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Pesticide residues in water and sediment from the Densu River Basin in Ghana

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ABSTRACT

The Densu River is a typical river used for drinking water source, flowing through agricultural areas in Southern Ghana. Surface water and sediment samples at 45 sampling sites from the river and its tributaries were collected and analyzed for pesticides residues using GC with ECD/FID. Sampling was over a period of 24 months covering both the wet and dry seasons of 2007 and 2008. Pesticide residues and metabolites detected in water and sediment samples were organochlorines (dieldrin, DDT, DDE, endosulfan sulphate, α-endosulfan, γ- HCH, δ- HCH, aldrin, γ - chlordane ,endrin, endrin ketone, endrin aldehyde, methoxychlor and heptachlor). In an average of 13.69% of sediment and 3.30% of water samples, at least one pesticide residue was detected per sample. A total of 8 different pesticides residues were detected in water samples with concentrations ranging between 0.1μgL^{-1} and 48.6μgL^{-1} whiles in sediment samples 14 different types were detected with concentrations ranging between 0.10μgL^{-1} and 163.00μgL^{-1}. The maximum levels of residues were found in downstream section of the basin. Aldrin and dieldrin levels detected were above the recommended limit of 0.03 μgL^{-1} with endosulfan, endrin and chlordane registering levels above their recommended limits of 20.0μgL^{-1}, 0.6μgL^{-1} and 0.2 μgL^{-1} respectively for drinking water. The ratio of DDE/DDT in environmental matrix were quite high indicating there old input of DDT and significant degradation. The results implicitly revealed that improper land use in the basin has led to poor water quality.

Introduction

The use of pesticides to control pests has been in practice in Ghana for over 40 years (*Ntow, 2005*). Without pesticides, the quantity and quality of food produced would have diminished. Despite the benefits of pesticides for agriculture production and public health, the increased higher application of pesticides have resulted in soil, water and food contamination (*Wilson et al, 2007*). The presence of pesticide residues in water and sediment are extremely important because of their potential impact on human health, the aquatic ecosystem and implications on drinking water sources. Pesticides gained notoriety for their high toxicity, persistence in the physical environment and their ability to bioaccumulate in food chains since the mid 1960's (*Tanabe and Kawata, 2009; Liu et al, 2002; Jiries et al, 2002*). At low concentrations, pesticides cause suppression of immune response and hypersensitivity to chemical agents, breast cancer, reduced sperm count and male sterility (*Carvalho, 2006*). Death cases and pesticide poisoning have been reported around the world particularly in developing countries (*Tariq et al, 2007*). Studies in Ghana indicated the presence of pesticide residues in water, sediment, vegetables, fruits, fish and soil at locations unknown for pesticide usage (*Amoah et al, 2006; Mensah et al, 2004; Aboagye, 2002; Osafo et al, 1998*). Growth in agro-based industries, increase in agriculture and increased human population in Ghana have resulted in a rapid increase in input of large numbers of agrochemicals especially pesticides into the environment. Improper storage and lack of sound disposal facilities have caused contamination of soil, water, air, plants and animals (*FAO, 1998*). The short distance between agricultural fields and waterways also increase the probability of agrochemicals reaching waterways via agricultural runoff. Leachates from landfill site in the basin, improper disposal of empty pesticide containers and untreated effluent from agricultural, domestic, municipal and agro-based industries in the basin contribute significantly to the contamination of the Densu River basin.

The Densu River basin constitutes one of Ghana's most productive agro-ecological ecotones (*Attua, 2003; Gyasi et al, 1994*), the cradle of Ghana's cocoa industry and an active zone for the large scale cultivation of vegetables, citrus fruits, oil palm, pineapples and food crops for export. The modernization of agricultural practices in the basin has led to an increased use of pesticides some of which are known to be highly persistent and toxic to non-target organism. Accurate records concerning pesticide usage in the basin do not exist though the nationwide consumption for 2008 available at EPA-Ghana was 27,886 metric tons (*Ghana- EPA, 2008*). Ghana's vast water resources especially the Densu River and its tributaries are among those most affected by environmental stress imposed by human population growth, anthropogenic activities, agriculture, industrialization, improper disposal and management of waste (*WRC, 2003*).

