Journal of Ecology and the Natural Environment Vol. 3(12), pp. 392-399, 26 October, 2011
Available online at http://www.academicjournals.org/JENE
ISSN 2006 - 9847©2011 Academic Journals

Full Length Research Paper

Organochlorine and organophosphorus pesticide residues in water and sediment from Yala/Nzoia River within Lake Victoria Basin, Kenya

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Accepted 29 September, 2011

This study set out to survey pesticide usage and concentrations of their residues in lower Yala/Nzoia catchment areas of Lake Victoria, Kenya during the dry and rainy seasons of 2009. Water and sediment samples were analyzed for selected organochlorine and organophosphorus pesticide residues using gas chromatography equipped with electron capture detector. The findings of the survey showed that the banned organochlorines are still being used in the catchment. Pesticide residue levels of organochlorines in water samples from Yala/Nzoia Basin were below detection limit (BDL) both during the rainy and dry seasons. The residue levels detected in sediment samples collected during the rainy season ranged from 0.05 to 59.01 µgkg^{-1}, whereas during the dry season, they ranged from BDL-24.54 µgkg^{-1}. The concentrations of dieldrin and p,p'-DDD were notably higher ($p < 0.05$) than aldrin and p,p'DDT, respectively, in most of the samples. No organophosphates were detected in any of the water and sediment samples. Organochlorine residues detected in sediment in both seasons were below the World Health Organization recommended guidelines. Therefore, it was concluded that there were neither environmental nor human health hazards posed by these compounds in the water and sediments.

Key words: Organochlorine, organophosphorus, residues, Lake Victoria.

INTRODUCTION

The use of pesticides has certainly increased agricultural production, and improved longevity and quality of life. Coupled with these successes are a number of side effects. Pesticide use is still indispensable in Kenya in the area of agricultural production and public health vector control. However, the toxicity of these compounds and their presence in the environment pose grave issues that obliges the development of methods that will increase agricultural productivity and disease vector control with minimal environmental contamination and side effects to non-target species. Previous research conducted on sediments, micro-invertebrate organisms from both marine and freshwater ecosystems in Kenya have continually revealed contamination by pesticides (Barasa, 1998; Everaarts et al., 1997; Getenga et al., 2004; Mugachia et al., 1992; Munga, 1985; Wandiga et al., 2002). Levels of pesticide contamination at the top of the food chain in the basin have been exhibited by presence of residues in the cow and human milk, and bird eggs (Kahunyo et al., 1986; Kanja, 1988; Kituyi et al., 1997; Wandiga and Mutere, 1988).

Potential risks of pesticide use in the tropics may differ from those in the widely studied temperate regions. It is

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generally assumed that the climatic conditions in the tropics facilitate the breakdown of bioactive compounds, thus avoiding most of the side effects of pesticides. Kenya, like most other developing countries, is at the crossroads of environmentally sustainable agricultural practices and ostensibly economically sustainable practices, characterized by high usage of agricultural chemicals. The current rural to urban migration necessitates the use of herbicides as an easier alternative to the age-old labour intensive manual weed control. Also, the pressure of a fast growing population calls for more intensification of agriculture with an attendant increased use of agricultural chemicals, in Kenya as well as in other tropical countries. Pesticide use in Kenya is already one of the highest in sub-Saharan Africa with a market share of approximately USD 40.4 million by 2003 (Gonzalez et al., 2003). However, several chemical contaminants from the agricultural fields, comprising of pesticides and other agrochemicals have been reported in the drainage systems and are likely to jeopardise the quality of the water bodies that support the fishery industry and are used for domestic human consumption. The use of the pesticides poses a great challenge to the country to develop satisfactory techniques that can combine optimal agricultural productivity and environmental protection.

Currently, information on pesticide residues in the Lake Victoria catchment and Yala/Nzoia Basin in particular, is fragmentary and inadequate. There is therefore the need for data on the major contaminants in the drainage system of Lake Victoria for proper management of the lake water quality and the Nile River and the sustainability of the Nile Basin ecosystem. Thus, the objective of the present study was to determine the levels of the contaminants; mainly organochlorines and organophosphorus in Yala/Nzoia Basin and its respective catchment. The findings of this study was intended to provide vital information needed in coming up with informed catchment management plans to mitigate the impacts of micropollutants to aquatic organisms and man in the Lake Victoria Basin.

