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Distribution of Diazinon in Water, Sediment and Fish from Warri River, Niger Delta Nigeria

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Abstract

This paper presents the first attempt to quantify the levels and distribution pattern of diazinon in surface water, sediment and fish (*Chrysichthys furcatus* and *Tilapia zilli*). The samples were collected from three stations (Ovwian, Ekakpamre and Ovu) of Warri River in the western Niger Delta of Nigeria in 2006 during the dry(January-April) and wet seasons (May – August) . A total of 96 samples made up of 24 samples each for water, sediment and fish were analyzed in this study. The pesticide levels were analyzed using high performance liquid chromatography (HPLC model CECIL 1010) to elucidate its distribution in various environmental compartments. The concentrations of the pesticide in the matrices ranged from; 0.01-3.61µg/L (water), 0.01-3.64µg/gdw (sediment), 0.01-7.51µg/gdw (*C. furcatus)* and 0.01-1.13 µg/gdw (*T. zilli*) . From this result, decreasing order of occurrence of the pesticide is as follow; fish > sediment > water. The concentrations observed in fish (*C. furcatus*) were higher than the levels observed in sediment and water suggesting bioaccumulation of the pesticide by the fish. Spatial variations occurred with downstream stations having statistically higher concentrations in all matrices at $P < 0.05$. Seasonal variations occurred with higher concentrations in dry season for water and sediment only, while the fish species had higher concentrations in the wet season. The observed values were above the ecological bench marks (0.02µg/L) recommended by Nigeria Environmental Protection Agency and European Union. They were also relatively higher than previous studies on the Nigerian environment, an observation that calls for regular monitoring of the Niger Delta water bodies.

الملخص

تتناول هذه الورقة أولى المحاولات لقياس مستويات و أنماط توزيع الديازينون في المياه السطحية و التربة و الأسماك كريسختش فيركاس و تلابيا زيلي . جمعت العينات من ثلاث محطات (أوفيان و ايككابامير و أفو) من نهر واري في دلتا النيجر الغربية خلال موسم الجفاف عام ٢٠٠٦ (كانون الثاني – نيسان) و خلال الموسم الرطب (أيار – آب) . تم جمع ٩٦ عينة تتكون من ٢٤ عينة من الماء و التربة و نوعي السمك . تم تحليل مستوى المبيد الحشري بواسطة الكروموتوغرافيا عالي الجودة . بلغت تراكيز المبيد من ٠١. – ٣,٦١ مايكروغرام / اللتر في الماء ، و ٠١. – ٣,٦٤ مايكروغرام / غرام جاف في التربة ،و ٠١. – ٧,٥١ مايكروغرام / غرام جاف في سمكة كريسختش ، و ٠١. – ١,١٣ ميكروغرام / غرام جاف لسمكة التلابيا ، لذا يمكن ترتيب التركيز من العالي إلى المنخفض : سمك > التربة > الماء . يدل التركيز العالي في السمكة على التراكم الحيوي للمبيد في السمك . أما التوزيع المكاني للمبيد فكان التركيز عالياً في أسفل النهر ، أما التوزيع الوقتي فكان التركيز في الموسم الجاف أعلى منه في الموسم الرطب في الماء و التربة و على العكس في السمك . كانت التراكيز أعلى من التراكيز التي حددتها وكالة البيئة النيجيرية و الاتحاد الأوروبي (٠١. ميكروغرام / ليتر) و هذا يستدعي مراقبة مياه دلتا النيجر .

Keywords: C. furcatus; Diazinon; Niger Delta; Sediment; Surface water; *T.zilli*; Warri River;

1. Introduction

Pesticides play an important role in agriculture and in pest control worldwide. In order to feed the ever - increasing human population in Nigeria, it has become necessary to use pesticides. Pesticides usage in Nigeria dates back to 1948.By 1974, 21 types of organophosphates and carbamate insecticides had been introduced into Nigeria. There was a phenomenal increase in this number in 1979, when the Federal Government introduced the Green Revolution Programme (Badejo and Sosan, 2005).

