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Organotin compounds in surface and pore waters of Ganga Plain in the Kanpur-Unnao industrial region, India

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Abstract

Organotin compounds (OTCs) belong to those chemicals most toxic to the aquatic organisms which are deliberately introduced into the aquatic system through anthropogenic activities. Various species of organotin compounds were detected in surface and pore waters of the Ganga Plain in Kanpur-Unnao industrial region in pre- and post-Monsoon periods of 1995. The extraction of these compounds was performed using a method of direct aqueous phase in situ ethylation with sodium tetraethylborate ($NaBEt_4$). After extraction into hexane, they were detected by GC-MIP-AED. The water of this area is contaminated with dimethyltin (DMT), monobutyltin (MBT), dibutyltin (DBT) and tributyltin (TBT) compounds. Concentrations of these compounds in surface water of the pre-Monsoon period of 1995 range from 2.1 to 70.1 ng Sn/l for MBT, 1.7–101.1 for DBT and 2.9–19.8 for TBT, whereas in pore water; 9.7–23.5 ng Sn/l for MBT, 11.2–18.0 for DBT and 8.7–32.6 for TBT. However, in the post-Monsoon period of 1995, surface water shows considerable decrease in concentrations: DMT below detection–1.8 ng Sn/l, DBT 3.0–5.4, TBT 3.1–3.6 and MBT is below detection. This study is a preliminary documentation of water pollution by OTCs in the Kanpur-Unnao region of the Ganga Plain and suggests the necessity of further detailed OTCs studies in other regions of the Ganga Plain.

Keywords: Ganga Plain; Organotin compounds (OTCs); Monomethyltin (MMT); Dimethyltin (DMT); Monobutyltin (MBT); Dibutyltin (DBT); Tributyltin (TBT); Tetrabutyltin (TeBT); Surface water; Pore water

1. Introduction

Wide industrial and agricultural use of organotin compounds (OTCs) have been a continuous cause of environmental pollution by OTCs. They are the most acute toxic chemicals to aquatic organisms (Thompson et al., 1985; Maguire, 1987; Alzieu et al., 1989; Kuballa et al., 1996). They are more toxic even at very low levels than the inorganic species of Sn because of high bioavailability of the former. The toxicity of organotin compounds depends on the number and nature of

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organic groups, e.g. trialkyltin compounds (R_3SnX) are much more toxic to mammals and aquatic organisms than the monoalkyltin compounds ($RSnX_3$) (Kuballa et al., 1996). Tributyltin (TBT), an active ingredient in antifouling paint formulations, is the most acute toxic chemical to the marine organisms especially molluscs (Maguire, 1987). TBT is reported to induce shell malformation in oysters at a concentration of only 2 ng Sn/l in the water which additionally caused drastic decrease of the oyster population at the French Atlantic coast in the early 1980s (Alzieu et al., 1989). TBT has been put on the black and grey lists of several international treaties on water pollution by dangerous substances (Vrijhof, 1985). The European Commission has listed eight organotin compounds as priority pollutants (Soniassy et al., 1994). In relation to other toxins, e.g. toxic heavy metals, OTCs are rated highly toxic to biota because of their simultaneous hydrophobic and lipophilic characters which enhance their bioavailability.

For the last few decades, the Ganga Plain (Fig. 1) has been overexploited and improperly used for the disposal of domestic and industrial wastes which adversely affected the quality of water, sediments and soils. It is documented that 40% of the pollution in the Ganga River is due to sewage discharge and 13% owing to chemical wastes released from the factories (Anonymous, 1985). The Yamuna River (a tributary of the Ganga River) is the most sewage polluted river of India. The level of pollution has already reached such alarming proportions that the High Court ordered the closure of many industries in several metropolitan cities of this plain. Industrial and domestic wastes are discharged into the Ganga River and its various tributaries and thus pollute the water and sediments of the rivers (Ajmal et al., 1985; Subramanian et al., 1987a,b; Kumar, 1992; Chander et al., 1994; Ansari et al., 1996; Singh, 1996; Singh et al., 1997).

