Impact of wastewater irrigation on groundwater in the Lahore region and contamination source identification
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ABSTRACT
Organic pollutants in groundwater samples were analyzed for the very densely populated ‘Lahore’ city and its surrounding ‘Mangamandi’ areas of Punjab-Pakistan, where composite (industrial and urban) wastewater is used for cultivation of crops and vegetables. The samples were pre-concentrated using the Solid Phase Extraction (SPE) technique and analyzed by using High Pressure Liquid Chromatography (HPLC) and a Gas Chromatograph Mass Spectrometer (GC-MS). Organic contaminants like Dichlorodiphenyltrichloroethane (DDT), Dichlorodiphenyldichloroethylene (DDE), Dichlorophenol (DCP), Trichlorophenol (TCP), endrin, and dieldrin were found in certain samples above the permissible limits. Stable isotopes like $^{13}$C, $^{15}$N and $^{18}$O were analyzed to assess the source of shallow groundwater channel contamination. Chemical and isotopic data reveal that contamination of shallow groundwater channels in the area 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, filters through the soil to contaminate the shallow groundwater channels.

Key words | chemical and isotopic data, contamination source, groundwater, organic pollutants

HIGHLIGHTS
- Describes contamination of groundwater channels by wastewater irrigation and seepage of unlined wastewater drains.
- Identifies sources of contamination.
- Evaluates wastewater mixing.

INTRODUCTION
Groundwater is a significant source of water for many municipal water systems (Bralower & Bice 2014). In urban areas, people mainly rely upon municipal water systems (Rail 2000). 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 (NESPAK 1991; Zuberi & Sufi 1992; Ahmad 1993; Ahmad et al. 2002a, 2002b; Yeping 2013). Industries are releasing untreated effluents in domestic wastewater drains and consequently posing a threat to the quality of ground water resources. Poor wastewater drainage systems and wastewater irrigation practices in the area have caused contamination of groundwater sources (Chilton & Chapman 1996; Rail 2000; Oren et al. 2004). In Pakistan, about 80% of the population is dependent on groundwater for household use (Bhutta 1999; PCRWR 2002; WWF 2007). Access to drinking water is reduced either by a shortage in the quantity of water available or by the contamination of the groundwater quality of aquifers. Furthermore, porous soil facilitates contamination of...
shallow aquifers (Rail 2000; Tariq et al. 2004), through seepage phenomena (Kanwal et al. 2013). Lahore and adjacent areas like ‘Mangamandi’ have a huge industrial infrastructure, with a wide variety of industries like pharmaceuticals, herbicide and pesticide manufacturing industries, soft drinks, chemicals, steel products 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, with farmers using wastewater from 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 (Ahmad et al. 2002a). Furthermore seepage from wastewater drains is also affecting nearby groundwater under hydraulic gradient and leading to contamination (Malarkodi et al. 2007; Deepali & Gangwar 2010). The shallow aquifer of the Lahore and Mangamandi area is contaminated with nitrate in high concentrations (Khan & Malik 2000).

This research was designed to investigate the impact of wastewater irrigation and wastewater drains on shallow and shallow to deep groundwater in the region. The groundwater samples were collected from near to wastewater drains in residential areas and agriculture land being irrigated by wastewater. Groundwater samples from Lahore and Mangamandi areas were analyzed for organic pollutants and stable isotopes ($\delta^{13}$C, $\delta^{18}$O and $\delta^{15}$N) to evaluate the possible impact of wastewater irrigation and wastewater drain recharge to groundwater channels. Using the characteristic of $\delta^{18}$O and $\delta^{15}$N, nitrate supplied by precipitation can be distinguished from nitrate produced by microbial activity in soils or added to soil as fertilizer. Nitratification 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 aquifers with depth less than 100 m (shallow groundwater) and greater than 120 m (deep groundwater) (Wang et al. 2013). The sampling points were chosen on the basis of previous studies on the organic profile of drains (Sumera 2013). Sampling points were categorized based 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 2018 (pre-monsoon). Before collecting the water samples, the water was pumped out from bore holes in enough quantity to remove stagnant water. Groundwater samples collected from Lahore were labeled as LGW and from Mangamandi as MGW as shown in Figures 1 and 2.

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 purposes. Physicochemical parameters such as the pH, electrical conductivity (EC) and total dissolved solids (TDS) of samples were measured in situ. Dilute HNO$_3$ was added to each sample until the pH was <2 for major cations, then the sample bottles were stored at about 4 °C. 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 a 0.45 μm membrane filter using a filtration assembly equipped with a vacuum system. The groundwater samples were pre-concentrated prior to analyze on a Gas Chromatograph Mass Spectrometer (GC-MS) and using High Pressure Liquid Chromatography (HPLC). Pre-concentration of samples was carried out by using solid phase extraction method using a C18 cartridge (Supelco) and elution was carried out using extra pure solvents like acetone, hexane and ethyl acetate (4 ml each) with a flow rate of 1 mL/Min (Tanabe et al. 2000).

