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DETERMINATION OF SOME PHYSICO-CHEMICAL PARAMETERS OF SEDIMENTS FROM ETHIOPE RIVER, DELTA STATE, NIGERIA.

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ABSTRACT

The physico – chemical properties of sediments collected from River Ethiope, Delta State, Nigeria were examined. Sediments were collected from eight sampling locations along the river which are Umutu 1, Umutu 2, Abraka 1, Abraka 2, Eku (Igun) 1, Eku (Igun) 2, Sapele 1, Sapele 2. pH, electrical conductivity, total organic carbon (TOC), total nitrogen, total phosphorus, potassium, calcium and magnesium were examined. Findings from this study revealed that pH ranged from 6.07 ± 0.00 to 8.73 ± 0.00; electrical conductivity 27.90 ± 0.00 to 110.05 ± 0.01µS/cm; temperature ranged from 28.60 ± 0.01 to 29.80 ± 0.01; total phosphorus 0.020 ± 0.00 to 0.163 ± 0.00; TOC range from 0.50 ± 0.02 to 8.80 ± 0.00%; total nitrogen 0.004 ± 0.00 to 0.030 ± 0.01; Potassium 0.69 ± 0.07 to 5.18 ± 0.02mg/kg; calcium 1.92 ± 0.01 to 7.88 ± 0.0 mg/kg, and magnesium 5.05 ± 0.01 to 12.32 ± 0.01 mg/kg. The results of the present investigations suggest that the present increasing pollution load resulting from rapid industrialization along the Sapele axis may not have affected sediments, but maylead to subsequent effects in the aquatic environment. The experimental data suggests a need to start implementing common objectives, compatible policies and programmes to discourage the discharge of untreated effluent into rivers and also for improvement in the industrial and domestic waste water treatment methods.

Running Title: Determination of Physico-Chemical Parameters of Sediments from Delta State
Key Word: Determination, Physical, Chemical, Properties, Sediment, River Ethiope,

INTRODUCTION

It is evident that natural earth materials consisting of soil particles of all sizes ranging from the smallest, "silt (mud) and sand", to larger sizes which include the gravels, cobbles, boulders, bedrock and clay are materials that succumb to the forces of erosion from water, wind and pressures caused by expanding and contracting forces (Okoh*et. al*. 2016). This natural earth materials are called Sediments. They could also be described as the breaking down of organic and inorganic materials (Bortone, 2007).Sediment deposited in Delta's and reservoirs are generally fine-grained - sand, silt, and clay (Edjere and Okpidi, 2016). Sediments contain nutrients vital for plant maintenance and growth, including nitrogen, phosphorus, calcium and magnesium (GoedkoopandPettersson, 2000). Organic carbon in sediments supports diverse microbial populations that are responsible for transforming and retaining nutrients (Hyland *et al*.,2005; Thompson & Lowe 2004). Okoh*et. al*., (2016) reported that sediment is a potential source of pollution for the aquatic body. They further reported that loose surface degraded sediments that are not held in place by plants could be moved into nearby aquatic systems, where they are then conveyed downstream to other land and water ecosystems. This thus affects the quality of water available for use. While Onwughara*et. al*., (2013) reported that contaminants such as bacteria, viruses, heavy metals, nitrate sand salt could also pollute water supplies as a result of inadequate treatment and disposal of waste from humans, livestock, industrial discharges, domestic discharge and extensive use of limited water resources. General survey reveals cases of typhoid, diarrhoea and other water borne diseases arising from the consumption of contaminated water. Different works have been reported by many researchers on water quality assessment, physico-chemical parameters of sediments and the assessment of metal concentration of water and sediment. Though, little work is done on the physico-chemical parameters of sediments and water quality index from Ethiope River, Delta State, Nigeria. Physico-chemical properties of the water gets varied season-wise and in addition, anthropogenic activities such as agriculture, urbanization, domestic sewage in the catchment area resulting to the deterioration of water quality (verma*et. al*., (2012). Temperature, turbidity, nutrients, hardness, alkalinity and dissolved oxygen are some of the important factors that play a vital role for the growth of living organisms in the water body (Pelczar*et al*., 2005). Water quality indicates the relation of all hydrological properties including physical, chemical and biological properties of the water body. Hence, water quality assessment involves analysis of physico-chemical, biological and microbiological parameters that reflects the biotic and abiotic status of ecosystem (Qureshimatva, *et. al.,* 2015). They further reported that the water quality index (WQI) provides a nominal number that represents overall water quality at a certain location and time, based on several water quality parameters. Hence, the objective of water quality index is to turn complex water quality data into detailed information useful for the public. WQI indicates the water quality in terms of index number and offers a useful presentation of overall quality of water for public or for any intended use as well as in the pollution abatement programmes and in water quality management. Thus, this study is aimed at assessing the physic-chemical of sediments from River Ethiope, Delta State, Nigeria.

