

Original Research Article

LEVEL OF ORGANOCHLORINE PESTICIDES RESIDUE IN FISH, WATER AND SEDIMENT IN BIU DAM (RESERVOIR) BORNO STATE NIGERIA

ABSTRACT

Health risks posed by organochlorine compounds to the total environment have continued to

elicit concern among researchers in developing countries worldwide. Hence, this study focused on investigating the levels of Organochlorine pesticides residues in water sediment and fish samples in Biu dam reservoir. The pesticides residues in the fish and sediment samples were extracted by soxhlet extraction process using a mixture of hexane and acetone while water sample was extracted using dichloromethane liquid- liquid extraction method. The extracts were cleaned and analysed using a gas chromatograph mass spectrometer (GCMS) equipped with electron capture detector. Endrin, aldrin, dieldrin, endosulfan I, endosulfan II, alpha BHC and heptachlor were detected in the study area. While chlordane, methoxychlor, other isomers of BHC, DDT and its degradation products were not detected in all the samples analysed. Endrin had the highest value of 14.758 mg/Kg in catfish while the lowest endrin value of 0.017mg/Kg was found in sediment. The findings provide evidence of ~~accumulation~~ *pollution* of some the organochlorine pesticides residue in Biu dam reservoir which is slightly contaminated with organochlorines pesticide. Therefore serious need for the monitoring of these pesticide residues in water, sediments, fish and the environment, as this will go a long way towards preventing various environmental and public health hazards.

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Key words: Organochlorines, Pesticides, Sediments, Reservoir, Biu dam, water

INTRODUCTION

Pesticides are substance used to kill, repel, or control certain forms of plant or animal life that are considered to be pests. Pesticides include herbicides for destroying weeds and other unwanted plants, insecticides are used for controlling a wide variety of insects, fungicides used to kill or prevent the growth of molds and mildew, disinfectants for preventing the spread of bacteria, and compounds used to control mice and rats. Because of the widespread use of agricultural chemicals in food and agricultural production, people are exposed to low levels of pesticide residues through their diets.

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34 Fishes are suitable indicators for environmental pollution monitoring because they concentrate
35 pollutants in their tissues directly from water and also through their diet [1]. Pesticides are
36 frequently applied in agricultural, forestry, and urban settings. There are tens of thousands of
37 pesticides in use, many of which are synthetically produced. Pesticides will break down in the
38 environment forming by-products, some of which are toxic whereas others are relatively non-
39 toxic [2, 3]. Acute (immediate) toxic effects can influence the survival or reproduction of aquatic
40 species leading to the disruption of predator-prey relationships and a loss of biodiversity. If
41 aquatic organisms are not harmed immediately, they may ~~concentrate~~accumulate chemicals
from their
42 environment into their tissues. This bio-concentration can lead to bio-magnification, a process in
43 which the concentrations of pesticides and other chemicals are increasingly magnified in tissues
44 and other organs as they are transferred up in the food chain. Terrestrial predators that feed on
45 aquatic species may also be affected [2,4]. People are exposed to pesticides through aquatic
46 systems either by ingesting fish or shellfish that have stored these compounds in their tissues or
47 directly by drinking contaminated water. Reports have shown that pesticide exposure has been
48 linked to cancer, neurological damage, immune system deficiencies, and problems with the
49 endocrine system [4,5,6, 7].

50 Organochlorine pesticides (OCPs) are a class of non polar toxic chemical compounds classified
51 as dichlorodiphenylethane cyclodienes and chlorinated benzenes [8]. OCPs are ubiquitous
52 environmental contaminant which have spread globally and have been detected in food stuffs,
53 meat, drinking water and sediments as well as wide range of biota including fish [9]. Numerous
54 studies on both human and laboratory animals provide strong evidence of the toxic potential of
55 the exposure to OCPs. The health effects associated with OCPs include reproductive failures,
56 birth defect, endocrine disruption immune system dysfunction and cancer [10, 11, 12]. Other

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investigations confirmed that OCPs have strong potential to cross placental barriers even in minute concentration and cause serious neonatal damage [13].

