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Study on Organochlorine and Organophosphorus Pesticide Residues in Surface and Underground Water Samples of Daudkandi Upazila in Bangladesh

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Abstract: The farmers of Bangladesh mainly rely on pesticides for pest control to boost the crop production. This study was carried out to ascertain the presence and subsequent amount of some organochlorine and organophosphorus pesticide residues in water samples collected from pond, ditch and tube-well of Daudkandi upazila. Using the High Performance Liquid Chromatography (HPLC) a total of thirty water samples was examined to evaluate the suspected pesticide residues. Among the organophosphorus pesticides, diazinon was detected only in one water sample at a concentration of 651 µg/l, whereas, eleven water samples were found to be contaminated with malathion ranging from 1000 to 8200 µg/l. Chlorpyrifos was also detected only in one water sample at a concentration of 31 µg/l. None of the tested water samples was found to be contaminated with suspected organochlorine pesticide residues. On the other hand, tube-well water samples were totally free from suspected pesticide residues. Appropriate measures should be taken to lessen the over dependence on pesticide in agriculture and thus decrease the possible health risk of the people.

Key words: Organochlorine · Organophosphorus · HPLC · Water Sample · Pesticide Toxicity

INTRODUCTION

Agricultural sector, on which the economy of Bangladesh is mostly dependent, has derived a remarkable benefit in the last few decades using pesticides. Pesticides have been widely applied to protect agricultural crops since the 1940s, and since then, their use has increased steadily [1]. Endrin was the first imported pesticide in Bangladesh of which three metric tons (MT) was first imported for agricultural purpose in 1995 [2]. The government of Bangladesh provided pesticides to the farmers free of cost until 1974 to increase the crop production then full price was imposed in 1979. At present, 173 active ingredients with 2,947 trade names have been registered as agriculture pesticides whereas; other 73 active ingredients with 415 trade names and 3 active ingredients with 4 trade names have been registered as Public Health Pesticide (PHP) and Bio-Pesticide, respectively [3]. The use of organochlorine insecticides (except heptachlor) for all agricultural and public health purposes was banned in Bangladesh by late 1993 [4]. But illegal trans-boundary entry has made the banned product available to the farmers and is still being used in agriculture.

The economy of Daudkandi upazila is mainly agro-based. It is a flood-prone and the most intensive agricultural area in Bangladesh. It is one of the potential agricultural areas of the country. The main crops of this upazila are paddy, wheat, jute, sugarcane, potato, mustard plant, pepper etc. The population density of this upazila is 1111 per square kilometer (sq km) [5]. To provide food security to this highly dense population as well as to

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other parts of the country, farmers use various types of pesticides and techniques to boost the crop production.

Pesticides have been promoted and increased in Bangladesh to intensify the crop production for overly growing population [6]. According to Bangladesh Bureau of Statistics [7], the pesticide consumption in Bangladesh increased from 11610.66 to 40882.94 metric tons (MT) from the year of 1998 to 2012. But the use of pesticides in Bangladesh is still low in comparison with other countries of the region [4]. According to Bangladesh Crop Protection Association (BCPA) [8], a negative trend in pesticide consumption in Bangladesh has been observed from 2008, when 48688.02 metric tons (MT) of pesticide was consumed but in 2014, it has been decreased to 35801.68 MT.

The estimated loss in yields due to attacks from pests and diseases annually ranges from 15 to 25 percent [9]. Pests globally consume food estimated to feed an additional one billion people [10]. The loss of crops due to pests is around 30-40 per cent, which is equivalent to the food consumed in the country in three or four months [11].

Due to inadequate knowledge and instruction about pesticide application, farmers use excessive amount of pesticides than required. It has been estimated that, only about 0.1% of the applied pesticides reach the target organisms where the remaining portion contaminates the surrounding environment [12]. The residues of these applied pesticides are drained to the water bodies by various means and pollute them easily. After spraying, pesticides may move downward in the soil and either adheres to particles or dissolve, taken by plants, vaporize and enter the atmosphere, reach surface and underground water through runoff and leaching [13].