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Insecticides play an important role in aquatic ecosystems as documented by the accumulated data on their detrimental effects to community structure, reproduction, and developmental process among several taxa including macro invertebrates, amphibians, birds, and other wildlife (Colborn et al., 1999; Thompson et al., 1996)

Diazinon is an organophosphate insecticide; common trade names include Spectracide,Basudin^{TM},Knoxout ^{TM}.Globally the pesticide is the fifth most commonly used pesticide used by homeowners, with two to four million pounds used annually (Cox Caroline, 1992).It originated with Ciba- Geigy in 1952. Various diazinon formulations are widely used in agriculture and for structural pest control, besides being used on lawn, and home gardens. Important target pests for diazinon applications include cockroaches, fleas, ticks, aphids, scales, mites, ants, crickets, flies, and grubs (Extension Toxicology Network,1996). Diazinon may be found in formulations with a variety of other pesticides. Depending on form, the EPA has classified diazinon as a toxicity class II or III pesticide, based on a scale of I to IV, I being the most toxic class (Meister , 2000).

Diazinon is a moderately acutely toxic broad-spctrum insecticide,with a LD_{50}of350 to 400mg/kg for humans (Doherty et al.,1982). Like other organophosphate pesticides, diazinon affects the nervous system through the inhibition of AchE, an enzyme needed for proper nervous system function. Diazinon is easily absorbed through the skin, and can be synergistic with other chemicals (meaning that the two together are more toxic than either alone), including pyrethrins and certain chemicals used in pharmaceuticals (Plott ,1988). The pesticide is highly toxic to fish, aquatic invertebrate, predatory or parasitic insects, soil microbes, and mites. (Coupe et al., 2000).

The United State Geological survey data show that "diazinon is the most commonly found insecticide in surface water nationally"(Hoffman et al .,1994). Diazinon was one of the five pesticides found to be able to concentrate in fog droplets. In 1995, the U.S. Geological survey began a study to determine the occurrence and temporal distribution of 49 pesticides and pesticide metabolites in air and rain samples from an urban and an agricultural sampling site in Mississippi. The study found that every rain and air sample collected from the urban and agricultural sites had detectable levels of multiple pesticides. Diazinon was found to have the highest concentration at the both sites (Glotfelty et al., 1987)

There is currently widespread concern about the concentration and effects of chemical pollutants such as diazinon on aquatic resources in the western Niger Delta (Egborge, 2001; Ezemonye,2005). This is not unconnected with indiscriminate use of pesticides to control water-inhabiting pests and disease vectors in the region (Badejo and Sosan, 2005)

Warri River, a major navigable channel of the Niger Delta, southern Nigeria .It takes its origin from around Utagba Uno and flows through zones of freshwater swamps, mangrove swamps, and coastal sand ridges .The river is a relatively large water body which stretches within latitudes $5^{0}21^{1}$ - $6^{0}00^{1}$ N and longitude $5^{0}24^{1}$ - $6^{0}21^{1}$E, covering a surface area of about 255 sq. km with a length of about 150km (Tetsola, 1980; Gabriel, 1986;Netherlands Engineering Consultants, 1954) .It drains various tributaries and empties into the brackish Forcados River that in turn empties into the Atlantic Ocean (fig 1).

To the author's knowledge this represents the first evaluation of diazinon concentrations in Water, sediment and fish samples from the Warri River.

2. Study Area and Sampling Schedule

The study area is the Warri River with stations established at Ekakpamre, Ovwian and Ovu (Fig. 1). Ekapamre and Ovwian stations were chosen to reflect possible sources of pesticides contamination while Ovu Station served as control. Two dominant fish species were selected for this study; *C. furcatus* and *T.zilli*. Catchments of intensive agricultural use are drained into the stations with the exception of the control. The sampling stations were visited on monthly basis. During this period, water, sediment, and fish samples were collected and analyzed for the pesticide residues

2.1. Ovwian Station

This station is surrounded by vegetable and cassava farms, the river receives effluents from Niger Benue Transport Company (NBTC) and Delta Steel Company (DSC), which are located very close to the river

2.2. Ekakpamre Station

The station has poultry farms where diazinon is regularly used for fumigation. The River also drains catchments of intensive agricultural land.

