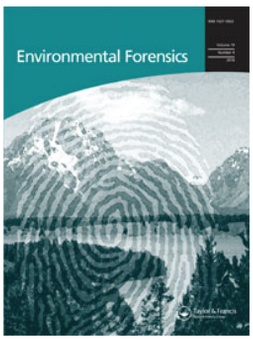

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Assessment of trace metal contamination and its anthropogenic influence in the sediments of an urban water body in Kozhikode, Kerala, India

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ABSTRACT

Urban water bodies in India receive pollution loads from numerous sources. Identification of the polluter in such cases is a difficult process. An investigation of forensic nature is needed for this. The present study targets the Conolly canal (aka Canolly Canal in local parlance) passing through the centre of Kozhikode city, constructed for inland navigation during the British reign of India. It is a typical urban water body that is exposed to different point and non-point sources of pollution, directly receiving untreated municipal and industrial sewage, in addition to storm water. For the identification of the sources polluting the Canal, an investigation following the guidelines regarding the collection of samples in the 'Pollution Crime Forensic Investigation Manual' published by the INTERPOL, was carried out. The human influence on the heavy metals (Cd, Cr, Cu, Hg, Fe and Pb) concentration in the canal sediments was analysed using the following indices – Enrichment factor (EF), contamination factor (CF) and geo-accumulation index (I_{geo}). Sediment samples for the study were collected from the different locations and depths of the canal bed and were analysed using atomic absorption spectrometry. All the three indices showed human influence on the heavy metals contamination of the canal. The results indicated that the EF and CF for the metals analysed are in the order of Cd > Cu > Cr > Hg > Pb. The Igeo showed high pollution level for the metals Cd and Cr. All three indices showed maximum values for the top (surface) sediments, which indicated the ongoing waste disposal into the canal. The study also revealed that the contamination levels are very high at the location where a major drain discharges waste water into the canal and the level of contamination was above the limiting value set by the World Health Organisation, and hence a cause for serious concern.

KEYWORDS

Canolly Canal; heavy metal contamination; Pollution Crime Forensic Investigation Manual; sediments; heavy metal contamination; enrichment factor; contamination factor; geo-accumulation index

Introduction

Heavy metals are one of the major pollutants in the water body, particularly in the urban setting. They are partitioned in various environmental components–water, suspended solid, biota and sediments. Unlike other pollutants, the heavy metals are non-biodegradable and will finally deposit on the sediments (Perin et al., 1997; Abbas and Shakweer, 2005). The mechanism of metal transfer from water to sediments could be by adsorption on the sediment surface (Du Laing et al., 2009; Peng et al., 2009), coprecipitation (Gaur et al., 2005; Varol, 2011) and consequent deposition on sediments or complex formation (Sorvari and Sillanpää, 1996; Weng et al., 2002; Awofolu et al., 2005; Senesi & Loffredo 2005; Okafor and Opuene 2007). Sometime the elements may form oxides or hydroxides of Fe, Mn (Bradl, 2004) that settles down and join the sediments. Also, the trace metals which interact and attach to organic matters in aqueous phase, finally settle down (Bahnasawy et al., 2011) and become part of bottom sediments. The deposited metal is retained by the soil (Evan, 1989) and over a time this depositions will reach to a level that will be toxic to the aquatic life (Bai et al., 2011; Mohiuddin et al., 2011; Sundaray et al., 2011; Yi et al., 2011; George et al., 2012; Tao et al., 2012; Li et al., 2013).

Both natural and anthropogenic activities are the sources of the trace metal in the aquatic environment. The anthropogenic sources are mainly from the industry (Krishna & Govil 2007; Gowd et al., 2010) and mining activities (Jian-Min et al., 2007; Mishra et al., 2008), which discharges their waste to the water resources like lake, river, canal and estuaries. Untreated municipal, domestic and agricultural effluents also have a major role in the contribution of heavy metal in the

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aquatic environment (Smith et al., 1996; Koch and Rotard, 2001; Rattan et al., 2005). The heavy metals may reach the water sources through natural phenomenon like weathering of rocks (Muwanga & Barifaijo 2006; Sekabira et al., 2010), also. In order to control the heavy metals in the aquatic sediments, it is important to know their sources.