MATERIALS AND METHODS

Pesticide use survey

Inventory of types of pesticides used in Yala/Nzoia catchment was conducted using a structured questionnaire observation and focused group discussions (FGD) among large-scale and small-scale farmers, and stockist shops. More information was also gathered from offices of the Ministry of Agriculture, Livestock and Ministry of Health. The interviewees were categorized into large scale and small scale and randomly sampled using random numbers, where at least 40 respondents were interviewed in each category. Secondary data was obtained from the ministries of agriculture and health on pesticide usage and crops farmed.

Field sampling

Sampling was carried out at 9 selected sites at Yala/Nzoia Basin (Figure 1) between January to March (Dry season) and October to December (Rainy season) of 2009. The sampling was focused on locations influenced by urban areas, agricultural areas and river mouths. The water was sampled by grab method into 2.5 L amber bottles by making several composite samples. Sediment was sampled using pre-cleaned Ekman grab sampler and packed in a black polythene bag. The samples were kept in icebox containing wet ice during the sampling trip and later stored in a refrigerator at -4°C after sampling trip prior to extraction. Sampling from the field was done thrice during the rainy season (October, November and December) and thrice during the dry season (January, February and March).

Reagents

All solvents (n-hexane, acetone, dichloromethane, di-ethyl ether) as well as other analytical materials used (anhydrous sodium chloride, Florisil) were of analytical grade quality obtained from Dr EHRENSTORFER GmbH, (Augsburg, Germany) and the solvents double distilled to ensure purity. Florisil and individual pesticide standards were available at the Kenya Plant Health Inspectorate Services (KEPHIS) laboratories, Nairobi.

Sample extraction and cleanup

Extraction and cleanup followed previously described protocol (NRI, 1991). 2 L of water sample was transferred into a separatory funnel and pH measured. A 50 ml portion of 0.2 M disodium hydrogen phosphate buffer was added to the sample, and pH adjusted to 7 by adding drops of 0.1 N sodium hydroxide and HCl solutions. The neutralized sample was treated with 100 g sodium chloride to salt out the pesticides from the aqueous phase. A 60 ml aliquot of triple distilled dichloromethane was added and shaken for 2 min while releasing pressure. The sample was allowed to settle for 30 min to enhance separation of the phases. The organic layer was collected in 250 ml Erlenmeyer flasks and stored at 4°C in a refrigerator. The extractions were repeated twice using 60 ml portions of dichloromethane, the organic phase combined and cleaned by passing through florisil column topped with anhydrous sodium sulphate. Pesticide residues were sequentially eluted with 200 ml of 6%, 15% and then 50% diethyl ether in hexane. The elutes were combined and concentrated to near dryness using a rotary evaporator at 60°C, and reconstituted in 5 ml HPLC hexane for GC analyses.

The sediment samples were allowed to thaw for 4 h in the lab prior to mixing. Triplicates of 25 g sediment samples were placed in 150 ml teflon vials, treated with 3.5 ml of 0.2 M ammonium chloride and allowed to settle for 15 min. A 50 ml aliquot of hexane-acetone 1:1 mixture was added to each sample vial and extracted for 12 h on orbital shaker model SO1, whereas separation was achieved using a centrifuge at 4000 rpm for 30 min. The extracts were decanted into 250 ml Erlenmeyer flasks, covered with aluminum foil, and kept in a refrigerator at 4°C. The sediments were re-extracted twice using 25 ml of hexane-acetone (1:1) mixture. The extracts were cleaned by passing through a florisil column. The clean extracts were concentrated on a rotary evaporator to near dryness and reconstituted in HPLC hexane to 5 ml.

Chromatographic analyses

Analysis of organochlorine pesticide residues were carried out using the Varian Chrompack CP-3800 GC based at the Department of Chemistry, Masinde Muliro University, Kenya. The GC was equipped with Ni^{63} ECD and CP-SIL 8CB-15 m, 0.25 mm (ID) and