2.3. Ovu Station

This is the upstream station and is located away from agricultural farms. There is no evidence of pesticide usage around this station, which therefore served as the control station.

3. Materials and Methods

3.1. Sample Collection

All samples were collected from designated stations (Ovwian, Ekakpamre and Ovu) of Warri River between Januarys – August 2006. A total of 96 samples made up of 24 samples each for water, sediment and fish (*C. furcatus* and *T. zilli)* Were collected and analyzed for diazinon.

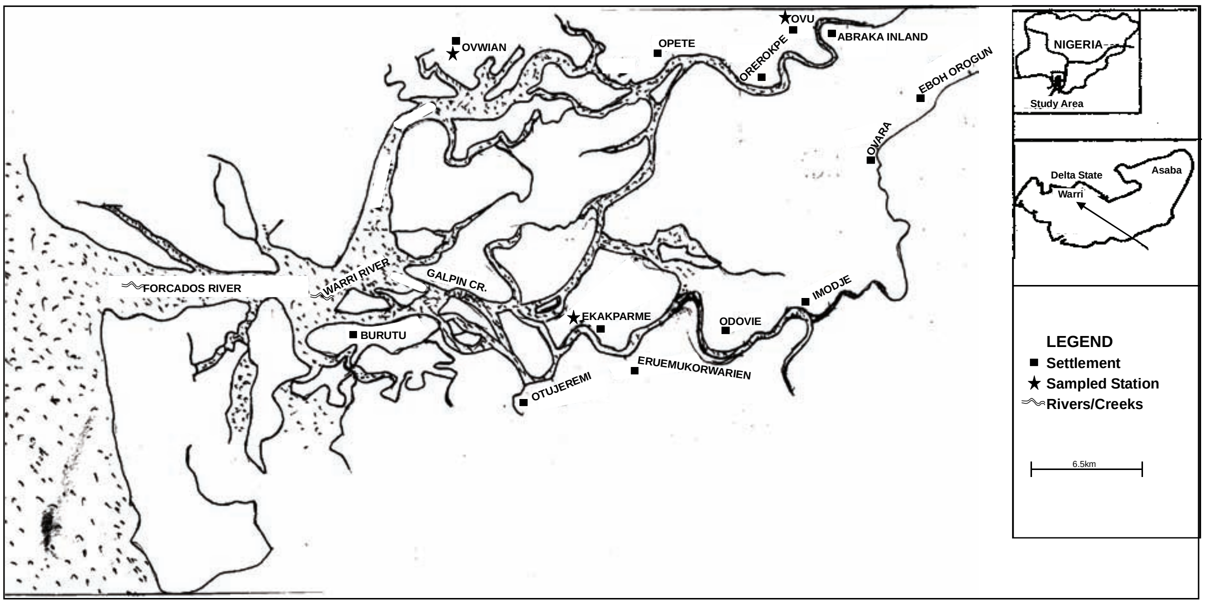

Fig. 1: Map of entire Warri River; **Source:** Ministry of land and survey, Asaba Delta state (Modified after Ezemonye, 2005)

3.1.1. Water Samples

One liter of water samples were taken from 0.3m below the water surface with a pre – cleaned glass bottle using hydrobios sampler. For sampling, turbulent midstream positions of water bodies were chosen to approximate mean concentration of river water. All foreign bodies were removed and the samples thoroughly homogenized. After collection, the samples were stored on ice during transport (<6hrs) and were kept at 4^{O}C in the laboratory (<2 days) until extraction.