OCPs are widely used by farmers because of their effectiveness and their broad-spectrum activities [14,15]. Moreover, OCPs showed very resistant characteristic ~~es~~ to microbial degradation and are employed to control ecto-parasites of farm animals and pests. However, this chemical and several other 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, irrigation, industrial, domestic and human consumption. Africa has therefore been suggested as a highly relevant area for increased research activities to establish possible links between the increasing number of unexplained cancer cases and exposure to anthropogenic chemical pollutants in food, air, water and soil [16].

Organochlorine pesticides such as DDT are still frequently in use all over Africa both as a means for effective and cheap vector control health and for agricultural purposes [17]. One of the most controversial pesticides of all time, dichlorodiphenyltrichloroethane (DDT) is today even being introduced on a broader scale to defeat malaria, despite being banned in most countries of the world [18]. From an African perspective this might be understandable, since malaria still is a tremendous problem killing one child every 30 minutes in sub-Saharan Africa [19].

The contamination of the environment and food chain by chlorinated organic pesticides has become a topical issue of considerable concern in many parts of the world, and has led many researchers to investigate their occurrence, distribution and concentrations in commercially sold fruits, vegetables, milk, fish and water [20, 21, 22, 23 & 24]. Organochlorine pesticides have been used extensively worldwide since the early 1950s [25,26]. Dichlorodiphenyltrichloroethane

80 and HCH or BHC which had been produced for use in agriculture, forestry, and public health for
 81 more than four decades, are considered to be the most important OCPs in China [27]. Soil is
 82 likely the largest sink in the environment for OCPs and the release of these contaminants from
 83 __soils ~~continues~~continue to be a source to other environmental compartments [28]. Most of
 84 organochlorinated pesticides have been progressively restricted and then banned in the 1970s in
 85 most industrialized countries, since their uses have resulted in a widespread environmental
 86 pollution [29]. Rain water and irrigations can take away pesticides from their place of application
 87 areas to the surface waters and even ground water. Organochlorinated pesticides solubility in
 88 water is limited, because they are mostly lipophilic compounds so they tend to connect with
 89 the suspended matter, to precipitate in sediments, to accumulate and concentrate in biota of
 90 aquatic systems [30, 31]. Literature data on the concentrations of OCP residues in the Nigerian
 91 __environment especially in north east Nigeria are inadequate. There is need for continuous
 92 monitoring to identify the occurrence and the levels of OCPs in fishes in Nigeria. In order to
 93 obtain more information about the pathways along which bioaccumulation occurs, it is important
 94 to investigate the distribution of pesticide residues in biota where fish use water to survive and
 95 some feed on sediments. This study was undertaken to determine the levels of organochlorine
 96 residues in two species of fish, water and sediment in Biu dam reservoir, Borno state.