Study Area

River Ethiope could be accessed from Warri through Abraka and from Asaba through Agbor, Umutu to Umuaja. It has its source at Umuaja in Ukwuani Local Government Area of Delta state. It then flows through communities such asUmuaja, Umutu,obi – Iloh,Ebedei-Ukwale, Owa-Abbi, Obinomba, Obiaruku, Umeghe, Urhuoka, Abraka P.O., Ajalomi, Urhuovie, Erho,Oria, Sanubi, Eku,Igun,Okpara Waterside, Ekpan-Ovu , Aghaiokpe, Arabga-Okpe, Adarweran, Egbeku, Ibada, Eko , Amukpe, Okirigwhre, Sapele, Jesse , Ogharain the State. It is ninety-six kilometers long.Fishing is one of the main activities that take place. The adjoining land which do not consist ofbuildings are use for crop farming. Some of the crops produced in this area are Maize, Yam, cassava, tomatoes and vegetables. (Okumagbaand Ozabor, 2014; Akporido and Kadiri, 2014)

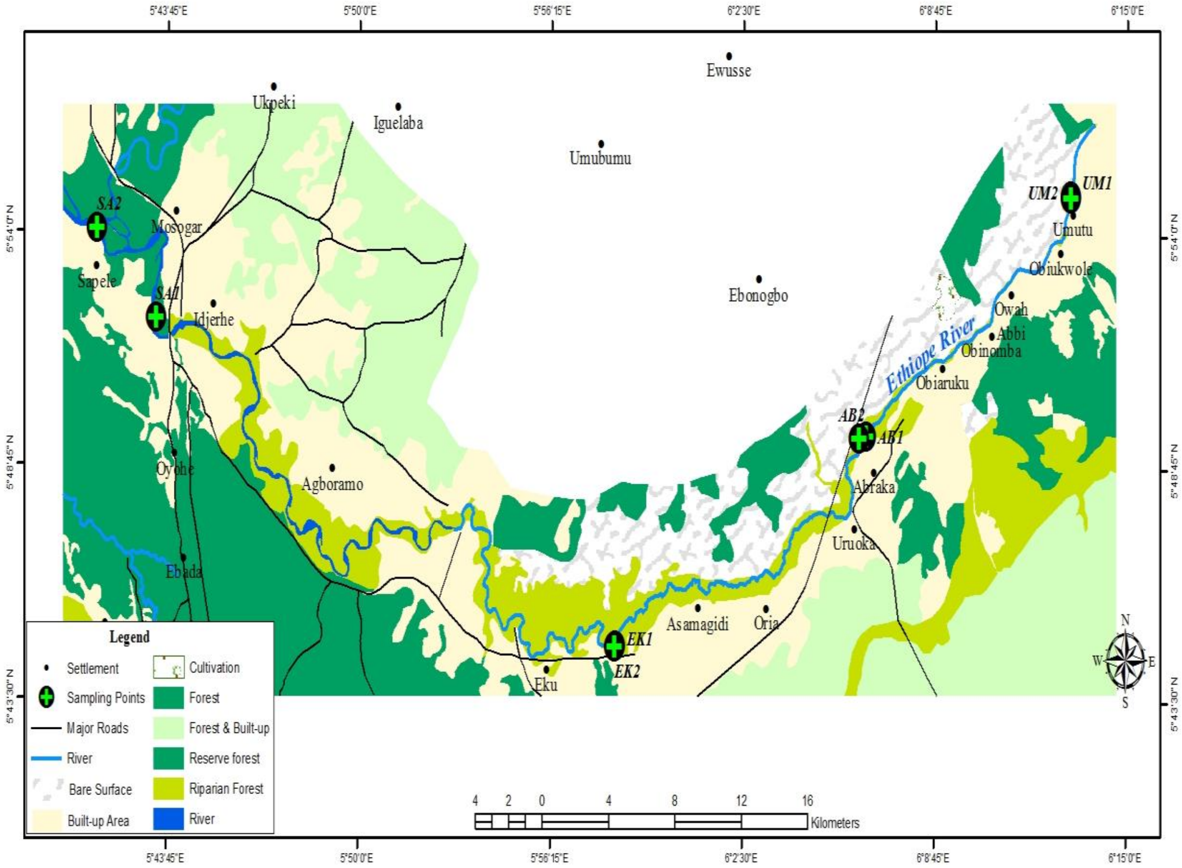

Figure 1: Map showing sampling locations

KEY:

UM = UMUTU
EK = EKU (IGUN)
AB = ABRAKA
SA = SAPELE

MATERIALS AND METHODS

Sampling

Sediment samples were collected in duplicate at eight points along the river which are Umutu1, Umutu 2, Abraka 1, Abraka 2, Eku (Igun) 1, Eku (Igun) 2, Sapele 1, Sapele 2. This was done using a grab sampler and kept in labelled polythene bags. The sediment cores retrieved in the field were sliced on arrival to the lab at 1cm depth intervals for the first 15 cm, 2-cm depth intervals from 15–25 cm, and then every 5 cm for the deeper sections of the cores. The sediments were kept cool in an icebox during the transportation to the laboratory. They were then ground manually to fine powder in an alumina mortar; it was passed through a 2-mm mesh screen and stored in polyethylene bags for analysis.

Sand separation (CANADA-MANITOBA SOIL SURVEY,2006)

8cm diameter sieve with openings of 53µm (270 mesh) was placed in a large funnel held above a cylinder. The sediments were washed on the sieve until the water became clear then the sand was transferred to a 100mL beaker and dried at 105°C, cooled and weighed.

$$\% Sand = \frac{Total\,Sand}{\mathrm{Recovered}\;Weight\,of\;Sample(g)}\,x100 \quad ------------(1)$$

Separation of silt and clay

The cylinder containing the silt and clay suspensions was made up to100 mL by adding distilled water, covered with a watch glass and placed in a water bath. It was then allowed to equilibrate and checked for flocculation before sampling. A perforated plunger was inserted to the bottom of the cylinder and stirred vigorously for several seconds to loosen the sediment. The plunger was removed and 25mL aliquot was drawn off a 10cm depth into a 50mL beaker and dried at 105^{o}C. Cooled in a dessicator and weighed as weight of silt, clay and dispersing agent.

