

# Physico-Chemical Quality of Reservoir Sediments Under Long-Term Amendment with Organic Manure

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## Abstract

Physico-chemical parameters of sediments samples collected from a 1.5 ha reservoir which was amended with organic manure over a fifteen year period were analysed and mean pH, conductivity, organic matter, nitrate and total nitrogen had values of  $7.57 \pm 0.80$ ,  $355.7 \pm 115.71 \mu\text{Scm}^{-1}$ ,  $8.78 \pm 1.83 \%$ ,  $12.12 \pm 5.53 \text{ mg kg}^{-1}$  and  $49.70 \pm 21.81 \text{ mg kg}^{-1}$ , respectively. Total phosphorus, available phosphorus and cation exchange capacity had mean values of  $74.61 \pm 41.50 \text{ mg kg}^{-1}$ ,  $12.46 \pm 5.84 \text{ mg kg}^{-1}$  and  $16.48 \pm 4.72 \text{ mmol cm}^{-1}$ , respectively. Total heavy metals were determined in the samples and mean Zn concentration was significantly higher ( $P < 0.05$ ) than the mean concentrations of other metals. The profile of mean metal concentrations in the bed sediments was  $\text{Zn} > \text{Cu} > \text{Pb} > \text{Ni} > \text{Cr} > \text{Cd}$ . Mean Cd, Cu and Zn concentrations exceed the USEPA (1997) threshold effect levels in sediments and this is due to the long term application of manure without the dredging of the reservoir.

## Keywords

Reservoir, Sediment, Manure, Physico-Chemical, Metals

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## 1. Introduction

The Integrated Fish Farming Reservoir of the National Institute for Freshwater Fisheries Research (NIFFR) has been in operation since 1983 and poultry (chicken and guinea fowl) farming is integrated with fish farming. It was observed that after a 15-year amendment with organic manures, aquatic primary productivity in the reservoir was not improving. The reservoir was not dredged within this fifteen year period, despite the annual loading of cattle and chicken manure.

Heavy metals easily get into animal feedstuff and are consequently discharged during excretion and defecation. Thus, sewage and animal manure serve as sources of heavy metals in water bodies either when applied directly or via surface runoff when used as soil manure. A study found high levels of Zn, Cu, Mn and Fe in poultry feed samples with Pb

concentrations exceeding the permissible limit of  $< 1 \text{ mg kg}^{-1}$  in the United Kingdom [1] while another study on poultry and livestock feeds and manure in Jiangsu province, China, reported that Cu, Zn, Pb, Cd and Cr concentrations in manures were high with Cu concentration in a manure sample as high as  $1726.3 \text{ mg kg}^{-1}$  [2]. Dissolved metals in waterbodies adsorb on particulate phase (sediments and suspended matters) due to the density of electrons on the surface of the particles or due to chemical reactions [3, 4, 5]. Metals are also remobilized from sediments into the overlying water column depending on pH and other conditions. Bottom sediment analysis offers certain advantages over water analysis for the control and detection of metal pollution and records the history of contamination and the types of contaminants present [6, 7]. The quality of bed sediment plays a crucial role in influencing the biological productivity of waterbodies since the overlying water is in dynamic equilibrium with bed sediment [8]. This study was

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conceived to determine the physico-chemical quality and heavy metal concentrations in the bed sediments of NIFFR integrated reservoir after a 15-year amendment with organic manure.

## 2. Materials and Methods

### 2.1. Study Area and Site

The experimental site was the 1.5 ha Integrated Fish Farming Reservoir of the National Institute for Freshwater Fisheries Research (NIFFR), New Bussa which has a national mandate to conduct research into all aspects of freshwater fisheries. New Bussa is in the semi-arid zone of Nigeria and is located between latitude 9°51'N – 10° 55' N and longitude 4°23'E – 4°45'E. It has a tropical continental climate characterized by a shorter wet season (May to September) and a longer dry season (October to April) with a temperature range of 15°C to nearly 40°C. Annual rainfall is about 1000 mm. The town is bordered by the Kainji Lake created by the damming of River Niger at Kainji. The vegetation in New Bussa is guinea savanna and the soil is generally alluvium but is highly variable in physical composition, low in phosphorus and nitrate but rich in potassium [9]. The annual manure loading into the reservoir was: chicken manure, 9600.84 ± 247.18 kg and cattle manure 451.60 ± 90.44 kg.

