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Research Article

Source Identification of Organic Pollutants in Wastewater Irrigated Areas of Lahore and Mangamandi; Pollutants Source Identification

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Abstract

Organic pollutants like DDT, DDE, DCP, TCP, Endrin, and Dieldrin were investigated in the shallow groundwater samples for most densely populated "Lahore" city and its surrounding "Mangamandi" areas of Punjab-Pakistan. In these areas, composite wastewater, mainly consisting of industrial and urban wastewater, is being used for cultivation of crops and vegetables. Solid Phase Extraction (SPE) technique was used to extract the organic pollutants and then analyzed by High Pressure Liquid Chromatography (HPLC) and Gass Chromatogram equipped with Mass Spectrometer (GC-MS). Organic pollutants like DDT, DDE, DCP, TCP, Endrin, and Dieldrin were found in certain samples above the permissible limits. Stable isotopes like $1^{3}C$, ^{15}N and ^{18}O were applied to assess the source of groundwater contamination. Chemical and isotopic data reveal that contamination of groundwater is mainly due to wastewater irrigation and, to some extent, by seepage through unlined wastewater drains in nearby areas. The wastewater containing organic pollutants used for cultivation is filtered through soil to contaminate the shallow groundwater.

Keywords: Groundwater, Organic pollutants, Chemical and isotopic data, Contamination source

Introduction

Groundwater is significant source of water for many municipal water systems [1]. In urban areas, people mainly rely upon municipal water systems [2]. However, withdrawing water from tube wells and hand pumps is also common practice. In these areas, unnecessary pumping by tube wells and recharge by drain water and agricultural activities are causing groundwater contamination [3-8] The industries are releasing untreated effluents in domestic wastewater drains and consequently posing threat to the quality of ground water resources. Owing to poor wastewater drainage systems and wastewater irrigation practices in area has caused contamination of groundwater sources [9,2,10]. In Pakistan, about 80% of the population is dependent on groundwater for household use [11, 12, Afzal et al. 1999, 13]; 1Access to drinking water is reduced either by a shortage in the quantity of water or by the contamination of the groundwater quality of aquifers (WB 2013). Furthermore, porous soil facilitates contamination of shallow aquifers [14; 15], through seepage phenomena [16]. Lah ore and adjacent areas like "Mangamandi" have huge industrial infrastructure, wide variety of industries like pharmaceuticals, herbicide and pesticide manufacturing industries, chemicals etc. These industries are discharging their effluents directly into wastewater drain without any prior treatment. In many areas of this region, like many other developing countries, wastewater irrigation is normal practice where farmers are using wastewater of drains to irrigate fields for different agriculture activities and consequently making areas more prone to contamination by means of seepage of contaminated water through the soil [17]. Furthermore seepage from wastewater drains is also affecting nearby ground water under hydraulic gradient and lead to contamination [18, 19]. The shallow aquifer of the Lahore and Mangamandi area is contaminated in respect of nitrate of high concentrations [20].

Present research work was designed to investigate the impact of wastewater irrigation and wastewater drains on shallow and shallow to deep groundwater of region. The groundwater samples were collected from nearby wastewater drain residential areas and agriculture land being irrigated by wastewater. Groundwater in Lahore and Mangamandi areas. Samples were analyzed for organic pollutants and stable isotopes ($\delta^{13}C$, $\delta^{18}O$ and $\delta^{15}N$) to evaluate the impact of wastewater irrigation and wastewater drains possible recharge to groundwater channels. Using characteristic of $\delta^{18}O$ and $\delta^{15}N$, the nitrate supplied by precipitation can be distinguished from the nitrate produced by microbial activity in soils or added to soil as fertilizer. Nitrification of ammonium and/or organic-N in fertilizer, precipitation, and organic waste can produce a large range of d values. The data of $\delta^{13}C$ was used to evaluate the wastewater mixing.

Materials and Methods

Sampling

Samples were collected from shallow aquifer with depth less than 100m and greater than 120m referred as shallow to deep groundwater [21]. The sampling points were chosen on the basis of previous studies on organic profile of drains [22]. Sampling points were categorized basis on deep water and shallow water aquifers of agricultural, industrial and residential areas. Groundwater samples were collected from hand pumps, boreholes and tube wells in duplicate in pre-cleaned air tight polypropylene bottles in the month of May-June 2022 (pre-monsoon). Before collecting the water samples, the water was pumped out from bore holes in enough quantity to remove lines stagnant water. Co-ordinates of groundwater samples collected from Lahore (LGW) and Mangamandi (MGW) as given in Table 1 & 2.