At present, knowledge about the occurrence of organotin compounds in river water of the Ganga Plain is very limited. This study is aimed at investigating the occurrence and distribution of various species of OTCs in river water and pore water in sediments of the Ganga Plain in the Kanpur-Unnao industrial region.

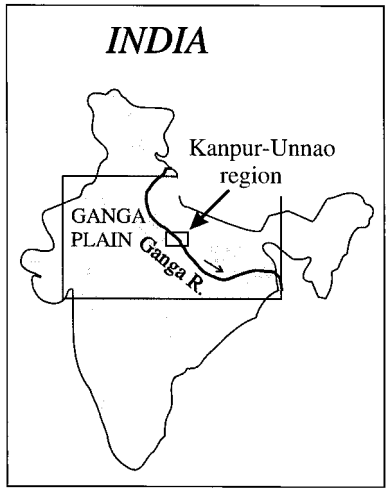

Fig. 1. Map showing location of study area of the Kanpur-Unnao industrial region.

2. Site description

The Kanpur-Unnao industrial region (4.6 million population) is a part of the Central Alluvial Ganga Plain (Fig. 1). Kanpur, the eighth largest metropolis in India is the largest industrial town of the state Uttar Pradesh. This area is traversed by the Ganga River and its tributaries Pandu and Loni rivers (Fig. 2). The river Pandu enters the city from the west and joins the Ganga River after a distance of 156 km downstream from Kanpur city. The waste water from the industries and sewage water of the Kanpur area are transported by various drains which finally meet the Pandu and Ganga Rivers (Table 1). Its industrial basis includes cotton and wool textile mills, leather tanning and leather making industries, large fertilizer companies and several arms factories. Additionally, light engineering factories for rerolling and casting, and the manufacture of chemicals, paints, plastics (PVC — polyvinylchloride), varnish and other miscellaneous items dominate Kanpur's small and medium scale industrial sectors. In the Kanpur-Unnao industrial region, OTCs are used mainly in plastic industry (especially PVC) for the stabilization against heat and UV light and they are also used in agricul-

Table 1
Amounts of the waste water in various urban drains of the Kanpur city during the summer period of pre-Monsoon, 1993 (after CPCB, Central Pollution Control Board, Zonal Office, Kanpur)

No.	Name of drain	m^3/day	% count
Waste water disposed off into Ganga River			
1	Ranighat drain	851	0.21
2	Azadnagar drain	3404	0.85
3	Muir Mill drain	3108	0.77
4	Police line drain	199	0.05
5	Jail drain	673	0.16
6	Dabka drain	1555	0.38
7	Wajidpur drain	432	0.10
8	Bangalighat drain	6169	1.53
9	Seesmau drain	98 712	24.60
10	Pump house drain	29 804	7.43
11	Jajmau irrigation network	122 947	30.64
Waste water disposed off into Pandu River			
12	Panki thermal power drain	17 021	4.24
13	LML drain	31 147	7.76
14	Vijaynagar drain	54 171	13.50
15	Usmanpur drain	22 801	5.68
16	COD drain	8180	2.04
Total		401 174	100

ture as fungicides and insecticides. Their industrial and agricultural use are the sources of OTCs in the area of study. A location map with sample stations and drainage pattern of the study area is shown in Fig. 2.

3. Hydrography of the study area

The climate of the area is divided into three seasons: the cold season (November–February), the hot season (March–June) and the Monsoon season (July–October). Heavy rainfall occurs during the months of July, August and September. Usually 70–80% of the total annual rainfall occurs during this period (Chowdhury et al., 1982), which causes large-scale transfer of water and sediments. For the greater part of the year, high temperature conditions prevail over the plain and a large amount of the water is lost through evapotranspiration. The role of Monsoon rain on heavy metal concentrations and their dispersion patterns in sediments and soils of the Ganga Plain has been demonstrated (Ansari et al., 1998).

The annual hydrography of the Ganga River in Kanpur is characterized by low discharges during pre-Monsoon (March–May) and post-Monsoon (October–November) seasons, and by extremely high ones during the Monsoon season (Fig. 3).