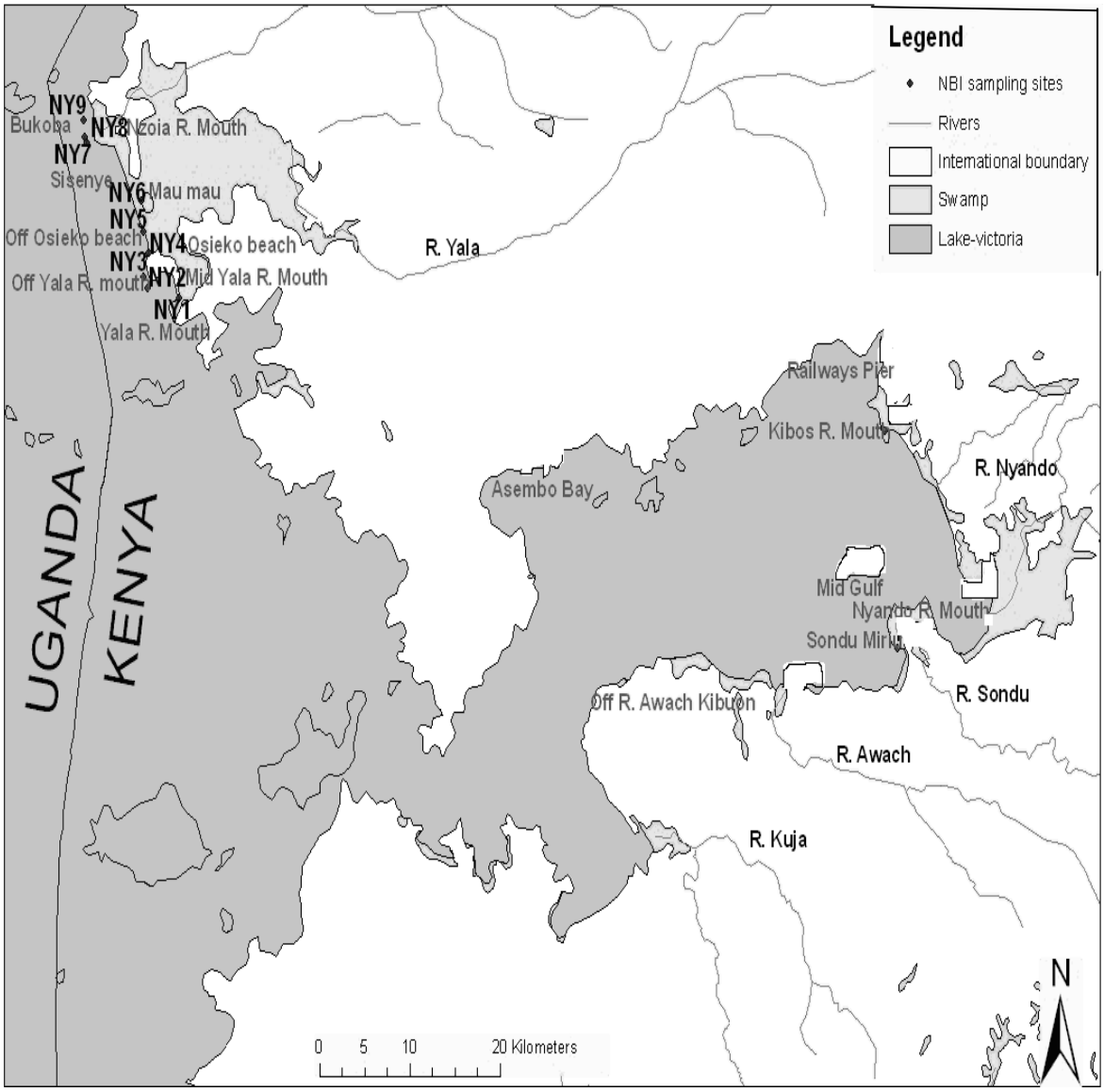

Figure 1. Map of Lake Victoria showing the Nzoia/Yala Nile Basin Initiative (NBI Project) sampling sites, sites under which this study was undertaken, in the Kenyan part of the lake. NY represents NBI sampling sites on Nzoia/Yala Basin.

