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Determination of Extractable and Bound Organochlorine in Sediment by Instrumental Neutron Activation Analysis

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Authors' contributions

This work was as an aspect of the corresponding author's project, supervised by authors JAMA and SO. Author SA did the sampling, laboratory works and prepared the manuscript. Author JAMA reviewed the scientific background while author SO explained the data and dissected the results involved in the preparation of the manuscript. Authors EE, MT, GAM and BP helped with field and laboratory analysis. All authors read and approved the final manuscript.

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ABSTRACT

Extractable organochlorine (EOC) and bound organochlorine (BOC) compositions in sediments from Lake Bosomtwi have been determined with instrumental neutron activation technique. Within the limit of experimental errors, the precision and accuracy of the method for the determination were within 3% and 4% respectively. The mean levels of EOC and BOC were in the range of 1.71 mg/kg to 5.18 mg/kg and 0.31 mg/kg to 0.68 mg/kg respectively. Analysis of organochlorine data showed the EOC content on the average was about seven times more than those of BOC. Washing of chloride from sediment fortified with an insoluble chloride, thus $PbCl_2$, with various concentrations of sodium nitrate ($NaNO_3$) solutions was investigated and washing with 0.1 M solution of $NaNO_3$ gave the optimum yield of chloride. Organic carbon (OC) and BOC compositions in the sediments were subjected to correlation analysis and results showed that the two correlated very well with corrected coefficient of 0.65. OC in sediments could therefore, be one of the carrier phases of BOC in sediments.

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1. INTRODUCTION

Organochlorine compounds are produced synthetically by the action of elemental chlorine on hydrocarbon obtained from petroleum. Carbon-chlorine bonds are therefore, typically associated with persistent organochlorine pollutants [1]. These synthetic organochlorines are mainly pesticides, toxaphenes, PCBs, and dioxins [2]. The carbon-chlorine bonds are characteristically difficult to break, and the presence of chlorine atoms essentially reduces the reactivity of the other bonds in organic molecules [3]. This lack of reactivity is a distinct advantage in many applications. However, this same property means that once organochlorines enter the environment, they are slow to degradation and instead tend to accumulate. The lack of efficient sink for organochlorine compounds in addition to their hydrophobicity, has led to their accumulation in living organisms, including fish, humans and other animals [4]. Indeed, the entire planet, including all living things are said to have undergone some level of contamination by these chemicals [5]. Organochlorines are noted for their adverse human effects including reproductive failures, birth defects, immune malfunction, endocrine disruption and carcinogenic effects [6].

Analysis of organochlorines (OCs) in the sediments and other environmental samples typically involves vigorous extraction with suitable organic solvents or solvent systems. During extraction of OCs from the sediment and related matrices, extractable organochlorines (EOC) are those that come into the organic phase. As OCs age in the environment, they tend to bind to some environmental matrices. Indeed, in the terrestrial environment, they typically bind to soil matrices whereas in water bodies, they are likely to bind to sediments. The binding is such that during extraction of OCs, irrespective of the extraction method, they remained bound to the matrices. Such OCs which remained bound to the matrix after solvent extraction is referred to as bound or non-extractable organochlorine. Clearly, the formation of bound or non-extractable OCs in the environment ought to be appreciated as their formation eventually reduces the proportion of the OCs available for environmental re-cycling. This in turn potentially reduces the amounts that enter the food chain [7].

Characterization and quantification of individual organochlorine in the extracts using gas chromatograph-electron capture detector (GC-ECD) technique has witnessed and will continue to witness numerous applications [1,8,9]. Chromatographic method cannot however, be useful in the quantification of total extractable and bound organochlorine. Such determinations require the use of analytical technique such as instrumental neutron activation analysis (INAA). Although Kwablah et al. [6] and Gustavson and Johnson [10] for instance, determined extractable organohalogen by INAA, this method for determination of extractable organochlorine (EOC) and bound organochlorine (BOC) has not witnessed wide application. It is against this background that INAA was validated and applied for the assessment of EOC and BOC in sediment. It must however, be stressed that neutron activation analysis has been a proven and excellent technique for the determination of halogens such as Cl, Br and I [10]. Neutron activation analysis can therefore, be very useful for the determination of total halogen, extractable organohalogen and total organically bound halogen compounds.