4. Material and methods

4.1. Collection and storage of samples

Surface water samples were collected from the river channels and stored in fluor-ethylene-propylene (FEP, Nalgene) bottles. Pore water was taken by decanting the wet river sediments. Samples were frozen at $-20°C$ to conserve their original concentrations and to minimise the loss of OTCs during the storage (Schebek et al., 1991). They were covered and stored in darkness to avoid their exposure to light, as OTCs breakdown whenever they are exposed to UV radiation (Thompson et al., 1985). Both surface water and pore water were filtered using a cellulose membrane filter with a 0.45-μm pore size. Extraction of OTCs was also attempted on few unfiltered samples, but it was difficult to obtain the extract from the unfiltered samples. Differences in the values from filtered and unfiltered samples were noticed. Therefore filtered samples were preferred for the extraction and better comparison of the results. Precise Eh values of the water and sediments were not measured, however, high contents of organic carbon and foul smell of the samples at the time of sampling were the indication of the moderate reducing state. Nevertheless, any comment on preservation of species under reducing state can not be made due to lack of Eh values.

4.2. Extraction of organotin compounds

Organotin cations were derivatised by direct aqueous phase in situ ethylation with sodium tetraethylborate ($NaBEt_4$) and extraction into hexane. The extract was used for the detection and measurement of OTCs. Detailed information

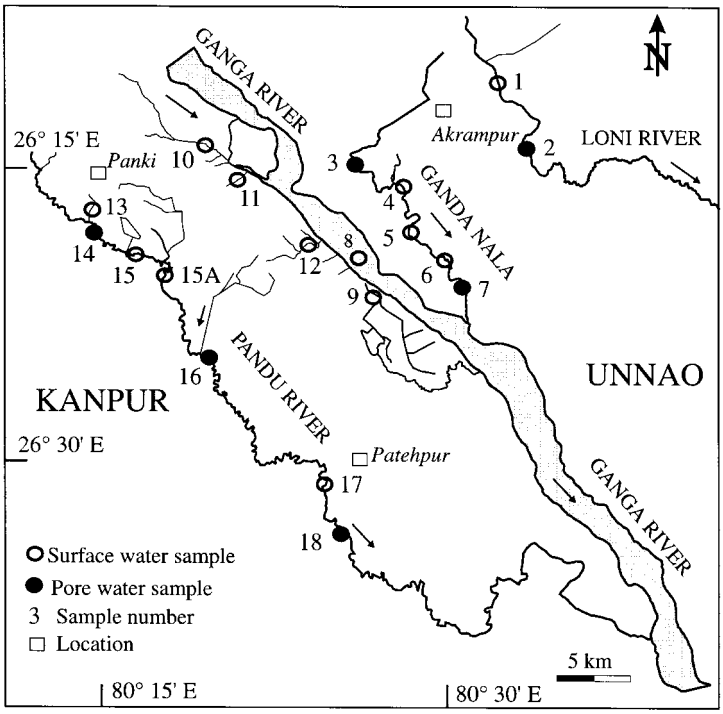

Fig. 2. Drainage map of Kanpur-Unnao industrial region of the Ganga Plain showing sample stations.

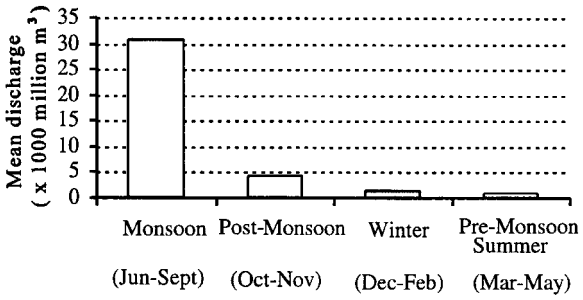

Fig. 3. Seasonal variability of water discharges in Ganga River in Kanpur (after Dasgupta, 1984).

about the extraction method applied for the analysis of the OTCs in the present study is given by several workers (Jantzen and Wilken, 1991; Cai and Bayona, 1995; Prange and Jantzen, 1995). The content of organic carbon in sediment samples was also determined by the titration method of Gaudette et al. (1974). Of the sediments < 20 μm fraction (clay, fine and medium silt) was considered for the analysis of organic carbon because of its high adsorbing and retaining capacity of the pollutants (Gibbs, 1977).