0.25 µm film. Column temperature was programmed at 150°C (1 min), changed at 4°C/min to 200°C (0 min) and 4.5°C/min to 300°C. Injector temperature was maintained at 250 and 300°C for the detector, whereas flow pressure of 30 Psi of nitrogen gas was applied as carrier gas. Sample size of 1 µl and split ratio of 1:20 was used for all the samples, whereas data processing was done using Star Version 5.4. Organophosphorus pesticide residues in the field sample were analyzed by GC Varian Chrompack 3400 based at the Department of Chemistry, Masinde Muliro University, Kenya. The instrument was equipped with TSD detector and column type of OV-1701 (30 m x 0.32 mm x 0.5 µm); temperature was programmed at 90°C (1 min), changed at 30°C (0 min) and 4°C/min to 250°C. The injector temperature was set at 250°C, while the detector was at 300°C. The carrier (N_2) gas flow rate of 5 ml/min, make up (H_2) at 25 ml/min and air flow rate of 175 ml/min were applied. Sample analysis was carried out by injecting 2 µl sample size into the GC.

Identification and quantification were accomplished by external standards method, whereas processing was done using Star workstation version 5.5. External standard calibration was used to determine peak areas from the samples. Quality control and quality assurance procedures included replicate sampling, extraction and analysis for all the samples. Extraction of the water samples also incorporated studies of spiked samples to determine the recovery rate of the method used. Pure distilled water samples were also incorporated as blanks, and these together with external standards were used to determine the detection limit of each pesticide investigated. High recovery rates were obtained using solvent-solvent extraction method. The average recovery rates for the analysed pesticides were α-HCH 95.55%, β-HCH 94.23%, γ-HCH

96.62%, p,p'-DDT 97.53%, o,p'-DDE 97.21%, p,p-DDD 98.32%, aldrin 88.62% and dieldrin 97.23%. These were good recoveries in relation to the recommended rate that ranges between 70 to 120%.

The data obtained was recorded in Microsoft excel sheets and subjected to analyses in STATISTICA version 8.0 and statistical package for social sciences (SPSS for Windows version 10.0). Frequency of occurrence of pesticide were done using SPSS frequency table. Lack of significant variations for pesticide residues within stations ($p > 0.05$) led to the pooling of data at a basin level. The ranges and means (± SE) for the pesticides were obtained for comparisons with WHO/NEMA recommended guidelines. Variations among pesticides and seasons were done using two-way ANOVA at $p < 0.05$.

RESULTS

Most commonly used pesticides

The most commonly used pesticides in Yala/Nzoia catchment were organophosphates, and organochlorines, in that order, with very few pyrethroids (Table 1). The survey also found out that the largest pesticide users in the catchment were sugarcane growers, followed by tea plantations. Based on the World Health Organization (WHO, 1984) classification of pesticides, farmers used mostly pesticides in categories II and IV which are classified as highly toxic and slightly toxic respectively.

Recovered pesticide residues

Organochlorine residues

Pesticide residue levels of organochlorine in water samples from Yala/Nzoia Basin were below detection limit (BDL) both during the rainy and dry season (Tables 2 and 3). The residue level detected in sediment samples collected during the rainy season ranged from 0.05 to 59.01 μgkg^{-1}, whereas during the dry season, it ranged from BDL-24.54 μgkg^{-1}. Dieldrin constituted the highest residue ($p < 0.05$) detected during the dry and rainy season in sediment samples. The concentration of dieldrin and p,p'-DDD were notably higher ($p < 0.05$) than aldrin and p,p'DDT, respectively, in most of the samples. Organochlorine residues detected in sediment both during the rainy and dry season were below the World Health Organization (WHO) recommended guidelines in the sampling points.

Organophosphorus residues

No organophosphates were detected in any of the water and sediment samples, with exception of diazinon and malathion which were detected at Yala and Nzoia river mouths sediment samples respectively. The concentrations were found to be less than 0.03 and 0.01 μgkg^{-1} at Yala and Nzoia River mouths respectively. Consequently, no tables could be derived.

Seasonal variations of the pesticide residues

Water samples from Yala/Nzoia Basin did not record any of the tested organochlorine pesticide residues both during the rainy and dry season. Similarly, most of the samples did not record any of the tested organophosphorus pesticide residues. Consequently, no geographical trends could be derived. Analysis of seasonal variations and pesticide residues detected in sediment samples collected from Yala/Nzoia Basin showed that samples collected during the rainy season contained higher organochlorine pesticide residues than those collected during the dry seasons (Figure 2).

