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ORIGINAL ARTICLE

Pesticide residues analysis in water samples of Nagarpur and Saturia Upazila, Bangladesh

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Abstract

Pesticides used to protect the crops from pest attack in the agricultural fields pose harmful effect to the non-target organisms such as human and many other aquatic and terrestrial organisms either directly or indirectly through food chain. The present study was conducted to monitor a total of seven pesticide residues under organochlorine, organophosphorus and carbamate pesticides in three different sources of pond water, paddy field water and tube-well water from Nagarpur Upazila and paddy field water in the company of Dhaleshwari and Gazikhali river water from Saturia Upazila, Bangladesh. A total of 40 water samples were analyzed using high-performance liquid chromatography equipped with ultraviolet detector. Among the organophosphorus pesticides, diazinon was detected in eight water samples at a concentration ranging from 4.11 to 257.91 µg/l whereas, malathion was detected only in one water sample at a concentration of 84.64 µg/l and chlorpyrifos pesticide was also detected only in one water sample and the concentration was 37.3 µg/l. Trace amount of carbaryl was identified but it was below the detection limit. None of the tested water samples was found to be contaminated with DDT or its metabolites (DDE and DDD). The water samples contaminated with the suspected pesticides were above the acceptable limit except for the fish pond samples of Sahabatpur and Dubaria union. To control the misuse of pesticides and to reduce the possible health risk, appropriate control systems of pests such as integrated pest management system should be implemented immediately by the authorities of the country.

Keywords Pesticide · Water sample · HPLC · Organochlorine · Organophosphorus · IPM

Introduction

Bangladesh is an agricultural country. About 18.70% of the total GDP of Bangladesh is contributed by agriculture (BBS 2013). About 80% of the people depend on agriculture for their livelihood (Chowdhury et al. 2011). The GDP in agriculture is 3.04% in FY 2014–15 with great contribution of 22.60% in total agricultural income by Fisheries alone (MoF 2015).

Globally, there are an estimation of 70,000 different pest species together with insects and mites, plant pathogens and weeds which causes an estimation of 14, 13 and 13% agricultural crop damage, respectively (Pimentel 2009a). In Bangladesh, about 40% of the crop loss is caused by pests and insects attack which is a considerable loss (Bagchi et al. 2009). Crop loss from pests can be reduced to 35–42% using pesticides (Pimentel 1997) although risks of using pesticides are serious as well (Pimentel 2009b). Because of inadequate knowledge and instruction about the application of pesticide, farmers spray excessive amount of organophosphate and carbamate pesticides in the cultivated lands (Bhattacharjee et al. 2012).

The surface and ground water bodies are easily being contaminated by runoff water or by rain for the indiscriminate use of pesticide. According to Rahman (2000), the widespread use of pesticide may contaminate the environment and freshwater fish. It is obligatory of entering the fractions of applied insecticides into the aquatic ecosystems due to the

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application techniques for crop protection (Van Wijngaarden et al. 2005).

From water bodies, pesticide residues directly or indirectly pass through the food chain and eventually cause harm to human and other aquatic or terrestrial organisms. Several problems can be caused in human body due to pesticide exposure. Organophosphorous pesticides (OP) may affect sperm chromosome segregation and augment the risk for genetic syndromes (Recio et al. 2001). Lee et al. (2004) reported an association between chlorpyrifos use and incidence of lung cancer in Iowa and North Carolina. Pahwa et al. (2011) observed the association of soft-tissue sarcoma with specific pesticides such as aldrin and diazinon in six regions of Canada. Organochlorine (OC) pesticide use is related with an increase in cancer risk (Xu et al. 2010). A positive association of pesticide exposure with Parkinson's disease (PD) was shown by Hancock et al. (2008).

In Bangladesh, endrin was the first imported pesticide of which 3 metric tons (MT) were imported in 1955 for agricultural pest control (Rahman 2004). Primarily, the government of Bangladesh provided a 100% financial assistance for pesticides and were supplied free of cost to the farmers but the pesticide industry was privatized in 1979 when 100% price was imposed (GoB, MoEF 2005).