The presence of pesticides in the environment, water and food may affect the aquatic ecosystem, human health and create barriers to trade in agricultural commodities. The presence of pesticides in water consumed by residents may have public

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health implications since some of these pesticides have carcinogenic and mutagenic effects. This study therefore seeks to assess the types and concentrations of pesticide residues in water and sediment samples as well as their seasonal variation in the Densu River basin. This is to establish the extent of spatial distribution of pesticide residues in various environmental matrices in order to establish the extent of pesticide pollution of the Densu River and its tributaries.

Materials and Methods

The study Area

The Densu River Basin covers approximately 2,565 km2 and lies between latitude 5° 30' N - 6° 20' N and longitude 0° 10' W - 0° 35' W (Fig. 1). The basin is predominantly underlain by Precambrian granitoids comprising mostly Cape Coast granite and granodiorites with associated gneisses. The study area is a low lying plain with forest plateau and generally undulating with scattered steep hills and largely covered with deciduous forests. The Densu Basin has two different climatic zones (wet semi-equatorial and dry equatorial climate zones). The wet semi-equatorial climatic zone occurs in the north where the annual rainfall is approximately 1,700 mm. On the contrary, the coastal area experiences the dry equatorial climate where the annual rainfall is approximately 800 mm. Both climatic zones, however, experience homogeneous temperatures in the range 23–32◦C with mean annual value of 27◦C (WRI 2003; Dickson and Benneh 1980).

Fig. 1: Location of some sampling points in the Densu River Basin

Sampling

Sampling was restricted to the forest zone where industrial crops such as cocoa, oil-palm, coffee, cola nut, fruits and food crops are produced on large scale and the semi urban agricultural areas where large scale commercial vegetables, fruits especially pineapple, legumes, cereals, root and tubers crop farms are located. Sample locations for surface water (Sites 1-45) and sediment (Sites 1-45) targeted tributaries of the Densu River and the River Densu itself. Sampling was done over a period of 24 months covering both the wet and dry seasons in 2006-2007. Surface water samples were taken from 0 – 5 cm below the water surface into acid cleaned amber 1-L glass bottles following sampling protocol as described by FDA (1994) and Akerblom, (1995). Sediment samples were taken from sites of water collection points in rivers and streams with polypropylene spoon. Composite samples were homogenized and wrapped in aluminum foil. All samples were immediately stored on ice after collection, transported in ice cooler and stored in the laboratory at 4 ^{0}C prior to analysis.

Sample Extraction

Extraction of pesticides from water samples was performed by solid phase extraction (SPE) method while that of sediment was by solid dispersion method (Laabs et al, 2002; Zhou et al, 2000; Tanabe et al, 2009; Akerblom, 1995). Filtered water sample (1 L) was passed through conditioned C-18 SPE cartridges at a flow rate of 10 ml/min. The cartridges were then dried in an air stream for 15 min. Pesticide residues collected on the cartridges were eluted with 6ml (2x3ml) ethyl acetate under reduced pressure. The combined eluates were concentrated to 2ml under a stream of nitrogen. Sediment samples (wet) (approx. 25g) were mixed with anhydrous Na_2SO_4 (30g) and ground to form free flowing powder. The powdered sample was extracted using a mixture of ethyl acetate and acetone (1:1 v/v) (50 ml). After extraction, they were centrifuged and the sample extract decanted. The remaining water phase was liquid – liquid extracted with dichloromethane (3 x 25ml) successively. The combined organic phase extracts were then dried with anhydrous sodium sulphate (25g) and concentrated to 5ml with rotary evaporator at 40^{0}C.

Clean – up

Water extracts in all cases were deemed sufficiently clean and were thus not subjected to clean-up procedures. Conditioned 8-ml C-18 solid phase extraction (SPE) cartridges were used for the sample clean-up (Zhou et al, 2000). Each sediment extract was percolated through the cartridges at a flow rate of approximately 5 ml /min under vacuum pump. The pesticides trapped in the cartridges were eluted with 6 ml (2 x 3ml) ethyl acetate. The sample extract was concentrated using the rotary evaporator aided with a water chiller to 2ml for GC analysis.