Neutrons interact with elemental nuclei over a wide range of incident energies. The neutron having no charge can penetrate the coulomb field surrounding the nuclei. When a sample is exposed to a flux of thermal neutrons (Φ) for a period of time t, small fractions of the various stable isotopes of the elements present will capture some of the neutrons and the mass number of the element will be increased by one and the element get to the excited state. When it de-excites, it emits gamma ray (γ-ray) of characteristic energies [11]. Thus elements have their characteristic γ energies. This can be represented by hypothetical equation below:

$$ {}^{A}_{Z}X + {}^{1}_{0}n \rightarrow {}^{A+1}_{Z}Y + \gamma \quad (1) $$

The product Y is radioactive and therefore, it will decay. Thus, at the end of irradiation, the product nuclides decay as per their half-lives. The decay time is termed as the cooling time.

In this study, INAA was validated using two reference standard materials, and the validated method was applied for the determination of total extractable and bound organochlorine in sediment samples from Lake Bosmotwi. Lake Bosomtwi was selected to establish the burden of organochlorine contamination in the basin and the sediment was used for the study as sediment readily serves as sink for organochlorine and therefore, can be used as a monitor to indicate organochlorine environmental contamination.

2. MATERIALS AND METHODS

2.1 Study Area

The sediment samples for this study were collected from Lake Bosomtwi (Fig. 1). The Lake is situated within an ancient meteorite impact crater approximately 8 km across and the only natural Lake in Ghana [12] with a combined population of about 70,000 people [13]. Lake Bosomtwi is a natural in Ashanti Region of Ghana, and the local people consider it as a sacred Lake. The Lake exhibits a radial drainage system of 106 km^2, a diameter of about 11 km at its widest part and a maximum depth of 78 m. Lake Bosomtwi covers an area of about 52 km^2 [14]. The Lake has no outlet, although it has apparently overflowed in recent geologic past [15]. Lake Bosomtwi is fed by ground water which drains into it, by rainfall and run-off. It is also believed to be fed by river Aberewa.

2.2 Sampling and Sample Treatment

Sampling was done in 2013 from January to February and from September to October for the purpose of duplicating measurements. Sediment samples were collected at various locations as shown in Fig. 1 using Ekman grab from a depth of about 30 cm. Sediment samples were wrapped in pre-cleaned aluminium foil to prevent cross contamination and then bagged in polyethylene bags. The sediment samples were air dried and milled with pestle and mortar and sieved using 500 µm mesh size sieve to remove stones and other foreign materials. The sieved samples were then wrapped in aluminium foil and kept at room temperature in a clean cupboard.