Presently, there are a total of 141 active ingredients represented by 425 trade names, registered as public health pesticides (PHP) and agricultural pesticides (AP) in Bangladesh (GoB, MoEF 2005). Out of the total pesticides use, over 80 pesticides are used in rice fields alone (Ara et al. 2014). On the other hand, the consumption of pesticides in Bangladesh increased from 11,610.66 to 40,882.94 MT from the year 1998–2012 (BBS 2013).

Pesticides through food chain can be stored in the human body and may cause several health hazards because of prolonged consumption. The present study was conducted to reveal the original scenario of pesticide application by the farmers and to assess the possible health risk through identification and quantification of the pesticide residues in water samples in these regions.

Materials and methods

The present study was conducted for monitoring of pesticide residues in three different sources of pond water, paddy field water and tube-well water from ten unions of Nagarpur Upazila under Tangail district and paddy field water in the company of Dhaleshwari and Gazikhali river water from nine unions of Saturia Upazila under Manikganj district, Bangladesh.

To carry out the present study, a total of 30 water samples from Nagarpur Upazila and ten water samples from Saturia Upazila were collected in plastic bottle having 500 ml for each sample and were labeled as well as instantly carried to the laboratory and kept at − 20 °C for further analysis. For determination of pesticide residues, 250 ml of water sample and 100 ml of double-distilled (DD) *n*-hexane (Merck, Germany) as solvent was taken into a 1000-ml separating funnel and shaken by mixing well for about 10 min and then kept standing for 10 min for settling down. Then, lower aqueous layer and upper hexane layer were collected in separate conical flasks. The aqueous layer was re-extracted twice by adding 50 ml solvent (DD *n*-hexane) for each re-extraction and then the solvent layer (upper) was collected. Combined extract was collected with anhydrous sodium sulfate (Na_2SO_4) (Merck, Germany) for removing water (if any). The collected extract was then concentrated using a rotary vacuum evaporator (R-215, Buchi, Switzerland). 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, and making the volume of 7 ml.

Cleanup of the extract was done over florisil (magnesium silicate, mesh 60–100, active at 1250 °F, Janssen Chimica) packed with anhydrous sodium sulfate at the top of the column. The extract was eluted with 100 ml DD *n*-hexane in the company of 2% diethyl ether (BDH, England) (double-distilled) 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 evaporated by nitrogen gas using N_2 blower (PU 90003, Alfa industry, England) and dried completely. Final volume was made adding 1 ml of HPLC-grade acetonitrile (Merck, Germany) prior to injection.

For pesticide residues analysis, aliquot (20 µl) was injected by microliter syringe into the high-performance liquid chromatography (Waters Company, England) fitted with ultraviolet (UV) detector. The mobile phase was a combination of 65% acetonitrile and 35% distilled water. Column C_{18} (Nova Pack) along with the output device at 254 nm absorbance was used for determination of the level of organochlorine (DDT, DDE and DDD), organophosphorus (malathion, diazinon and chlorpyrifos) and carbamate (carbaryl) pesticides.

Identification of the suspected pesticide residues were done in relation with retention time (RT) of pure analytical standards which were purchased from GmbH (D-86199 Augsburg, Germany). The quantity was measured with formation of calibration curve from standard samples of different concentrations. The calibration curves for organochlorine, organophosphorus and carbamate pesticides were prepared at three different concentrations of 5, 10 and 20 µg/l of the standard solutions. The mean percentage recoveries with standard deviation (± SD) of DDT, DDE, DDD, malathion, diazinon, chlorpyrifos and carbaryl in the spiked positive controls of the water samples with the florisil cleanup system were 90.13 ± 1.36, 80.07 ± 0.61, 86.28 ± 0.53, 89.33 ± 3.29, 82.57 ± 2.21, 83.84 ± 1.43, and 89.48 ± 1.65%, respectively, which is shown in Table 1.