Analysis of Pesticides

Water and sediment samples were analyzed in a gas chromatograph (Varian CP-3800 gas chromatograph) equipped with Ni-63 electron capture detector (ECD) and Flame ionization detector (FID). Extracts of samples were interspersed with analytical standards of interest, placed on auto-sampler with standards at the start, between every 15 samples and the last of the GC sample run. The pesticide residue components were identified by comparing their retention times with those of the standard mixture of the pesticides. Quantification was based on comparison of peaks with that of calibration standards in the concentration range of 0 to 200 µg/L. The ECD working conditions were as follows: injector temperature, 225 ^{0}C; injector mode, splitless; oven temperature, programmed from 70 ^{0}C, held for 2 min to 180 ^{0}C at a rate of 25^{0}C/min, then from 180^{0}C to 290^{0}C at a rate of 10^{0}C/min, the detector temperature 300^{0}C, injector volume 1.0 µL. The FID working conditions were oven temperature, 60^{0}C held for 2min to 180^{0}C at a rate of 35 ^{0}C/min, then increased to 270^{0}C at a rate of 2^{0}C/min, and finally increased to 300^{0}C at a rate of 5^{0}C/min and held for 10 minutes, the detector temperature was 310^{0}C. The injector mode was splitless and the injection volume 2 µL (*USEPA, 1994*).

Quality Control/Quality Assurance

Quality control and quality assurance as prescribed by the CODEX Alimentarus Committee were incorporated in the analytical scheme. Quality assurance measures applied in the laboratory included rigorous contamination control procedures (strict washing and cleaning procedures), monitoring of blank levels of solvents, equipment and other materials, analysis of procedural blanks, recovery of spiked standards, monitoring of detector response and linearity. During extraction, blanks and duplicates were included in the analysis and re-calibration standards run frequently to check the integrity of the calibration curve. Aliquot (100 ml) of each solvent was concentrated to 2 ml and analyzed to check the contamination from the reagents (*FAO/WHO, 2007*). Percentage recoveries in spiked samples

were 68.5 - 102% hence the results of the study were not corrected for recoveries since all were within the normal acceptable range of 65 - 120% (*FAO/WHO, 2007; Hill, 2000)*

Results and Discussion

The percentage positive detection, minimum and maximum concentrations of pesticide residues detected in sediment and water samples are summarised in Table 1. The results of the study indicate that all the detected residues and metabolites in water and sediment samples from the Densu River Basin were organochlorines (OCs). This is most probably due to the persistent nature of this class of pesticides to the other classes. The organochlorine pesticides were extensively used in the past in the cocoa, oil palm, citrus and coffee plantations as well as pineapple, vegetable and food crop farms (*MOFA, 2003*).

In an average of 13.69% of sediment and 3.30% of water samples, at least one pesticide residue was detected per sample. The occurrence and concentration of pesticides and metabolites varied widely between samples of different matrices analyzed with the lowest levels detected in water samples. A total of 8 different types of pesticide residues and metabolites were detected in varying concentrations ranging between 0.1µgL^{-1} and 48.6µgL^{-1} in water samples whiles 14 different types with concentrations ranging between 0.10µgL^{-1} and 163.00µgL^{-1} were detected in sediment samples. Of the numerous pesticides evaluated, γ-HCH (21.6%), DDT (21.6%), recorded the highest percentage detection in sediment samples while in water samples DDT (4.4%), γ- HCH (4.4%) and γ – chlordane (4.4%) registered the highest percentage detection (Fig. 2).