Table 1: Coordinates of Saming points of Lahore areal.

1	LGW-1	31°23’46.3”N	74°21’47.1”E
2	LGW-2	31°23’46.5”N	74°21’53.5”E
3	LGW-3	31°23’47.9”N	74°22’7.3”E
4	LGW-4	31°23’86.4”N	74°22’19.8”E
5	LGW-5	31°23’44.3”N	74°21’38.7”E
6	LGW-6	31°23’33.7”N	74°21’25.3”E
7	LGW-7	31°23’30.7”N	74°21’20.6”E
8	LGW-8	31°23’20.3”N	74°21’6.0”E
9	LGW-9	31°23’32.9”N	74°21’29.1”E

Table 2: Coordinates of Sampling points of Mangamandi area.

1	MGW-1	31°15’8.1”N	74°10’7.7”E
2	MGW-1	31°15’2.6 ”N	74°10’4.2”E
3	MGW-1	31°14’44.8”N	74°9’46.0”E
4	MGW-1	31°14’41.5”N	74° 09’23.1”E
5	MGW-1	31°14’52.8”N	74°09’5.3”E
6	MGW-1	31°15’36.0”N	74°10’22.0”E
7	MGW-1	31°15’23”N	74°10’11.5”E
8	MGW-1	31°15’24.2”N	74°10’7.4”E

Quality parameters like clarity, odor, and color were recorded in the field. All the samples were colorless and odorless as they were being used for drinking purpose. Physicochemical parameters such as the pH, electrical conductivity (EC) and total dissolved solids (TDS) of samples were measured in situ. Dilute HNO3 was added to each sample until the pH was <2 for major cations, then the sample bottles were stored at about 40C. Measurement of pH was done using a digital pH meter (Adwa, Model AD1030). The EC and TDS of collected samples were measured with a portable conductivity meter (WTW-Model LF 95) calibrated with standard solutions from Hanna instruments (Italy). Water samples were filtered through 0.45 µm membrane filter using filtration assembly equipped with vacuum system. The groundwater samples were pre-concentrated prior to analyze on GC-MS and HPLC. Pre-concentration of samples was carried out by using solid phase extraction method using C18 cartridge (Supelco) and elution was carried out using extra pure solvents like aceton, hexane and ethyl acetate (4 ml each) with flow rate of 1mL/Min [23].

Analysis and Measurement

Organic compounds were analyzed using HPLC-UV (Waters 1525) for qualitative analysis and a GC-MS (HP 5890; Hawlett Packard series II) equipped with DB-5m column (30m x 0.25m x 1µm) and a quardrupole mass spectrometer (JEOL). Ionization was performed under 70eV electron impact conditions (300µA, 400V) where the initial temperature of the column was 35°C, raised at 15°C/min to 150°C and then at 3°C/min to 280°C for quantitative analysis.

The stable isotope analyses were performed by using a modified Varian Mat GD-150 Mass Spectrometer. The ammonium distillation method was used to measure the nitrogen isotopes of nitrate in water sample (Sigman et al., 1997). This method involves the reduction of nitrate to ammonium, which is distilled and concentrated as an ammonium sulphate salt and then combusted to produce nitrogen gas for isotope measurement using isotope ratio mass spectrometer (IRMS). For ^{13}C isotope analysis on mass spectrometer, total dissolve inorganic carbon in water samples was converted into gaseous phase. Sample was poured into the pyrex reaction flask and reacted with H_3PO_4 acid [24]. The reaction flask assembly was connected to the vacuum line. About 5 ml H_3PO_4 was added to the pre-evacuated reaction flask. The CO_2 gas was evolved as a result of reaction between inorganic carbon component/fraction of sample and the phosphoric acid as shown in equation:

$$H_3PO_4 + \text{Carbonate mineral} \rightarrow CO_2 + H_2O$$

The moisture produced during the reaction was removed by cryogenic trap of -80°C. The CO_2 gas was solidify in liquid nitrogen cryogenic trap. Other undesired gases were evacuated to get pure CO_2. The pure CO_2 was collected in ampoule for ^{13}C analysis on Varian Mat GD-150 Mass Spectrometer. Isotope ratio (δ‰) of ^{15}N, ^{13}C and ^{18}O were calculated by using following relation:

$$\delta\%V_s[std] = [\frac{R_{sample}}{R_{std}} - \frac{R_{std}}{R_{std}}](1000\delta\%)$$

The overall analytical errors ±0.01 ‰ ($\delta^{13}C$ & $\delta^{18}O$) and ±0.1‰ ($\delta^{14}N$) were recorded for measurements. To ensure precision, standard deviation of the mass spectrometer has been computed and the standard deviation of each sample has been ensured to be within permissible limit.

