



Asian Journal of Scientific Research

ISSN 1992-1454

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Distribution of Pesticides in Sediments from Municipal Drains in Delhi, India

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ABSTRACT

A study was conducted to assess the levels of organic pollutants i.e. organochlorine pesticides, (OCPs), organophosphate pesticides (OPPs) and herbicides, in sediments from nine major municipal drains in Delhi, India. Sediment samples from Delhi's municipal drains were extracted with acetone/dichloromethane (1:1v/v) and analyzed using GC and HPLC. The average concentration of Σ OCPs, Σ OPPs and Σ herbicides was 27.26 ± 9.7 ng g⁻¹ (dry wt.), 80.89 ± 22.4 ng g⁻¹ (dry wt.) and 16.20 ± 4.45 ng g⁻¹ (dry wt.), respectively. Among OCPs Σ HCH alone accounts 63% followed by Σ DDT (16%), Σ endosulphan (13%) and drins 8%. The ratio of p,p'-DDT/ Σ DDT (0.23), p,p'-DDT/p,p'-DDE (0.26) and o,p'-DDT/p,p'-DDT (<0.01) indicates the aged mixture or past usage of DDT and no dicofol type DDT. Among OPPs Chlorpyrifos was the dominant pollutant with 89% of total OPPs. Pendimethalin accounts 77% of total herbicides followed by Butachlor (14%) and Alachlor (9%). The level of herbicides, OCPs and OPPs in this study were compared with Canadian Sediment Quality Guidelines, Interim Sediment Quality Guidelines (ISQG) and Probable Effect Level (PEL) and found much lower than fresh water sediment guideline values. However, it is recommended that regular intensive assessment for persistent organic pollutants and heavy metals to be conducted, due to environmental concerns.

Key words: Organochlorine pesticides (OCPs), organophosphate pesticides (OPPs), herbicides, sediment, gas chromatograph (GC), high performance liquid chromatography (HPLC)

INTRODUCTION

The pesticides are extensively used for control of agricultural pests and vector borne diseases. Hundreds of pesticides including organochlorine, organophosphate, carbamate and synthetic pyrethroid insecticides, fungicides and herbicides are commonly used for pest control in agriculture. Environmental pollution by pesticides is a matter of great concern because of their prolonged persistence, lipophilic nature and tendency to accumulate in animal and plant tissues. These compounds are typically persistent in the environment and are known to accumulate in soil, sediment, plants, animals and human beings (Iwata *et al.*, 1994; Kannan *et al.*, 1995; Senthilkumar *et al.*, 2001). Pesticides are among the hazardous contaminants and can be persist in soil and sediments for decades even after banned (Shegunova *et al.*, 2007). These compounds

have a wide range of acute and chronic health effects, including cancer, neurological damage, reproductive disorders, immune suppression, birth defects, stress and are also suspected endocrine disruptors (Kodavanti *et al.*, 2008; Wang *et al.*, 2008; Al-Sarar *et al.*, 2009; Sharma *et al.*, 2010).

Furthermore, pesticides have an affinity for particulate matter and main sinks are river and marine sediments through drainage system mixed with municipal waste. Sediment is a matrix of materials which is comprised of detritus, inorganic and organic particles and is relatively heterogeneous in terms of its physical, chemical and biological characteristics (Hakanson, 1992). These are usually regarded as the ultimate sink for persistent pollutants discharged into the environment, because pesticides are highly stable in sediments (Al-Masri *et al.*, 2009). Some of these pesticides have been banned in developed and developing countries including India; however, some third world countries are still using these compounds because of their low cost versatility in industry, agricultural and public health practices (Gupta, 2010; CAPE, 2005; Zhang *et al.*, 2008; Chakarborty *et al.*, 2010).

In India a number of pesticides were used in various purposes for a long time. Currently, 165 pesticides registered for use in India and there is a sequential rise in the production and consumption of pesticides during last three decades. India is the fourth largest pesticide producer in the world after US, Japan and China. During 2003-2004, the domestic production of pesticides was approximately 85 TMT (thousand metric tones) and about 60 TMT used annually (Anonymous, 2005), where 71% accounts for insecticides (Bhattacharyya *et al.*, 2009; Gupta, 2010; Narula and Upadhyay, 2010). However, the consumption of pesticides in India is 0.5 kg ha^{-1} , comparatively low (only 3.75% of global consumption) against 12.0, 7.0, 6.6 and 3.0 kg ha^{-1} in Japan, USA, Korea and Germany, respectively (Gupta, 2004; Chauhan and Lokesh, 2006).