$$\% Silt = \frac{Total\,Silt}{\mathrm{Recovered}\;Weight\,of\;Sample(g)}\,x100 \quad ------------(2)$$

The cylinder containing the silt and clay suspensions in the temperature controlled water bath was maintained until the silt greater than 0.002 mm settles below 10 cm depth.25 mL aliquot was removed from a 10 cm depth below the suspension surface into a 50mL beaker with arsine water from the pipette and then dried, cooled and weighed as weight of clay and dispersing agent.

$$\%\ Clay = \frac{Total\ \ Clay}{\mathrm{Recovered}\;Weight\,of\;Sample(g)}\,x100 \quad ------------(3)$$

Correction Factor: To allow for the weight of dispersing agent in the sample, 10 mL of the dispersing agent was diluted to 1000 mL in a graduated cylinder. 25 mL of the liquid was removed and dried, cooled and weighed as with the silt and clay fractions (D.A. = dispersing agent)

Calculations

Wt. of silt & clay in aliquot (corrected) = Wt of silt & clay & D.A. – Wt. of D.A.

Wt. of clay in aliquot (corrected) = Wt of clay & D.A. – Wt. of D.A.

Total clay in sample = Wt. of clay in aliquot (corrected) x 40

Total silt in sample = [Wt. of silt & clay (corrected) – Wt. of clay (corrected)] x 40

Total sand in sample = Wt of sand fraction

Recovered Wt. of sample = Total Sand (S) + Total Silt (Si) + Total Clay

pH Measurement/ Electrical Conductivity

The pH of the sediment was determined in the laboratory by using an Elico pH meter. Before pH determination, all laboratory wares used in the experiment were washed, soacked in dilute nitric acid then rinsed with distilled water several times. A 1:2 sediments - distilled water suspension are thoroughly mixed for 30 minutes on automatic shaker machine, then pH of this suspensions were recorded. Before measuring the pH of the sediments suspension, the pH meter was standardized. First the pH instrument was switched on for 15-20 minutes for warm up, the temperature adjusted to room temperature, then the combined electrode dipped in the buffer solution of pH = 4.0.The pH knob was moved to pH=4.0. After the calibration of the pH meter, pH of sediment samples were taken.(Singh *et al.,* 1999). For electrical conductivity, the 30 minutes stirred suspension was allowed to settle and the clear layer was measured by HI9819 multiparameter waterproof meter.