The toxic effect of pesticides to the non target organisms is a common incident. Fishes are the main non target species adversely affected by application of hazardous pesticides [14]. Natural insect enemies like parasitoids and predators are the most prone to insecticides which are severely affected [15]. Pesticides also pose negative effect on pollinators which are prerequisite for crop production. They are also the culprit to cause mortality of birds, frogs, fishes and other aquatic and terrestrial organisms.

Increased accumulation of pesticide residues in the food chain and drinking water have been reported to pose serious human health hazards [16]. Due to pesticide exposure, several problems can be caused in human body. An augment in cancer risk is coupled with the use of organochlorine (OC) pesticides [17]. There is a positive association between pesticide exposures with Parkinson's disease (PD) [18]. Pesticides exposure during pregnancy may lift up the risk of hypertensive disorders [19].

In this study, some selected organochlorine and organophosphorus pesticide residues were evaluated in water samples to obtain the current information about their contamination in Daudkandi upazila and also to assess the possible health risk so that necessary steps can be taken.

MATERIALS AND METHODS

A total of thirty water samples were monitored to evaluate the presence and quantity of organochlorine (dichlorodiphenyltrichloroethane (DDT), dichlorodiphenyldichloroethylene (DDE) and dichlorodiphenyldichloroethane (DDD) and organophosphorus (diazinon, malathion and chlorpyrifos) pesticide residues in water samples of pond water, ditch water and tube-well water from five different unions of Daudkandi upazila in Bangladesh (Fig. 1).

For conducting the present study, water samples were collected in glass bottle having 1000 ml capacity for each sample and were labeled as well as instantly carried to the laboratory of Agrochemical and Environment Research Division and kept at -20°C for further analysis.

To carry out the study, all the pure analytical standards for the suspected pesticides were purchased from GmbH (D-86199 Augsburg, Germany). Acetonitrile (HPLC grade), n-hexane (HPLC grade) and anhydrous sodium sulfate ($Na_2SO)_4$ were purchased from Merck (Darmstadt, Germany) whereas, diethyl ether were from BDH, England. On the other hand, rotary vacuum evaporator (R-215) was purchased from BUCHI, Switzerland; florisil (magnesium silicate, mesh 60-100, active at 1250 °F) from Janssen Chimica; N_2 blower (PU 90003) from Alfa Industry, England; High Performance Liquid Chromatography from Waters Company, England and the column C_{18} was of Nova Pack.

For determination of pesticide residues, 250 ml of water sample was taken into a 1000 ml separating funnel in the presence of 100 ml double distilled (DD) n-hexane as solvent and was shaken for mixing well for about 15 minutes. The mixture was then kept standing for 10 minutes for settling down. After that, lower aqueous layer and upper hexane layer were collected in two different conical flasks. The aqueous layer was re-extracted twice by adding 50 ml solvent (DD n-hexane) for each re-extraction and then the upper layer (solvent) was collected. Combined extract was