3.1.2. Sediment Samples

Sediment samples were taken from the positions where an accumulation of fine – texture substrate took place. The upper 2cm of bed sediment at each site were collected with a teflon-coated spoon and wrapped in aluminum foil. Samples were immediately stored on ice (<6hrs) after collection and stored at – 20^{O}C in the laboratory until analysis

3.1.3. Fish Samples

Fish samples were captured (1.5-2.5kg/sample, 6samples from each station) wrapped in aluminium foil and kept at 20^{O}C in the laboratory until analysis

3.2. Sample Extraction

The procedure applied for the extraction of pesticides was similar to those reported by; Laab et al., (2000), Steinwandter (1990), and Von Duszeinj (1988).

3.2.1. Water Sample

One-liter water sample were extracted using 20ml of hexane: dichloromethane (3:1) for 30 minutes. The extract was concentrated with the aid of rotatry evaporator. Pre-elution was carried out with the HPLC methanol.The concentrated solvent extract was then analyzed for diazinon.

3.2.2. Sediment Samples

Wet sediment samples were homogenized air dried and passed through a no 32 mesh sieve. 15 gram of the sediment were spiked with a solution of surrogate standard (d8-naphthalene, d10-acenaphthene, d12-chrysene and d12-perylene) and extracted with a mixture of dichloromethane and n – hexane in a ratio 2:3, having been subjected to a vigorous shaking in a sonication bath for 5hrs. The solvent was separated and concentrated with a rotatory evaporator. Pre – elution was carried out with HPLC methanol. The concentrated extract was then analyzed for diazinon.

3.2.3. Fish Samples

Sections of the dorsolateral muscle of fish samples were prepared according to Steinwandter (1990) .To estimate the lipid content of the fish, four fish samples were dissected and the lipid tissues isolated .The fish tissues were freeze dried at 60^{O}C Celsius for 4 hours and one gram of dried tissue was weighed into a clean extraction bottle. Diazinon was extracted using acetone, water and dichloromethane. After subjected to vigorous shaking in a solication bath for 5hrs, the solvent was separated, concentrated in a rotatory evaporator and eluted using HPLC methanol The concentrated extract was then analyzed for diazinon.

3.3. Sample Preparation for Analysis

3.3.1. Chemicals/Reagents

Methanol (HPLC analytical grade), Diazinon (98.5% purity), which were used as internal standard in HPLC analysis were obtained from chemical Service, West Chester, U.K.

3.3.2. Equipment/Glass wares

Cecil High performance Liquid Chromatography (HPLC) system comprised of CE 1200 High performance variable wavelength monitor and CEII00 liquid chromatography pump, UV detector with variable wavelength and stainless steel column (C_{18} Reverse phase) packed with Octasilica; vacuum pump and ultrasonic check.

3.3.3. Preparation of External Standard Stock Solution

One milligram per gram stock solutions of the external standard was prepared by measuring 0.1g of diazinon standard into 100ml volumetric flask. A small quantity of methanol was then added to the volumetric flask to dissolve the standard. Distilled water was then used to fill the flask to 100ml mark. The following concentrations (80, 60, 40,20and 10) mg/g were later prepared from the 1mg/g stock solution. Then from the 1mg/g concentrations, lower concentrations up to 0.01mg/g were prepared.

3.3.4. Activation of the HPLC System

The target wavelength for the analyses was determined by UV/visible equipment. A small quantity of the stock solution was diluted with methanol and its wavelength of 202nm was determined by scanning. The instrument wavelength was then set at 202nm, with a sensitivity of 0.05nm and a flow rate of 1ml/min.The instrument was purged to remove air and charge the column. Purging was conducted using a washing solution of 30% methanol and 70% distilled water.

3.3.5. Degassing the Mobile Phase Solution

Helium gas was bubbled into the solution to degassing the mobile phase. The mobile phase was then injected into the instrument and allowed to run through the system for 20 minutes. The system was then separated following the procedures outlined in the instrument operating manual.