The concentrations of individual pesticides measured in surface water and sediment samples from majority of sampling sites were below detection limit. It is interesting to note that pesticide residues were detected in only a few water samples (Sites 5 and 16). These sites were located in vegetable growing communities where pesticide usage was intensive. The concentrations of individual pesticides measured in about 90% of surface water in the study area never exceeded 0.1 µgL^{-1} limit established by the International Community for drinking water (*WHO, 2004; US EPA, 2000*). Out of the 90 water samples analysed, 2.2 % were found to contain detectable levels of dieldrin, endosulfan sulphate, aldrin, DDE and endrin while 4.4 % contain detectable levels of DDT, γ-HCH (lindane) and γ-chlordane (Fig. 2). Surface water samples did not record significant levels of pesticide residues as most of them were below detection limit. Sampling Sites 1-20 located in the forest zone of the basin had no residue detected except Sites 5 and 16 where intensive vegetable farming along the banks of the river and its catchment occurs.

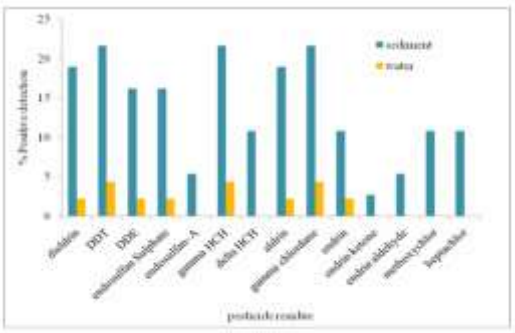

Fig. 2: Percentage positive detection of pesticide residues in water and sediment samples

Sampling locations 25 – 37 recorded appreciable levels of pesticide residues in water samples. The levels recorded ranged from 0.10 µgL^{-1} to 48.6 µgL^{-1}. The highest pesticide concentration was found for endosulfan sulphate at sampling Site 16 (Mangoase). Significant levels of γ- HCH, aldrin and γ – chlordane were detected at sites 26 – 30. Concentration of γ-HCH ranged between 0.2 µgL^{-1} and 1.4 µgL^{-1}, aldrin 0.1µgL^{-1} to 1.4 µgL^{-1} , γ – chlordane ranging from 0.8 µgL^{-1} to 1.9 µgL^{-1}, DDT between 0.40 µgL^{-1} and 0.60 µgL^{-1} , DDE was between 0.10 µgL^{-1} and 0.70 µgL^{-1}. The highest concentrations of endrin (1.30µgL^{-1}), γ-HCH (1.40 µgL^{-1}) and γ-chlordane (1.90 µgL^{-1}) were registered in analyzed water samples from sites 26 – 32.

Sediment samples registered the greatest number and highest concentrations of pesticide residues. α-endosulfan recorded the highest maximum concentrations in sediment samples though it was detected in only 5.4 % of analyzed sediment samples. The levels fluctuated between 48.00 µgkg^{-1} and 163 µgkg^{-1} with a mean of 105.50 µgkg^{-1}. The most frequently detected pesticides in sediment samples were DDT, γ - chlordane and γ- HCH while dieldrin, DDE, aldrin and endosulfan sulphate were detected in more than 15% of analyzed sediment samples. Site 32 (Oboadaka) recorded the highest level of pesticide residues especially α-endosulfan, endosulfan sulphate and heptachlor. Concentration ranges and mean (in bracket) registered were endosulfan sulphate 6.00 µgkg^{-1} - 48.6 µgkg^{-1}, (22.767 µgkg^{-1}) while endrin aldehyde registered maximum levels of 17.3 µgkg^{-1} (Fig 3). DDT concentration in sediment samples ranged between 0.20 µgkg^{-1} and 1.10 µgkg^{-1} with a mean of 0.563 µgkg^{-1}, heptachlor 4.00 µgkg^{-1} to 96.00 µgkg^{-1} with a mean of 34.50 µgkg^{-1} while endrin aldehyde levels varied between 0.30 µgkg^{-1} to 17.30 µgkg^{-1} and a mean of 8.80 µgkg^{-1}.

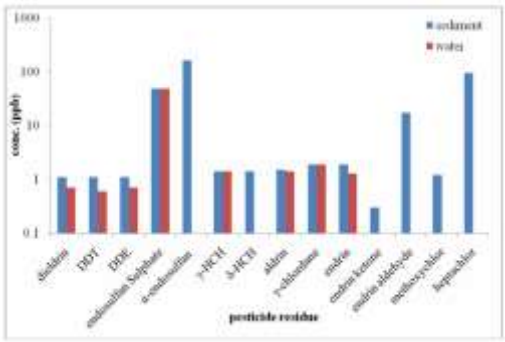

Fig. 3: Maximum concentration of pesticide residues in water and sediment samples.