97 MATERIALS AND METHOD

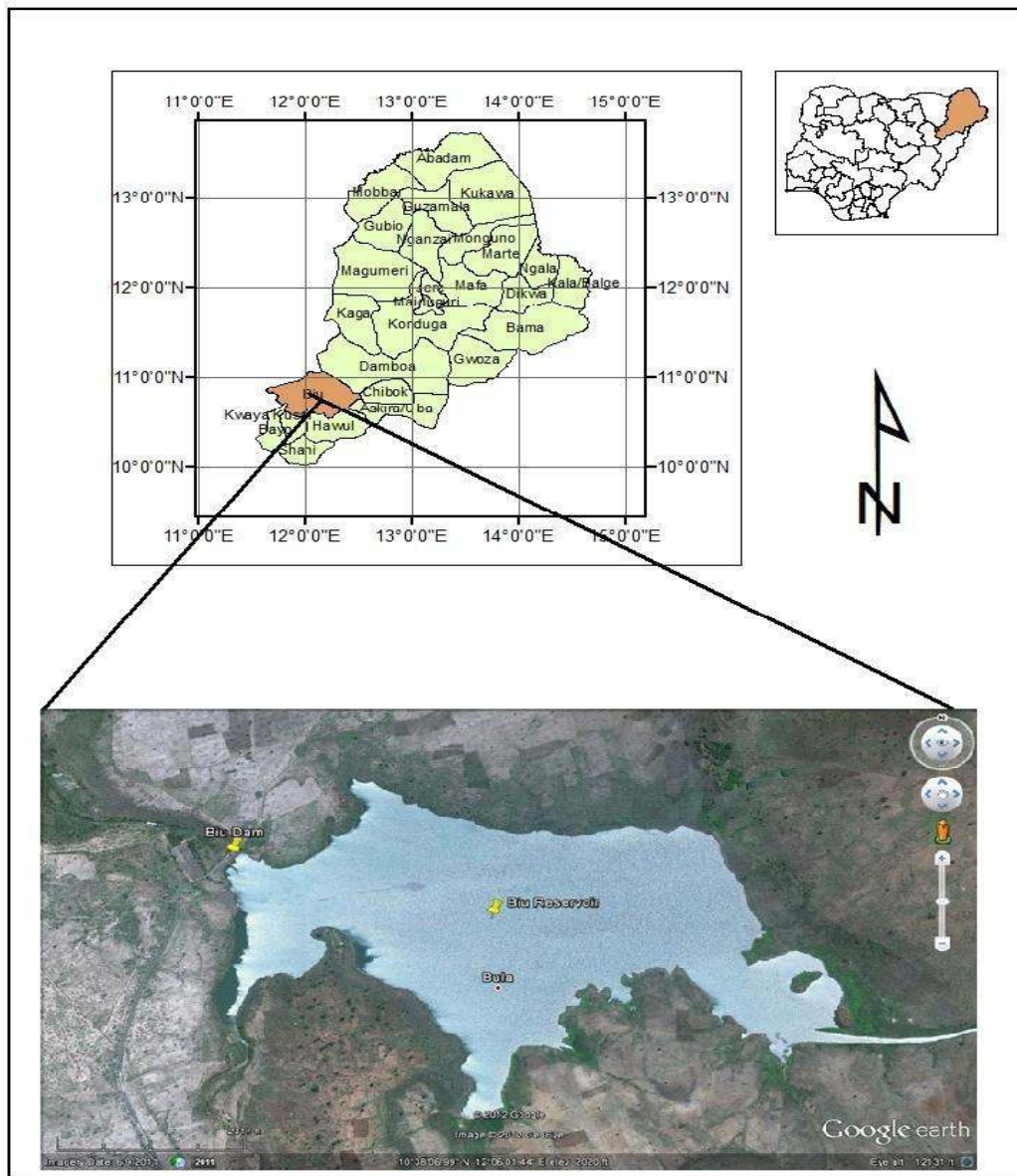
98 **Study area:** Biu dam is situated in Biu local government in Borno state and located on latitude
 99 $10^{\circ}38'00''$ N and longitude $12^{\circ}05'31''$ E. The dam was constructed purposely to supply drinking
 100 water to Biu community being the second largest town in Borno state and the reservoir is to be
 101 used for irrigating the fertile agricultural land around the dam but this project was abandoned

102 be successive government. The water from the dam reservoir is primarily use in irrigation,
103 fishing and source of water large cattle farmer in the area.

104 **Sample collection:** Samples of the environmental matrices-sediment, water and biota (fish) were
105 collected at four locations from the study area for a period of July, 2013 - May, 2014 and
106 prepared into composite samples during the dry and wet seasons. ~~The~~ Fish species were
collected
107 alive from their normal environment, martyred and stored in an ice packed container before
108 transporting to the laboratory where they were stored at temperature of -20°C until ready for use.
109 The fish species collected were *Tilapia zilli* (Tilapia) and *chrysichthys nigrodigitatus* (Catfish).
110 They were identified by a specialist from Department of fisheries and Aquaculture Adamawa
111 state university, Mubi.