Identifying the source(s) of pollution and providing a technical framework for allocating the responsibility for cleaning up the pollution to the polluter(s) using scientific methods, are fundamental purposes of the emerging field of Environmental Forensics (Varghese and Alappat, 2012). Environmental forensic tools are now increasingly being used to identify potentially responsible parties in the event of environmental pollution (Morrison, 2000, Raghunath S/o Rakhamji Lokhane Vs MPWPB & Ors (Original Application No. 11/2013(THC)(WZ), Janardan Pharande Vs MoEF and Ors (Original Application No. 7/2014 (THC) (WZ)) and Vinesh Madanyya Kalwal Vs State of Maharashtra Ors. (Original Application No. 30(THC)/2013(WZ)). Recognising the importance of forensic environmental investigations in legally establishing responsibility of pollution, the International Police Organisation (INTERPOL) has published a manual for environmental forensic investigation (INTERPOL, 2014). The manual gives guidelines for collecting environmental samples for forensic investigation. Compliance with such guidelines improves the acceptability of the results of investigation.

As contamination/pollution can be natural or manmade, the first challenge before an environmental forensic expert is to ascertain if the pollution is manmade or natural. After that only, questions of responsibility arise. This is true in the case of heavy metal pollution of aquatic sediments, also. There are existing tools, in the form of some indices, which can be used in such instances to establish the human influence on pollution.

The current study combined the guidelines of the INTERPOL manual on 'Pollution Crime Forensic Investigation' for sample collection and the above-mentioned indices to demonstrate the influence of human activity on the heavy metal concentration in the sediments of Conolly Canal, an urban water body in the Kozhikode Corporation area of the State of Kerala, India.

Material and methods

Description of study area- Conolly Canal, Kozhikode

The Canolly Canal, a manmade canal constructed in 1848, connects the Korapuzha River in the north and the Kallai River in the south and flows through the Kozhikode Corporation in Kerala, India (Figure 1).

The Kozhikode district falls within latitudes 11°08' and 11°42' and longitudes 75°31'48" and 75°49'30" and is situated along the southwest coast of India. The district has a humid tropical climate and an average annual rainfall of approximately 3,000 mm. The Conolly canal is 11.4 km long and the width ranges from 6 to 20 m. The water depth in the peak rain period varies from 0.5 to 2m. The areas along the whole stretch of the canal are heavily urbanised except for the most northern part. There are a lot of industrial activities such as coir retting, log setting and other kinds of timber industries around the southern end of the canal. Most residential areas and several

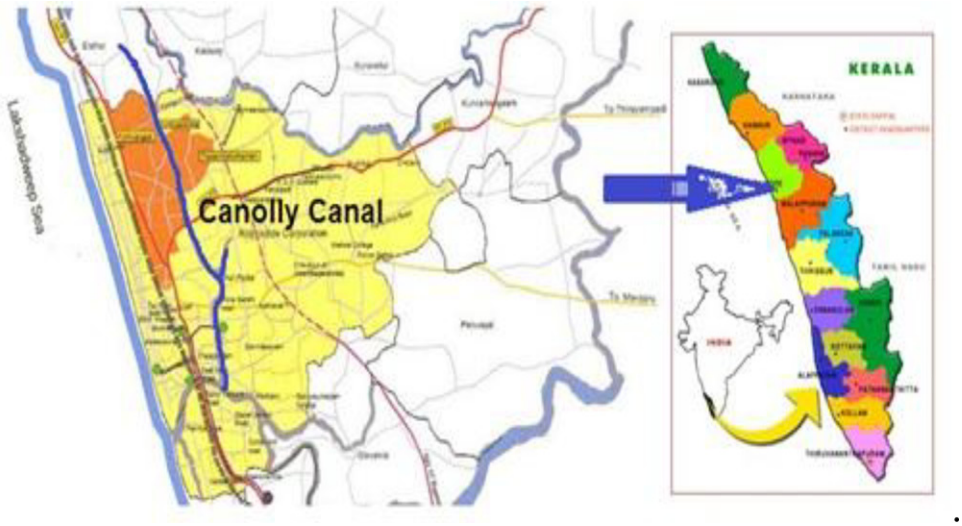

Figure 1. Study area.