Available Nitrogen

Available nitrogen was estimated by alkaline potassium permaganate method. In kjeldhal flask 20 g of air dried crushed sediment sample was taken and added to 20 ml of distill water to moisten the soil. 1 ml of liquid paraffin and few glass beads were also added to avoid frothing and bumping. Also added to it was 100 ml of 0.32% $KMnO_4$ solution followed by 100 ml of 2.5 % NaOH solution. The flask was closed immediately. The contents in the Kjeldhal flask was distilled at a steady rate and the liberated NH_3 was collected in a receiving flask containing 20 ml Boric acid solution with mixed indicator.With the absorption of ammonia, the pink colour of Boric acid solution turned to green. 100 ml of the distillate was is collected in the receiving flask and then titrated against standard hydrochloric acid till the colour changed from green to pink (Thakur *et al.*, 2012).

Organic Carbon

Total organic carbon content of the sediment was determined using the Walkey - black method (Krishan,*et al.*, 2009). This method is based on the theory that the colour of a soil sample determines the organic carbon content of that soil. 0.5 g of sediment samples was sieved through a 2 mm mesh-size sieve and weighed into 250 mL conical flask. 10ml of potassium dichromate (K_2Cr_2O7) and 20 mL H_2SO_4 were added and left to stand for 30 min on asbestos after intermittent swirls. 100ml of distilled water (spectator ion) was added. To this was added 3-4 drops of ferrous indicator and it was titrated with 0.5N $FeSO_4$. $7H_2O$. If the soil sample is rich in organic carbon it will assume a greenish colour on adding all reagents and indicators but if it is not rich in organic carbon it will assume an orange colour. Upon titration, an organic carbon rich soil goes from green to light green and finally to maroon red or brown; that is the end point. Total organic carbon was then calculated thus:

$$Organic\ Carbon(\%) = \frac{Titre\ value\ 2 - Titre\ value 1\ x\ 0.195\ (factor)\ of\ blank\ of\ sample}{Weight\ of\ Soil\ Sample(g)}$$

Available Phosphorous

2.5g of sediment sample was put into a 50ml conical flask and to it was added 0.5g Dacro-G-60 activated charcoal (P-free). 50 ml of $NaHCO_3$ (0.5M) solution was added and the flask was shaken for 30 minutes on mechanical shaker and then filtered through Whatman No. 1 filter paper. 5ml aliqout of the extract was putin a 25ml volumetric flask and acidified with 5N H_2SO_4. Some drops of distilled water was added and 4ml of reagent B (ascorbic acid). After 10mins the intensity of the solution (blue colour) was read on the spectrophotometer at 600nm wavelength (Krishan,*et al.*, 2009). The available phosphorus was calculated using the equation below:

$$Available\ P\ (kg/ha) = \frac{R\ x\ F\ x\ 50\ x\ 2.24}{5\ x\ 2.5}$$

Where:
Weight of sample = 2.5g
Volume of extractant used = 50ml
Volume of filtrate used = 5ml
Absorbency = R
Absorbency from standard curve =A
Concentration of P from absorbency A = B ppm
Column conversion factor = 2.24
F (factor) = B/A

Available Potassium

The available potassium was analysedusing a flame photometer. 10 g of air dried sediment sample was put in a 250 ml conical flask and then 50 ml of 1 N neutral ammonium acetate solution was added to it and the flask was shaken for 10 minutes.It was filtered through Whatman filter paper No. 40. The sample wasthen filtered out and the volume was made up to 100 ml with ammonium acetate solution. Standard solutions of potassium weremeasured by the flame photometer and the readingsof absorbance were drawnagainst concentration. The ammonium acetate extract (leachate) of sediments were then measured in order to get the concentration of potassium from the standard curve.