Delhi is the national capital of India covering 1485 sq km area, with population of approximately 19 million (NCRPB, 1999; Rohilla *et al.*, 1999) and consumes about 66 MT of technical grade pesticides for agriculture and public health practices (CIBRC, 2000). Drains in Delhi receive municipal, industrial waste water and agricultural runoff and discharge into Yamuna River and other water bodies. The Stockholm Convention requires national monitoring of Persistent Organic Pollutants (POPs) (Fiedler, 2007). In this study, we conducted a monitoring of multiresidues organic pollutants i.e. organochlorine pesticides, (OCPs), organophosphate pesticides (OPPs) and herbicides in sediments from nine major municipal drains in Delhi, India.

MATERIALS AND METHODS

Sampling: Twenty seven sediment samples were collected in duplicate and analyzed for pesticides contamination. Nine major drains in Delhi (Najafgarh, Shahadra, Barapulla, Sen Nursing Home, Power House, Gunchi, Civil mill, Nizamuddin and Drain No. 2) selected for sampling. Approximately 1 kg of soil sample was collected using Van-Veen sediment grab sampler and after removing pebbles and wood sticks the sample was mixed thoroughly to homogenized, then an aliquot was transferred to clean wide mouth amber glass bottle. All the collected samples were kept at -20°C until chemical analysis.

Chemicals and solvents: Chemicals and solvents were purchased from Merck India. Silica gel 60 (0.063-0.100 mm) was from Sigma-Aldrich. Prior to use, silica gel and anhydrous sodium sulphate was cleaned separately with methanol, dichloromethane and acetone in Soxhlet extractor for 8 h each and stored air tight at 130°C . The preparation of acid silica, basic silica and silver nitrate impregnated silica gel were described elsewhere (EN: 1948;1-3, 1996). Pesticide standard

solutions were obtained from Supelco (Sigma, USA) and Dr. Ehrenstorfer (GmbH, Germany), respectively.

Extraction and clean-up: The 20 g wet sample was homogenized with sodium sulphate and transferred to cellulose extraction thimble. The extraction of pollutants was carried out in Soxhlet apparatus using dichloromethane: acetone (1:1 v/v). The extracts were concentrated to 1-2 mL by a rotary evaporator (Buchi, Switzerland). The multilayered column (combined sulfuric acid silica and florisil and at top sodium sulphate) chromatography was performed to remove sulfur and other aliphatic compounds. The column was pre-rinsed with 50 mL n-hexane before sample was loaded. The elution of analytes was subsequently carried out using 170 mL hexane and concentrated to 2.0 mL. The concentrated extracts were divided into two aliquots, one for OCPs and OPPs analysis by gas chromatograph and second for herbicides analysis after solvent exchange to acetonitrile by HPLC. Moisture content was determined to report data on dry weight basis.

Instrumental analysis: In the present study seven organochlorine pesticides, ten organophosphate pesticides and herbicides analyzed in sediments, using GC and HPLC.

Separation and quantification of OCPs were carried out using gas chromatograph (Perkin Elmer Clarus 500, Singapore) attached with auto sampler and equipped with an electron capture detector (^{63}N , ECD). A 25 m \times 0.20 mm ID, Elite-1 column with coatings of 0.33 μm of 5% diphenylpolysiloxane and 95% dimethylpolysiloxane, was used for all the separations. The column oven temperature program was as follows: The oven temperature was initially maintained at 170°C and programmed to increase at 7°C min⁻¹ to 220°C and again ramped to 250 at 5°C min⁻¹ and held for 7.0 min. The injector and detector temperature were maintained at 250 and 350°C respectively. A purified nitrogen gas was used as carrier at the flow rate of 1.0 mL min⁻¹.

The OPPs compounds were quantified on Shimadzu gas chromatograph (Shimadzu 2010, Japan), equipped with autosampler and flame photometric detector (FPD). Rtx 1701 (Restek, USA) column of 30 m \times 0.25 mm ID with 0.25 μm particle coating (14% cyanpropylphenyl and 86% dimethylpolysiloxane) was used for all the separation of the compounds. The column oven temperature was programmed as: Initial temperature was 160°C for 1 min, temperature was increased from 160 to 220°C at the rate of 3°C min⁻¹ and again ramped to 260°C at the rate of 10°C min⁻¹, keeping the final temperature for 5.0 min. Carrier gas was purified nitrogen at the flow rate of 1.20 mL min⁻¹. Injector and detector temperature was maintained at 250 and 290°C, respectively.