#### Analysis and measurement

Organic compounds were analyzed using HPLC-UV (Waters 1525) for qualitative analysis and a GC-MS (HP 5890; Hewlett Packard series II) equipped with DB-5 m column (30 m × 0.25 m × 1 μm) and a Quadrupole mass spectrometer (JEOL). Ionization was performed under
Figure 1 | Lahore groundwater sampling points along a wastewater drain.

Figure 2 | Mangamandi groundwater sampling points along a wastewater drain.
**RESULTS AND DISCUSSION**

**Physico-chemical parameters**

In Lahore TDS samples were found between 939 to 1,530 mg/L and EC varied between 1,439 and 2,054 μS/cm. In Mangamandi, TDS values ranged between 1,001 to 3,313 mg/L, whereas EC values were observed between 1,662 to 5,052 μS/cm (above the permissible limits), as shown in Table 1. Groundwater with a TDS above 500 mg/L and EC > 2.25 to 4 mS/cm is considered not safe for consumption (Harter 2003).

**Analysis of organic compounds in Lahore groundwater**

The concentrations of organic pollutants in the Lahore area are shown in Table 2 and Figure 3. Sample LGW-1 collected from a borehole in an industrial area alongside the wastewater drain showed high concentrations of endrin, dieldrin and Dichlorophenol (DCP). The LGW-2 sample collected from shallow groundwater in an industrial area near the wastewater drain showed concentrations of DCP, Table 1 | Physicochemical parameters of groundwater samples

| Sample | Depth (m) | TDS (mg/L) | EC (μS/cm) | pH | Cl (ppm) |
|--------|-----------|------------|------------|----|-----------|
| LGW-1  | 150       | 959        | 1,593      | 7.6| 102.9     |
| LGW-2  | 80        | 995        | 1,651      | 7.2| 142.0     |
| LGW-3  | 200       | 939        | 1,559      | 7.6| 74.5      |
| LGW-4  | 180       | 949        | 1,576      | 8.0| 85.2      |
| LGW-5  | 80        | 1,530      | 2,054      | 7.6| 237.8     |
| LGW-6  | 120       | 1,206      | 2,000      | 7.6| 142.0     |
| LGW-7  | 90        | 1,063      | 1,764      | 7.6| 138.4     |
| LGW-8  | 40        | 868        | 1,439      | 7.3| 67.45     |
| LGW-9  | 50        | 1,132      | 1,879      | 7.6| 131.3     |
| MGW-1  | 150       | 3,313      | 5,052      | 7.0| 102.9     |
| MGW-2  | 190       | 1,052      | 1,750      | 7.4| 330.1     |
| MGW-3  | 40        | 1,797      | 2,099      | 7.3| 344.3     |
| MGW-4  | 140       | 1,084      | 1,784      | 7.2| 315.9     |
| MGW-5  | 80        | 1,573      | 2,062      | 8.22|252.0 |
| MGW-6  | 160       | 400        | 1,662      | 7.62|205.9 |
| MGW-7  | 120       | 1,536      | 2,056      | 7.6| 305.3     |
| MGW-8  | 70        | 1,480      | 3,069      | 7.7| 450.8     |

The moisture produced during the reaction was removed by a cryogenic trap of −80 °C. The CO₂ gas was solidified in a liquid nitrogen cryogenic trap. Other undesired gases were evacuated to get pure CO₂. The pure CO₂ was collected in an ampoule for ¹³C analysis on a Varian Mat GD-150 Mass Spectrometer. The isotope ratio (δ₁₃C) of ¹⁵N, ¹³C and ¹⁸O were calculated by using the following relation:

\[
\delta^{13}C_{\text{Vs}}[\text{std}] = \frac{R_{\text{Sample}} - R_{\text{std}}}{R_{\text{std}}} \times 1000 \delta_{\text{Vs}}^{13}C
\]

The overall analytical errors ±0.01‰ (δ¹³C & δ¹⁸O) and ±0.1‰ (δ¹⁵N) were recorded for measurements. To ensure precision, standard deviation of the mass spectrometer results were computed and standard deviation of each sample was ensured to be within permissible limits.
Trichlorophenol (TCP), Dichlorodiphenyltrichloroethane (DDT), endrin and dieldrin. Both LGW-1 and LGW-2 were taken from locations in the industrial areas along the drain but a high concentration of organic pollutants in the shallow groundwater sample (LGW-2) as compared to the deep groundwater sample (LGW-1) suggested that

Table 2  | Concentrations of organic pollutants in groundwater samples

| S. No. | Sample | Endrin | Dieldrin | TCP  | DCP  | DDT  | DDE  |
|--------|--------|--------|----------|------|------|------|------|
| 1      | LGW-1  | 0.012  | 0.030    | 0.023| 0.036| 0    | 0    |
| 2      | LGW-2  | 0.021  | 0.038    | 0.031| 0.050| 0.051| 0    |
| 3      | LGW-3  | 0.030  | 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| 0.010| 0.064|
| 9      | LGW-9  | 0.028  | 0.028    | 0    | 0.039| 0    | 0    |
| 10     | MGW-1  | 0.027  | 0.027    | 0    | 0.040| 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  | 0.010  | 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 (2008) and USEPA (2015)

| Compound                  | WHO   | USEPA |
|---------------------------|-------|-------|
| Endrin                    | 0.002 | 0.0006|
| Dieldrin                  | —     | 0.00003|
| Phenolic compounds        | <0.002| —     |
| DDT and metabolites       | —     | 0.001 |

Figure 3  | Concentration (mg/L) of organic pollutants in the Lahore area.
the shallow groundwater channel is more vulnerable to contamination.

