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Research Paper

LevelsofOrganochlorineandOrganophosphorus Pesticide Residues in Water,Sediment and Soil in Kithinu and Mutonga Rivers(Kenya)

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ABSTRACT.
Thisstudyaimedatassessingthelevelsoforganochlorineandorganophosphoruspesticideconcentrationsinwater,sediments and soil along rivers Kithinu and Mutonga catchments areas, where small holder irrigation projects exists.Organochlorinepesticideresiduespersistintheenvironment,whileorganophosphoruspesticidesarehighlytoxic.Laboratory assessments of water quality at points upstream, within and downstream of the irrigation projects were done.Water and sediment samples were collected seasonally between the months of April 2016 and September 2016. Soil sampleswere collected in the month of September 2016. From the study, organochlorines pesticide residues comprising of a-HCH, b-HCH, g-HCH, d-HCH, Heptachlor, Aldrin, Heptachlor epoxide, a-Endosulfan, pp-DDE, Dieldrin, Endrin, b-Endosulfan, pp-DDD, Endrin Aldehyde, pp-DDT, Endosulfan sulfate, and Methoxychlor were detected in water, sediment and soil samples inthe study area. Some ranging from below detection limits (BDL) to 116.376 µg/Kg. Significant differences were recorded inlevels of methoxychlor ($p<0.05$). Organophosphorus pesticides were not detected in all samples in both the wet and dryseason.
***KEYWORDS**:Agrochemicals,residues,organochlorine,organophosphorus.*

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I. INTRODUCTION.

Watercontaminationwithpesticideresidues,associatedwithdecreasedwaterqualityandincreasedexposureofhumanandwildlife has been detected in lakes, rivers and streams in various locations, especially in areas with intensive and prolongedpesticideapplications[1].

Agriculture has been enumerated as a major cause of water pollution [1], [2]. This pollution tends to arise over a widegeographical area and is dependent on what happens on the surface of the land. Agricultural wastes include the pesticides thatare sprayed on crops, as well as sediment, fertilizers and plant and animal debris that are carried into waterways duringperiods ofrainfallorasrunoffandduringtheirrigationoffarmland[3].

Several studies carried out in Kenya have shown agrochemicals used in agricultural activities as one of the sources ofsurfacewatercontamination[4],[5],[6],[7],[8],[9].Thiswouldrenderthewaterbodyunsafefordomesticpurposes.Many organochlorine compounds are very persistent in the environment and have a tendency to bioaccumulate significantlythroughfood chains [10].Organophosphateshaveseveraladvantagesoverother typesofpesticides,includinghighacutetoxicity to target organisms, but they are not persistent in the environment as are organochlorines, as they decompose to non-toxic products. However, their acute toxicity is of concern [11]. Due to the undesirable effects on environmental quality andanimal health, the production and usage of organochlorine compounds was banned or severely restricted during the 1970sand1980sinmostdeveloped countries[12].However,thedemandfororganochlorine pesticideshasbeenincreasinginsomedeveloping countries in Africa, Latin America and Asia due to the ever increasing demand for food as a result of populationgrowth[13].

Kithinu and Mutonga rivers are reliable sources of domestic as well as irrigation water in Meru and TharakaNithicountiesinKenya.Smallholderirrigationisalsopractised withintheriverscatchmentareas.Agriculturalactivities ifnotwellmanagedmayhaveanegativeimpactonsoil, surfacewaterandgroundwaterqualityhencetheneedtoconstantlymonitorthewaterqualityasaresultoftheseincreasedagriculturalactivities.

II. MATERIALSANDMETHODS.

2.1. Studydesign

Thestudyinvolvedlaboratoryanalysisofwater,sedimentsandsoilforthepresenceoforganochlorinesandorganophosphoruspesticideresidues.

2.2. Sampling

Random selectionof points forwaterand sediment sample collectionwas done. A total of9sampling stations wereestablished. Three of these points (K1, K2,and M1) are located before, fourpoints (K3, K4a, K4b, and M3) are locatedwithin and the other two points (K5, and M5) are located after the various irrigation projects as shown in the map (Fig.1).Water samples were collected in two seasons; dry season and rainy season. The samples for the dry season were collected inthe months of August and September 2016, while samples for the wet season were collected in the months of April and May2016.