Quantification of herbicides was performed with HPLC system using UV Diode Array Detector (DAD, λ = 210 nm). Isocratic mobile phase of acetonitrile: water (70:30 v/v) at 0.5 mL min⁻¹ flow rate was used. Injections of extracts with 20 μL sample loop were chromatographed on a 150 \times 4.6 mm internal diameter, C18 reversed phase, ZORBAX Eclipse XDB (Agilent) column with 5.0 μm particle.

Analytical quality control: The concentrations of target compounds were determined by external standard method using the peak area of the samples and the five level calibration curves of the standards. The peak identification was conducted by the accurate retention time of each standard. A procedural blank consisting of all chemicals and solvents was run to check for interferences and cross contamination. Appropriate quality assurance quality control (QA/QC) analysis was performed, including analysis of procedural blanks (analyte concentrations were

<MDL 'method detection limit'), random duplicate samples (Standard deviation<5), calibration curves with the r^2 value of 0.999. Each sample was analyzed in duplicate and the average was used in calculations.

Recovery study was undertaken to demonstrate the efficiency of the method. Three separate replicates of sediment sample were spiked with known working standard solutions of OPPs, OCPs and herbicides, then extracted and analyzed in the same way as the real samples. The percent recoveries were in range of 80-111 (± 6 -12), 76-116 (± 8 -14) and 79-119 (± 3 -12) at the fortification levels of 10.0 and 50.0 ng g^{-1} for OCPs, OPPs and herbicide, respectively.

Statistical analysis: The results of the analysis are reported in ng g^{-1} dry weight (dry wt.) basis. A reporting limit of $>0.01 \text{ ng g}^{-1}$ dry wt was taken for calculation. Levels below reporting limit or below MDL ($<0.01 \text{ ng g}^{-1}$ dry wt.) were taken as zero (0) in the calculations. Arithmetic mean was calculated and reported with standard error (\pm). The standard error was calculated from standard deviation divided by square root of number of samples. The ratio of HCH and DDT isomers were calculated and presented in Table 2.

RESULTS AND DISCUSSION

The concentration of Organochlorine (OCPs), Organophosphate Pesticides (OPPs) and herbicides were presented in Table 1, 3 and 4, respectively. The average concentration of OCPs, OPPs and herbicides was 27.26 ± 9.7 , 80.89 ± 22.4 and $16.20 \pm 4.45 \text{ ng g}^{-1}$ (dry wt.), respectively. The observed pattern of insecticides during the study was as: Organophosphate (OPPs) > Organochlorine (OCPs) > herbicides (Fig. 1). Among the OCPs, HCH isomers alone account 63%, where β -HCH was the dominant pollutants with $17.29 \pm 4.5 \text{ ng g}^{-1}$ (dry wt.) mean concentration. DDTs (16%) were the second highest OCPs with $4.40 \pm 1.2 \text{ ng g}^{-1}$ (dry wt.), followed by endosulphan (13%) $3.52 \pm 2.0 \text{ ng g}^{-1}$ (dry wt.) and dieldrin (8%) $2.05 \pm 1.2 \text{ ng g}^{-1}$ (dry wt.) (Table 1). Aldrin was not detected in all the samples. HCH is available in two formulations: technical HCH and lindane. Technical HCH contains isomers in the following percentage: α , 55-80%; β , 5-14%; γ , 8-15%; δ , 2-16%; ϵ , 3-5% (Nhan *et al.*, 2001; Fu *et al.*, 2001) and lindane contains >90% of γ -HCH. Because in the text α -HCH and γ -HCH could not detected in any sample. So HCH ratio was not calculated. Therefore, fresh usage of technical HCH and lindane formulation in the study area could not