112 The sediment samples were collected using the triple sediment corer made by Uwitec fitted with
113 Plexiglas tubes of 50cm height. Sediment samples were collected at 2 and 8 cm depths from the
114 dam reservoir and brought to the laboratory as soon as possible under temperature-controlled
115 conditions. In the laboratory, sediment cores were weighed dried at 40°C until constant dry
116 weight samples were obtained. Samples were then homogenized using Agatha mortar and pestle
117 and sieved with 0.5mm mesh sieves. All samples were stored in sealed plastic bags at -4°C until
118 further analysis.

Comment [a1]: Plastic bags are usually avoided in case of pesticides residuals may be due to PCBs contamination interrupting in detection of organochlorines.



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120 _____ Figure 1: Map of Borno State showing the location of Biu
121 dam/reservoir

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124 Water sample from Biu dam reservoir were collected using 2L amber glass bottles. Water
125 samples were pre-filtered through 0.45 μ m fiber glass filters (Whatman) to remove debris and
126 suspended material [32]. The samples were preserved by refrigeration at -10°C until analysis was
127 undertaken

128 ***Extraction of water samples***

129 Liquid-liquid extraction method was used to extract organochlorine pesticides from the 2 L water
130 samples earlier collected. About 500 mL of water sample was put into a 1 L separatory funnel at
131 a time. This was extracted with 10 mL (3 portions) of dichloromethane (DCM) by vigorous
132 shaking for 30 minutes for each of the triplicate extraction. The combined extracts were dried
133 with anhydrous sodium sulphate $\text{Na}_2\text{SO}_4(\text{s})$ and concentrated to about 2 mL under a pure stream
134 of nitrogen of 99.999% percentage purity.

135 ***Extraction of sediment samples ;***

136 Dry Sediment sample was extracted according to Darko *et al* [32], with some slight
137 modifications. About 10 g of sediment samples were weighed and transferred into extraction
138 thimble that had been previously washed with n-hexane and acetone and oven dried. The sample
139 was extracted using 100 mL of n-hexane acetone mixture 4:1 v/v for eight hours using Soxhlet
140 extractor. The extract was evaporated to dryness using a rotary evaporator at 40°C . Each extract
141 was dissolved in 10 mL n-hexane and subjected to clean-up procedure.

142 ***Extraction of sediment-fish samples;***

143 The frozen composite whole-body of fish tissue samples were homogenized using an agate
144 mortar and pestle [33, 34]. Approximately 20 g of the properly chopped sample was further
145 mixed with 10 g of anhydrous sodium sulphate. This was Soxhlet extracted for eight hours using

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146 dichloromethane/n-hexane mixture. The extract was dried over anhydrous sodium sulphate and
147 concentrated to about 2 mL as described earlier in readiness for the clean-up procedure.

148 ***Clean-up procedure:***

149 A column of about 15 cm (length) × 1 cm (internal diameter) was packed with about 5 g
150 activated (at what degree?) silica gel prepared in a slurry form in n-hexane. About 5g of
anhydrous sodium
151 sulphate was placed at the top of the column to absorb any water in the sample or the solvent.
152 The column was pre-eluted with 15 mL of n-hexane without the exposure of the sodium sulphate
153 layer to air. The reducedEvaporated volume of extract was placed in the column and allowed to
sink below the sodium

154 sulphate layer. Elution was done with 2 × 10 mL portions of the extracting solvent (DCM). The
eluate was then collected, dried with anhydrous sodium sulphate and then evaporated to dryness

156 under a stream of analytical grade nitrogen (99.999%). The dried eluate above was then

146—157 reconstituted with 1mL spectra grade n-hexane and 0.5 mL of 20 mg/kg mixture of

under a stream of analytical grade nitrogen (99.999%). The dried
eluate above was then
reconstituted with 1mL spectra grade n-hexane and 0.5 mL of
20 mg/kg mixture of
157

158 organochlorines pesticides (Mention number or name of standard) we added as an internal

158—standard. 1.0 µL of the mixture was injected

159 into the GC-MS column for analysis.