DDT and its metabolite DDE, endosulfan sulphate, γ-chlordane, endrin, γ-HCH as well as aldrin and dieldrin were detected in both the water and sediment samples. The water samples recorded a maximum level of 1.4 µgL^{-1} aldrin while that of dieldrin was 0.7 µgL^{-1}. The presence of dieldrin in the basin may be due to the degradation of aldrin. The numbers were all below the WHO recommended guideline value (10µgL^{-1}) for aquatic environments (*WHO, 2003*). The Ghana Standard Board and WHO guidelines for drinking water quality recommend a health-based limit of 0.03 µg/l for aldrin and deldrin residues, either separately or together (*GSB 2005; WHO, 2004*). These recommended levels were exceeded in the study area. The elevated levels registered might be attributed to contamination arising from agricultural land since sites 16 – 45 were located in semi-urban agricultural areas where intensive vegetable and large scale fruit (pineapple) farming were practiced.

Although agricultural use of DDT has been banned in Ghana by the Environmental Protection Agency, it was detected in sediment and water samples along with its metabolite DDE. This degraded product is assumed to take place in aerated soils, thus it is suggested that DDE is transferred from treated soils to water bodies. This demonstrated the well-known environmental persistence of these substances even in tropical environments (*Laabs et al, 2002; Kidd et al, 2001*) and justifies its prohibition from agricultural use in Ghana. Relatively high levels of total DDT and other organochlorine pesticides in environmental samples can be related to past use of the pesticides in agricultural fields and the health sector as well as increased dispersion of these volatile compounds in the studied tropical environment. Generally, DDE is more persistent in the environment than DDT, when the use of DDT in a country ceases, the levels of this compound decreases more rapidly than the levels of DDE thereby producing an increasing DDE/DDT ratio (*Marco et al, 2004*). The DDE/DDT ratios were quite high in both water and sediment samples. The ratio ranges between 0.75 to 1.17 indicating old input of DDT and significant degradation.

The distribution of pesticides in the Densu River basin showed an interesting trend. The pesticide residues were detected mostly in analyzed samples from sites 16 -37. They were detected from tributaries draining rapid urbanized commercial areas and non-forest intensive agricultural communities. Large scale commercial vegetables and fruit farming especially pineapple are practiced in these areas where the use of agrochemicals is high. The Northern part of the study area (sites 1-15) located mainly in the forest zone registered insignificant levels of pesticide residues. Most farmers in these areas use pesticides especially insecticides in their tree crop farms under strict supervision of Extension Officers from the Ministry of Agriculture and Ghana Cocoa Board. Good agricultural practice employed in the basin especially the upper sections which are major cocoa, oil palm and citrus growing areas and the fast dissipation of pesticides in tropical soils after application might have contributed to the low contamination level of the Densu River and its tributaries in the upstream section.

The levels of DDT, DDE and γ-HCH, detected in surface water were far below the WHO guideline limit set for drinking water quality. The limit recommended by Ghana Standards Board and WHO for DDT and γ-HCH are 1.0μgL^{-1} and 2.0 μgL^{-1} respectively (*GSB, 2005; WHO, 2004*). Endosulfan, endrin and chlordane registered levels above their recommended limits of 20.0μgL^{-1}, 0.6μgL^{-1} and 0.2 μgL^{-1} respectively for drinking water quality (*WHO, 2004*). Although most of these organochlorines in the samples were well below the allowable maximum limits, they might still pose a potential risk for public health in that they are highly toxic, persistent and bioaccumulative in the environment.