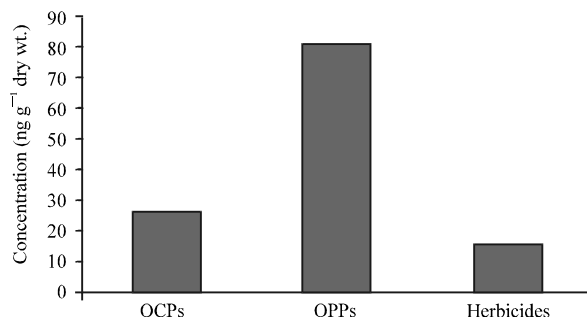


Fig. 1: Distribution of total organochlorine, organophosphate pesticides and herbicides in sediments from municipal drains, Delhi

Table 1: Range and mean±standard error* concentrations (ng g⁻¹ dry wt.) of organochlorine pesticides (OCPs) in sediments from municipal drains, Delhi

Compound	Range		Mean	%**
	Minimum	Maximum		
α-HCH	<0.01		<0.01	<0.01
β-HCH	<0.01	67.28	17.29±4.5	63
γ-HCH	<0.01	<0.01	<0.01	<0.01
ΣHCH	<0.01	67.28	17.29±5.9	63
Aldrin	<0.01		<0.01	<0.01
Dieldrin	<0.01	18.47	2.05±1.2	8
β-endosulphan	<0.01	14.78	1.64±0.9	6
γ-endosulphan	<0.01	16.91	1.88±1.1	7
Σendosulphan	<0.01	31.69	3.52±2.0	13
p,p'-DDE	<0.01	14.16	2.79±1.0	10
o,p'-DDT	<0.01		<0.01	<0.01
p,p'-DDT	<0.01	5.90	1.61±0.5	6
ΣDDTs	<0.01	14.16	4.40±1.2	16
ΣOCPs	<0.01	131.60	27.26±9.7	100

<0.001: Below detection limit, *Standard error = SD / \sqrt{n} **% of ΣOCPs

Table 2: Range and mean±standard error* ratio of HCH and DDT isomers in sediments from municipal drains, Delhi

Compound	Range		Mean
	Minimum	Maximum	
p,p'-DDT/ΣDDT	<0.01	1.00	0.23±0.1
p,p'-DDT/o,p'-DDE	<0.01	1.53	0.26±0.1
o,p'-DDT/p,p'-DDT		<0.01	<0.01

<0.01: Below detection limit, *Standard error = SD / \sqrt{n} Table 3: Range and mean±standard error* concentrations (ng g⁻¹ dry wt.) of organophosphate pesticides (OPPs) in sediments from municipal drains, Delhi

Compound	Range		Mean	%**
	Minimum	Maximum		
Monocrotophos	<0.01	7.74	1.22±0.5	2
Dimethoate	<0.01	27.11	3.01±1.7	4
Chlorpyrifos	<0.01	286.56	72.28±21.5	89
Methyl parathion	<0.01		<0.01	<0.01
Phosphomidon	<0.01		<0.01	<0.01
Malathion	<0.01		<0.01	<0.01
Quinolphos	<0.01	2.76	0.31±0.2	<0.01
Profinophos	<0.01	3.03	0.82±0.2	1
Ethion	<0.01	8.38	2.21±0.6	3
Phorate	<0.01	5.17	1.04±0.3	1
ΣOPPs	<0.01	301.52	80.89±22.4	100

<0.01: Below detection limit, *Standard error = SD / \sqrt{n} , **% of ΣOPPs

Table 4: Range and mean±standard error* concentrations (ng g⁻¹ dry wt.) of herbicides in sediments from municipal drains, Delhi

Compound	Range		Mean	%**
	Minimum	Maximum		
Alachlor	<0.01	4.52	1.46±0.30	9
Butachlor	<0.01	12.96	2.19±0.89	14
Pendimethalin	<0.01	53.19	12.53±3.91	77
ΣHerbicides	<0.01	66.16	16.20±4.45	100

<0.01: Below detection limit, *Standard error = SD / \sqrt{n} **% of Σherbicides

conclude. However, in the past studies the use of technical HCH as well as lindane was anticipated (Zhang *et al.*, 2008; Kumari *et al.*, 2008; Kumar *et al.*, 2009; Chakarborty *et al.*, 2010). The technical mixture of HCH was produced and used in India until it was banned in 1997 and lindane formulations are registered for use in public health practices to control vector borne diseases and for pest control in selected crops (Gupta, 2004; CAPE, 2005).