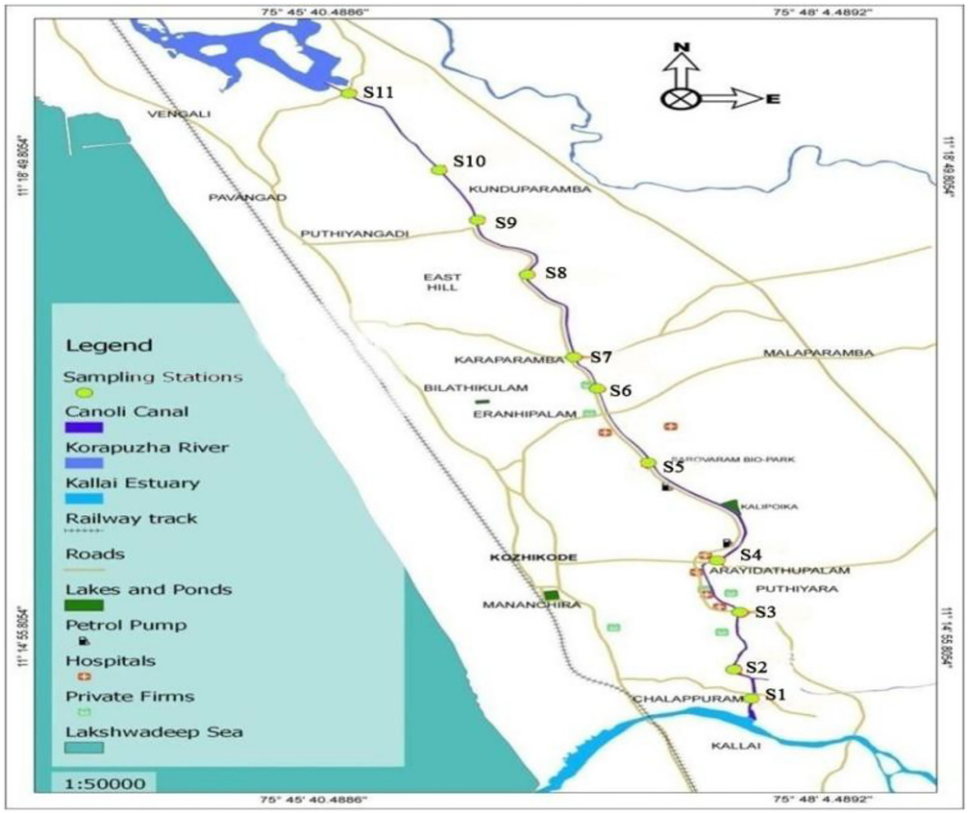

Figure 2. Map of Conolly Canal with sampling locations.

hospitals along the canal are letting out all their waste water into the canal via ditches. In addition to the liquid waste, there are also considerable amounts of solid waste dumped into the Conolly Canal, both domestic as well as industrial.

The Conolly canal, which receives sewage from different parts of Kozhikode Corporation, has been a focus of many studies intended to analyse the water quality issues. Megha et al. (2015) studied the bacterial contamination of Conolly canal and observed that the canal is contaminated with pathogenic organisms and the contamination had spread to nearby wells. Bindhyamol and Harikumar (2013) analysed water from 11 locations within the canal and reported that the southern stretch of the canal is more polluted than the northern stretch. Though previous studies have reported water quality problems in the canal, no studies were conducted on the level of pollution of canal sediments.