Results and Discussion

Physico-chemical parameters

In Lahore samples TDS was found between 939 to 1530mg/L and EC varied between 1439 and 2054 µS/cm. While, in Mangamandi TDS values ranged between 1001 to 3313mg/L whereas, EC values were observed between 1662 to 5052 µS/cm (above the permissible limits) as given in Table 3. Ground water with a TDS above 500 mg/L and EC >2.25 to 4mS/cm is considered not safe for consumption [25].

Table 3: Physiochemical parameters of groundwater samples.

Sample	Depth(ft.)	TDS(mg/l)	EC(µS/cm)	pH	Cl (ppm)
LGW-1	150	959	1593	7.6	102.9
LGW-2	80	995	1651	7.2	142
LGW-3	200	939	1559	7.6	74.55
LGW-4	180	949	1576	8	85.2
LGW-5	80	1530	2054	7.6	237.8
LGW-6	120	1206	2000	7.6	142
LGW-7	90	1063	1764	7.6	138.4
LGW-8	40	868	1439	7.3	67.45
LGW-9	50	1132	1879	7.6	131.3
MGW-1	150	3313	5052	7	770.3
MGW-2	190	1052	1750	7.4	330.1
MGW-3	40	1797	2099	7.3	344.3
MGW-4	140	1084	1784	7.2	315.9
MGW-5	80	1573	2062	8.22	252
MGW-6	160	1001	1662	7.62	205.9
MGW-7	120	1536	2056	7.6	305.3
MGW-8	70	1480	3069	7.7	450.8

Analysis of Organic Compounds in Lahore Groundwater

The concentration of organic pollutants in Lahore area is shown in Table 4 and Fig.1. Sample LGW-1 collected from borehole in industrial areas alongside the waste water drain showed high concentrations of Endrin, Dieldrin and DCP. The LGW-2 sample collected from shallow ground water in industrial area near the waste water drain showed concentrations of DCP, TCP, DDT, Endrin and Dieldrin. Both points LGW-1 and LGW-2 were located in the industrial areas along the drain but high concentration of organic pollutants in shallow groundwater sample (LGW-2) as compare to deep groundwater sample (LGW-1) suggested that shallow groundwater channel is more vulnerable to contamination.

Endrin and Dieldrin were found in LGW-3 and LGW-4 samples which were collected from agricultural area near the wastewater drain (upstream). Whereas, LGW-5, sample collected from agricultural area at wastewater drain (downstream) showed e TCP, DCP, Endein, Dieldrin and DDT, Presence of organic contaminants in deep ground water sample (LGW-3) depicts the contamination of soil has reached to its saturation point and contaminants are seeping down in deep water aquifers.

In LGW-6 only Endrin and Dieldrin were recorded, this sample was collected from industrial area whereas more organic pollutants were found in LGW-7, LGW-8 and LGW-9 as these samples were collected from agricultural area along the wastewater drain. The results suggested that shallow groundwater channel in agricultural areas is being more affected by organic contamination, as compare to industrial area, due to wastewater irrigation.

Overall Endrin and Dieldrin were found in nine, DCP in six, TCP in five, DDT in four and DDE in two samples. Contamination of both shallow groundwater (high level) and deep groundwater (low level) is an alarming situation. Shallow groundwater channels are being suffered from wastewater irrigation infiltration and seepage of unlined wastewater drains. Deep Groundwater channels contamination suggested the overloading of organic pollutants on deep soil through wastewater irrigation where the buffering and degradation potentials of soils exhibited low organic carbon retention which might be due to variable and changing nature of organic matter and clay contents in soils [26].

Table 4: Concentrations of organic pollutants in groundwater samples.