### 2.2. Sample Collection and Pre-treatment

Sediment samples were collected with Eckman grab from 10 randomly selected sites located within the 1.5 ha reservoir. Extraneous materials were removed from sediment samples. Each sample was divided into two parts and one part was air dried while the other was oven dried at 110°C. The air dried and oven dried samples were passed separately, through 2 mm and 0.2 mm sieves. They were then stored in dry acid washed polyethylene bottles with screw caps, at room temperature, prior to analyses. The 2 mm samples were used for the determination of pH, EC, total phosphorus, available phosphorus, total nitrogen and nitrate while the 0.2 mm samples were used for organic matter, CEC and metal analyses.

### 2.3. Quality Assurance

Glassware, plastic containers, crucibles, pestle and mortar were washed with liquid soap, rinsed with distilled-deionised water and then soaked in 10% HNO<sub>3</sub> solution for 24 hrs. They were washed with distilled-deionised water again and dried in a Gallenkamp drying oven at 80°C for 5 hrs. Analytical grade reagents and chemicals were used for this study. Distilled-deionised water was also used in all relevant analyses. All digestion and analyses were done in triplicate. Procedural and reagent blanks were used and a clean

laboratory environment was ensured during the analysis and preparation of solutions. The Shimadzu (model AA – 650) double beam digital Atomic Absorption Spectrophotometer (AAS) was calibrated with Multi-element standard Solution (MESS) and the calibration standards were analysed after 10 sample runs to ensure that the instrument remained calibrated.

### 2.4. Physico-Chemical Analysis

The pH of the dry sediments was determined by inserting a pre-calibrated digital pH meter into a 1:2.5 sediment-distilled water slurry and taking the reading. Electrical Conductivity was determined with the sodium hexa-metaphosphate method using a digital conductivity meter and taking the reading in  $\mu\text{Scm}^{-1}$  [10]. Organic Matter was analysed by ignition of the sample in a muffle furnace at 500°C for 4 hrs and subsequently cooling in a desiccator to achieve constant weight [11]. Nitrate-Nitrogen, NO<sub>3</sub><sup>-</sup>-N in sediment samples was determined with the Boric acid method which involved extraction, steam distillation and titration [10]. Total nitrogen (TN) was analyzed using the Kjeldahl method [10]. Available Phosphorus in the samples was determined with the Bray-1 colorimetric method [12] while total Phosphorus was analysed in each sample by digestion with perchloric acid, dilution of digest with distilled-deionised water, filtration of the resulting solution and utilization of the stannous chloride method to determine P in  $\text{mgkg}^{-1}$  [10,13]. Cation Exchange Capacity (CEC) in the sediment samples was determined with the ammonium chloride method which involves leaching with NH<sub>4</sub>Cl and NaCl solutions, washing with ethanol and distillation over boric acid. CEC was then calculated in  $\text{mmol kg}^{-1}$ . All analyses were done in triplicate.

### 2.5. Digestion of Samples for Total Metal Determination

Samples and blanks were digested in triplicate with a modification of the method of [14]. 5 g of sample was placed in a 250 ml conical flask and 3 ml of 30 % H<sub>2</sub>O<sub>2</sub> was added and allowed to stand for 1 h for the vigorous reaction to abate. 75 ml of 0.5 M solution of HCl was added and the flask was gently heated at 80°C for 2 h. The flask was allowed to cool, contents were filtered with Whatman No. 42 filter paper into a 50 ml volumetric flask and made up to mark with distilled/deionised water prior to analysis.