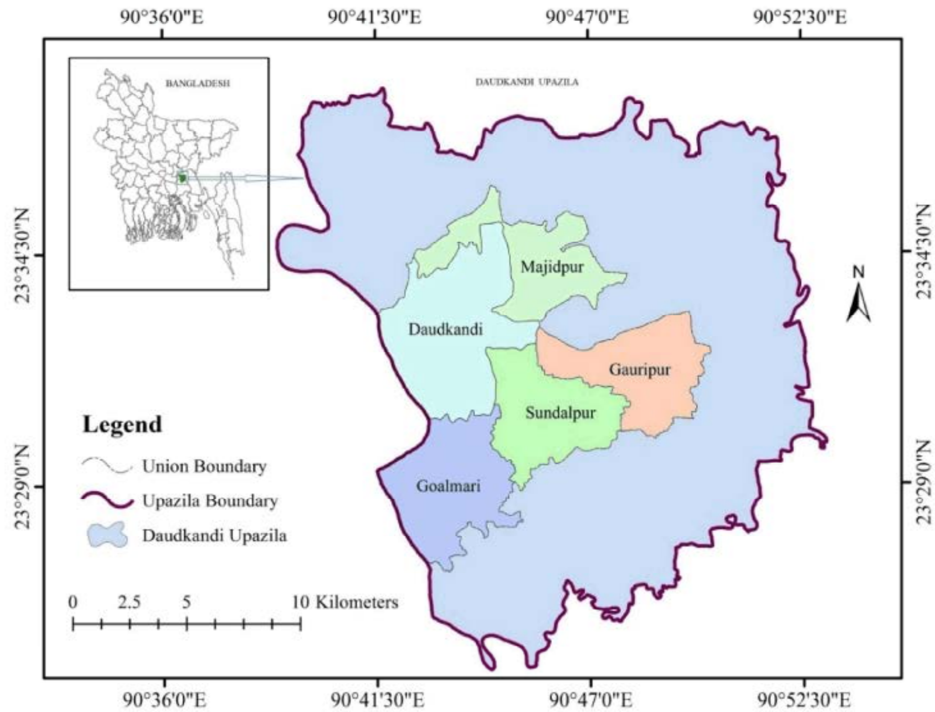

Fig. 1: Sample sites of Daudkandi upazila

collected with anhydrous sodium sulfate for removing trace of water (if any). The collected extract was then concentrated using a rotary vacuum evaporator. The concentrated extract was then transferred to a vial ringing the round bottom flask for three times with DD n-hexane with a volume of 3, 2 and 2 ml, respectively making the final volume of 7 ml.

Clean-up was done according to the procedure described by Matin *et al.* [4]. The clean-up of the extract was performed over florisil (magnesium silicate, mesh 60-100) where, the top 1.5 cm of the column was packed with anhydrous sodium sulphate. The extract was eluted with 100 ml DD n-hexane in the company of 2% double distilled diethyl ether in a flow rate of 5 ml/min. Again the extract was evaporated at 40°C by vacuum rotary evaporator and transfer into vials. The extracts were then completely evaporated by nitrogen gas using N_2 blower (PU 90003). Final volume was made adding 1 ml of HPLC grade acetonitrile before injection.

For pesticide residues analysis, 20µl of aliquot was injected by micro liter syringe into the High Performance Liquid Chromatography (HPLC) fitted with Ultra Violet (UV) detector whereas, acetonitrile (65%) was the mobile phase. Column C_{18} along with the output device at 254 nm absorbance was used for determination of the level of organochlorine (DDT, DDE and DDD) and organophosphorus (diazinon, malathion and chloropyrifos) pesticides.

Identification of the suspected residues was done in relation to retention time (RT) of pure analytical standards. The quantity was measured with formation of calibration curve from standard samples of different concentrations. The calibration curves for organochlorine and organophosphorus pesticides were prepared at five different concentrations of the standard solutions. The mean percentage recoveries of the pesticides were calculated with the following equation-

$$P_i = (S_i / T_i) \times 100$$

Here; P_i is the percent recovery, S_i is the analytical results from the laboratory control standard and T_i is the known concentration of the spike.

Recovery test has been done three times for each pesticide and the mean value of recovery test for each pesticide has been calculated. The mean percentage recoveries for DDT, DDE, DDD, diazinon, malathion and chloropyrifos (after spiking in the distilled water and florisil clean-up) were 85.59, 82.11, 89.33, 81.50, 84.54 and 90.51%, respectively (Table 1).

Table 1: Outcome of the recovery experiment

	Mean amount (µg) in HPLC*		
Pesticides	Spiked	Measured	Mean recovery (%)
DDT	300	256.76	85.59
DDE	300	246.34	82.11
DDD	300	267.98	89.33
Diazinon	300	244.51	81.50
Malathion	300	253.62	84.54
Chloropyrifos	300	271.54	90.51

*Mean value of triplicates

RESULTS AND DISCUSSION

In Daudkandi upazila, there is a total of 803 acres of permanent cropped area with 31203 acres of temporary cropped area. On the other hand, there are 4132 ponds with 3 river flows in this study area [5]. As a result, these water bodies are easily contaminated by pesticides applied to the crop fields which have been revealed in the present study.