Sampling and analysis for heavy metal determination

Thirty three samples were collected form eleven locations of Conolly canal (Figure 2). From each sampling location, the sediments were collected at three depth ranges 0–5 cm (top), 5–15 cm (middle) and 15–30 cm (bottom) by means of Van Veen grab sampler. The sampling locations are selected based on the ecological condition and human activity near the canal and also considering the guidelines given by the Interpol Pollution Crime Investigation Manual-Vol-1 (INTERPOL, 2014). The collected sediments were stored in pre labelled polythene bags. Analysis of metal (Fe, Cu, Cr, Pb and Cd) using Flame Emission Absorption Spectrophotometer (FL-AAS, model: 3300) was done taking 5 g sample, after triacid total digestion (HNO_3, HF and $HClO_4$). Hg was tested in Absorption Spectrophotometer using cold vapour technique. Analytical blanks were run in the same way as the samples and concentrations were determined using standard solutions prepared in the same acid matrix. Suspensions were triplicate with results as mean values. Results were expressed in mg/kg dry sediment. The precision and accuracy were checked in standard reference material (CRM 277). The analysed value showed good agreement with the certified value (Table 1).

Indices to determine human influence on heavy metal concentration of sediments

To establish the human influence on the presence of heavy metals in the sediment samples, researchers have used indices like *Enrichment Factor (EF)* (Hilton

Table 1. Comparison of analytical result of reference material (CRM277) with certified data.

Element	Cd	Cu	Cr	Hg	Pb
Certified	11.90 (± 0.06)	101.90 (± 1.6)	192 (± 7.0)	1.77 (± 0.06)	149 (± 3.0)
Measured (n = 6)	11.6	104.8	189	1.70	145
Recovery (%)	97.4	102.8	98.4	96.0	97.3
Accuracy (%)	−2.6	+2.8	−1.6	−4	−2.7

et al., 1985; Kaushik et al., 2009; Ghrefat et al., 2011; Mohiuddin et al., 2011; George et al., 2012; Nowrouzi & Pourkhabbaz, 2014), *Contamination factor (CF)* (Loska et al., 1997; Loska et al., 2004; George et al., 2012) *and Geo-accumulation index (I_{geo})* (Ghrefat et al., 2011; George et al., 2012; Nowrouzi & Pourkhabbaz, 2014). Brief descriptions of these indices are given below.

i. *Enrichment Factor (EF)*, is a normalisation method proposed by Simex & Helz (1981), to assess the concentration of the metals. It normalises metal concentration as a ratio to another constituent of the sediments (Nowrouzi & Pourkhabbaz, 2014)

$$\mathrm{EF} = \frac{\left(\frac{M}{Fe}\right)sample}{\left(\frac{M}{Fe}\right)b} \quad \text{(i)}$$

$(M/Fe)_{sample}$ is the ratio of metal and Fe concentration in the sample and $(M/Fe)_b$ is the world shale average of the metal. Shale averages proposed by Turekian and Wedepohl (1961) were used in this study.

ii. *Contamination factor (CF),* is usually used to express the contamination level of sediments (Hakanson, 1980),

$$\mathrm{CF} = \frac{M_S}{M_B} \quad \text{(ii)}$$

M_s is the metal content of the sediment and the M_B represents the world shale average (Turekian and Wedepohl, 1961).

iii. *Geo-accumulation index (I_{geo})* (Muller, 1969), work by comparing the concentration of the selected heavy metal with that of a component which is unaltered by human influence.

$$\mathrm{I_{geo}} = \log_2 \frac{C_n}{1.5B_n} \quad \text{(iii)}$$

Where C_n is the measured concentration in the sediments for the metal 'n', B_n the background value for the metal 'n' and the factor 1.5 to accommodate the variations in the background data. The world average shale data from Turekian and Wedepohl (1961) was used as the background value of metals.