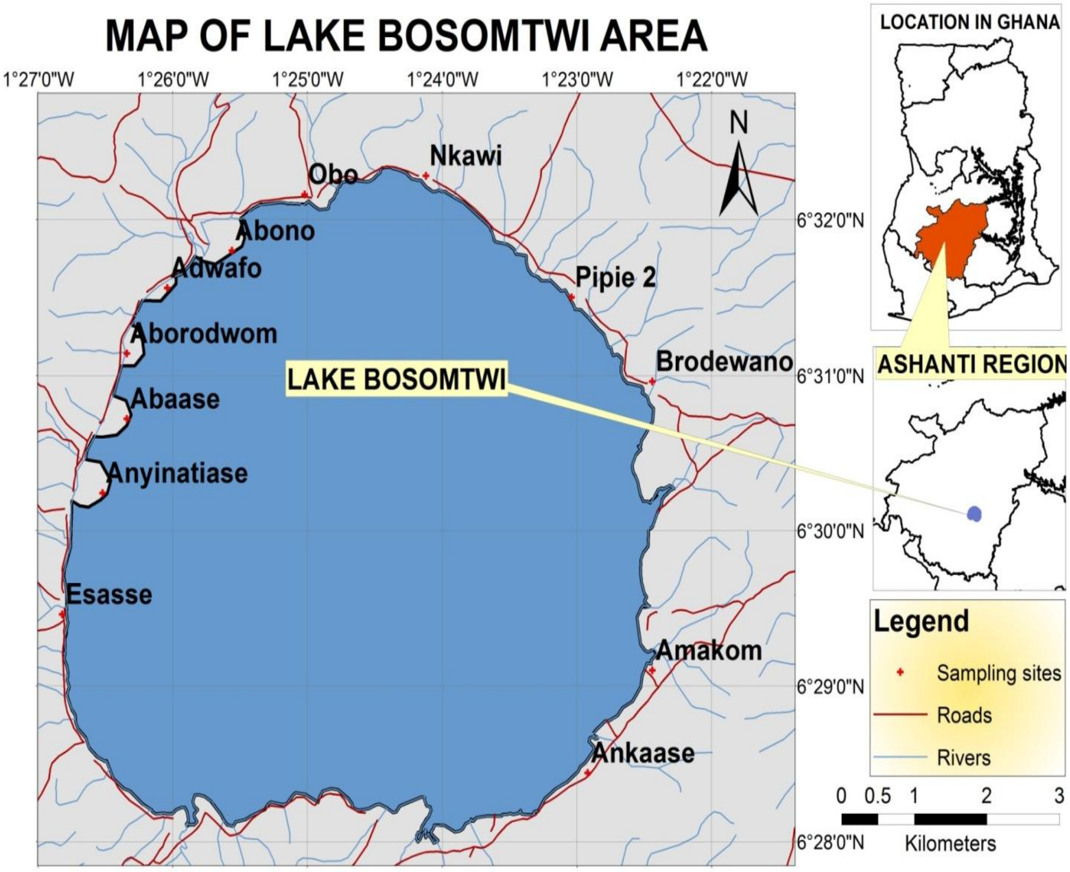

Fig. 1. Map of Lake Bosomtwi showing the sampling location

2.3 Determination of Organic Carbon and Organic Matter of Sediments

Analysis of organic carbon was based on the method of Walkley and Black modified by Page [16]. About 1 g of the sediment sample was weighed into 250 ml conical flask and 10 ml of 1 M $K_2Cr_2O_7$ solution was added to the sample in the flask. Twenty milliliter (20 ml) of concentrated H_2SO_4 was then added to the content of the flask. After allowing the mixture to stand for about thirty minutes, 10 ml of de-ionized water and 10 ml orthophosphoric acid were added. Finally, 2 ml barium diphenyl amine was added as indicator. The content was titrated against 0.2 M $A = \pi r^2$ ammonium ferrous sulphate $[Fe(NH_4)_2(SO_4)_2.H_2O]$ solution until the colour changed to green and finally to reddish brown. Blank determination was carried out similarly, but without the sample. Percentage organic matter was obtained by multiplying the percentage of organic carbon by the Van Bemmelen factor, 1.724 [17]. Van Bemmelen factor is used because organic matter contains 58% carbon. The method is basically the reduction of $Cr_2O_7^{2-}$ by organic carbon and subsequent reduction of unreduced $Cr_2O_7^{2-}$ by redox titration with Fe^{2+}. The reaction involved is as shown by the chemical equation 2:

$$2Cr_2O_7^{2-} + 3C + 16H^+ + Fe^{2+} \rightarrow 3CO_2 + Fe^{3+} + 8H_2O + 4Cr^{3+} \ldots \quad (2)$$

The percentage organic carbon was calculated using equation below:

$$\%\ \text{Organic carbon} = \frac{\text{Vb} - \text{Vs x M x 0.39}}{\text{Weight of sample}} \quad (3)$$

V_b = titre volume (ml) of blank, V_s = titre volume (ml) of sample.
M = molarity of hydrated ferrous ammonium sulphate.

The empirical factor 0.39 = 3 x 10^{-3} x 100 x 1.3. 3 is the equivalent weight of carbon, 1.3 is the due to 77% recovery of carbon.

2.4 Validation of INAA Method

The INAA method was validated by determining the precision and accuracy of the method using two certified standard reference materials, NIST – SRM 1547 (peach leaves) and NIST – SRM 1556^b (oyster tissue). Accuracy was calculated as percentage relative error while, precision was computed as percentage relative deviation. Two hundred milligram (200 mg) of the SRM was weighed unto polyethylene sheets, wrapped and heat sealed. Samples were irradiated in the Ghana Research Reactor – 1 (GHARR-1) at the Ghana Atomic Energy Commission, operating at a power of 15 kW at a neutron thermal flux of 1 x 10^{11} ncm^2 S^{-1}. Samples were transferred into irradiation sites via pneumatic transfer system at a pressure of 60 psi. The irradiation was categorized according to the half-life of the element of interest. The reaction of interest for this work is as presented by the equation 4:

$${}^{37}_{17}\text{Cl} + {}^{1}_{0}\text{n} \rightarrow {}^{38}_{17}\text{Cl} + \gamma \ldots\ldots \quad (4)$$

Since ^{38}Cl is a short lived radionuclide with half-life of about 37.3 minutes; samples were therefore, irradiated for 120 seconds after which radioactivity measurement of induced radionuclide was performed by a PC-based γ - ray spectrometry and counting was done for 10 minutes.