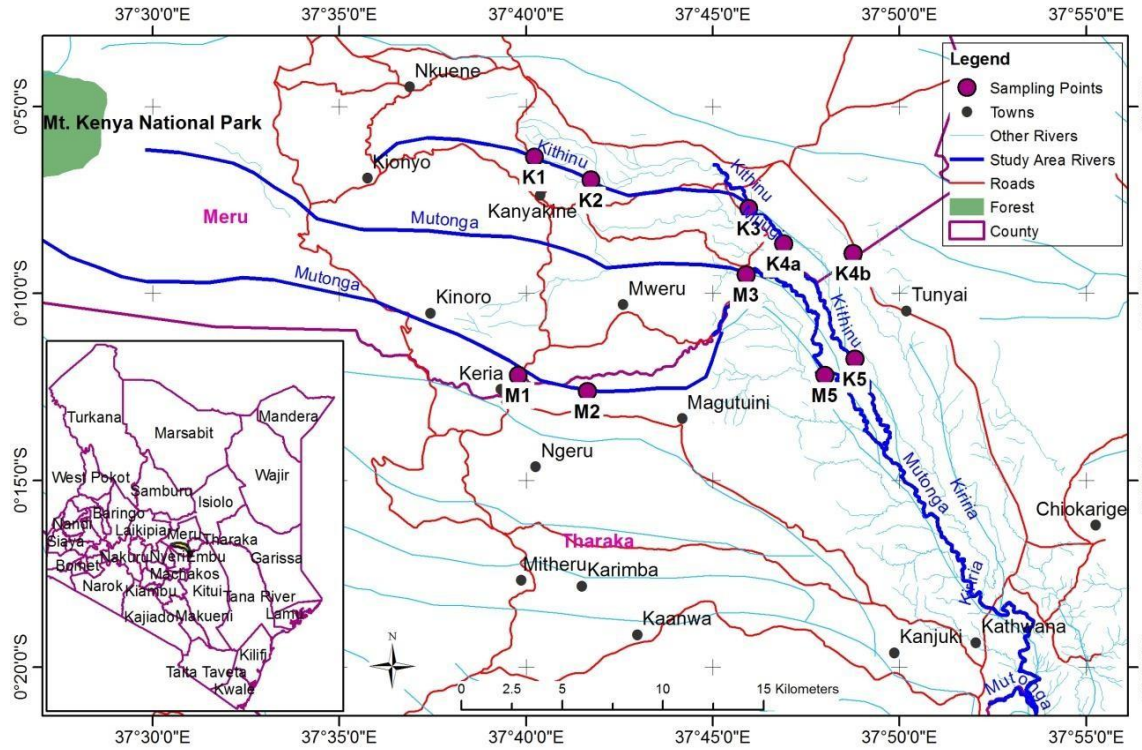

Figure1;AmapshowingRiverMutonga andRiverKithinocatchmentsandlocationof thesampling sites.

2.3 Water,sediment andsoilsamples

2.3.1 Watersamplescollectionandpreservation

Sample collection and handling was done as per the standard methods and procedures for examination of water and wastewateroftheAmericanPublichealthAssociation[14].

Threereplicatesofwatersamplesat eachsitewerecollected.Grabwatersampleswerecollected50cm belowthewatersurface.

Water samples (2.5litres)collected werepreserved withapproximately100 gof10%NACLin2.5litreamberglassTeflon-stoppered samplingbottles.Thewatersampleswerestoredin coolerboxesatatemperatureof4^{0}C.

2.3.2 Sedimentsamplescollectionandpreservation

Sedimentsampleswerecollected atthesamepointsofwater samplecollection.Sedimentsampleswereobtained byuseofacoresampler(0-30cmdepthfromtheriverbed)[15].

Aweightof500gofsedimentsampleswerecollectedfromeachsite,carefullywrappedinaluminiumfoils,then placedinaself-sealingbagandstoredincoolerboxesatatemperatureof-10^{0}C.

2.3.3 Soilsamplescollectionandpreservation

Soilsampleswerecollected fromfarmsadjacenttothewatersampling sites.Thefieldsitesselected,were3mfromtheriverbank.

The soil cores were dug to a depth of 15- 30 cm using a 2 cm internal diameter soil corer [15], carefully wrapped inalluminium foils, then placed in self-sealing bags and stored in cold boxes at a temperature of -10 ^{0}C. The samples weretransferredtoUniversityofNairobipesticidelaboratoryforpreparationandanalysis.