Exchangeable Ca and Mg

Calcium and Magnesium(Page *et al.*, 1982) were determined by using EDTA titrimetric method. 0.5g of the sediment was dissolved in 20ml dilute HClin a beaker, this was left for 1 hour 30 minutes and the acid was neutralised by a dilute NaOH until the pH was almost 7. The solution was transferred to a 100ml volumetric flask and made up to mark by distil water.

Calcium and Magnesium ions (Ca + Mg) were determined by taking 25 ml of the digested sample into 100 ml of conical flask and diluted by adding about 25 ml of distilled water. Added to it was 4 ml of NH_4Cl + NH_4OH buffer. This solution was warmed to about 60^oC. Drops of EBT indicator were added and it was titrate with 0.01 N EDTA to a pure turquoise blue without any traces of red. This titre value was considered as “A”.

To determine the calcium ion, 10 ml of the digested sample was put in 100 ml conical flask and the content was diluted by adding 25 ml of distilled water. Also added to it was 5 ml of 10% NaOH solution to raise the pH to 12.0. and the solution was warmed to about 60^oC. Drops of hhnna indicator was added titrate the solution was titrated with 0.01N EDTA to a pure turquoise blue without any traces of red. This titre value was considered as “B”.

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The equations below were then used to calculate the values

$$Ca^{2+} + Mg^{2+}\ milli\ equivalents\ per\ litre\ (me/L) = \frac{ml\ of\ EDTA\ x\ Normality\ of\ EDTA\ x\ 1000}{Volume\ of\ sample\ used}$$

$$= \frac{A\ (titre\ value) x\ 0.01\ N\ x\ 1000}{Volume\ of\ sample\ used}$$

(1 ml of 0.01NEDTA = 0.01 me of Ca or Ca+Mg or Mg in givenaliquot).

$$Ca^{2+}(me/L)\ = \frac{ml\ of\ EDTA\ x\ Normality\ of\ EDTA\ x\ 1000}{Volume\ of\ sample\ used}$$

$$= \frac{A\ (titre\ value) x\ 0.01\ N\ x\ 1000}{Volume\ of\ sample\ used}$$

$$Mg^{2+}\ (me/L) = me\ /\ L\ (Ca^{2+} + {}^{Mg2+}) - me\ /\ L\ (Ca^{2+})$$

Quality Control/Assurance

Sediment samples were collected with plastic-made implements to avoid contamination. Samples were kept in polythene bags that were free from heavy metals and organics and well covered while transporting from field to the laboratory to avoid contamination from the environment. All reagents were standardised against primary standards to determine their actual concentrations. All instruments used were calibrated before use. Tools and work surfaces were carefully cleaned for each sample during grinding to avoid cross contamination. Duplicate samples were analyzed to check precision of the analytical method and instrument.

RESULTS AND DISCUSSIONS

The experimental data on physico-chemical properties of sediment samples collected from eight sampling location along river Ethiope, Delta State, Nigeria are presented in Table 1.

Table 1: Physical and chemical properties of the sediments.

STATION	% Sand	% Silt	% Clay	pH	Electrical Conductivity (µS/cm)	Temp. (^{0}C)	Total Phosphorus (%)	TOC (%)	Total Nitrogen (%)	K (mg/kg)	Ca (mg/kg)	Mg (mg/kg)
Abraka 1	68±0.30	21±0.51	10±0.11	7.46±0.02	43.10±0.08	29.2±0.02	0.020±0.00	0.50±0.02	0.020±0.01	1.27±0.05	3.21±0.01	9.94±0.02
Abraka 2	72±0.61	17±0.43	9±0.72	6.59±0.01	27.90±0.00	28.9±0.6	0.020±0.00	8.80±0.02	0.005±0.03	1.42±0.00	1.92±0.01	10.49±0.02
Umutu 1	66±0.09	20±0.25	13±0.60	6.07±0.00	63.00±0.01	28.8±0.04	0.040±0.00	2.90±0.00	0.023±0.00	5.18±0.02	7.88±0.01	8.55±0.05
Umutu 2	73±0.41	15±0.66	10±0.08	7.01±0.00	45.35±0.05	28.9±0.01	0.103 ±0.02	2.90±0.01	0.030±0.01	1.39±0.02	5.26±0.01	8.56±0.01
Eku 1	78±0.60	10±0.03	11±0.27	6.52±0.02	35.45±0.01	29.8±0.01	0.163±0.00	8.80±0.00	0.026±0.01	0.69±0.07	1.92±0.01	7.43±0.02
Eku 2	72±0.82	14±0.09	12±0.02	8.73±0.00	110.05±0.01	28.9±0.02	0.089±0.00	7.20±0.00	0.005±0.00	1.58±0.03	2.57±0.04	12.32±0.01
Sapele 1	69±0.31	21±0.15	9±0.20	8.33±0.06	70.55±0.02	28.6±0.01	0.031±0.02	7.30±0.01	0.029±0.00	1.89±0.01	2.57±0.00	7.39±0.03
Sapele 2	73±0.18	19±0.20	8±0.09	7.8±0.01	64.90±0.02	29.5±0.00	0.117±0.01	6.10±0.04	0.004±0.00	2.35±0.00	2.56±0.02	5.05±0.01
Mean	71.4±0.41	17.13±0.29	10.25±0.26	7.31±0.15	57.54±0.015	29.07±0.02	0.073±0.01	5.56±0.01	0.018±0.01	1.77±0.03	3.49±0.01	8.71±0.02