4.3. Technique

Detection and speciation of the OTCs in surface and pore waters were performed by capillary gas chromatography (HP 5890 Series II plus equipped with a HP 7673 autosampler) coupled to a microwave-induced plasma (MIP) atomic emission detector (AED) (HP 6921 A).

Detection intensities of organotin species depend on various instrumental parameters of the GC-MIP-AED (Scott et al., 1991; Lobinski et al., 1992; Frimmel et al., 1993). In order to get the improved detection conditions of the GC-MIP-AED, optimized instrumental parameters (Leipprand et al., 1996) were used, i.e. injection: 1 μl splitless; temp.: 290°C; oven: 50°C for 3 min, rate 28°C/min to 250°C; transfer line and cavity temp.: 250°C; helium makeup flow: 210 ml/min; reagent

gas H_2: 4 bar, O_2: 1.1 bar; wavelength: Sn 326.234 nm.

4.4. Detection limit

Detection limits of GC-MIP-AED for each OTC species were calculated with the following formula. For the calculation of the detection limit, standard samples were used (Leipprand, 1996).

σ — Standard deviation of background emission line;
f — sensitivity of the detector; and
M_g — mean of background emission line.

Detection limits for each species of OTC are given in Table 2.

4.5. Accuracy and precision of the data

Confirmation of peaks of various OTC species was done by comparing them with the peaks of the standards which were parallel analysed (Fig. 4). Recovery standard (TeBT) was added to all the samples. The certified reference samples are commercially available for some of the OTCs, however, they were not available at the time of analysis, therefore the quality of analysis was controlled by the known values of the standard samples purchased from various firms (Merck, Aldrich, Fluka and Alfa).

For the precision, standards with the lowest and the highest concentrations were measured 10 times. Their relative standard deviations (R.S.D. in %) were calculated for each OTC species: 3–10% (from the highest to the lowest concentrations) for DMT, 3–7% for MBT, 3–4% for DBT, 3–6% for TBT and 1–3% for TeBT. The precision of the instrumental technique lies within 10% R.S.D. Quantification of various species of OTCs was calculated with the help of the known concentrations of the standards and spiked internal standard in the sample.

Table 2
Absolute detection limit (ng Sn/l) of GC-MIP-AED for various species of organotin compounds

Peak height (326 nm)	DMT	MMT	MBT	DBT	TBT	TeBT
Detection limit	0.46	0.40	0.31	0.35	0.29	0.30

Abbreviations. DMT, dimethyltin; MMT, monomethyltin; MBT, monobutyltin; DBT, dibutyltin; TBT, tributyltin; TeBT, tetrabutyltin.

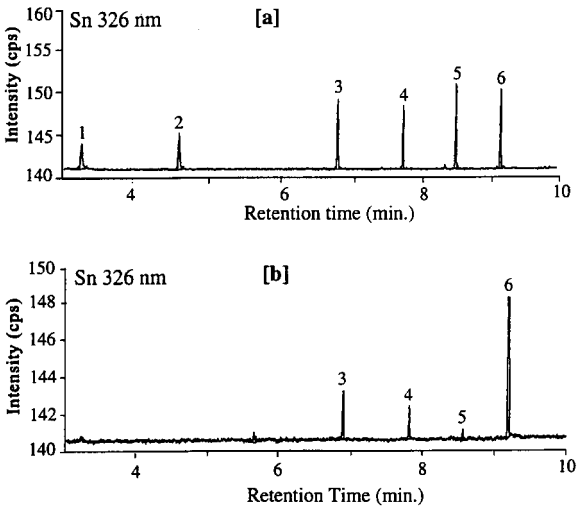

Fig. 4. Chromatograms of (a) a standard (b) a water sample of the Ganda Nala stream of the Kanpur-Unnao region. 1 = dimethyltinethyl (DMTE), 2 = monomethyltintriethyl (MMTE), 3 = mono-*n*-butyltintriethyl (MBTE), 4 = di-*n*-butyltindiethyl (DBTE), 5 = tri-*n*-butyltinethyl (TBTE), 6 = tetrabutyltin (TeBT).