### 2.6. Statistical Analysis

Statistical analysis was done using the SPSS (version 15.0) for Windows software package. Mean concentrations and standard deviations were calculated for each parameter. The results were also subjected to analysis of variance (ANOVA) and means were compared using Duncan Multiple Range Test.

### 3. Results and Discussion

Physico-chemical parameters are shown in Table 1. The pH of sediment samples ranged from 5.60 to 8.25 and was mostly alkaline. pH expresses the intensity of acidity or basicity of a sample and sediment pH influences chemical reactions at the sediment water interface [15]. Sediment with slightly acidic to alkaline pH is favourable for fish farming. Electrical Conductivity (EC) of sediment samples had a range of 211 – 522  $\mu\text{Scm}^{-1}$ . Manure commonly contains 4 to 5% soluble salts (dry weight basis) and this may be as high as 10% in some cases. Application of manure to waterbodies will lead to the increase in the concentration soluble salts and the adsorption of same on sediments and this may account for the high values of EC in the samples [16]. Organic matter (OM) content of sediment had a range of 6.50 – 12.49 %. Total nitrogen (TN) ranged from 24.92 to 83.55  $\text{mg kg}^{-1}$ . Mean TN was significantly different ( $P<0.05$ ) from OM content but mean nitrate content, which had a range of 6.71 –

23.44  $\text{mg kg}^{-1}$  was similar ( $P>0.05$ ) to mean OM content. Total phosphorus (TP) and available phosphorus (AP) had ranges of 26.20 – 129.53  $\text{mg kg}^{-1}$  and 5.23 – 23.09  $\text{mgkg}^{-1}$ , respectively. Total phosphorus refers to the total concentration of P species in the sediment while available phosphorus refers to the labile pool of sediment phosphorus from which plants and animals draw to meet their phosphorus requirements. Mean TP content in the sediment was significantly higher ( $P<0.05$ ) than mean AP. Cation exchange capacity (CEC) of sediment samples ranged 10.80 to 21.50  $\text{mmol cm}^{-1}$  with a mean of  $16.48 \pm 4.72 \text{ mmol cm}^{-1}$ . Cations are held in the sediment by the negatively charged clay and organic matter particles through electrostatic forces and these cations are easily exchanged with other cations making them available for use by organisms. The cation exchange capacity (CEC) of a sediment sample represents the total amount of exchangeable cations that the soil can adsorb [17]. CEC increases as the amount of clay and organic matter of soil/sediment increases.

**Table 1.** Physico-chemical Parameters of Sediment Samples.

Parameters	Sampling Points										Mean $\pm$ SD
	1	2	3	4	5	6	7	8	9	10	
pH	7.62	7.50	7.74	5.60	6.89	7.95	8.21	8.06	7.90	8.25	7.57 $\pm$ 0.80 <sup>a</sup>
EC ( $\mu\text{Scm}^{-1}$ )	280.00	351.00	522.00	401.00	190.00	318.00	211.00	320.00	450.00	514.00	355.70 $\pm$ 115.71 <sup>b</sup>
Organic Matter Content (%)	9.32	9.14	8.09	7.11	6.50	8.20	7.44	9.31	10.20	12.49	8.78 $\pm$ 1.83 <sup>a</sup>
$\text{NO}_3^-$ ( $\text{mg kg}^{-1}$ )	7.59	8.40	6.71	8.36	10.11	12.44	10.30	14.92	23.44	18.90	12.12 $\pm$ 5.53 <sup>a</sup>
TN (%)	25.37	29.64	24.92	34.25	41.70	52.31	59.22	70.75	75.27	83.55	49.70 $\pm$ 21.81 <sup>c</sup>
TP ( $\text{mg kg}^{-1}$ )	26.20	28.72	27.70	45.50	100.16	89.00	129.53	111.52	125.70	62.08	74.61 $\pm$ 41.50 <sup>d</sup>
AV ( $\text{mg kg}^{-1}$ )	7.52	5.23	7.05	8.80	16.17	11.60	17.02	18.11	23.09	10.04	12.46 $\pm$ 5.84 <sup>a</sup>
CEC ( $\text{mmol cm}^{-1}$ )	14.30	16.50	10.80	20.90	21.50	14.80	16.20	18.10	20.40	11.30	16.48 $\pm$ 4.72 <sup>c</sup>