2.5 Counting of Irradiated Samples

The irradiated certified SRM were counted by using a computer based gamma-ray spectroscopy system, consisting of an N-type High Purity Germanium (HPGe) Detector model GR 2518 mounted on liquid nitrogen as a coolant, high voltage power supply model 3103, spectroscopy amplifier model 2020, ACCUSPEC multi-channel analyzer simulation software card (all manufactured by Canberra industries inc.), and a micro computer for data acquisition, evaluation and analysis.

2.6 Determination of Extractable Organochlorine in Sediment

The method of Ho-Sang et al. [18] with modifications was adopted for the determination of EOC. The EOC in the samples were extracted by sonicating 2.5 g of the sample on sonication bath for 3 hours at 40°C with 50 ml of 3:1 hexane/acetone solvent system. The residual sediment after filtration was dried at room temperature and packed in pre-cleaned aluminium foil for determination of BOC. Extracts were subjected to INAA as described in section 2.3 and 2.4 for determination of EOC.

2.7 Determination of Bound Organochlorine (BOC)

The samples that were kept for determination of BOC were washed several times with distilled water and 0.1 M $NaNO_3$ solution [19] to remove any residual inorganic chlorine which might be present in the samples. The samples were then dried at room temperature and then subjected to irradiation and neutron activation analysis.

2.8 Optimization of the $NaNO_3$ Washing Method

The $NaNO_3$ washing procedure was optimized by determining its concentration that would give optimum yield for chloride. Five (5) gramme of the sediment was weighed and transferred into filter paper placed in a funnel. The sample was washed with deionized water several times to ensure the removal of soluble inorganic chloride. The sample was then dried at room temperature overnight after which it was treated with 0.5 g $PbCl_2$. Treatment was done by mashing the sediment and the $PbCl_2$ with pestle and mortar. The lead chloride treated sediment sample was then washed with the various prepared concentrations of $NaNO_3$. The chemical reaction which takes place during the washing process can be represented by the equation below:

$$\mathrm{PbCl_2} + 2\mathrm{NaNO_3} \rightarrow 2\mathrm{NaCl} + \mathrm{Pb(N0_3)_2} \quad (5)$$

The chloride content in the washed solution was then determined by titrating with $AgNO_3$ solution using potassium chromate (K_2CrO_4) as indicator.

2.9 Data Analysis

Statistical analyses used for the data analysis include subjecting the measured extractable and bound organochlorines to descriptive statistics for the deduction of minimum, maximum and mean concentrations of organochlorines. Data was further subjected to variance to determine the differences in organochlorine concentrations. All tests were regarded as statistically significant when $p < 0.05$.

2.10 Analytical Quality Assurance/Control

A number of quality control measures were followed from initial sampling process to the final analyses of the samples using INAA equipment. Precautions were taken to reduce contamination during the handling and preparation of samples. All reagents were of analytical grade and sample containers and apparatus were washed and rinsed thoroughly prior to their use. Since reagents could be reliable sources of contamination in analytical work, high purity reagents and distilled water were used in this work. Blank samples were prepared and analyzed in the same manner as the samples in each batch of analysis in order to check the possibility of interference or contamination. Sampling and sample analyses were performed in duplicate. The INAA was validated by analyzing standard reference materials (SRMs), NIST – SRM 1547 (peach leaves) and NIST – SRM 1556^b (oyster tissue) and the accuracy and precision of the INAA method were then computed.