5. Results and discussion

5.1. Sediment characteristics

The fluvial sediments of the study area are mostly fine sand to mud. In few samples, contents of different size fractions (<20, 20–63, 63–125, 125–200 and >200 μm) were determined by grain size analysis (Table 3). Sediments contain high amounts of 20–63 and 63–125 μm fractions which are 26.7 and 24.0 wt.% (median values), respectively. Fractions <20, 125–200 and >200 μm constitute 14.7, 8.3 and 5.6 wt.% (median values), respectively. The proportion of various grain size fractions in sediments varies due to deposition under conditions of changing energies in different geomorphic settings, e.g. river channel, channel bar, natural levee, floodplain.

Table 3
Contents (wt.%) of various grain size fractions in selected sediment samples of the study area in the pre-Monsoon period of 1995

Sample no.	Total (wt.%)	<20 μm (wt.%)	20–63 μm (wt.%)	63–125 μm (wt.%)	125–200 μm (wt.%)	>200 μm (wt.%)
1	100	35.5	39.5	11.5	6.2	6.8
3	100	31.8	36.5	16.7	8.9	2.1
6	100	15.2	20.3	31.4	27.0	4.4
8	100	4.3	4.6	4.0	4.9	82.6
9	100	3.7	9.6	15.6	15.2	51.9
16	100	4.9	23.2	36.2	21.2	13.0
17	100	14.5	39.9	38.2	5.4	2.4
18	100	15.0	30.2	43.4	7.7	2.2
	Median ($n=9$)	14.7	26.7	24.0	8.3	5.6
	Mean	15.6	25.5	24.6	12.1	20.7

Mineralogically, coarse fractions of the sediments are composed of mica, quartz and feldspar, while clay fraction is made up of illite, mica, kaolinite, chlorite and calcite.

Content of organic carbon in sediments (<20 μm fraction) and the weight amount of <20 μm fractions are given in Table 4. The contents of organic carbon in sediments vary from 0.2 to 8.8 wt.% and the values of <20 μm fraction to the total weight of the bulk sediments range from 3.1 to 36.9 wt.%.

5.2. OTCs in surface and pore waters

Various species of methyl- and butyltin compounds were detected in surface and pore waters of the study area during pre- and post-Monsoon periods of 1995. The waters are contaminated with dimethyltin (DMT), monobutyltin (MBT), dibutyltin (DBT) and tributyltin (TBT). Concentrations of detected organotin compounds are given in Table 4.

5.2.1. Concentration of OTCs in surface water

During the pre-Monsoon period of 1995, the concentrations and regional dispersion pattern of various organotin species in surface water are highly variable showing high concentrations at the point sources of OTCs containing industrial wastes (Fig. 5). Pandu River, Ganga River and Ganda Nala are main polluted areas of OTCs. TBT is found in almost all the samples. The maximum concentrations of MBT, DBT and TBT in Pandu River at the point of industrial waste discharge (sample no. 15) are 26.0, 101.1 and 13.1 ng Sn/l, respectively. High OTCs concentrations at this point of the river is due to high input of the industrial waste discharged into the Pandu River through various drains, e.g. Panki Power drain and LML drain (Table 1). There are many plastic producing industries operating around this area. In the downstream part of the Pandu River, the concentrations and species of the OTC decrease substantially (Fig. 5).

In Ganda Nala, the maximum concentrations of MBT, DBT and TBT at the point of industrial waste discharge (sample no. 4) are 70.1, 26.0 and 19.8 ng Sn/l, respectively. High concentrations of OTCs in Ganda Nala at this point is due to the presence of plastic producing industries. Moreover, intense agricultural activities are carried out in this area where several kinds of insecticides and fungicides are used on a large scale. In the downstream part of the Ganda Nala, concentrations and species of the OTC decrease (Fig. 5).