The mean percentage recoveries for the various pesticides were calculated using the following equation:

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

where 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. Statistical analysis was done using SPSS (Version 11.5).

Results and discussion

A total of forty water samples from Nagarpur and Saturia Upazila were collected to identify and quantify the presence of organochlorine, organophosphorus and carbamate pesticides. The results of the tested water samples contaminated with suspected pesticide residues are shown in Table 2.

Among organophosphorus pesticides, diazinon was detected in eight water samples at a concentration ranging from 4.11 to 257.91 µg/l which are above the Canadian maximum acceptable concentration (MAC) of 20 µg/l except for the results of water samples from Sahabatpur and Dubaria union (water sample no. 14 and 26, respectively) for diazinon (Health Canada 2014). The result is also above the European guideline value of 0.1 µg/l for individual pesticides (EU 1998). The detected mean concentration with standard deviation (mean ± SD) of diazinon was 16.58 ± 49.13 µg/l (Table 2). Previously, diazinon was detected by Uddin et al. (2013) in Meherpur region at a concentration ranging from 32.8 to 79 µg/l. Hasanuzzaman et al. (2017) reported the presence of diazinon at the concentration of 31.497 µg/l in a water sample of Dhamrai Upazila, Bangladesh. Chowdhury et al. (2012a) also reported the presence of diazinon at the concentration of 0.9 µg/l in Savar. Diazinon was reported at the concentration of 7.86 µg/l in the lake of Savar (Hossain et al. 2014) and at the concentration of 0.027 µg/l in the paddy field of Manikganj (Bhattacharjee et al. 2012). Brigham (1994) found diazinon from less than 0.004–0.008 µg/l in stream-water samples of USA.

During the study period, malathion was detected only in one water sample at a concentration of 84.64 µg/l with mean (± SD) concentration of 2.12 ± 13.38 µg/l and the result is higher than the Australian health-based guideline value of 70 µg/l for malathion (NHMRC, NRMMC 2011). The concentration is also above the European guideline

Table 1 Results of recovery experiment

Pesticide	Spiked (µg)	Measured value (µg)	Mean measured value (µg)	SD	Recoveries (%)	Mean recoveries (%)	SD
DDT	200	177.15	180.25	± 2.73	88.58	90.13	± 1.36
	200	181.32			90.66		
	200	182.28			91.14		
DDE	200	160.5	160.13	± 1.21	80.25	80.07	± 0.61
	200	161.12			80.56		
	200	158.77			79.39		
DDD	200	171.5	172.56	± 1.06	85.75	86.28	± 0.53
	200	173.61			86.81		
	200	172.57			86.29		
Malathion	200	184.11	178.65	± 6.59	92.06	89.33	± 3.29
	200	180.51			90.26		
	200	171.33			85.67		
Diazinon	200	160.15	165.13	± 4.42	80.08	82.57	± 2.21
	200	168.61			84.31		
	200	166.63			83.32		
Chlorpyrifos	200	168.21	167.67	± 2.86	84.11	83.84	± 1.43
	200	164.58			82.29		
	200	170.22			85.11		
Carbaryl	200	175.28	178.96	± 3.31	87.64	89.48	± 1.65
	200	179.92			89.96		
	200	181.68			90.84		

SD standard deviation

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

Water sample no.	Location	Diazinon (μg/l)	Malathion (μg/l)	Chlorpyrifos (μg/l)	Carbaryl (μg/l)	DDT (μg/l)	DDE (μg/l)	DDD (μg/l)
2	Nagarpur (M), FP	257.91	BDL	BDL	BDL	BDL	BDL	BDL
3	Nagarpur (M), PF	134.95	BDL	BDL	BDL	BDL	BDL	BDL
6	Pakutia (U), PF	74.61	BDL	BDL	BDL	BDL	BDL	BDL
8	Mukna (U), FP	108.65	84.64	BDL	BDL	BDL	BDL	BDL
9	Mukna (U), PF	29.73	BDL	BDL	BDL	BDL	BDL	BDL
11	Bhadra (U), FP	45.34	BDL	BDL	BDL	BDL	BDL	BDL
12	Bhadra (U), PF	BDL	BDL	37.3	BDL	BDL	BDL	BDL
14	Sahabatpur (U), FP	8.03	BDL	BDL	BDL	BDL	BDL	BDL
26	Dubaria (U), FP	4.11	BDL	BDL	BDL	BDL	BDL	BDL
Mean		16.58	2.12	0.93	0	0	0	0
Standard deviation (± SD)		49.13	13.38	5.9	0	0	0	0