As indicated, the metal concentrations in unpolluted soil used in the calculation of these indices are the world shale averages. But, in order to validate the use of world shale averages for the instant case, metal concentrations were measured in the soil from an area nearby where the chances of pollution appeared minimum based on site investigation. If the metal concentrations in the soil measured are more than the world shale average, the uses of world shale averages in the calculations are not justified.

The procedure followed for the determination of heavy metals in canal sediments was used for determining background metal concentrations in nearby unpolluted soil.

Result and discussion

Metal concentration in samples

The minimum and maximum of total metal concentration observed at top, middle and bottom for the whole stretch were: Cu (1.200-1189, 3.90-1095, 5.30-853.30); Cd (0.08-12.06, 0.01-10.90, .010-9.96); Cr (50.50-1138, 42.70-1008, 56.40-993); Pb (0-111.90, 0-72.50, 0-80.50); Hg (0.33-1.41, 0.52-2.15, 0.34-1.21); Fe (23,400–48,020, 29,513–66,465, 30,500–74,109). Details of the test result are shown in the Table 2.

The Cu, Cd and Cr concentration at the location S6 was found to be very high. This location is in the city centre and is close to the discharge point of a major drain. The metal concentrations of this location were much higher than the world shale average value (Turekian & Wedepohl, 1961) of the metals. At most of the locations the Fe concentration was less than the average shale value, which shows that there is less anthropogenic Fe in the study area (Ghrefat et al., 2011).

The test results are first checked for the linear correlation between the metal pairs in terms of the significant positive correlation coefficient. The correlation analysis was done separately for the three layers sample (Tables 3–5). A strong positive correlation was observed for Cu–Cd, Cu–Cr and Cr–Hg.

Table2. Test results of trace metal concentration of Conolly Canal.

Sampling Location	Cu(mg/kg)	Cd (mg/kg)	Cr(mg/kg)	Pb(mg/kg)	Hg(mg/kg)	Fe(mg/kg)
$S1_t$	142.9	1.26	566	0	0.933	29,085
$S1_m$	125.3	0.92	476	0	0.841	29,513
$S1_b$	102.4	0.56	420	0	0.462	33,539
$S2_t$	76.5	0.08	64.9	0	0.810	23,400
$S2_m$	74	0.01	42.7	0	0.826	29,820
$S2_b$	86.9	0.01	76.9	0	0.578	30,500
$S3_t$	211.6	2.02	884	0	1.200	42,099
$S3_m$	235.5	2.47	882	0	1.520	48,631
$S3_b$	246.9	2.56	879	0	1.100	54,186
$S4_t$	98.2	0.56	507	0	1.200	31,141
$S4_m$	86.1	0.38	499	0	1.452	38,752
$S4_b$	63.7	0.26	459	0	1.200	40,161
$S5_t$	134.2	1.02	964	0	1.410	28,380
$S5_m$	102.4	0.51	649	0	1.450	35,742
$S5_b$	89.9	0.44	519	0	1.010	43,967
$S6_t$	1,189	12.06	1,138	0	1.120	29,085
$S6_m$	1,095	10.90	1,008	0	2.145	66,465
$S6_b$	855.3	9.96	993	0	1.210	74,109
$S7_t$	5.7	6.28	68.7	0	0.996	27,523
$S7_m$	7.3	2.25	71.6	0	0.885	29,513
$S7_b$	49.4	1.36	56.4	0	0.342	32,412
$S8_t$	2.1	0.78	94.7	111.9	0.806	48,020
$S8_m$	3.9	1.19	85.7	72.5	0.980	54,010
$S8_b$	5.3	1.20	103.7	80.5	0.680	66,460
$S9_t$	61.7	1.58	62.1	5.7	0.412	39,100
$S9_m$	51.8	0.81	63.3	2.9	0.530	41,170
$S9_b$	171	0.23	66.5	0.7	0.482	44,990
$S10_t$	249.3	1.83	89.5	0	0.332	36,070
$S10_m$	251.7	0.75	77.5	0	0.542	43,460
$S10_b$	239.2	1.00	85.1	0.45	0.620	64,740
$S11_t$	1.2	1.30	50.5	0	0.443	35,500
$S11_m$	40.1	1.28	61.1	0	0.523	35,868
$S11_b$	9.3	1.57	62	0.32	0.712	62,767

b, bottom; m, middle; t, top.