DDT has been banned in India for use in agricultural sector but, permitted to use up to 10,000 tonnes per year, under the Stockholm Convention, until an alternative can be found to combat vector borne diseases which are particularly prevalent in monsoon season (Zhang *et al.*, 2008; Chakarborty *et al.*, 2010). Approximately 85% of DDT produced in India is used for mosquito control; therefore the levels of DDT observed could be attributed to municipal waste water inflow from residential areas to the adjoining drains in Delhi. The elevated concentration of p,p'-DDE (2.79±1.0 ng g⁻¹ dry wt) and p,p'-DDT (1.61±0.5 ng g⁻¹ dry wt) as compared to o,p'-DDT (<0.01 ng g⁻¹ dry wt) was observed in analyzed drain sediments (Table 1). The vapor pressure of o,p'-DDT is 7.5 times greater than p,p'-DDT (Spencer and Cliath, 1972), leading to greater volatilization of o,p'-DDT to the atmosphere, suggesting the recent use of DDT and degradation of older DDT. p,p'-DDT metabolizes much faster in subtropical soils (Talekar *et al.*, 1977). So the amount of p,p'-DDT volatilized from the soil surface may be relatively small compared to the amount of o,p'-DDT. After DDT applications were discontinued, much of the DDT may be converted to p,p'-DDE (Baxter, 1990). Higher concentration of p,p'-DDE have been interpreted as a result of DDT conversion to p,p'-DDE by UV radiation after prolong exposure in the environment (Atlas and Giam, 1988). The residence time of p,p'-DDT could be estimated using the ratio of p,p'-DDT to ΣDDTs. The ratio for technical DDTs was reported to be 0.77 (WHO, 1989). The mean ratio of p,p'-DDT to ΣDDTs in present study was 0.23 (Table 2) which indicates that these areas have not been sprayed with DDTs more recently. The ratio of p,p'-DDT and p,p'-DDE can be used to estimate the existence of technical DDT in recent inputs. A ratio of 0.33 or less is considered as aged mixture, while a relatively high p,p'-DDT/p,p'-DDE ratio implies a recent input. In present study the ratio of p,p'-DDT/p,p'-DDE was 0.26 indicates the aged mixture DDT usage and not fresh technical DDT input in the drains of Delhi. Similar observations were reported by Zhang *et al.* (2008) and Chakarborty *et al.* (2010) for DDTs in Delhi region. The ratio of o,p'-DDT/p,p'-DDT can be used to distinguish technical DDT from "Dicofol-type DDT". The o,p'-DDT/p,p'-DDT ratio was reported to be 0.2~0.26 in technical DDT and ~7.5 in dicofol products (Qui *et al.*, 2005). In present study, the ratio of o,p'-DDT/p,p'-DDT is less than unity (<0.01) which is different from the scenario in China where dicofol usage is a major source of DDT (Liu *et al.*, 2006). Thus, the DDT contaminations in this region may be from technical DDT and obviously not from dicofol usage.

During 1995-2000, the average annual consumption of endosulphan in India was 3600t (Usha and Harikishna, 2005) and accounts for 10% of the total insecticide consumption. Endosulphan has the ability to affect the various endocrine system, spermatogonia in goat (Sharma *et al.*, 2010) and can affect the soil microbial respiration which reduces bioremediation of some pesticides from environment for pollution control (Nare *et al.*, 2010; Adhikari, 2010). Endosulphan consists in two isomers, α - and β -, in the ratio of 2:1-7:3. In the present study, the sum of endosulphan ranged from <0.01 to 31.69 ng g^{-1} (dry wt.), with the mean of $3.52 \pm 2.0 \text{ ng g}^{-1}$ (dry wt.). The α - and β -isomers of endosulphan were detected in selected soil samples. Average values of α -endosulphan and β -endosulphan isomers were observed as $1.64 \pm 0.9 \text{ ng g}^{-1}$ dry wt. and $1.88 \pm 1.1 \text{ ng g}^{-1}$ dry wt. (Table 1). Since β -endosulphan is less soluble in water and more strongly bound to the soil particles than α -isomer (Beyers *et al.*, 1965), α -isomer is carried by run off to the aquatic environment. Thus the presence of β -endosulphan seems to be a useful indicator of time since exposure to endosulphan (Nowak *et al.*, 1995). The overall average of α/β endosulphan in the present study is 1, suggests ongoing use of technical endosulphan which is in the agreement with the observations by Chakarborty *et al.* (2010). Concentrations of OCPs observed in this study were compared with Canadian Sediment Quality Guidelines (ISQG) and Probable Effect Level (PEL) (CCME, 2006) and found lower than guideline values.