S.No.	Sample	Endrin	Dieldrin	TCP	DCP	DDT	DDE
1	LGW-1	0.012	0.03	0.023	0.036	0	0
2	LGW-2	0.021	0.038	0.031	0.05	0.051	0
3	LGW-3	0.03	0.031	0	0	0	0
4	LGW-4	0.021	0.025	0	0	0	0
5	LGW-5	0.029	0.022	0.024	0.039	0.029	0
6	LGW-6	0.026	0.025	0	0	0	0
7	LGW-7	0.026	0.026	0.028	0.043	0.0061	0.0002
8	LGW-8	0.028	0.024	0.023	0.038	0.01	0.064
9	LGW-9	0.028	0.028	0	0.039	0	0
10	MGW-1	0.027	0.027	0	0.04	0	0.0018
11	MGW-2	0.011	0.013	0.021	0.035	0	0.0001
12	MGW-3	0.025	0.015	0	0.048	0	0.0054
13	MGW-4	0.022	0.012	0	0.038	0	0.0002
14	MGW-5	0.01	0.012	0	0	0	0.0009
15	MGW-6	0.09	0.089	0.023	0.038	0	0.0004
16	MGW-7	0.052	0.042	0	0.036	0	0.0001
17	MGW-8	0.045	0.045	0.02	0.038	0	0

*Permissible Limits set by WHO and U.S EPA respectively

Compound	WHO	U.S EPA
Endrin	0.002	0.0006
Dieldrin	----	0.00003
Phenolic compounds	< 0.002	-----
DDT and metabolites	-----	0.001

Analysis of Organic Compounds in Mangamandi Groundwater

The concentration of organic pollutants in Mangamandi area is shown in Fig. 2. MGW-1 sample was collected from borehole in agriculture area located near the waste water drain whereas MGW-2 sample was collected from borehole located in industrial area on opposite side of drain. It is important to mention that there was agricultural land at one side of the drain and industrial area at the other side. High concentration of organic pollutants in agricultural areas (MGW-1) as compare to non-agricultural area (MGW-1), within same grid, advocate more vulnerability of groundwater channel in agricultural areas towards organic pollutants.

MGW-3 9shallow groundwater sample) was collected from agricultural area near wastewater drain whereas MGW-4 was also collected from same area but it was deep water sample. Comparative high concentration of organic pollutants in MGW-3 as compare to MGW-4 clearly suggest that impact of wastewater irrigation become more worst from shallow to deep groundwater channels.

MGW-5 (shallow groundwater) and MGW-6 (deep groundwater) sample was collected from residential area surrounded by agricultural land. The presence of comparative high concentration of organic pollutants in MGW-5 as compare to MGW-6 indicates shallow groundwater deterioration. MGW-7 and MGW-8 samples were collected from industrial area near agricultural land. Endrin, Dieldrin, DDE and DCP were detected in these samples as well. It might be due to mobility of organic pollutants along groundwater flow patterns.

Overall it was observed that Endrin and Dieldrin were found in eight samples, DCP and DDE in seven, TCP in three samples. It might be due to infiltration of wastewater along with pesticides to contaminate the groundwater [27]. Contamination of even shallow to deep ground water shows that soil has reached to its saturation point and contaminants are seeping down into ground water for its contamination. It is fact that the soil provides a potential pathway of pesticide transport to contaminate water, through runoff and subsurface drainage; interflow and leaching [28-30].

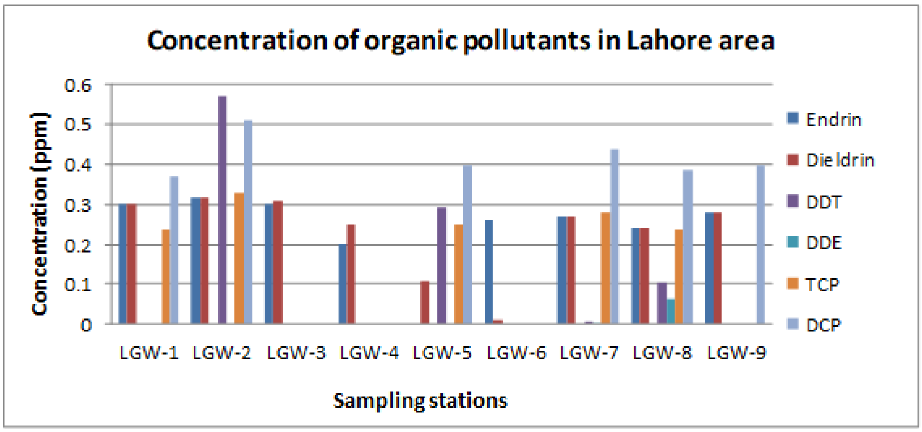

Figure 1: Concentration (mg/l) of organic pollutants in Lahore area.