DISCUSSION

It was noted that farmers used mostly pesticides in categories II and IV which are classified as highly toxic and slightly toxic, respectively. A similar observation was also reported by Gitau (1994) in another study in Lake Naivasha catchment, an indication that most of the pesticides being used are harmful to the environment. It could also indicate lack of regulation program within the country (Abuodha and Hecky, 2005). Organochlorine pesticide residues comprising of aldrin, dieldrin, p,p'-DDT, o,p'- DDE, p,p'-DDD, α- endosulfan, β- endosulfan, endosulfan sulfate, endrin, α- HCH, β- HCH, γ- HCH, heptachlor, heptachlor epoxide, and methoxychlor, were detected at varying concentrations in water during the rainy and dry seasons. The disparities were attributed to differences between sites, seasons, environmental factors, previous and current use of the compounds, and physico-chemical characteristic of the pesticides. The concentration of dieldrin and p,p'-DDD were notably higher than aldrin and p,p'DDT, respectively, in most of the samples. Since the latter are their degradation products, this indicated possible transformation process taking place on p,p'-DDT and aldrin previously used in the region. Sunlight and bacteria changed aldrin to dieldrin which in soil and water degrades slowly (ATSDR, 2002). The dieldrin concentrations were relatively higher compared to the p,p'-DDT concentrations. This is an indication that probably dieldrin is still in use within the basin.

Even though not established in the survey, the presence of isomeric residues of HCH and endosulfan in the samples suggested the use of technical products in the region. The residue levels of γ- HCH were found to be higher than α- HCH, and α- HCH higher than β- HCH in most of the samples, whereas the higher residues of α-HCH isomers compared to β- HCH could be attributed to degradation of γ- HCH and the use of technical CH,

Table 1. Pesticides most commonly used in Yala/Nzoia catchment area during the study period. I = Insecticide, A = acaricide, H = herbicide, N = nematicide, R = rodenticide. I, II, III and IV= WHO category.

Agricultural farms		
Family	**Trade Name**	**Active Ingredient**
Organophosphates	Diagran (I)	Diazinon(II)
	Basudin (I)	Diazinon (II)
	Diazole (I)	Diazinon (II)
	Fenom (I)	Diazinon (II)
	Perfekthion (I)	Dimethoate (II)
	Attain (F)	Malathion (III)
	Lysol (I)	Malathion(III)
	Roundup (H)	Glyphosate Acid(II)
	Steladone (A)	Chloropyrifos(II)
	Dursban (A, I)	Chloropyrifos(II)
	Actelic Super (I)	Primiphos-methyl(IV)
	Mboga dust (I)	Fenitrothion(III)
	Sumithion (I)	Fenitrothion(III)
	Skanar Super (I)	Malathion(III)
	Sulban (I)	Chloropyrifos(II)
	Kelthane (I)	Pirimiphos-methyl(IV)
	Velpar (H)	Hexazinone(IV)
	Diazole (H)	Diazinon(II)
	Gesapak Combi (H)	Ametryn + Atrazine(IV)
	Dual gold (I)	Pirimiphos-methyl(IV)
Organochlorines	Alanex (H)	Alachlor(IV)
	Akarint (I)	Dicofol(II)
	Pentac (I)	Dienochlor(IV)
	Mitigan(I)	Dicofol(II)
	Chlortox (I)	Chlordane(III)
	Velsicol (I)	Heptachlor(II)
	Novadrin (I)	Aldrin(II)
	Murtano (I)	Lindane(I)
	Nendrin (I)	Endrin(II)
Pyrethroids	Karate (I)	Lambdacyhalothrin(III)
	Brigade (I)	Bifenthrin(II)
	Bulldock (I)	Niclosamide(IV)
Small-scale shops in catchments		
Family	**Trade Name**	**Active Ingredient**
Organophosphates	Diazole (I)	Diazinon(II)
	Diagran(I)	Diazinon(II)
	Basudin(I)	Diazinon(II)
	Fenom (I)	Diazinnon(II)
	Attain (F)	Malathion(III)
	Skanar Super (I)	Malathion(III)
	Roundup (H)	Glyphosate Acid(II)
	Dursban (I)	Chloropyrifos(II)
	Steladone (A)	Chloropyrifos(II)
	Actelic Super (I)	Primiphos-methyl(IV)

Table 1. Contd.