The Ganga River (sample no. 8) shows concentrations of 3.9 for MBT, 12.1 for DBT and 3.4 ng Sn/l for TBT. MMT (monomethyltin) and DMT are below detection limit (Fig. 5). Industrial waste water and sewage water are discharged into the Ganga River through various urban drains (Table 1). Concentrations of OTCs in Ganga River water is relatively low as compared to those in the Pandu River and Ganda Nala. This is probably due to the dilution of OTCs in Ganga River water, as the Ganga River system is compara-

Table 4
Concentrations (ng Sn/l) of various species of organotin compounds in surface and pore waters of the Ganga Plain in the Kanpur-Unnao industrial region in the pre- and post-Monsoon periods of 1995

Sample no.	Grain size (< 20 μm) in wt.%	C-org in wt.%	DMT	MBT	DBT	TBT
Surface water, pre-Monsoon period, 1995						
1	27.7		–	–	–	–
4	3.8	1.3	–	70.1	26.0	19.8
5	17.3	1.5	–	38.6	27.2	8.9
6	15.2	2.2	–	3.6	3.9	3.6
8	5.5	4.6	–	3.9	12.1	3.4
9	3.1	7.3	–	–	1.7	2.9
10	9.9	1.6	–	–	–	3.6
11	5.3	3.0	–	9.5	22.3	19.1
12	21.9	3.6	–	2.9	–	3.5
13	10.6	0.5	–	2.1	7.0	3.1
15	11.3	3.2	–	26.0	101.1	13.1
17	14.5	1.2	–	–	–	3.3
Pore water, pre-Monsoon period, 1995						
2	29.5	1.4	–	–	–	13.1
3	36.9	8.8	–	–	–	10.9
6	15.2	2.2	–	9.7	11.2	8.7
7	29.0	1.0	–	–	–	9.4
14	14.1	0.2	–	–	–	32.6
16	9.5	2.5	–	–	15.7	24.7
18	15.0	1.4	–	23.5	18.0	29.4
Surface water, post-Monsoon period, 1995						
3	36.9	na	1.8	–	3.0	3.1
4	3.8	3.4	–	–	–	–
8	5.5	0.9	–	–	5.4	3.2
9	3.1	na	–	–	–	3.2
15A	15.3	1.7	–	–	–	3.6

Abbreviations. DMT, dimethyltin; MBT, monobutyltin; DBT, dibutyltin; TBT, tributyltin. (–) indicates concentrations below detection limit, na, data not available.
Notes. At sample location 6, both surface and pore water samples were taken. At sample location 3, pore water in the pre-Monsoon, surface water in the post-Monsoon period were taken.

tively a large system carrying large amounts of water and sediments.

No significant correlation with organic carbon in sediments and OTCs in surface water has been noticed. The high concentrations of OTCs at certain points are therefore not linked with organic carbon, but due to point source influx of waste effluents containing high contents of OTCs from the urban industries.

In the post-Monsoon period of 1995, the concentrations decrease considerably (Table 4). The concentrations range from below detection limit to 1.8 for DMT, 3.0–5.4 for DBT, 3.1–3.6 ng Sn/l for TBT. MBT is below detection limit. Probably, large scale water movement during Monsoon time dilutes the concentrations of OTCs.

5.2.2. Concentration of OTCs in pore water

In pore water, the concentrations of OTCs in pre-Monsoon of 1995 are also variable (Table 4). The distribution pattern of OTCs in pore water is shown in Fig. 6. The concentrations of OTCs in pore water range from 9.7 to 23.5 ng Sn/l for MBT, 11.2–18.0 for DBT and 8.7–32.6 for TBT. TBT is found in all the samples, however, MBT and DBT are below detection limit in few samples. MMT and DMT are below detection limit. The concentrations of MBT, DBT and TBT in Pandu River range from below detection (bd) −23.5 ng Sn/l, bd −18.0 ng Sn/l and 24.7–32.6 ng Sn/l, respectively. Whereas in Ganda Nala, the concentrations range from bd −9.7 for MBT, bd −11.2 for DBT and 8.7–10.9 ng Sn/l for TBT.

An attempt was made to see correlation of organic carbon in sediments with the concentrations of OTCs in pore waters. Concentrations of OTCs do not show any significant correlation with the contents of organic carbon. Increase in OTCs concentrations in pore water at some sample points are due to point source input of OTCs containing waste from the industries and agricultural areas.

In downstream parts of the Pandu River and the Ganda Nala, the concentrations of OTCs remain more or less the same with additional species (MBT and DBT). This indicates long distance transportation and preservation of OTCs bound to sediments. However, in the distant downstream part of the Ganda Nala, MBT and DBT again decrease (Fig. 6).