159—160 Organochlorine pesticides reference standard was obtained

from Resetek,USA. The modern

160—161 Shimadzu GC-MS QP-2010 was employed in analysing the

161 standards and the calibration curve

for each organochlorine compound was prepared

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163 RESULT AND DISCUSSION

164 The distribution of various organochlorine pesticides in water, sediment and fish from Biu dam
165 reservoir during dry and wet season is summarized in Tables 1&2 respectively. Organochlorine
166 pesticides were detected in water, sediment and fish samples. Endrin had the highest residue
167 value of 14.758 mg/Kg obtained in *chrysichthys nigrodigitatus* (cat fish) followed by aldrin
168 residue value of 1.464 mg/Kg found in *Tilapia zilli* (Tilapia) while lowest pesticides residue

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Table 1: Shows the level of Organochlorines (mg/kg) in water, sediment, and Fish found in Biu dam reservoir in dry season

Samples					
Pesticides	Water (mg/L)	Sediment (mg/Kg)	Tilapia(mg/Kg)	Catfish(mg/Kg)	
Gamma BHC	ND	ND	ND	ND	
beta-BHC	ND	ND	ND	ND	
alfa BHC	ND	ND	5.389	0.056	
Endosulfan I	0.033	ND	ND	0.042	
Endosulfan II	ND	ND	ND	0.122	
Heptachlor	ND	0.036	ND	ND	
Aldrin	0.183	0.090	1.464	ND	
Dieldrin	ND	ND	0.044	ND	
Endrin	ND	0.017	ND	14.758	
Lambda-Cyhalothrin	ND	ND	0.147	0.010	
Gamma-chlordane	ND	ND	ND	ND	
p,p'-DDT	ND	ND	ND	ND	
pp'-DDE	ND	ND	ND	ND	
DDD	ND	ND	ND	ND	
Methoxychlor	ND	ND	ND	ND	

ND : Not Detected (at the limit of 1×10^{-5} mg/kg)

Table 2: Shows the level of Organochlorines (mg/kg) in water, sediment, and fish found in Biu dam reservoir in wet season

Samples					
Pesticides	Water (mg/L)	Sediment (mg/Kg)	Tilapia(mg/Kg)	Catfish(mg/Kg)	
Gamma BHC	ND	ND	ND	ND	
beta-BHC	ND	ND	ND	ND	
alfa BHC	ND	ND	2.389	0.026	
Endosulfan I	0.033	ND	ND	0.032	
Endosulfan II	ND	ND	ND	0.022	
Heptachlor	ND	0.016	ND	ND	
Aldrin	0.083	0.040	0.164	ND	
Dieldrin	ND	ND	0.024	ND	
Endrin	ND	0.010	ND	1.758	
Lambda-Cyhalothrin	ND	ND	0.347	0.030	
Gamma-chlordane	ND	ND	ND	ND	
p,p'-DDT	ND	ND	ND	ND	
pp'-DDE	ND	ND	ND	ND	
DDD	ND	ND	ND	ND	
Methoxychlor	ND	ND	ND	ND	

ND : Not Detected (at the limit of 1×10^{-5} mg/kg) means LOD of instrument is 0.00001 mg/kg

value for endrin is found in sediment sample during dry season. Aldrin was detected in all the sample analysed except in cat fish while, chlordane, Methoxychlor, other isomers of HCH, DDT and its degradation products were not detected in all the samples analysed. In the rainy or wet season there's a significant decrease in the residue level of this pesticides, these decrease may be attributed to dilution of these chemicals in water, sediment and fish samples analysed as shown in table 2. Endrin is an organochlorine pesticide that was primarily used as an insecticide, as well as rodenticides. The compound endrin became infamous due to its persistence in the environment and for this reason it is banned in many countries. The majority of endrin (about 80%) was consumed as a spray to control insect pests of cotton. The remaining 20% was also used on rice and for seed treatment [35]. Cotton, maize and rice are commonly cultivated during wet and dry