A total of three suspected pesticide residues namely diazinon, malathion and chlorpyrifos were detected in the pond and ditch water samples with detection limit of 10 µg/l which have been shown in table 2 with water sample number and locations (union). The tube-well water samples were free from the suspected residues.

Among the thirty water samples, none of the samples was contaminated with organochlorine (DDT, DDE and DDD) pesticides (Table 2), although different concentrations of organochlorine pesticides were reported in water samples from several regions of Bangladesh by Bagchi *et al.* [20], Chowdhury *et al.* [21], Islam *et al.* [22] and Matin *et al.* [4] which have been shown in Table 3.

The absence of organochlorine pesticides in the present study may be due to its banning for agricultural purpose in Bangladesh. Kafilzadeh *et al.* [30] reported the presence of DDT and DDE in an average of 0.016 and 0.055 µg/l, respectively in Lake Parishan, Iran. The mean total concentrations of organochlorine residues in water samples of Dehzir, Tashk, Gonban and Midstream sites were 0.028, 0.041, 0.082 and 0.018 µg/l, respectively [31]. In surface and groundwater samples along El Rahaway drain of Egypt, □DDTs ranged from 0.003 to 0.239 µg/l during rainy season and from 0.008 to 0.066 µg/l during dry season [32].

Among the organophosphorus pesticides, diazinon was detected only in one water sample (4-P) of Goalmari union with the concentration of 651 µg/l (Table 2). The diazinon contaminated sample was a pond water sample. This concentration is far above the Canadian maximum acceptable concentration (MAC) of 20 µg/l for diazinon [33] and also above the Australian health-based guideline value of 4 µg/l for diazinon [34]. Previously, diazinon was detected at different concentrations in the water samples from various regions of Bangladesh (Table 3). Diazinon was also detected by Hasanuzzaman *et al.* [35]

Table 2: Results of the suspected pesticide residues detected in the water samples

Water Sample No	Location (Union)	DDT (µg/l)	DDE (µg/l)	DDD (µg/l)	Diazinon (µg/l)	Malathion (µg/l)	Chlorpyrifos (µg/l)
1-D	Daudkandi	BDL	BDL	BDL	BDL	82000	BDL
2-P	Daudkandi	BDL	BDL	BDL	BDL	62000	BDL
3-D	Goalmari	BDL	BDL	BDL	BDL	7000	BDL
3-P	Goalmari	BDL	BDL	BDL	BDL	48000	BDL
4-P	Goalmari	BDL	BDL	BDL	651	BDL	31
6-D	Sundalpur	BDL	BDL	BDL	BDL	6000	BDL
6-P	Sundalpur	BDL	BDL	BDL	BDL	54000	BDL
7-D	Majidpur	BDL	BDL	BDL	BDL	3000	BDL
7-P	Majidpur	BDL	BDL	BDL	BDL	7000	BDL
8-P	Majidpur	BDL	BDL	BDL	BDL	8000	BDL
8-D	Majidpur	BDL	BDL	BDL	BDL	1000	BDL
9-P	Gauripur	BDL	BDL	BDL	BDL	1000	BDL

D = Ditch, P = Pond and BDL = Bellow detection limit, Limit of detection = 10 µg/l

Table 3: Some previously reported concentrations of the suspected pesticide residues in water of several regions of Bangladesh