Endrin and dieldrin were found in samples LGW-3 and LGW-4 which were collected from an agricultural area near the wastewater drain (upstream), whereas sample LGW-5, collected from an agricultural area at the wastewater drain (downstream), revealed TCP, DCP, endrin, dieldrin and DDT. Presence of organic contaminants in a deep groundwater sample (LGW-3) depict that the contamination of soil has reached its saturation point and contaminants are seeping down in deep water aquifers.

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

Overall endrin and dieldrin were found in nine samples, DCP in six, TCP in five, DDT in four and Dichlorodiphenyl-dichloroethylene (DDE) in two samples. Contamination of both shallow groundwater (high level) and deep groundwater (low level) is an alarming situation. Shallow groundwater channels are suffering from wastewater irrigation infiltration and seepage of unlined wastewater drains. Deep groundwater channel contamination suggests the overloading of organic pollutants in deep soil through wastewater irrigation, where the buffering and degradation potentials of soils exhibited low organic carbon retention, which might be due to the variable and changing nature of organic matter and clay content in soils (Tariq et al. 2004).

Analysis of organic compounds in Mangamandi groundwater

The concentrations of organic pollutants in Mangamandi area are shown in Table 2 and Figure 4. Sample MGW-1 was collected from a borehole in an agriculture area located near the wastewater drain whereas sample MGW-2 was collected from a borehole located in an industrial area on the opposite side of the drain. It is important to mention that there was agricultural land at one side of the drain and an industrial area on the other side. High concentrations of organic pollutants in agricultural areas (MGW-1) as compared to non-agricultural areas (MGW-1), within the same grid, suggest more vulnerability of groundwater channels in agricultural areas towards organic pollutants.

MGW-3 (a shallow groundwater sample) was collected from an agricultural area near the wastewater drain whereas MGW-4 was also collected from the same area but was a deep water sample. Comparative high concentration of organic pollutants in MGW-3 as compared to MGW-4 clearly suggest that the impact of wastewater irrigation becomes worse from shallow to deep groundwater channels.

Samples MGW-5 (shallow groundwater) and MGW-6 (deep groundwater) were collected from a residential area surrounded by agricultural land. The presence of comparative high

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**Figure 4** | Concentrations (mg/L) of organic pollutants in the Mangamandi area.
concentrations of organic pollutants in MGW-5 as compared to MGW-6 indicates shallow groundwater deterioration. Samples MGW-7 and MGW-8 were collected from an industrial area near agricultural land. Endrin, dieldrin, DDE and DCP were detected in these samples as well. This 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, and TCP in three samples. This might be due to infiltration of wastewater along with pesticides to contaminate the groundwater (Turberg et al. 1994). Contamination of even shallow to deep groundwater shows that soil has reached its saturation point and contaminants are seeping down into groundwater to contaminate it. It is a fact that the soil provides a potential pathway of pesticide transport to contaminate water, through runoff and subsurface drainage, interflow and leaching (Abrahams 2002).

$\delta^{13}C$ values in groundwater samples

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

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

**Figure 5** | Plot of $\delta^{18}O$ and $\delta^{15}N$ values to assess the origin of pollutants.
The source of pollution in the groundwater was assessed by using stable isotope tools. Ratios of δ¹⁸O and δ¹⁵N were applied to determine the source of pollutants as shown in Figure 5 and Table 3.

The ratios of δ¹⁸O and δ¹⁵N of nitrates in water samples were placed in the assigned Kendall plot values and sources of organic pollutants induced in groundwater from Lahore and Mangamandi were assessed. The sources of pollutants assessed at sample points based on the isotopic data are anthropogenic and the nitrates in the groundwater mainly from fertilizers that were being used in fields. Nitrates from these fertilizers seeped through the soil and into the groundwater along with the wastewater that is used for cultivation.

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### CONCLUSIONS

i. Significant concentrations of organic compounds including endrin, dieldrin, DCP, TCP, DDE, DDT were found in groundwater samples from the Lahore and Mangamandi areas.

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

iii. The ratios of δ¹⁸O and δ¹⁵N of nitrates in water samples suggested that the nitrates in groundwater are mainly coming from fertilizers being used in fields.

iv. The depleted δ¹³C‰ value in some areas in the vicinity of wastewater drains suggested that wastewater drains, to some extent, are contributing to contaminating the shallow groundwater channels.

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

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

### DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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