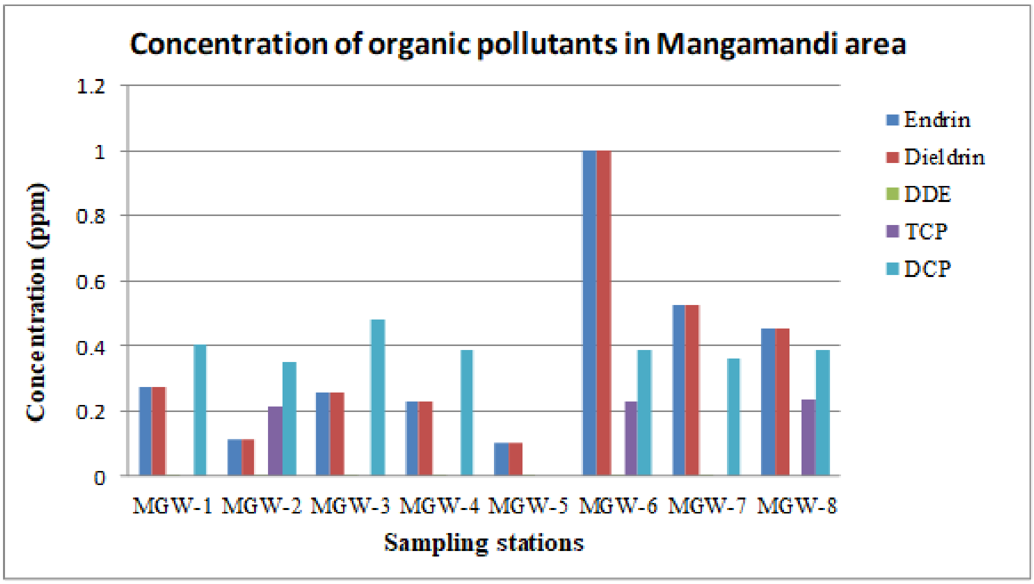

Figure 2: Concentration (mg/l) of organic pollutants in Mangamandi area.

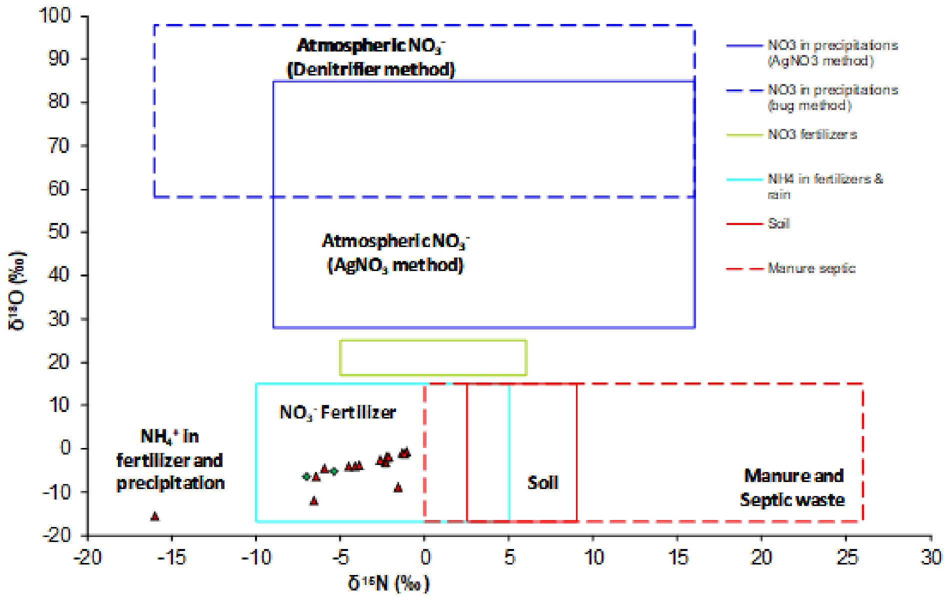

Figure 3: Plot of $\delta^{18}O$ and $\delta^{15}N$ values to assess the origin of pollutants.

$\delta^{13}C$ values in Groundwater Samples

Lahore groundwater samples showed $\delta^{13}C$ values ranged from -0.25 to -6.41 ‰ PDB. In Lahore samples, LGW-1 was collected from deep borehole and its value -6.35‰. The $\delta^{13}C$ depleted values shows that deep water has wastewater mixing. LGW-2 was collected from shallow water and it showed less depleted $\delta^{13}C$ values, it indicates that this point has almost no waste water mixing. LGW-3 was collected from borehole and it showed slightly depleted values which suggests that this water is slightly affected by wastewater. LGW-4 (deep channel) have minor depleted $\delta^{13}C$‰ values, however LGW-5 (deep channel) showed depleted $\delta^{13}C$ ‰ values. It suggested that shallow groundwater channels has more wastewater mixing. LGW-6 (shallow channel) has ignorable depleted values which might be due to sufficient aeration by plant roots respiration or oxidation conditions. LGW-7 and LGW- 8 were collected from shallow water and depleted $\delta^{13}C$ ‰ values suggest wastewater mixing. LGW-9 was collected from hand pump (shallow water) its more depleted $\delta^{13}C$ ‰ value proves more wastewater mixing.