One of the parameters of utmost importance is pH, since most of the chemical reactions in aquatic environments are controlled by any change in its value. Anything either highly acidic or alkaline would kill marine life. Aquatic organisms are sensitive to pH changes and biological treatment requires pH control or monitoring. Surface waters having a pH value below six can be hazardous to aquatic life (Fisher and Wood, 2004). From Table 1 above, the pH of sediment samples ranged from 6.07 ± 0.00 to 8.73 ± 0.00. The mean pH is 7.31±0.15. It was observed that the pH of sediment collected from Eku 2 was highest with 8.73±0.00, closely followed with 8.33 ± 0.06. The pH value was minimum in Umutu 1 with 6.07 ± 0.00. It was reported by Qureshimatva*et. al.* (2015) that the pH in aquatic bodies ranged from 7.0 to 7.85 and stated that the pH of water is important for the biotic communities as most of the plant and animal species can survive in narrow a range of pH from slightly acidic to slightly alkaline conditions.A work done by Gao*et al.*(2012) stated that low pH in water affects oxygen level, thus aquatic organisms survive more in a slightly alkaline water environment.

It is well known that electrical conductance is a good measure of dissolved solids. Conductivity is a measurement used to determine mineralization of water. Certain physiological effects on plants and animals are often affected by the number of available ions in the water. In the present investigation, the electrical conductivity ranges from 27.90 ± 0.00 from the Abraka 2 location point to 110.05±0.01 from the Eku location point 2 with the mean conductivity value of 57.54±0.025.

Temperatureis another important characteristics of an aquatic system, affecting dissolved oxygen levels. The solubility of oxygen decreases as water temperature increases.The temperature of the sediment from the different sample points ranged from 28.60 ± 0.01 to 29.8 ± 0.01. The temperature variation along the sample point is not much leaving a mean average temperature of 29.07±0.02. It is a known fact that the rate of chemical reactions increase with increasing water temperature hence this has a great effect on the biological processes such as metabolism, growth, and reproduction and also the species composition of the aquatic ecosystem because many aquatic species can survive only within a limited temperature range. Changes in water temperature may act as a signal for aquatic insects to emerge or for fish to spawn as it is a determining factor of nitrification and nutrient supply to the organisms. The sediment station of Abraka 2 and Eku 1 with TOC values of 8.80±0.02 and 8.80±0.00 containcomparatively higher organic matter than other stations ,and the sediments fromAbraka 1 (TOC= 0.50±0.02) had lower organic carbon than the other seven stations. Sedimentsare primarily attributed to the relatively higher supply of organic matter from the abundant vegetation.