Table 3. Pearson correlation matrix between different heavy metal pair (top layer).

	Cu	Cd	Cr	Pb	Hg	Fe
Cu	1.000	0.840	0.660	−0.199	0.244	−0.199
Cd		1.000	0.442	−0.178	0.216	−0.237
Cr			1.000	−0.261	0.770	−0.176
Pb				1.000	−0.089	0.670
Hg					1.000	−0.305
Fe						1.000

Table 4. Pearson correlation matrix between different heavy metal pair (middle layer).

	Cu	Cd	Cr	Pb	Hg	Fe
Cu	1.000	0.942	0.681	−0.203	0.707	0.743
Cd		1.000	0.623	−0.088	0.699	0.747
Cr			1.000	−0.258	0.901	0.510
Pb				1.000	−0.067	0.370
Hg					1.000	0.599
Fe						1.000

Table 5. Pearson correlation matrix between different heavy metal pair (bottom layer).

	Cu	Cd	Cr	Pb	Hg	Fe
Cu	1.000	0.927	0.698	−0.234	0.485	0.527
Cd		1.000	0.681	−0.066	0.498	0.620
Cr			1.000	−0.230	0.798	0.277
Pb				1.000	−0.092	0.362
Hg					1.000	0.409
Fe						1.000

Anthropogenic trace metal pollution

The background concentration of heavy metals determined in the soil of the nearby unpolluted site and its comparison with corresponding world shale averages, are given in Table 6.

All concentrations, except that of Cd are much less than the world shale average, for the unpolluted soil. Thus, it is clear that adoption of world shale averages will not over predict the human influence on pollution. This fact is very important when the pollution case is decided in a court of law. For all calculations in this study, the world shale average concentrations of metals were used as it is the widely accepted standard, and as it will NOT over predict the responsibility of the polluter, which is legally unacceptable.

Enrichment factor (EF)

The EF is a normalisation method proposed by (Simex & Helz, 1981) to assess the concentration of the metals. Metals Al or Fe are usually unaffected by the anthropogenic activity (Rejomon et al. 2016). Because of this, several researchers used Fe and Al as the normaliser (Neto et al. 2000; Zhang et al. 2007; Amin et al. 2009; Çevik et al., 2009). This study also

used Fe as conservative trace (normaliser) to identify the anthropogenic sources of metals using the equation (i). The plot was developed as shown in the Figure 3, with X- axis showing the sampling stations, S1 being the southernmost station and S12, the northernmost.

Table 7 shows the level of contamination based on EF (Birch, 2003). The average EF values in the canal sediments for the analysed metals on top, middle and bottom layers, respectively, were Cu (6.72, 3.95, 3.31), Cd (13.34, 6.30, 4.50), Cr (6.77, 4.32, 3.56), Pb (0.53, 0.30, 0.26), Hg (3.28, 3.05, 1.87). Thus, the average EF values showed a contamination level of moderately severe to severe enrichment, for most of the metals. The maximum EF values observed for Cu (42.70), Cd (64.96) and Cr (20.43) were at the sampling point S6, in top layer. The very severe enrichment at this location could be due to the discharge of the city drain.

It was also observed that the top layer of sediments (collected at a depth of 0–5 cm) have the highest EF value for all the analysed metals.

Contamination factor (CF)

Contamination factor is usually used to express the contamination level of sediments (Hakanson, 1980). The CF for all the test locations were calculated using equation (ii) and the plot was developed as shown in the Figure 4. Here also, X- axis shows the sampling stations with S1 as the southernmost station and S12, the northernmost.