3. RESULTS AND DISCUSSION

3.1 Validation of Instrumental Neutron Activation Analysis (INAA) Method

The results obtained from the validation of INAA method by analysis of certified standard reference materials (SRM) are presented in Table 1. Uncertainties associated with the measurements are mean deviations. Results of the present study were compared with the reported values for the two SRM. The reported values [20] and the measured values within the limit of experimental errors agreed favourably as shown in Table 1. The accuracy of the method for the determination of chlorine nuclide computed as percentage relative error was within 4% while that of the precision calculated as percentage relative deviation was within 3%. The method was, however, more precise for determination of chlorine content in NIST-SRM (Oyster tissue) with 1.35% precision compared to NIST-SRM 1547 (Peach leaves) with 2.80% precision. The nuclear data for determination is presented in Table 2.

Table 1. Analysis of SRM for determination of precision and accuracy of INAA

Reference material	Measured (mg/kg)	Reported (mg/kg)	Precision (%)	Accuracy (%)
NIST-SRM 1566b (Oyster tissue),	0.494±0.007	0.514±0.01	1.35	3.89
NIST-SRM 1547 (Peach leaves),	366.3±8.060	360.0±19	2.80	- 1.75

Table 2. Nuclear data for determination of chlorine nuclide

Nuclide characteristics	Nuclear data
Nuclide	^{38}Cl
Isotope abundance (%)	24.23
$T_{1/2}$ (min)	37
t_i (s)	120
t_d (min)	1 - 10
t_c (min)	10
Energy (kev)	1642.4, 2167.5

$T_{1/2}$ =half life, t_i =irradiation time, t_d =decay time, t_c =counting time

3.2 Optimization of the $NaNO_3$ Washing Method

Fig. 2 shows the results when the $NaNO_3$ method for washing of insoluble chloride was optimized by varying the $NaNO_3$ concentrations from 0.02 M to 0.25 M. The results as presented in Fig. 2 indicated that higher yield of chloride was achieved when 0.1 M $NaNO_3$ was used for washing. Further increased in the concentration of $NaNO_3$ from 0.1 M as shown in the figure progressively reduced the yield of chloride. Therefore, 0.1 M $NaNO_3$ was selected for the washing procedure. During the washing process with $NaNO_3$, the insoluble chloride is converted to sodium chloride which then comes into solution.

3.3 Extractable OC and Bound OC Compositions in the Samples

Statistical data showing the mean, the standard deviations as well as the range indicating the minimum and maximum compositions for extractable and bound organochlorine in the samples are presented in Table 3. Clearly, there were not significant differences in the levels of EOC in the samples. The mean levels of EOC ranged from 1.71 mg/kg to 5.18 mg/kg. The highest level of 5.18 mg/kg was recorded in sediments from Aborodwom. The mean level of BOC on the other hand ranged from 0.31 mg/kg to 0.68 mg/kg with sample from Aborodwom also recording maximum BOC composition. Close examination of the results show higher EOC content yielding corresponding higher BOC, while lower EOC yielded corresponding lower BOC, an indication that there may be relationship between the two OC types. In general, the results suggest that the higher the EOC composition, the higher the corresponding BOC content. Analyses of the results showed that the ratios of EOC to BOC were in the range of 4.39 to 9.77, approximately averaging about 7.0. Thus, the composition of EOC in the samples on the average was about 7.00 times higher than the corresponding BOC content.

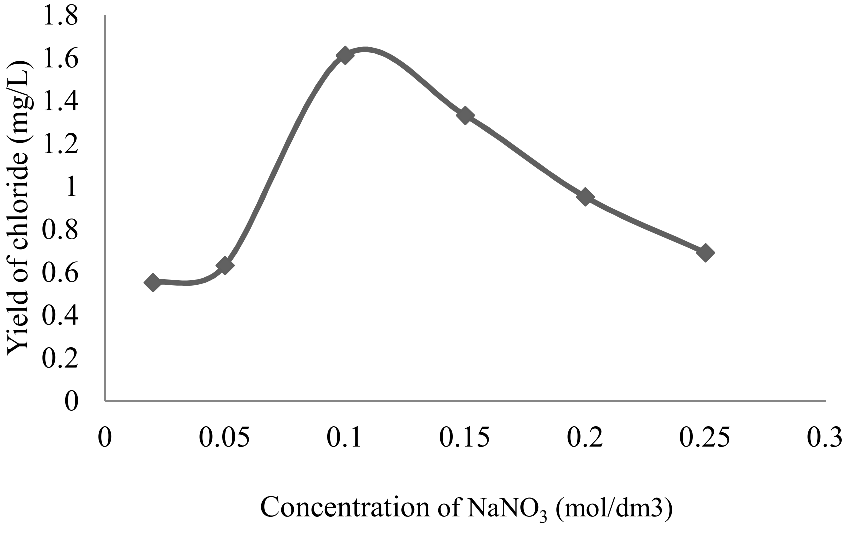

Fig. 2. Yield of chloride at various concentrations of $NaNO_3$

Table 3. Extractable and bound organochlorine (mg/kg) in the sediment compartment