2.3.4 Watersamplespreparationandextraction

The dissolved organic matter in the water samples were extracted by Liquid- Liquid Extraction (LLE) method [14]. Eachsample (2 L) was quantitatively transferred into a 3 L beaker and pH recorded. A volume of 50 ml of 0.2 M dipotassiumhydrogen phosphate buffer was added to the sample, stirred and the pH recorded. The pH of the sample was adjusted byeitheraddingdropsof0.1Mhydrochloricacidor0.1Msodiumhydroxidesolutionsandcarefullystirringtoadjustthep Hto

7.0. The neutral solution was transferred to a 2 L separating funnel and successively extracted with dichloromethane (3 x 50ml). The lower organic layer was then collected into a pre-cleaned and dry 250 ml conical flask. The combined extracts wasfiltered through a plug of glass wool containing 30 g anhydrous sodium sulphate (*ca*30 g) for drying. Cleaning was done byeluting through 20 g florisil, packed in 25 cm long and 1.5 cm internal diameter chromatographic column packed (at a flowrate of 2 ml/min.) with activated anhydrous sodium sulfate both at the bottom of the column and on top of florisil layer toremove any moisture present. The combined extracts for each sample was concentrated to about 3 ml using LABCONCOrotary evaporator. The sample was then stored in teflon-stoppered glass sample vials at 4 ºC awaiting gas chromatography(GC)analysis[14].

2.3.5 Extraction,cleanupandconcentrationofpesticides fromsedimentsandsoilsample

Residualpesticidesinsedimentandsoilsampleswere extractedbythe Liquid-SolidExtraction(LSE)methodusingamixture of hexane and acetone 3:1 v/v [14]. Triplicates of 20 g samples were dried with activated anhydrous sodium sulphate(Na_2SO_4) overnight before transferring to the soxhlet thimble and 100 µl of 0.01-ppm isodrin solution added as internalstandard. This was extracted with tripple distilled 175 ml of hexane: acetone (3:1 v/v) in a 200 ml round bottomed flask for atleast 16 hours in the soxhlet extractor set up. The extracts were then concentrated to 2 ml in isooctane with a LABCONCOrotary evaporator, transferred into a 10 ml glass sample vials pre-cleaned three times with two ml HPLC grade hexane andstoredinfridgepriortothecleanupprocess[14].

Samplecleanupwasdoneusingaluminachromatographic column25cm x1.5cmdiameterpackedwith1.0gofactivated anhydrous sodium sulphate (drying agent) followed by 15 g of deactivated alumina and finally another layer ofactivated anhydrous sodium sulphate. The column was pre-conditioned with 15 ml of HPLC grade hexane and discarded. Thesample extracts of sediments and soil were each introduced into the column and eluted with 165 ml of HPLC grade hexane. Avolume of 2 ml of iso-octane was added to each cleaned sample, as keepers then the sample were concentrated to about oneml using a rotary evaporator. The samples were then stored in teflon-stoppered glass sample vials at 4 ºC awaiting gaschromatography(GC)analysis[14].

2.3.6 Determinationofpesticideconcentrationandanalysis

The sample extracts previously stored in vials was analysed by an Agilet 6890N gas chromatography with an auto samplerequipped withelectron capturedetector(ECD)fororganochlorines andnitrogenphosphorusdetector(NPD) fororganophosphates. Non-polar (SE-30) and semi-polar (OV-1701) capillary columns of dimensions 30 m x 0.25 mm x 0.25µm liquid thickness was used. Nitrogen was used as both a carrier and make up gas, flowing at the rate of 30 ± 1 ml/min. Theinjector and detector temperatures were set at 250 ^{o}C and 300 ^{o}C, respectively. The column temperature was initially set at 90^{o}C, held for three minutes and then raised to 200 ^{o}C at the rate of 30^{o}C/ min (hold time 15 minutes). It was further raised to275^{o}Cattherateof30^{o}C/min(holdtimefiveminutes).Dataprocessingwas doneusingchemstationsoftware.

III. RESULTSANDDISCUSSION.

3.0 Organochlorinepesticidesinwater

Organochlorine pesticide residues detected during the wet season of April - May 2016 ranged between below detection limit(BDL) to 2.547 µg/L. α-HCH was the highest detected in Kithinu 3 (K3). Heptachlor and methoxychlor pesticides residuesweredetectedinmostsamplingsites.ThetotalOCPs inwaterinthewetseasonarerepresentedinFig.2.