6. Conclusions

The industrial waste effluents of the Kanpur-Unnao industrial region have polluted the surface

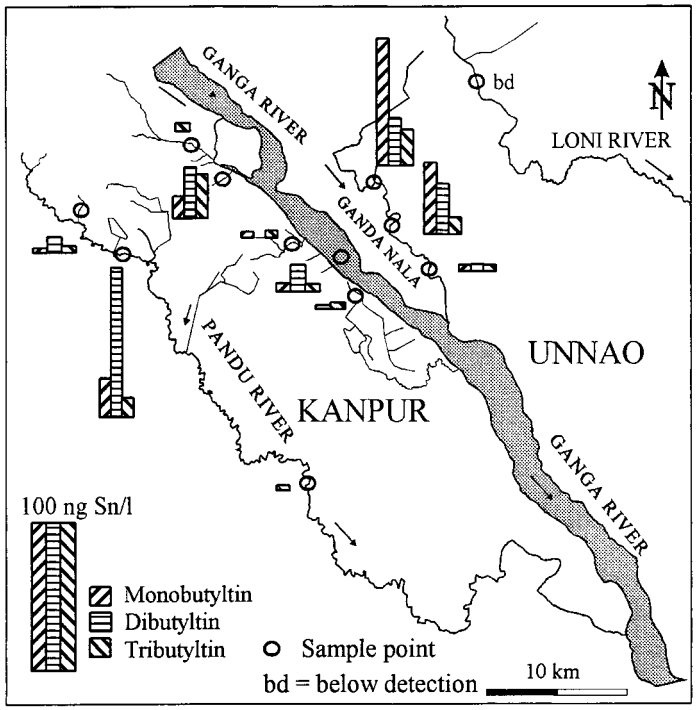

Fig. 5. Concentrations and dispersion pattern of various species of organotin compounds in surface water of the Ganga Plain in the Kanpur-Unnao industrial region during the pre-Monsoon period (April–May) of 1995.

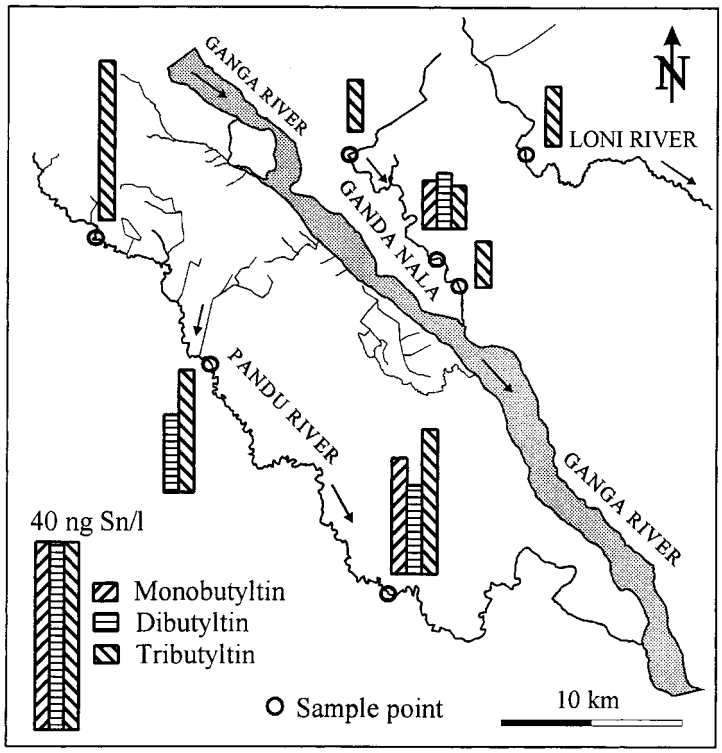

Fig. 6. Concentrations and dispersion pattern of various species of organotin compounds in pore water of the Ganga Plain in the Kanpur-Unnao industrial region during the pre-Monsoon period (April–May) of 1995.

and pore waters of the Ganga Plain with various species of organotin compounds (DMT, MBT, DBT and TBT). These preliminary data highlight the necessity of further detailed OTCs studies in other industrial sectors of the Ganga Plain.

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