3.3.6. Determination of Retention Time for External Standard

The internal diazinon standard was injected into the instrument to determine the retention time. A series of concentrations ranging from 0.025ppm to 100ppm where then injected. The resulting peak areas of the resulting chromatographs were plotted against concentrations to determine the linearity of the standard chromatographs .Using this approach, the retention time for the diazinon standard was 4.23 minutes

3.3.7. Pesticide Analysis

Each sample residue was dissolved in 1ml methanol. The extracted residue was then loaded and injected into the valve of the chromatograph system. The resulting chromatograph for each sample was printed out. The retention times noted, concentrations determined and recorded.

3.3.8. Data Analysis

The data were summarized separately for each sampled station using Description statistics (means, range, and histogram). The student's t-test and analysis of variance (ANOVA) was used to test for the level of significance at 0.05 level of probability for the seasons and the stations respectively.

4. Results

Results of spatial and seasonal variations in the concentrations of diazinon in surface water, sediment and fish (*C. furcatus* and *T. zilli)* from Warri River are presented in Tables 1 with further illustration in Figure 2-5.

4.1. Diazinon in Surface Water

The mean diazinon concentrations in the surface water were; 0.93μg/L ±1.05 (Ovwian), 0.41μg/L ± 1.09 (Ekakpamre) and 0.07μg/L±0.19 (Ovu), while the mean value for dry and wet seasons were; 1.76μg/L ± 0.85 and 0.09μ/L ±0.16 (Ovwian), 0.83μg/L±1.49and 0.01μg/L ±0.1 (Ekakpamre), 0.07μg/L ±0.02 and ND(Ovu). The results showed that the pesticide concentrations were significantly different only in the season at $P < 0.05$ ($F=7.61$).

4.2. Diazinon in the Sediment

The mean diazinon concentrations in the sediment were; 1.88μg / gdw ±1.26 (Ovwian), 0.78μg / gdw ± 1.45 (Ekakpamre) and 0.28μg/ gdw ± 0.42 (Ovu), while the mean seasonal values for dry and wet seasons were; 2.54μg / gdw ± 0.32 and 1.18μg / gdw ± 1.70 (Ovwian), 1.16μg / gdw ±1.44 and 0.04μg / gdw ±0.03 (Ekakpamre), 0.49μg / gdw ± 0.56 and ND (Ovu) respectively. The concentrations varied significantly both in time and space at $P < 0.05$ ($F = 3.13$).

4.3. Diazinon in C. Furcatus

The mean diazinon concentrations in this fish were; 2.23μg/ gdw ±2.80 (Ovwian), 1.00μg / gdw ± 0.80 (Ekakpamre) and 0.86μg / gdw ± 1.08 (Ovu), while the mean values for dry and wet seasons were 0.15μg / gdw ±0.03 and 4.29μg / gdw ± 2.62 (Ovwian), 0.11μg / gdw ± 0.08 and 1.66μg / gdw ± 0.39 (Ekakpamre), 0.09μg/ gdw ±0.14 and ND (Ovu) respectively. The pesticide concentrations varied significantly both in time and space at $P < 0.05$ ($F = 3.13$).

4.4. Diazinon in T.Zilli

The mean values of the pesticide were; 0.29μg / gdw ± 0.37 (Ovwian), 0.04μg / gdw ± 0.05 (Ekakpamre). However, diazinon was not detected in samples collected at station 3. The mean values for dry and wet seasons were; 0.04μg / gdw ± 0.03 and 0.54μg / gdw ± 0.34 (Ovwian), 0.02 μg / gdw ±0.01 and 0.07μg / gdw ±0.05 (Ekakpamre). The concentrations of the pesticide varied significantly both in time and space at $P < 0.05$ ($F = 0.17$).

Table 1. Diazinon concentrations during the dry and wet seasons in (a) River water (b) Fine-particle sediments and (c) Fish species in three named sites on the Warri River, Niger Delta, Nigeria sampled monthly from January to August, 2006. Means are based on the monthly observations. ND = Not Detectable.