242 season in Biu and along the entire bank of the dam reservoir. The levels of OCPs detected in the
 243 fish samples of the present study, were in most cases relatively higher than the values reported
 244 by Ogunfowokan [36], in freshwater and fish samples collected from various locations in
 245 Osogbo. Organochlorine pesticides tend to accumulate in living organisms especially in aquatic
 246 organisms and they substantially settle on the sediments [37]. Agricultural runoff is the primary
 247 source of these pesticides in aquatic ecosystems. Endosulfan I & II were detected in water and
 248 catfish respectively but not in sediment and Tilapia. Endosulfan has been shown to be highly
 249 toxic to fish and marine invertebrates and is readily absorbed in sediments. It therefore represents
 250 a potential hazard in the aquatic environment [38]. The value of endosulfan in this study
 251 0.033mg/L in water is lower than study carried out by Idowu *et al*, [39]
 252 The high level of endrin in catfish reported in this study shown in tables 1 & 2, may be due to
 253 agricultural runoff which may be attributed to an unethical used of this pesticides spray on cotton
 254 to control cotton pest, since the used of these pesticides are banned in Nigeria. The catfish tend
 255 to live in the turbid and cloudy water, and furthermore it is exposed to different types of
 256 environmental contaminations rather than another fish types. Endrin in this study is higher than
 257 values reported in fish by Ogunfowokan *et al*; [36]. The level of endrin in sediment reported in
 258 this **studystudy** is higher than those reported by Williams [40].
 259 Aldrin was detected in all the samples analysed except in catfish while dieldrin was detected
 260 only in Tilapia. The presence of these pesticides agrees with (Lanfranchi *et al*. [1], which
 261 reported that fish are suitable indicators for environmental pollution monitoring because they
 262 concentrate pollutants in their tissues directly from water and also through their diets. This study
 263 has clearly proved that this pesticides is still been used illegally in the study area since aldrin is
 264 used as a soil insecticide to control root worms, beetles, and termites which are common pest in

Comment [a3]:

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265 in the study area over the years. Farmers in the Biu local government over the years were having
266 problem of termite destroying their crops and affecting germination of cotton seed in the area.
267 Dieldrin has been used in agriculture for the treatment of soil and seed and in public health to
268 control disease vectors such as mosquitoes and tsetse flies [41]. The level of aldrin and dieldrin
269 in this study is higher than those of fish reported in Allau dam reservoir by Akan, *et al*; [42]. The
270 concentrations of aldrin and dieldrin in Tilapia fish sample were much higher than the WHO and
271 FAO [43], set maximum residue limit (MRL) of 0.2 µg/kg and the Acceptable Daily Intake
272 values (ADIs) of 0.0001µg/kg.

273 The commercial insecticide HCH sometimes referred to as BHC is a mixture of different isomers
274 mainly α , β and γ -HCH (Lindane). Other isomers in the group are delta and epsilon. Lindane (γ -
275 -HCH) has been used as an insecticide and is the most toxic. β -HCH is the most symmetric and
276 stable isomer; it is also the most persistent in nature. β -HCH is eliminated five times more
277 slowly from the body than other isomers and has a much higher ability to accumulate in the fat
278 tissue than lindane [44]. This may be the reason why some of this isomers were not detected in
279 all the samples analysed except alpha BHC in Tilapia and Catfish. The remaining two isomers β
280 and γ -BHC were not detected in the study. The non detection of some of these pesticides shows
281 that they are not in use in the surroundings of the dam reservoir which implies that the banned of
282 these pesticides usage is being enforced in Nigeria. Lindane is also rapidly degraded in the
283 environment [44], this explains the reason why lindane in all the samples analysis were not
284 detected. Generally, the result of this study aggress with the study carried out by Idowu *et al*;
285 [39], which reported the organochlorine pesticide residue levels in river water and sediment from
286 cocoa-producing areas of Ondo State.