In Mangamandi samples, MGW-1 and MGW-2 were collected from deep water (borehole) it showed depleted $\delta^{13}C$ ‰ values which suggest that these groundwater samples have wastewater mixing. MGW-3 was collected from shallow wastewater channel and has negligible depleted $\delta^{13}C$‰ values due to plant roots respiration or prevailing oxidation conditions. MGW-4 and MGW-5 have higher depleted $\delta^{13}C$‰ which suggest higher wastewater mixing. MGW-6 (deep channel) showed less depleted $\delta^{13}C$‰ values due to less wastewater mixing. MGW-7 and MGW-8 were collected from shallow water and they have depleted $\delta^{13}C$‰ values, which points towards wastewater mixing.

Table 5: Isotopic values (δ ‰) of groundwater samples.

S. No.	Sample	NO_3^- (ppm)	Cl^- (ppm)	$\delta^{13}C$	$\delta^{15}N$	$\delta^{18}O$
1	LGW-1	75	102	-3.05	-2.3	-3.01
2	LGW-2	55	142	-0.25	-1.08	-0.68
3	LGW-3	53	74	-2.12	-2.24	-2.01
4	LGW-4	51	85	-2.02	-2.16	-1.99
5	LGW-5	85	238	-6.41	-6.41	-6.57
6	LGW-6	56	142	-0.84	-1.35	-0.98
7	LGW-7	52	138	-9.75	-1.6	-8.78
8	LGW-8	48	67	-4.06	-4.51	-4.01
9	LGW-9	52	131	-2.65	-2.65	-2.59

10	MGW-1	90	770	-4.02	-4.1	-4.01
11	MGW-2	78	330	-5.03	-5.91	-4.56
12	MGW-3	82	344	-1.35	-1.18	-1.22
13	MGW-4	75	315	-15.77	-16.01	-15.49
14	MGW-5	68	252	-11.23	-6.56	-12.06
15	MGW-6	70	205	-3.82	-3.9	-3.78
16	MGW-7	74	305	-6.78	-6.99	-6.55
17	MGW-8	75	450	-5.38	-5.38	-5.29

Source of contamination in Groundwater

The source of pollution in the groundwater was assessed by using the stable isotope tools. Ratio of $\delta^{18}O$ and $\delta^{15}N$ were applied to determine the source of pollutants as shown in Fig. 3.

The ratios of $\delta^{18}O$ and $\delta^{15}N$ of nitrates in water samples were placed in the assigned plot values of Kendall and source of organic pollutants induced in groundwater of Lahore and Mangamandi were assessed. The sources of pollutants assessed at sample points based on isotopic data are anthropogenic source and that nitrates in groundwater is coming mainly from fertilizers that were being used in fields. Nitrates of these fertilizers seeped through the soil and become part of groundwater along with wastewater that is used for cultivation.

Conclusion

a. Significant concentration of organic compounds including Endrin, Dieldrin, DCP, TCP, DDE, DDT was found in groundwater sample of Lahore and Mangamandi areas.

b. Shallow groundwater channels of Lahore and Mangamndi areas are being contaminated mainly due to wastewater irrigation and, to some extent, through seepage from wastewater drains passing through the area.

c. The ratios of $\delta^{18}O$ and $\delta^{15}N$ of nitrates in water samples suggested that nitrates in groundwater is mainly coming from fertilizers being used in fields.

d. The depleted $\delta^{13}C$‰ value in some areas in vicinity of wastewater drains suggested that wastewater drains, at some extend, contributing to contaminate the shallow groundwater channels.

e. The shallow groundwater channels in the areas of Lahore and Mangamandi, are under high risk due to filtration caused by wastewater irrigation and seepage of unlined wastewater drains flowing through the areas.

f. Wastewater irrigation in Lahore and Mangamandi areas should be banned and wastewater drains should be lined to conserve the quality of groundwater.

Conflicts of Interest

None.

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