Ten commonly used organophosphate pesticides (monocrotophos, dimethoate, chlorpyrifos, methyl-parathion, phosphomidon, malathion, quinolphos, proflinophos, ethion and phorate), were analyzed in sediments from Delhi drains. The Σ OPPs were ranged between <0.01 to 301.52 ng g^{-1} (dry wt.) with the mean concentration of $80.89 \pm 22.4 \text{ ng g}^{-1}$ (dry wt.). Among studied OPPs the Chlorpyrifos is the dominant pollutant ($72.28 \pm 21.5 \text{ ng g}^{-1}$ dry wt.), in Delhi drains which account 89% of the total OPPs (Table 3). Chlorpyrifos, a phosphorothioate broad spectrum moderately toxic organophosphates (OP) insecticide remains one of the most widely used in agriculture and public health. Chlorpyrifos inhibits acetylcholinesterase (AChE) and alter hematological parameters (Ambali *et al.*, 2010). A much higher concentration of chlorpyrifos (172 ng g^{-1}) reported by Kumari *et al.* (2008). After Chlorpyrifos, ethion and dimethoate were high in concentrations with the mean of $2.21 \pm 0.6 \text{ ng g}^{-1}$ (dry wt.) and $3.01 \pm 1.7 \text{ ng g}^{-1}$ (dry wt.), respectively. Monocrotophos ($1.22 \pm 0.5 \text{ ng g}^{-1}$ dry wt.), quinolphos ($0.31 \pm 0.2 \text{ ng g}^{-1}$ dry wt.) and phorate ($1.04 \pm 0.3 \text{ ng g}^{-1}$ dry wt.) contributed 4% to the total OPPs. Other OPPs like methyl parathion, phosphomidon and malathion were not detected in all the samples. Similar to our results, residues of monocrotophos, dimethoate, methyl parathion and quinolphos have been reported by other worker (Kumari *et al.*, 2008; Kumar *et al.*, 2008).

India consumed about 600 tons of herbicides for weed control, mainly in irrigated crops and on plantations. The sediments from Delhi drains were analyzed for three herbicides (alachlor, butachlor and pendimethalin). The observed concentration of herbicides in were range between $<0.01 - 66.16 \text{ ng g}^{-1}$ (dry wt). The individual herbicide concentration range was $<0.01 - 4.52 \text{ ng g}^{-1}$ (dry wt.), $<0.01 - 12.96 \text{ ng g}^{-1}$ (dry wt.) and $<0.01 - 53.19 \text{ ng g}^{-1}$ (dry wt.) for alachlor (mean, $1.46 \pm 0.30 \text{ ng g}^{-1}$ dry wt.), butachlor (mean, $2.19 \pm 0.89 \text{ ng g}^{-1}$ dry wt.) and pendimethalin (mean, $12.53 \pm 3.91 \text{ ng g}^{-1}$ dry wt.) (Table 4), respectively. The pattern was Pendimethalin>butachlor>alachlor. After OPPs, pendimethalin was the predominant contaminant accounts 77% of the total herbicides studied. This herbicide is reported to be highly persistent in soil and sediments. Pendimethalin have been applied at 1000 and 2000 kg ha^{-1} as herbicide, either alone or in combination with one being in north India. Earlier reports have shown the elevated levels of pendimethalin in sediments and vegetables from north India (Arora and Gopal, 2004;

Kumar *et al.*, 2009; Kulshrestha *et al.*, 2002). The percent contribution of butachlor and alachlor was 14 and 9 percent to the total herbicides pollution in the Delhi drain sediments.

CONCLUSION

The study indicates the contamination with groups of pesticides which is a matter of concern. DDTs concentrations were higher than ISQG however, lower than PEL but dieldrin and γ -HCH concentrations were lower than ISQG and PEL values. No maxima were specified for herbicides and OPPs in sediments. Regular monitoring of pesticide residues in sediment is required to check the health and contamination level of water system.

ACKNOWLEDGMENT

The authors express their sincere gratitude to the Member Secretary and Chairman of Central Pollution Control Board, Ministry of Environment and Forest Government of India for encouragement and guidance to conduct the study.

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