TN = total Nitrogen, TP = Total Phosphorus, AP = Available Phosphorus, CEC = Cation Exchange Capacity  
Means with different letters are significantly different ( $P<0.05$ )

**Table 2.** Total Metal concentrations in Sediments ( $\text{mgkg}^{-1}$ , dry Weight).

Metals	Sampling Points										Mean $\pm$ SD	USEPA (1997) limits in sediments ( $\text{mg/kg}$ ) TEL
	1	2	3	4	5	6	7	8	9	10		
Cd	2.309	2.821	1.807	3.411	1.096	2.955	1.871	1.911	2.730	2.826	2.374 $\pm$ 3.833 <sup>a</sup>	0.676
Cr	5.701	4.821	7.900	3.640	2.650	2.319	4.733	3.500	3.190	3.117	4.157 $\pm$ 1.680 <sup>b</sup>	–
Cu	64.026	51.644	38.850	27.319	36.773	45.072	77.290	49.331	80.630	72.141	54.308 $\pm$ 26.531 <sup>c</sup>	18.7
Ni	4.307	6.630	3.912	7.840	3.255	1.728	4.910	6.320	7.883	2.910	4.970 $\pm$ 2.120 <sup>b</sup>	–
Pb	15.101	10.221	8.700	21.805	15.321	13.452	15.469	16.230	18.590	19.787	15.508 $\pm$ 5.089 <sup>c</sup>	30.2
Zn	114.822	150.300	107.214	92.330	97.560	123.941	110.660	160.206	208.797	234.115	139.995 $\pm$ 64.603 <sup>d</sup>	124

TEL = USEPA (1997) threshold effects level

Means with different letters are significantly different ( $P<0.05$ )

Table 2 shows the result of total heavy metal determination. Mean Cd concentration was the lowest with a value of  $2.374 \pm 3.833$ , while Cr and Ni had values of  $4.157 \pm 1.680$  and  $4.970 \pm 2.120 \text{ mgkg}^{-1}$ , respectively. Zn had the highest mean concentration of  $139.995 \pm 64.603 \text{ mgkg}^{-1}$  followed by Cu and Pb which had mean concentrations of  $54.308 \pm 26.531$  and  $15.508 \pm 5.089 \text{ mgkg}^{-1}$ , respectively. Mean Zn concentration was significantly higher ( $P<0.05$ ) than those of other metals

The profile of mean metal concentrations in the bed sediments was  $\text{Zn}>\text{Cu}>\text{Pb}>\text{Ni}>\text{Cr}>\text{Cd}$ . Mean Cd, Cu and Zn concentrations exceed the USEPA threshold effect levels in sediments [18] and this is probably due to the long term application of manure without the dredging of the sediments. Metal contamination in sediments is of great concern due to metal toxicity even at low concentrations on aquatic organisms [19]. Sediments are important sinks for metals and

also play a considerable role in metal remobilization into the water column under suitable conditions and high concentrations of heavy metals poses health risks to humans if present as dissolved inorganic or organic species in the water [20, 21].

## 4. Conclusion

The physico-chemical parameters of the sediment had high values as a result of long term manure application. In addition, Mean Cd, Cu and Zn concentrations were above recommended levels and it is inferred that long term application of manure will lead to the accumulation of heavy metals above recommended levels. It is recommended that dredging of the reservoir should be carried out and it should be dredged after being used for a maximum of 5 years to prevent deposition of excessive amounts of metals.

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