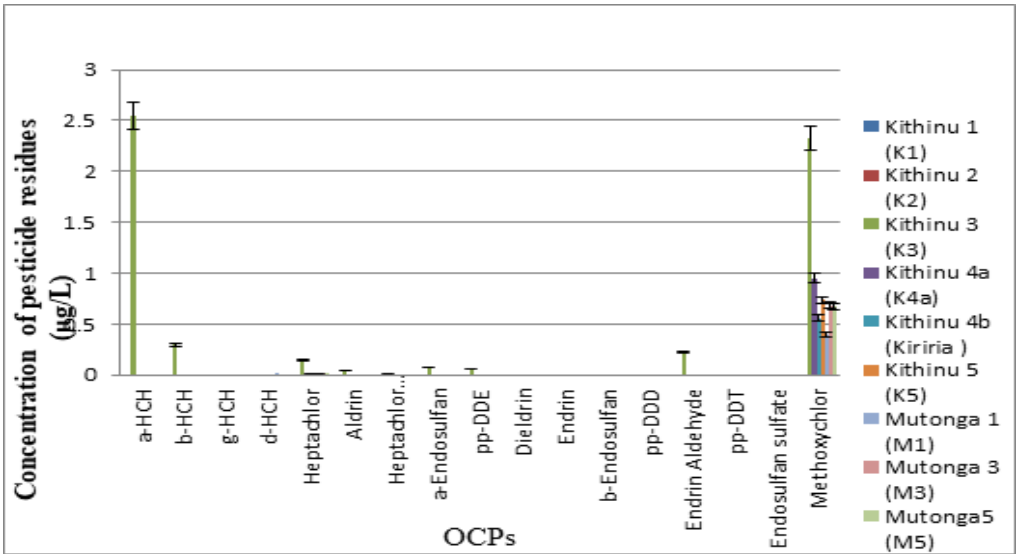

Figure 2: Concentrations of organochlorine pesticide residues in water samples during the wet season of April-May2016

Organochlorine pesticide residues detected during the dry season of August- September 2016 ranged from (BDL) to 2.031µg/L.Methoxychlorwasthehighestdetectedinsite Kithinu3(K3).Heptachlorandmethoxychlorpesticidesresidueswere detectedinmostsamplingsites.ThetotalOCPsinwaterinthedryseasonarerepresentedinFig. 3.

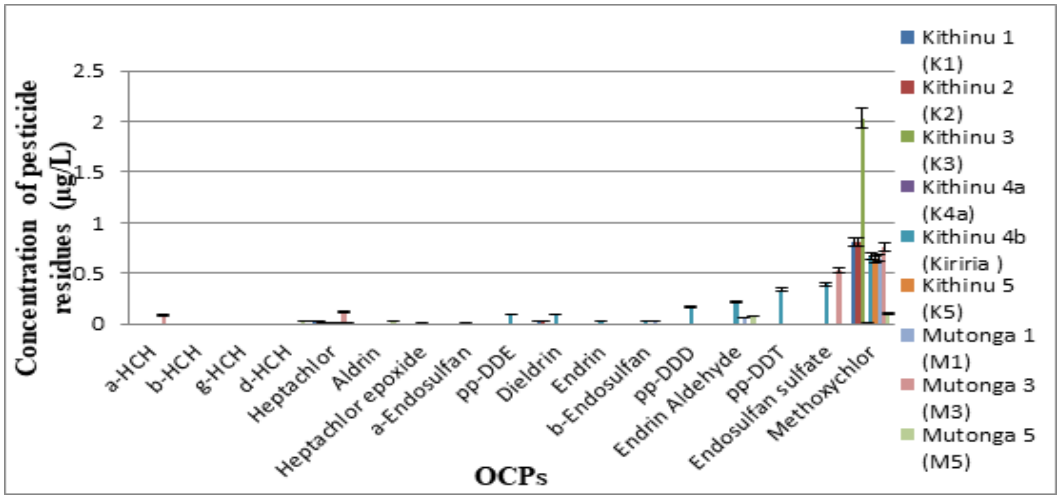

Figure 3: Concentration of organochlorine pesticide residues in water samples during the dry season of August-September2016

3.1 Organochlorinepesticidesinsediment

Organochlorine pesticide residues detected during the wet season of April - May 2016 ranged between (BDL) to 116.376µg/LMethoxychlor was thehighestdetected inKithinu5(K5).ThetotalOCPsinsedimentin thewetseasonarerepresentedinFig. 4.