		Surface Water (µg/gdw)		Sediment (µg/gdw)		*C. furcatus* (µg/gdw)		*Tilapia zilli* (µg/gdw)	
		Mean ± SD	Range	Mean ± SD	Range	Mean ± SD	Range	Mean ± SD	Range
Dry Season	Ovwian	1.76±0.85	0.50-2.27	2.54±0.32	2.16-2.89	0.15±0.03	0.12-0.19	0.04±0.03	0.01-0.07
	Ekakpamre	0.83±1.49	0.02-0.12	1.16±1.44	0.32-3.31	0.11±0.08	0.02-0.32	0.02±0.01	0.01-0.04
	Ovu	0.07±0.02	0.05-0.09	0.49±0.56	0.02-1.21	0.09±0.14	0.01-0.17	ND	
Wet Season	Ovwian	0.09±0.16	0.01-0.12	1.18±1.70	0.01-3.64	4.30±2.62	2.10-7.5	0.54±0.34	0.13-0.91
	Ekakpamre	ND		0.04±0.03	0.01-0.07	1.66±0.39	1.32-2.01	0.07±0.05	0.01-0.12
	Ovu	ND		ND		ND		ND	

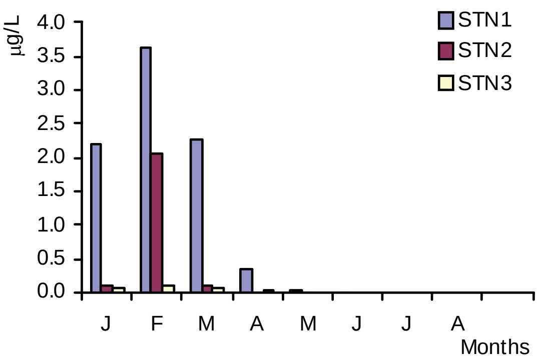

Fig. 2: Monthly variation in surface water diazinon concentrations (µg/L) in Warri River

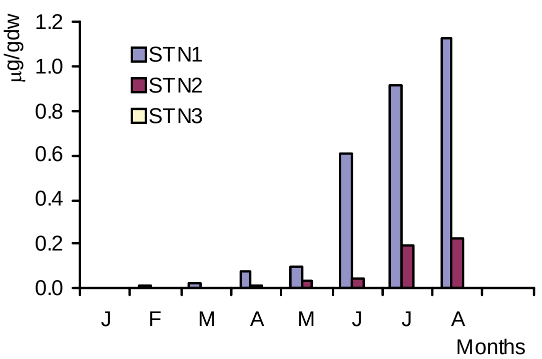

Fig. 5: Monthly variation in *T. zilli* diazinon concentrations (µg/gdw) in Warri River

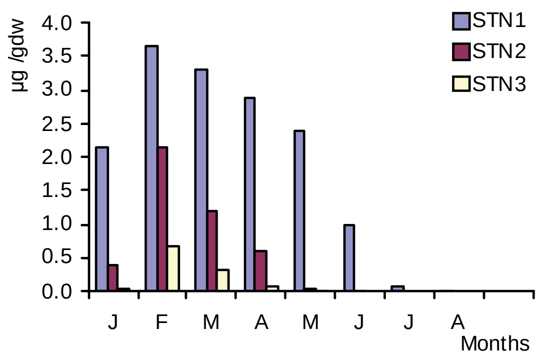

Fig. 3: Monthly variation in sediment diazinon concentrations (µg/gdw) in Warri River

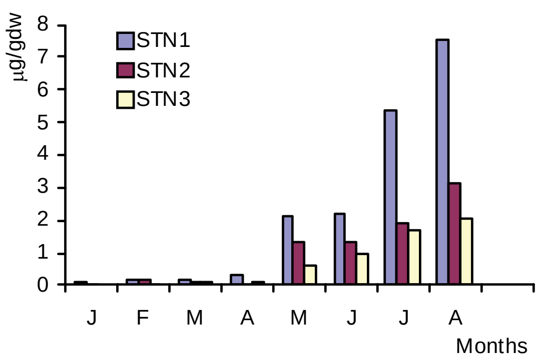

Fig. 4: Monthly variation in *Chrysichths furcatus* diazinon concentrations (µg/gdw) in Warri River