		Concentration/range of pesticide residues (in µg/l)					
References	Source of sample	Diazinon	Malathion	Chlorpyrifos	DDT	DDE	DDD
Ara *et al*. [23]	Pond	BDL	2000-64000	BDL	BDL	BDL	BDL
	Ditch	BDL	1000-22100	BDL	BDL	BDL	BDL
	Tube-well	BDL	4000-56000	BDL	BDL	BDL	BDL
Bagchi *et al*. [20]	Pond	-	-	146-609	316	14	52
Bhattacharjee *et al*. [24]	Paddy field	0.027	-	0.029-034	-	-	-
	Kaliganga River	BDL	-	0.01-0.12	-	-	-
Chowdhury *et al*. [25]	Paddy and vegetable fields	0.9	105.2	-	-	-	-
Chowdhury *et al*. [26]	Paddy field	-	-	0.477-1.189	-	-	-
	Lake	-	-	0.544-0.895	-	-	-
Chowdhury *et al*. [21]	Irrigated field	-	-	-	3.01-8.29	4.06	BDL
Hossain *et al*. [27]	Lakes in Savar	7.86	23.1-59.9	3.27-9.31	-	-	-
Islam *et al*. [22]	Agricultural field	-	-	-	0.115-0.54	-	-
Matin *et al*. [4]	Irrigated crop field, 1992	-	-	-	0.06-19.60	0.10-0.46	-
	Hand tube-well, 1992	-	-	-	Trace	BDL	-
	Surface water, 1993	-	-	-	0.069-0.669	0.094-2.510	-
	Surface water, 1994-95	-	-	-	0.051-0.068	0.013-0.06	0.0-14-0.038
	Ground water, 1994-95	-	-	-	0.027-1.204	0.010-0.084	0.014-0.365
Uddin *et al*. [28]	Pond	-	24.1-46.3	-	-	-	-
Uddin *et al*. [29]	Pond	33.79	-	10-471	-	-	-

BDL = Below detection limit, Trace = Identified but below the detection limit and "-" denotes not examined

at the concentration of 31.5 µg/l in a water sample of Dhamrai, Bangladesh. Brigham [36] also reported diazinon ranging from less than 0.004 to 0.008 µg/l in stream water samples of USA. Székács *et al.* [37] reported the presence of diazinon in the surface water samples of Hungary ranged between 0.01 and 0.9 µg/l from the year 1990 to 2015. On the other hand, the mean concentrations of diazinon in the surface water of Warri River, Niger delta ranged from 0.07 to 0.93 µg/l [38].

A total of eleven water samples was found to be contaminated with malathion having concentrations ranging from 1,000 to 82,000 µg/l (Table 2). The concentrations are far above the Australian health-based guideline value of 70 µg/l [34]. The result is also above the Canadian MAC of 190 µg/l for malathion [33].

The presence of malathion was also reported by Ara *et al.* [23], Chowdhury *et al.* [25], Bhattacharjee *et al.* [24], Hossain *et al.* [27] and Uddin *et al.* [28] in water samples from several regions of Bangladesh (Table 3). Hasanuzzaman *et al.* [35] detected the presence of malathion at the concentration ranging from 42.58 to 922.8 µg/l in water samples of Dhamrai. Braun and Frank [39] reported the concentration of malathion ranging from 0.24 to 1.8 µg/l in Southern Ontario agricultural watersheds.

On the other hand, chlorpyrifos pesticide residue was detected only in pond water sample (4-P) of Goalmari union at the concentration of 31 µg/l (Table 2). The concentration is above the Australian health-based guideline value of 10 µg/l for chlorpyrifos [34]. Previously, chlorpyrifos was detected in Bangladesh by different researchers like, Chowdhury *et al.* [26] in water samples from Rangpur district, Bagchi *et al.* [20] in pond water samples, Bhattacharjee *et al.* [24] in Kaliganga river, Hossain *et al.* [27] in lake water samples of Savar and Uddin *et al.* [29] in Meherpur region (Table 3). Average residue level of chlorpyrifos in Lagoon water sample of Ghana was 1545.1 µg/l [40].

CONCLUSIONS

From the present study it has been revealed that, the farmers of Daudkandi upazila are mostly dependent on malathion to protect the crops from pest attack because, a total of eleven water samples were found to be contaminated with malathion and the concentrations of the residue were high enough as assigned by different international organizations. The actual scenario may be more severe than the present result since organophosphorus pesticides are degraded within few days. This result is very dreadful for the people inhabiting in this area as well as for other aquatic and terrestrial organisms. To keep the environment healthy and free from pesticide contamination, accurate and instant measures should be practiced in agricultural sector. Regular observation is needed to understand the frequency of contamination so that appropriate measures can be taken to protect the environment.

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