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289 **Conclusion:**

290 This study has shown that water, sediment and fish samples from Biu dam reservoir are
 291 contaminated with some organochlorine pesticides residue in varied degrees in both dry and wet
 292 seasons. Higher levels of OCPs were recorded in fish, however, higher levels of OCPs were
 293 found in the fish samples in the dry season than during the rainy season may be possible due
 294 bioaccumulation tendency of OCPs in the fish species during the dry season. Generally, the
 295 seasonal levels of OCPs in the fish samples were higher in most cases, than the recommended
 296 WHO Maximum Residue Levels (MRLs) in food items and should give cause for concern. The
 297 occurrence of the OPCs in fish sediments and water from Biu dam reservoir is consistent with
 298 the agricultural activities of the study area due to pesticide usage by the farmers. Biu dam is
 299 slightly contaminated with organochlorines pesticides, there is therefore serious need for the
 300 monitoring of these pesticide residues in water, sediments, fish and the environment, as this will
 301 go a long way towards preventing various environmental and public health hazards.

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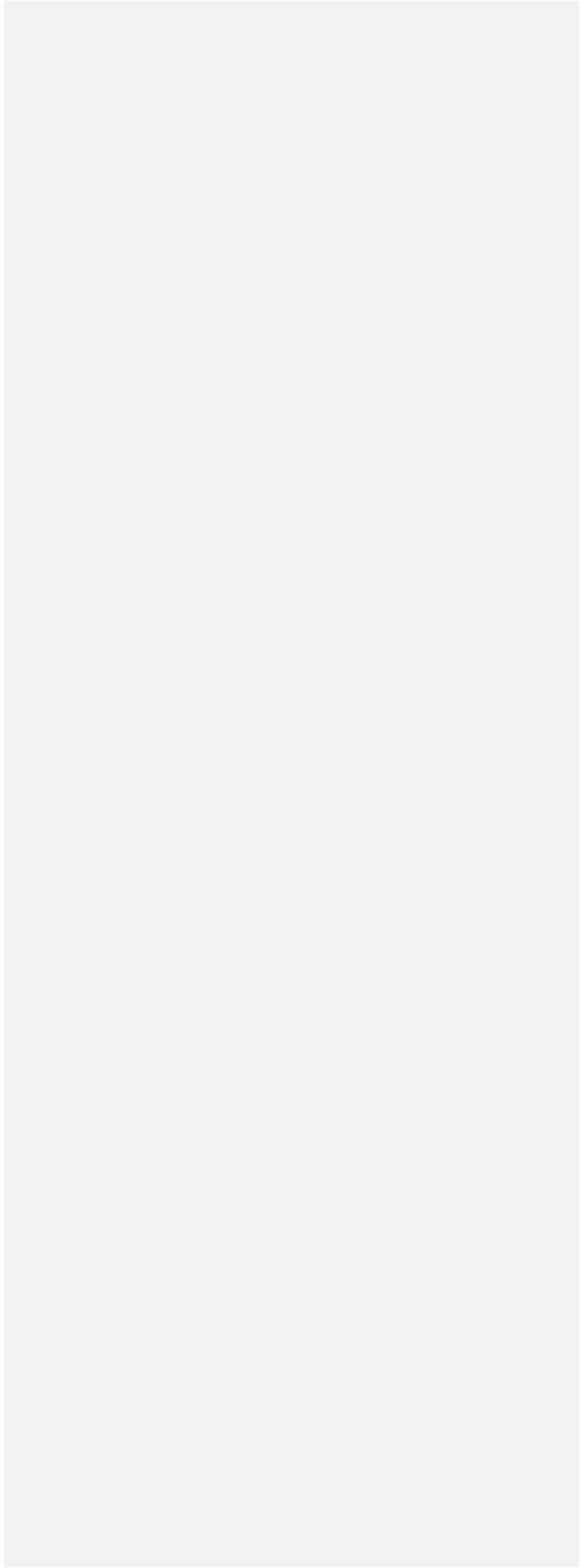
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