5. Discussion

This study reported the occurrence of varying concentrations of diazinon in surface water, sediment and fish (*C. furcatus* and *T. zilli*) samples from designated stations of the Warri River.Diazinon was detected in all the stations and matrices. It was observed in the station far from agricultural activities with no evidence of pesticide usage except in *T. zilli* tissue where the concentration was insignificant. This is indicative of the ubiquitous use of diazinon outside agricultural usage (Leonard et al., 1999)

Concentrations of diazinon in sediment were much higher than concentrations in water. In general, these results support that concept of sediments acting as a sink for pollutants. The observation corroborated the finding of Voss and Embrey, (2000) where they reported higher concentrations of dichlorvos and malathion in bottom sediment of a small streams toxicity/pesticide study in selected small streams in King and Snohomish Counties, Washington

Higher concentrations of diazinon detected in the fish (*C. furcatus*) from Ovwian and Ekakpamre Stations indicated the long-term effect of agriculture and other human activities. Schmitt et al., (1990), reported similar result during the biomonitoring program of pesticides residues in United State fresh water fish, sediment and water and observed that the concentrations of pesticides compounds detected in fish tissue from eastern Iowa stream and rivers were as a result of long term effect of previous human activities.

Concentrations of diazinon were higher in C. *furcatus* than sediment indicating possible bioaccumulation and poor elimination in the fish (Wilfred, 1995).Diazinon levels varied significantly between the two fish species (*C. furcatus* and *T. zilli*) at P< 0.05 (F= 4.90). This may be attributed to lipid content, size and species (Hosteller *and* Kidwell, 1990). The bottom detritus and predatory feeder *C. furcatus* had diazinon residues significantly higher than the herbivorous *T. zilli.* This variation is probably due to different feeding and living habits. Predatory fishes might bioaccumulate the pesticide by eating other fishes and the constant contact of the fish with the sediment allows their continuous exposure to pesticides. Kent and Johnson, (1974) found that Utah Sucker (*Castomus ardens*) a bottom feeder, contained the highest level of pesticide compounds in American fall Reservoir.

Higher values of diazinon were recorded in the dry season in surface water and sediment samples which is consistent with the observation of Osibanjo and Jensen, (1980) and may be attributed to the planting season (dry season) when farmers treat their farms before planting .Most of the crops grown in these areas (Ovwian and Ekakpamre) are seasonal and include leafy vegetable such as *Amaranthus* species, fluted pumpkin and bitter leaf. Other crops include tomatoes, Okra, sweet pepper, garden eggs and tuber crops like cassava. The concentrations of the diazinon observed in the water and sediment during the rainy season could be as a result of the inactivity during the period in the area.

Lower concentrations of diazinon were observed during the dry season. The pesticide concentrations in (*C. furcatus*) were 53% higher in the raining season than concentrations in the dry season. This could be attributed to; different living habits of the fishes between the seasons, breaking down of the diazinon in environment and absorption of the pesticide through the skin .According to Leight et al., (1999) the accumulation of organic contaminants in the tissue of aquatic organisms is a complex function of the physicochemical properties of the contaminants, its distributions in the aquatic system, the feeding behaviour and metabolism of the aquatic organism.

The concentrations of diazinon increased towards the downstream direction with Ovwian station having highest concentrations. The upstream station (Ovu) was comparatively low with the pesticide contamination; this condition may be due to very low to absence of agricultural activities in the area

6. Conclusion

This study presents the first site-specific data on diazinon concentrations in the Warri River of Niger Delta, Nigeria. It also provides a platform for developing regulatory measures to control contamination of aquatic environments in this region. This is against the background that diazinon levels in Warri river samples from Ovwian, Ekakpamre and Ovu stations exceeded The Environmental Protection Agency (EPA) recommended limit of 0.02µg/L (Hamilton, 2003)

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