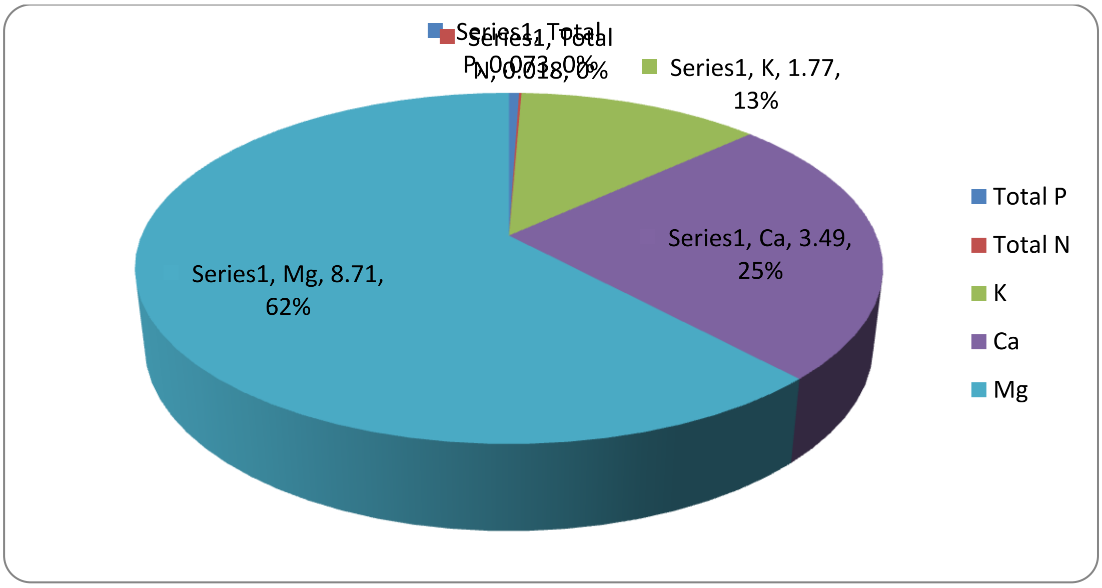

Fig.1: Average percentages of some trace elements in the sampling areas

The sediment from the different locations did not record significant concentrations of nitrogen. The mean concentration was 0.018±0.01. This could be attributed to the near zero domestic drainage nitrogenous fertilizers used bycultivators, industrial discharge, and also through moredecomposition and immobilization processes ofnitrogenous organic and inorganic matter within the region. The inhibition of nitrification with elevated pH, with decreases of 80 % at pH>9 relative to peaknitrification, has been observed in fine-grained sediments inthe Arika Sea (Isnansetyo*et al*., 2011).

The concentration of phosphate in sediments in all the locations was significantly very low. This amount could be attributed to low pollution discharge through streams from various tributaries into the Ethiope River. This is also attributed to the fact that the river is used as a resort and recreational center for neighboring and foreign institutions. Enhanced nitrogen and phosphorus fluxes may promote high levels ofphytoplankton biomass (Kemp *et al*., 2005). Such phytoplankton blooms lead to the sustained accumulation of phytodetritus in sediment, fueling nutrient recycling through organic matter remineralization (Cowan and Boynton, 1996;Nixon *et al*., 1996).The consequences, such as decreased water clarity, depletion of bottom-water oxygen and the decomposition of phytodetritus, may enhance sediment respiration,decrease redox potential, limit nutrient uptake by benthic microalgae, and generally increase nutrient fluxes (Kemp *et al*.,2005).The switch of sedimentary nutrient effluxes from high N to high P may reinforce N limitation andselectively support N_2-fixing cyanobacterial blooms. Givenhigher P demand for diazotrophs, the augmentation of Pflux with pH may boost the growth and persistence of algalblooms (Xie*et al*., 2003; Paerl, 2008).The eight sediment locations in this study had significant concentration of calcium and magnesium. The magnesium concentration ranged from 5.05±0.01 in the Sapele 2 location to 12.32±0.01 for the Eku 2 sediment location. The calcium concentration in the various sediment locations ranged from 1.92±0.01 from the Abraka 2 sediment location to 7.88±0.01 for the Umutu 1 location. The concentration of exchangeable calcium and magnesium may be due to the amount of exchangeable ionsin solution form of calcium and magnesium in sediment samples. It is also attributed to the weathering of minerals and their depositions in sediments.

A baseline study of the physic-chemical parameters of the sediments of Ethiope River in delta state Nigeria was conducted. Sediments collected from eight locations. The results of investigations suggest that the present increasing pollution load resulting from rapid industrialization along the Sapele axis may not have affected sediment. It may also be inferred that the sediment dwelling organisms have not yet been affected. The experimental data suggest a need to start implementing common objectives, compatible policies and programmes to discourage the discharge of untreated effluent into rivers and also for improvement in the industrial and domestic waste water treatment methods.

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