M municipality, *U* union, *FP* fish pond, *PF* paddy field, *BDL* bellow detection limit, below detection limits (BDL) which stand 0 for the calculation of mean and standard deviation, total number of water sample is 40 but only location-wise contamination status has been presented here

value of 0.1 μg/l for individual pesticides (EU 1998). The presence of malathion in seven water samples ranging from 42.58 to 922.8 μg/l in Dhamrai region, Bangladesh was reported by Hasanuzzaman et al. (2017). Chowdhury et al. (2012a) reported the similar result (105.2 μg/l) in a water sample from Dhamrai Upazila. Malathion was also detected by Hossain et al. (2014) at a concentration from 23.1 to 59.9 μg/l in lakes adjacent to agricultural fields of Savar, Bangladesh. Diazinon was reported from 0.24 to 1.8 μg/l in Southern Ontario agricultural watersheds (Braun and Frank 1980) and 0.99 μg/l in Ontario river basins (Frank and Logan 1988).

Among 40 water samples, chlorpyrifos pesticide was detected only in one water sample and the concentration was 37.3 μg/l. The mean concentration of chlorpyrifos with standard deviation was 0.93 ± 5.9 μg/l (Table 2). The result is above the Canadian maximum acceptable concentration (MAC) and WHO acceptable daily intake (ADI) value of 10 μg/l (Health Canada 2014; WHO 2008). The result is also above the desirable limit of Indian standard of 30 μg/l (BIS 2009). Chowdhury et al. (2012b) reported the water samples contaminated with chlorpyrifos ranging from 0.477 to 1.189 μg/l in Rangpur district. Chlorpyrifos was also detected in water samples of Savar Upazila at the concentration from 3.27 to 9.31 μg/l which were within the acceptable range.

Out of 40 water samples, trace amount of carbaryl was identified in several samples but were beyond the detection limit. Chowdhury et al. (2012a) reported carbaryl in the water samples of Dhamrai Upazila from 14.1 to 18.1 μg/l. Carbaryl was also detected in the water samples of paddy field ranging from 0.055 to 0.163 μg/l in Rangpur (Chowdhury et al. 2012b) and near of the vegetable fields of Savar Upazila ranging from 4.6 to 6.3 μg/l (Hossain et al. 2014).

Out of seven suspected pesticide residues, only malathion, diazinon and chlorpyrifos residues were detected in 9 water samples out of 40 tested samples. Within the 40 (100%) water samples, diazinon residue were detected in 8 or 20% of water samples, whereas both malathion and chlorpyrifos residues were detected in each for one water sample or 2.5% (Fig. 1). Malathion and diazinon have been detected in the same water sample (sample no. 8) as a result; the percentage of water sample contaminated with malathion (2.5%) is included within that of diazinon (20%). The residues of DDT, DDE, DDD, and carbaryl were not detected (0%) at any of the water samples. On the other hand, a total of 31 or 77.5% samples remained uncontaminated (Fig. 1).