	Extractable			Bound			
Locations	**min**	**max**	**mean±SD**	**min**	**max**	**mean±SD**	**EOC/BOC**
Esasse	2.20	4.76	2.47±0.23	0.20	0.65	0.48±0.04	5.15
Anyinatiase	2.06	3.70	2.44±0.21	0.21	0.45	0.39±0.04	6.25
Abaase	1.75	3.99	2.83±0.36	0.37	0.60	0.47±0.03	4.72
Aborodwom	4.62	6.66	5.18±0.56	0.43	0.68	0.73±0.11	9.77
Obo	2.55	4.85	3.88±0.34	0.26	0.59	0.53±0.08	6.57
Nkowi	1.04	3.61	2.37±0.29	0.24	0.47	0.36±0.06	5.04
Pipie 2	0.66	2.18	1.71±0.22	0.18	0.39	0.31±0.02	4.39
Abonu	1.51	4.32	3.02±0.32	0.20	0.51	0.39±0.05	5.92
Adwafo	2.23	3.83	2.96±0.25	0.28	0.60	0.59±0.06	5.02
Amakom	1.62	3.86	3.49±0.37	0.20	0.63	0.56±0.05	6.23
Ankaase	2.26	4.10	3.05±0.33	0.27	0.52	0.43±0.05	7.09

The results of ratios for EOC to BOC suggest that there might be a relation between the two OC types. The relation was determined by correlation plot of EOC against BOC as shown in Fig. 3. The plot suggests strong correlation between the two with coefficient of correlation (R^2) of 0.8. Thus, the plot is described by linear correlation as shown in equation 6:

$$\mathrm{EOC} = 6{,}7241\mathrm{BOC} - 0.1179 \quad \ldots \qquad (6)$$

Fig. 4 presents the percentage composition of extractable and bound organochlorine in the samples. EOC accounted for 82.0 to 91.0% of total organochlorine while BOC composition on the other hand, accounted for 9.0% to 18.0% of the total organochlorine content.

3.4 Relationship between Bound Organochlorine (BOC) and Organic Carbon (OC) in the Sediment

The movement of organic pollutants such as organochlorines (OCs) can be affected by factors such as relative concentrations of the pollutants and the carrier phases of the pollutants during transport [21,22,23]. Organochlorines being organic compounds are therefore, likely to interact with the carbonaceous materials such as organic carbon, black carbon and related substances in sediments. Against this background, the relationship between organic carbon and the corresponding BOC in the sediments was investigated. Result of plot of OC against BOC is presented in Fig. 5. The plot reveals correlation between OC and BOC with coefficient of correlation of 0.647. The correlation is a linear equation as shown below:

$$\mathrm{OC} = 1.755\mathrm{BOC} - 0.2691 \quad \ldots \qquad (7)$$

The results therefore, suggest that organic matter (58% of organic matter is organic carbon) in sediments to a higher degree could be one of the carrier phases of OCs. Organic matter in sediments could therefore, facilitate the formation of BOC.