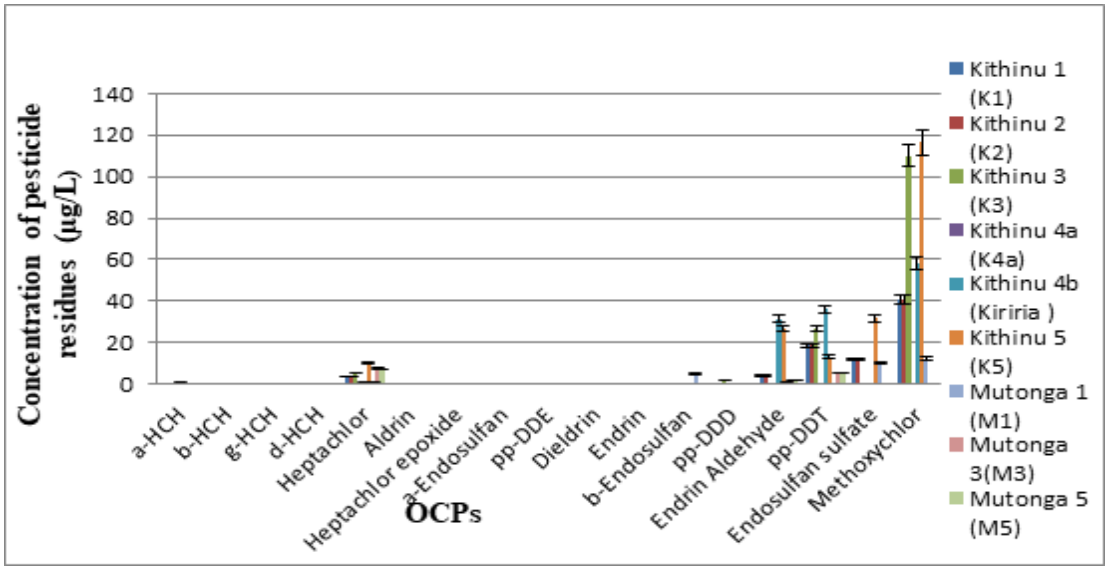

Figure 4: Concentration of organochlorine pesticide residues in sediment samples during the wet season of April-May2016

Organochlorine pesticide residues detected during the dry season of August - September 2016 ranged from (BDL) to 63.405µg/L. pp-DDT was the highest detected in Kithinu 4b (K4b). The total OCPs in sediment in the dry season are represented inFig. 5.

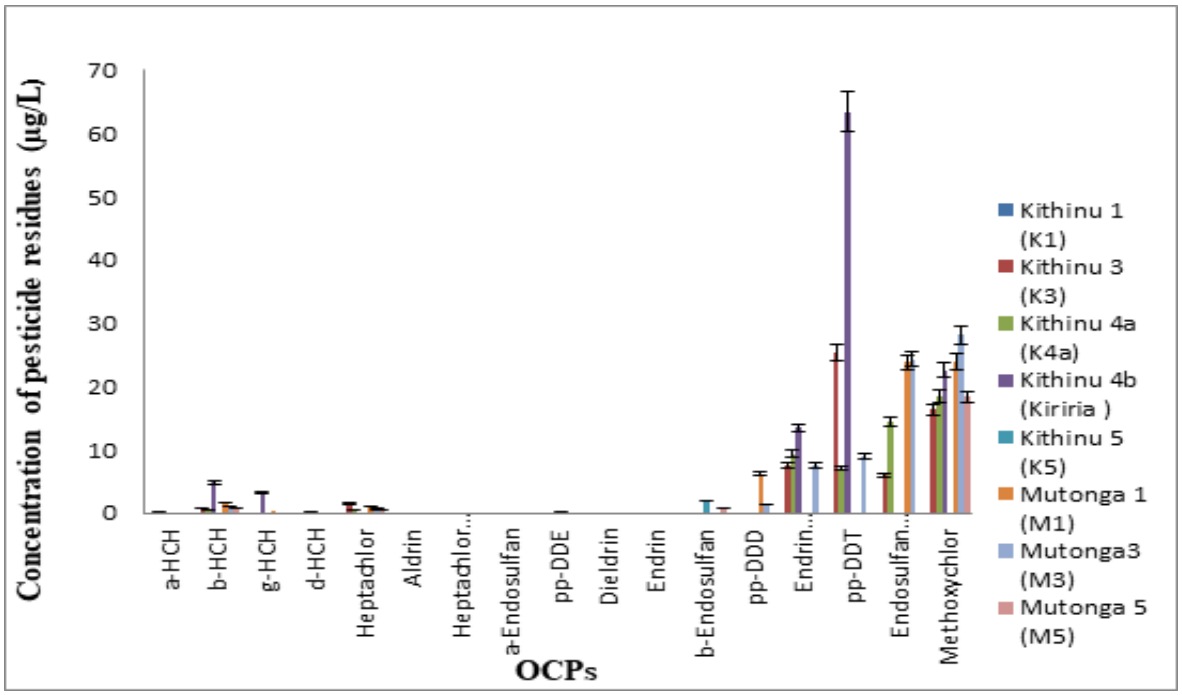

Figure 5: Concentration of organochlorine pesticide residues in sediment samples during the dry season of August -September2016

3.2 Organochlorinepesticidesinsoilsamples

Organochlorine pesticides residues in soil samples were analysed during the dry season of August-September 2016. Theyrangedbetweenbelowdetectionlimits(BDL) to33.037µg/L.Thepp-DDTwasthehighestdetected inKithinu4b(K4b).The a-HCH and methoxychlor pesticides residues were detected in most sampling sites. The total OCPs in soil in the dryseasonarerepresentedinFig. 6.