	Sulban (I)	Chlorophyrifos(II)
	Mboga dust (I)	Fenitrothion(III)
	Sumithion(I)	Fenitrothion(II)
	Velpar (H)	Hexazinone(IV)
	Kelthane (I)	Pirimiphos-methyl(IV)
	Hostathion (A, I, N)	Triazophos(II)
	Folimat (A, I)	Omethoate(II)
	Nemacur (N)	Fenamiphos(II)
	Miratex(I)	Bromadiolone(II)
	Orthene (A, I)	Acephate(IV)
	Diazole (H)	Diazinon(II)
	Dual gold (I)	Pirimiphos-methyl(IV)
	Gesapak Combi (H)	Ametryn + Atrazine(IV)
	Lysol (I)	Malathion(III)
Organochlorines	Alanex (H)	Alachlor(IV)
	Pentac (I)	Dienochlor(IV)
	Akarint (I)	Dicofol(II)
	Mitigan (I)	Dicofol(II)
	Chlortox (I)	Chlordane(III)
	Velsicol (I)	Heptachlor(II)
	Nendrin (I)	Endrin(II)
	Murtano (I)	Lindane(I)
	Novadrin (I)	Aldrin(II)
Pyrethroids	Karate (I)	Lambdacyhalothrin(III)
	Brigade (I)	Bifenthrin(II)
	Bulldock (I)	Beta-cyfluthrin(IV)
	Krismat (I)	Lambdacyhalothrin(III)

Table 2. Mean concentration (± SE) of organochlorine pesticide residues in water and sediment samples from Yala/Nzoia Basin during rainy season.

Pesticide name	Water (μgL^{-1})	Sediment (μgkg^{-1})
α-HCH	BDL	1.81±0.006
β-HCH	BDL	0.92±0.017
´Y-HCH	BDL	6.43±0.012
p,p'-DDT	BDL	7.16±0.012
o,p-DDE	BDL	6.21±0.006
p,p'-DDD	BDL	15.66±0.035
α-Endosulfan	BDL	5.96±0.023
Endosulfan sulfate	BDL	2.88±0.012
β-Endosulfan	BDL	0.05±0.006
Aldrin	BDL	12.67±0.023
Dieldrin	BDL	59.01±2.121
Endrin	BDL	18.13±0.012
Heptachlor	BDL	9.58±0.046
Heptachlor epoxide	BDL	3.44±0.023
Methoxychlor	BDL	14.78±0.040

Table 3. Mean concentration (± SE) of organochlorine pesticide residues in water and sediment samples from Yala/Nzoia Basin during dry season.

Pesticide name	Water (μgL^{-1})	Sediment (μgkg^{-1})
α-HCH	BDL	4.41
β-HCH	BDL	2.76
´Y-HCH	BDL	16.03
p,p'-DDT	BDL	9.48
o,p-DDE	BDL	4.21
p,p'-DDD	BDL	13.16
α-Endosulfan	BDL	13.2
Endosulfan sulfate	BDL	5.35
β-Endosulfan	BDL	BDL
Aldrin	BDL	12.71
Dieldrin	BDL	24.54
Endrin	BDL	8.64
Heptachlor	BDL	10
Heptachlor epoxide	BDL	8.76
Methoxychlor	BDL	2.52

