), Lead(Pb), Iron(Fe), Zinc(Zn), and Cadmium(Cd) were identified from the water and soil samples collected.[16] had reported the isolation of similar metals. All the eight isolated metals were identified in both water and soil samples collected and analyzed for heavy metals. Lead (Pb) was absent in soil sample A while Manganese (Mn) was not detected in water sample C,D & F and soil sample D,E &F . In reference to the control samples, all the water samples analyzed recorded Zinc (Zn) and Lead (Pb) with high significant difference($p<0.05$) while in the soil samples, Iron (Fe) was remarkably high significantly difference ($p<0.05$)in concentration followed by Lead (Pb). Of the eight isolated metals : Nickel(Ni), Chromium(Cr), Lead(Pb), Iron(Fe), Zinc(Zn), and Cadmium(Cd) were noted to occur above their respective permissible limits as approved by theFederal and State Environmental Protection Agencies in all sampling points.

The result of the paired sample t-test carried out to test for different in means of concentrations (ppm) between soil and water samples showed that only iron (Fe) has a highly significant difference ($P=0.001$, $t=-5.657$, $df=6$) in the mean concentrations of the soil and water samples across all the points analyzed. All other heavy metals showed no significant difference in their mean concentrations between soil and water samples respectively as compared to the control.

4. CONCLUSION

It has been established that Vegetable-oil-producing Industry effluent contains some hazardous substances especially heavy metals e.g. lead, cadmium and iron which have a huge impact on the environment they occur. It is obvious that the heavy metals extracted during the study were mostly introduced into the surrounding from the effluent of the vegetable-oil-producing industry and the increase in concentrations of the isolated heavy metals is also highly influenced by pond accumulation (point B) and mining and/or farming activities (points C, D & E) which resulted in mixing the various chemical content of the soil into the surrounding water bodies. This concentrations increase which was above the require FEPA permissible limits: for Oil and grease, Turbidity,BOD, Alkalinity,Pb, Fe, Zn, Cr, Cd and Ni all affects the physical and chemical nature of the streams associated with vegetable –oil-industrial effluent making it unfit for the survival of many aquatic life forms and most commercial and domestic use.

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