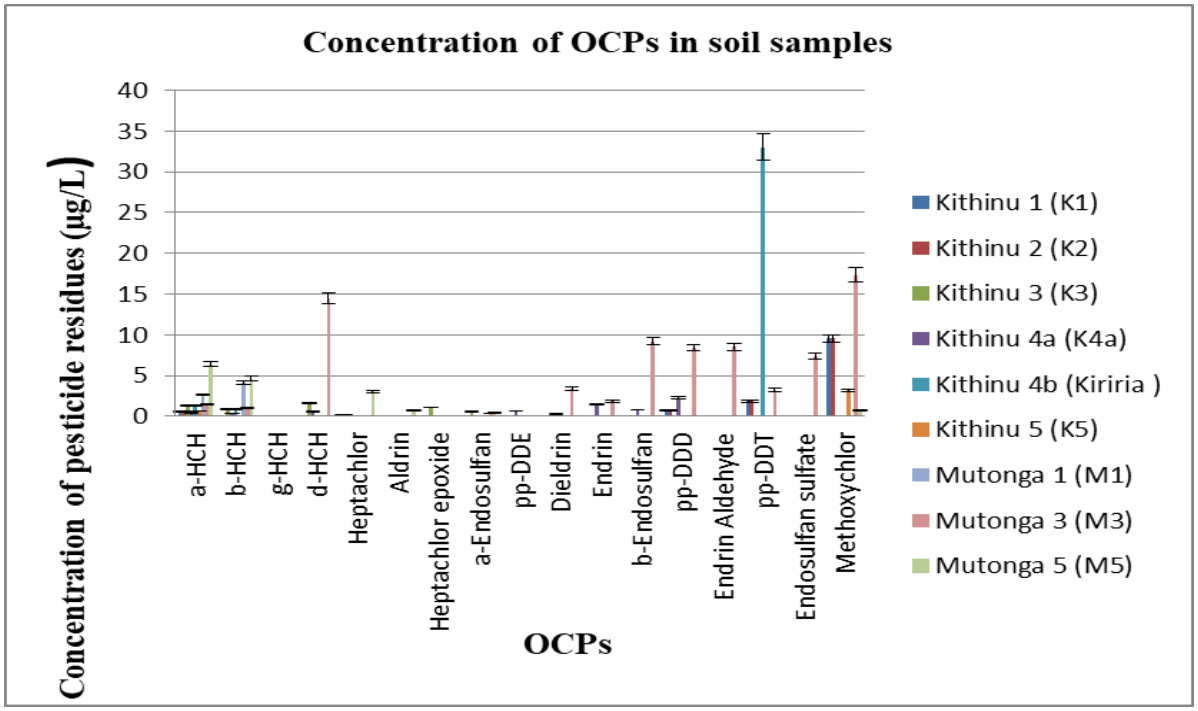

Figure 6: Concentration of organochlorine pesticide residues in soil samples during the dry season of August -September2016

3.3 Statisticaltestdatafororganochlorinepesticides

Statistical analysis for levels of organochlorine pesticide residues in the different river sections and across seasons showed nosignificantdifferences(P>0.05)

Significantdifferenceswererecordedonlyformethoxychlor(TABLE1)at 95%confidencelevel.