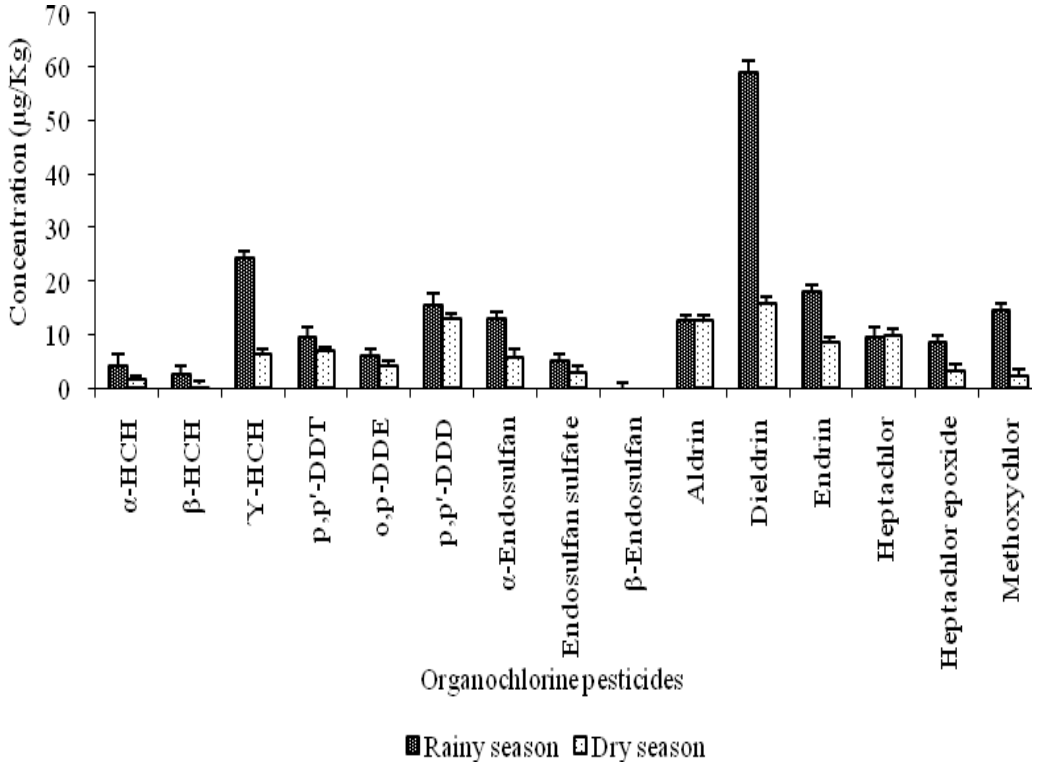

Figure 2. Seasonal variation in the concentration of organochlorine pesticide residues in sediment from Yala/Nzoia Basin during the study period.

which has higher amount of α- HCH than β- HCH. Lindane is known to degrade to α- HCH on exposure to sunlight in the environment. The γ- HCH was commonly used in Kenya for seed dressing to protect crops against ants, but it is currently under restricted pesticides due to its persistence and toxicity. The major sources of organochlorine pesticide residues in the Lake Victoria region are agricultural activities and aerial sprays for

vector control. DDT was extensively applied in aerial sprays against mosquitoes to control malaria (Mitema and Gitau, 1990), whereas aldrin, and dieldrin are used in termite control in building industry (Getenga et al., 2004). Lindane has been in long term use for seed dressing, whereas endosulfan, heptachlor, endrin and methoxychlor were used as insecticides (PCPB, 1998). The public use of these compounds was banned (heptachlor, endrin) or restricted (DDT, aldrin, dieldrin, lindane) in Kenya (PCPB, 1998). High residues of lindane, endosulfan and heptachlor in the environment indicates that some farmers are still applying them illegally, and hence more strict control measures against the use of these compounds needs to be put in place. They were established by the survey to be found in the stockiest shops and used in the farms.

Lack of organophosphate residues in samples does not necessarily indicate lack of impact of organophosphate compounds on the environment. They may still impact on the aquatic system in the short term, before they break down to non-toxic products. However, while the fate in the aquatic system of organophosphate compounds is not clear, the most serious concern relating to the organophosphates is that they are still suspected to have serious toxicological impacts upon terrestrial wildlife and man (Barlas, 1999). The survey established that a number of organophosphates including diazinon and malathion were present in the stockiest shop and are being used in the farms. The seasonal changes were observed to influence the concentration of pesticides in the field samples. The pesticide residues in various samples depicted a general trend of concentration levels in rainy season higher than in dry season. Since the major aspect of seasonal changes was rainfall, it would imply that most residues were washed off agricultural fields by the storm water into the rivers and the lake.

The present study has shown that the banned organochlorine pesticide compounds are still in use. However, concentrations of the tested organochlorines, diazinon and malathion found in Yala/Nzoia basin were below the minimum levels and may not pose environmental and human risks to consumers. However, there is an increasing need for constant monitoring of these compounds in the aquatic ecosystem to ensure the protection of the aquatic food source. There is also the need to establish maximum permissible levels of toxic substances for protection of aquatic biota.

ACKNOWLEDGEMENTS

We thank the Kenya Plant Health Inspectorate Services (KEPHIS) for the pesticide standards. Nile Basin Initiative (NBI) is acknowledged for funds, which contributed to the success of this work. We wish to thank Masinde Muliro University of Science and Technology Chemistry Laboratories for technical support. Kenya Marine and Fisheries Research Institute provided the logistical support.

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