Endosulfan sulphate, aldrin and dieldrin levels recorded in water samples exceeded both the acute (aldrin 0.3595 μgL^{-1}, dieldrin, 0.3595 μgL^{-1} and endosulfan 0.22 μgL^{-1}) and chronic (aldrin 0.651 μgL^{-1}, dieldrin 0.651 μgL^{-1}, and endosulfan 0.0056 μgL^{-1}) ambient water quality criteria for aquatic organisms in fresh water recommended by USEPA (*USFWS, 2003*). Although DDT, γ-HCH, chlordane and endrin values were below the acute limits, they all exceeded the chronic limits of 0.001 μgL^{-1}, 0.08 μgL^{-1}, 0.0043 μgL^{-1} and 0.61 μgL^{-1} for DDT, γ-HCH, chlordane and endrin respectively.

Comparatively, elevated levels of residues were registered in water samples from the Densu River and its tributaries than those registered in the Volta and Bosomtwi lakes in Ghana. Adrin (1.4 μgKg^{-1}), dieldrin (0.70 μgKg^{-1}), DDT (0.60 μgKg^{-1}), DDE (0.70 μgKg^{-1}) and endosulfan sulphate (48.60 μgKg^{-1}) were registered in surface water from the Densu Basin while endosulfan sulphate (0.23 μgKg^{-1}) and lindane (0.008 μgKg^{-1}) were recorded in the Volta lake (*Ntow, 2005*) with Lake Bosomtwi recording DDT (0.012 μgKg^{-1}), DDE (0.061 μgKg^{-1}), lindane (0.071 μgKg^{-1}) and endosulfan (0.064 μgKg^{-1}) in its surface water (*Darko et al, 2008*). The sediments showed a different trend in residue levels with the Densu river basin recording lower levels than the Bosomtwi and Volta lakes. DDT levels were 9.0 μgKg^{-1} in Volta Lake, 4.41 μgKg^{-1} in Lake Bosomtwi but 0.563 μgKg^{-1} was found in Densu River when compared respectively.

Conclusion

The Densu River Basin in Ghana have special economic significance, representing the country's greatest hydrostructure with freshwater and food basket. At the same time there are multiple anthropogenic influences that impacts on the basin. The results regarding pesticide residues indicate that several pesticides especially OCs were widely used in the past by farmers in the basin. Farmers also mix cock-tail of various pesticides to increase potency. The residues are believed to have originated from various pesticide rich sources, mainly agricultural and household uses. The natural processes of leaching and run-off are likely to enhance their transfer to the main course of the river. The highly impaired sites were located in waters with close proximity to the urbanized, agricultural and high density residential communities in the basin. Aldrin and dieldrin levels detected were above the recommended limit of 0.03 μgL^{-1} with endosulfan, endrin and chlordane registering levels above their recommended limits of 20.0μgL^{-1}, 0.6μgL^{-1} and 0.2 μgL^{-1} respectively for drinking water. Most of the detected compounds are generally persistent, volatile, lipophilic and bioaccumulative both in the environment and at each trophic level of the food chain hence they might still pose a potential risk for public health.

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Table 1: Detection frequency, pesticide and metabolite concentrations in surface water and sediment samples from the Densu River Basin

Substance	Positive detections (% of samples)	Concentrations (μgL^{-1}) Min	Max
Surface water samples, Sites 1 – 45 (n=90)			
dieldrin	2.2	0.1	0.7
DDT	4.4	0.4	0.6
endosulfan Sulphate	2.2	0.1	48.6
γ- HCH	4.4	0.2	1.4
aldrin	2.2	0.1	1.4
γ - chlordane	4.4	0.8	1.9
DDE	2.2	0.1	0.7
endrin	2.2	0.1	1.3
Sediment samples Sites 1 – 45 (n=45)		Concentrations (μgKg^{-1})	
dieldrin	18.9	0.10	1.10
DDT	21.6	0.20	1.10
DDE	16.2	0.20	1.10
endosulfan Sulphate	16.2	6.00	48.60
α-endosulfan	5.4	48.00	163.00
γ- HCH	21.6	0.20	1.40
δ- HCH	10.8	0.60	1.40
aldrin	18.9	0.20	1.50
γ - chlordane	21.6	0.10	1.90
endrin	10.8	0.40	1.90
endrin ketone	2.7	0.20	0.30
endrin aldehyde	5.4	0.30	17.30
methoxychlor	10.8	0.60	1.20
heptachlor	10.8	4.00	96.00