TABLE1:t-testfororganochlorinepesticideresidues inKithinuandMutongarivers

t- test

Organochlorine pesticides	Test Value = 0					
	t	df	Sig. (2-tailed)	Mean Difference	95% Confidence Interval of the Difference	
					Lower	Upper
a-HCH	1.286	35	.207	.0000923	-.000053	.000238
b-HCH	1.881	35	.068	.0002708	-.000021	.000563
g-HCH	1.011	35	.319	.0000912	-.000092	.000274
d-HCH	1.406	35	.169	.0000023	-.000001	.000006
Heptachlor	2.901	35	.006	.0012004	.000360	.002040
Aldrin	1.328	35	.193	.0000018	.000000	.000004
Heptachlor epoxide	1.156	35	.255	.0000004	.000000	.000001
a-Endosulfan	1.056	35	.298	.0000021	-.000002	.000006
pp-DDE	1.760	35	.087	.0000067	-.000001	.000014
Dieldrin	1.490	35	.145	.0000039	-.000001	.000009
Endrin	1.000	35	.324	.0000007	.000000	.000002
b-Endosulfan	1.456	35	.154	.0002186	-.000086	.000523
pp-DDD	1.488	35	.146	.0002673	-.000097	.000632
Endrin Aldehyde	2.542	34	.016	.003	.00	.01
pp-DDT	2.848	35	.007	.0063473	.001823	.010872
Endosulfan sulfate	2.776	35	.009	.0037248	.001001	.006448
Methoxychlor	3.064	35	.004	.0144112	.004862	.023961

Comparisons between organochlorine pesticide residue levels recorded in water and sediments, showed significant differences for b-HCH (p=0.03),heptachlor (p=0.00), dieldrin (p=0.03), aldrin aldehyde (p=0.00), endusulfansuphate (p=0.00), methoxychlor (p=0.00), and pp-DDT (p=0.00). (TABLE 2 asperlavenestest).

TABLE2:Levene'sequalityoftest forvariances

Source of variance		Levene's Test for Equality of Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
a-HCH	Equal variances assumed	2.447	.127	-.747	34	.460	-.0001079	.0001445	-.0004016	.0001857
	Equal variances not assumed			-.747	18.553	.464	-.0001079	.0001445	-.0004109	.0001950
b-HCH	Equal variances assumed	9.900	.003	1.822	34	.077	.0005082	.0002789	-.0000586	.0010750
	Equal variances not assumed			1.822	17.123	.086	.0005082	.0002789	-.0000799	.0010963
g-HCH	Equal variances assumed	4.515	.041	1.011	34	.319	.0001824	.0001803	-.0001841	.0005489
	Equal variances not assumed			1.011	17.000	.326	.0001824	.0001803	-.0001981	.0005629
d-HCH	Equal variances assumed	.861	.360	.407	34	.687	.0000013	.0000033	-.0000053	.0000080
	Equal variances not assumed			.407	24.733	.688	.0000013	.0000033	-.0000054	.0000081
Heptachlor	Equal variances assumed	35.460	.000	3.206	34	.003	.0023589	.0007359	.0008634	.0038543
	Equal variances not assumed			3.206	17.006	.005	.0023589	.0007359	.0008064	.0039113

TABLE2:Levene'sequalityoftest forvariances

Source of variance		Levene's Test for Equality of Variances		t-test for Equality of Means						
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
									Lower	Upper
Aldrin	Equal variances assumed	8.587	.006	-1.344	34	.188	-.0000035	.0000026	-.0000088	.0000018
	Equal variances not assumed			-1.344	17.000	.197	-.0000035	.0000026	-.0000090	.0000020
Heptachlor epoxide	Equal variances assumed	5.697	.023	-1.162	34	.253	-.0000008	.0000007	-.0000021	.0000006
	Equal variances not assumed			-1.162	17.000	.261	-.0000008	.0000007	-.0000022	.0000006
a-Endosulfan	Equal variances assumed	4.498	.041	-1.058	34	.298	-.0000042	.0000040	-.0000123	.0000039
	Equal variances not assumed			-1.058	17.000	.305	-.0000042	.0000040	-.0000126	.0000042
pp-DDE	Equal variances assumed	.758	.390	-.447	34	.658	-.0000034	.0000077	-.0000190	.0000121
	Equal variances not assumed			-.447	33.101	.658	-.0000034	.0000077	-.0000190	.0000121
Dieldrin	Equal variances assumed	10.261	.003	-1.518	34	.138	-.0000077	.0000051	-.0000181	.0000026
	Equal variances not assumed			-1.518	17.000	.147	-.0000077	.0000051	-.0000185	.0000030