During the study period organochlorine pesticides (DDT, DDE and DDD) were not detected in any of the water samples from Nagarpur Upazila and Saturia Upazila. This may be due to the banning the use of DDT by the government of

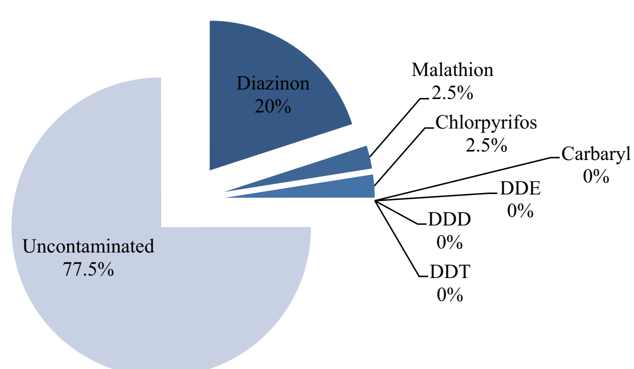

Fig. 1 Contamination status of the water samples Asterisk since, the water sample (sample no. 8) is contaminated with both malathion and diazinon residues; the percentage of water sample contamination by malathion (2.5%) is included within that of diazinon (20%)

Bangladesh. Bangladesh banned agricultural use of DDT in the year of 1997 (DoE 2007). Bangladesh signed to the Stockholm Convention on Persistent Organic Pollutants (POPs) held on 23 May 2001 in Stockholm, Sweden (DoE 2007). Protecting the human health and the environment from persistent organic pollutant is the objective of the convention. Case studies revealed the old stocks of some POPs pesticides at different places and continuous use of DDT for controlling mosquito vectors of malaria and dry fish insect in Bangladesh (ESDO 2005), as a result DDT and its metabolites (DDE and DDD) are detected at various concentrations in different locations of Bangladesh. Chowdhury et al. (2013) reported DDT in five several districts in Bangladesh ranging from 0.133 to 8.29 µg/l. Matin et al. (1998) detected 19.6 µg/l of DDT in Begumganj, Bangladesh. In an average 0.016 µg/l of DDT was reported in Lake Parishan, Iran (Kafilzadeh et al. 2012). Water sample of Lake Parishan was also contaminated with 0.055 µg/l of DDE (Kafilzadeh et al. 2012).

The present study has revealed that out of 40 water samples none of the water sample from Saturia Upazila was contaminated with pesticides although a trace amount of carbaryl was identified but was below the detection limit. It is also found that, organophosphorus pesticides are being used excessively in Nagarpur Upazila. Lacorte et al. (1995) observed a total of ten organophosphates including malathion, diazinon and some other pesticides which disappeared within 2 weeks. Since, organophosphorus pesticides are degraded very quickly and the samples have been collected randomly, the concentration of organophosphorus pesticides (prior to the collection) may be more than the detected value. The presence of these suspected pesticide residues are an alarming result for the people living in this area. With the increasing population along with increasing demand of food, the uses of pesticides have been increased a lot in the third world countries like Bangladesh. The prolonged consumption of these pesticides through food can be stored in the lipid of human body and may cause severe health hazards.

To reduce the increasing trends of pesticide use and, therefore, the increasing health effect, integrated pest management (IPM) can be a good solution to it. It is the process in which the pest population is suppressed below the level that causes economic injury using all suitable techniques and methods. A panel of experts from the Food and Agriculture Organization (FAO) set the concept of IPM in action in 1968 (Rajinder and Ashok 2009). IPM is the main strategy recommended for pest management under Agenda 21 of the United Nations Conference on Environment and Development (UNCED 1992). The concept of IPM consisted mainly of the use of insecticides in a manner that was compatible with biological control of insect pests but the focus of IPM began to shift to non-pesticidal strategy in the 1980s including expanded use of cultural control, introduction of resistant varieties and biological control (Norris et al. 2003). Implementation of IPM strategies saved USA agriculture from $500 million per year due to reductions in pesticide use (Rajotte et al. 1987). Norway started a pesticide reduction plan in 1988 which employed a levied banded tax system based on toxicity at the rate of $3.8 per hectare that resulted in a 54% reduction of pesticide use (PAN, Europe 2004). So, it is a great opportunity for the agro-based countries such as Bangladesh to reduce the possible health hazards as well as costs due to pesticide use applying the IPM strategy.

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