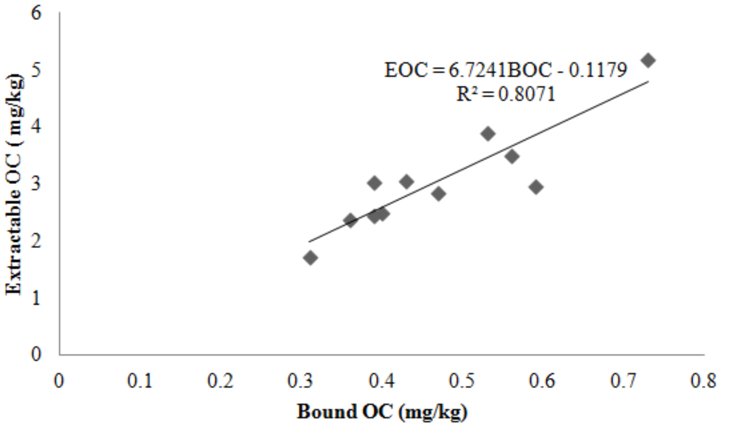

Fig. 3. Correlation plot between EOC and BOC

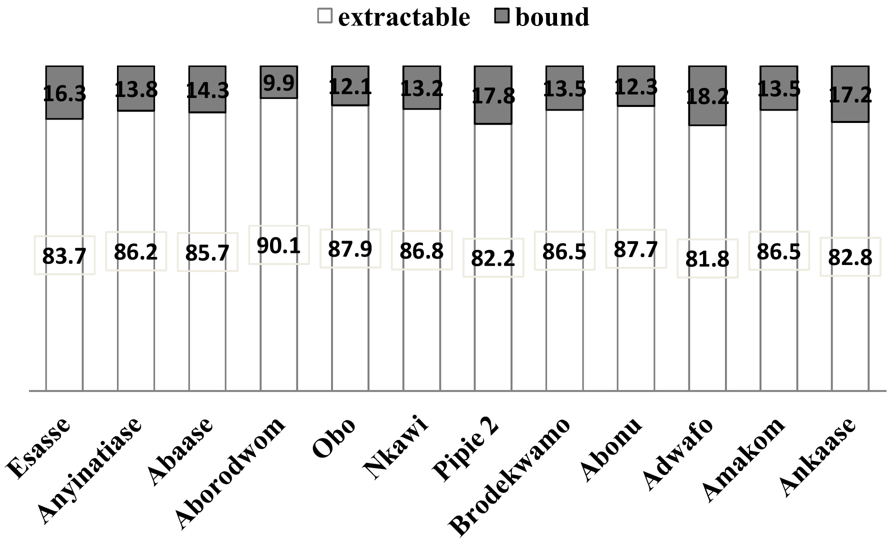

Fig. 4. Percentage composition of extractable and bound OC

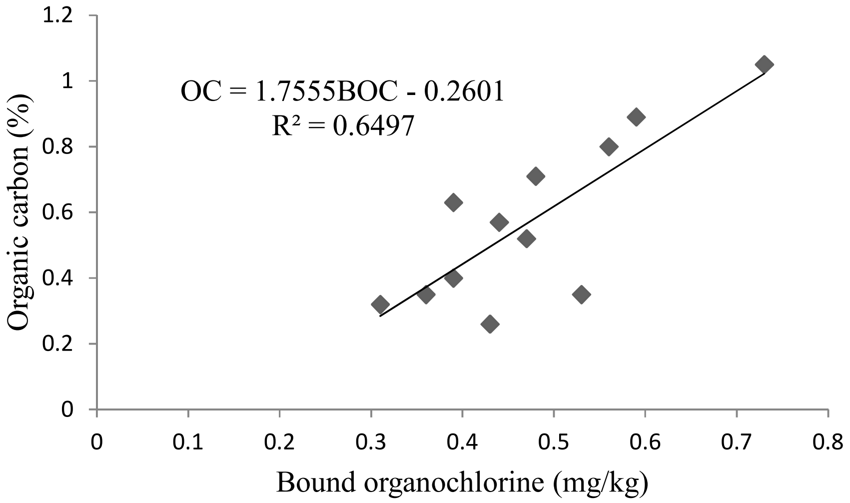

Fig. 5. Correlation plot of organic carbon and bound organochlorine

4. CONCLUSION

Instrumental neutron activation analysis (INAA) presented is very suitable and reliable for the determination of extractable and bound organochlorine in sediments. The composition of extractable organochlorine in the sediment on the average was about seven times bound organochlorine content. Extractable organochlorine accounted for 82.0 to 91.0% while bound organochlorine accounted for 9.0% to 18.0% of the total organochlorine content. 0.1 M $NaNO_3$ gave the optimum yield of chloride when the $PdCl_2$ fortified sediment was washed with various solutions of $NaNO_3$. Correlation analysis shows that extractable and bound organochlorine relate very well with coefficient of correlation which can be approximated to eighty percent. Organic carbon and bound organochlorine correlation investigation has shown that organic carbon in sediments could be one of the carrier phases of bound organochlorine in sediments.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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