TABLE2:Levene'sequalityoftest forvariances

		Levene's Test for Equality of Variances		t-test for Equality of Means						
									95% Confidence Interval of the Difference	
		F	Sig.	t	df	Sig. (2-tailed)	Mean Difference	Std. Error Difference	Lower	Upper
Endrin	Equal variances assumed	4.516	.041	-1.000	34	.324	-.0000014	.0000014	-.0000042	.0000014
	Equal variances not assumed			-1.000	17.000	.331	-.0000014	.0000014	-.0000044	.0000016
pp-DDD	Equal variances assumed	9.243	.005	1.451	34	.153	.0005166	.0003535	-.0002019	.0012351
	Equal variances not assumed			1.451	17.022	.162	.0005166	.0003535	-.0002292	.0012624
Endrin Aldehyde	Equal variances assumed	18.724	.000	2.650	33	.012	.006	.002	.001	.011
	Equal variances not assumed			2.729	17.002	.014	.006	.002	.001	.011
pp-DDT	Equal variances assumed	24.015	.000	3.191	34	.003	.0126574	.0039672	.0045951	.0207198
	Equal variances not assumed			3.191	17.001	.005	.0126574	.0039672	.0042873	.0210275
Endosulfan sulfate	Equal variances assumed	40.280	.000	3.045	34	.004	.0073478	.0024131	.0024437	.0122518
	Equal variances not assumed			3.045	17.007	.007	.0073478	.0024131	.0022567	.0124388
Methoxychlor	Equal variances assumed	18.959	.000	3.298	34	.002	.0274004	.0083086	.0105152	.0442856
	Equal variances not assumed			3.298	17.010	.004	.0274004	.0083086	.0098715	.0449293

3.4 Organophosphatesinwater,sedimentsandsoil.

All samples from the sampling sites were analysed for dimethiote and diazinon organophosphate pesticide residues. Thechoice for testing of the two organophosphates was based on the most commonly used pesticides as per a survey done in2016[16].Valuesfororganophosphatepesticideresidueswerebelowdetectionlimits(BDL) inboththewetanddryseason.

3.5 Discussion

3.5.1 Organochlorinespesticideresiduesinwater,sedimentsand soil

ThepresenceofMethoxychlor,α-HCH,endrin,heptachchlorandDDTinthewater,sedimentsandsoilsamplesindicateuseof some banned organochlorines [12] in the study area. A similar study conducted by Abongo et al., in river Nyandocatchmentarea,confirmedtheuseofbannedorrestrictedorganochlorinepesticidesbyfarmers[17].

Methoxychlor, heptachlor and pp-DDT pesticide residues were mostly detected in the study area with Methoxychlorrecording significant differences ($p<0.05$). Detection of methoxychlor is expected since its use is not restricted or banned [12]in Kenya. The presence of mostly heptachlor and not heptachlor epoxide indicates recent use of heptachlor. Methoxychlorwhich hasbeenwidelyusedas aninsecticidesettlesinthesoilanddegradesmorerapidlywithenoughsupplyof oxygen[17]. This explains the lower levels detected in the soil in comparison to the water and sediment samples. Their detection inthe water and sediment indicates a recent use in the agricultural activities in the study area [18] [19]. Similar studiesconducted in Lake Naivasha basin showed the presence of methoxychlor in the lake water, indicating its common use in thearea [4; 6]. Levels of heptachlor detected in rivers Kithinu and Mutonga were relatively lower than the values detected inrivers running through a sugarcane plantation in Kilimanjaro Tanzania [20]. Other studies conducted indicated thatmethoxychlor was reported to exert effects on human and experimental animals due to inhibited synthesis and increaseddegradationofthyroidhormones[21].

Withregardtolackofsignificantdifferences inorganochlorinepesticideresidueacross thesamplingregionsandseasons,their movement in not limited to being carried as runoff from the agricultural fields but can also be transferred by aerialdeposition[2].

High organochlorine residues in sediment than water is comparable tostudies conducted in Tana and Sabaki rivers inKenya. The studies indicating that while pesticide molecules can dissolve in water, a large proportion binds to suspendedparticles and settles at the bottom of the water body, producing contaminated sediments, hence higher values in sedimentsthaninwater[11].

3.6.2.Organophosphatepesticideresiduesinwater,sedimentsandsoil

Lack of organophosphate pesticides water, sediments and soil samples during the wet and dry season, may have been due tothe fact that they are easily degradable in water and do not persist in the environment [11],[22]. The lack of organophosphateresiduesinsamples doesnotnecessarilyindicatelackofimpactofthecompoundstotheenvironment.Theorganophosphatesmaystillaffecta quaticaswellasterrestrialorganismsbeforebreakdowntonon-toxicproducts[11]

IV. CONCLUSIONSANDRECOMMENDATIONS

All organochlorine pesticide residues detected in water were below the WHO guidelines of 0.005 ppm [23]. The presence ofthe banned and restricted organochlorine pesticides residues even at safe limits confirms their continued use in the country.Therefore strict control measure against the use of these compounds need to be put in place as well as awareness campaignsconducted to educate the public on the adverse environmental and human health impacts on use of fertilizers, manures andthepesticides.

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