The classification of contamination level based on CF (Hakanson 1980) is shown in Table 8. Average values of CF on top, middle and bottom layers, respectively, were Cu (4.39, 4.19, and 3.88), Cd (8.73, 6.52, 5.82), Cr (4.53, 3.96, 3.76), Pb (0.53, 0.74, 0.34), Hg (2.20, 2.66, 1.91) and Fe (0.71, 0.88, 1.06). CF values showed that the contamination level is high for the metals Cu, Cd and Cr. Contamination levels for Pb and Fe were low indicating that there is no anthropogenic influence in the case of these two metals. It is also observed that at the sampling point S6, the metals Cu, Cd, Cr and Hg have very high level of contamination, same as the trend seen with EF. This, points to the possibility of a point source of contamination in its vicinity. Also, the top layer sediments have the highest CF for 3 of the metals, Cu, Cd and Cr.

Geo-accumulation index

Present metal concentration in the sediments can be compared with pre industrial level using Geo-accumulation index (I_{geo}) (Muller 1969). The test resuts are applied in equation (iii) for finding the Igeo index. The values of I_{geo} plotted against the sampling stations is shown in Figure 5.

The I_{geo} has seven classes of contamination level (Muller, 1969) as shown in Table 9. The average value of the I_{geo} for different metals at the study area on top, middle and bottom layers, respectively, were Cu

Table 6. Metal concentrations measured in unpolluted soil.

Metal	Background concentration in nearby unpolluted soil (mg/kg)	World shale average (mg/kg) (Turekian and Wedepohl, 1961)
Cu	0.40	45
Cd	0.30	0.30
Cr	0.70	90
Pb	0.50	20
Hg	Not detected	0.40

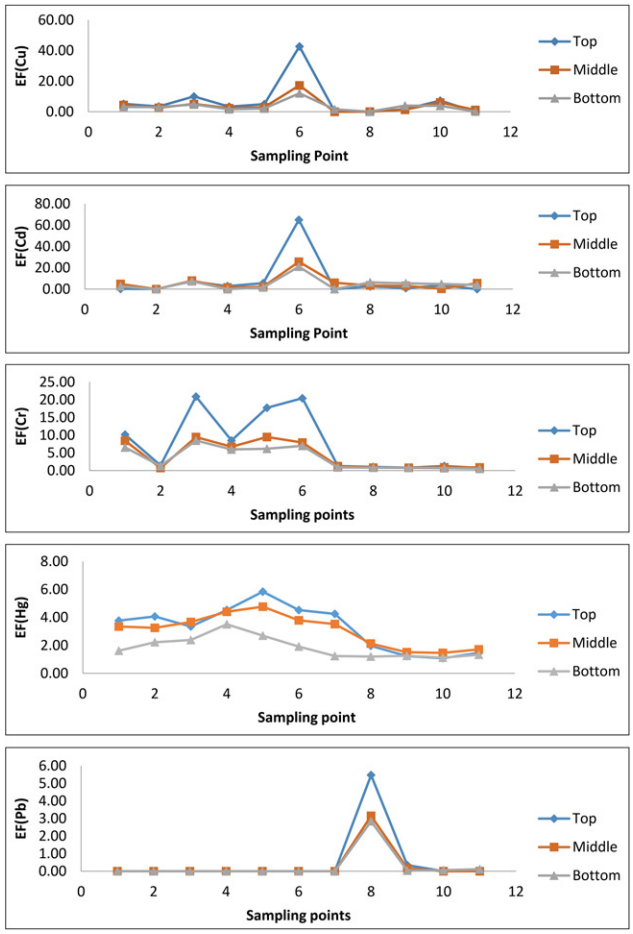

Figure 3. Spatial variation of enrichment along the Canolly Canal.

Table 7. Enrichment categories (Birch, 2003).

Value	Contamination level
EF < 1	No Enrichment
EF = 1–3	Minor Enrichment
EF = 3–5	Moderate Enrichment
EF = 5–10	Moderately severe Enrichment
EF = 10–25	Severe Enrichment
EF = 25–50	Very Severe Enrichment
EF > 50	Extremely Severe Enrichment

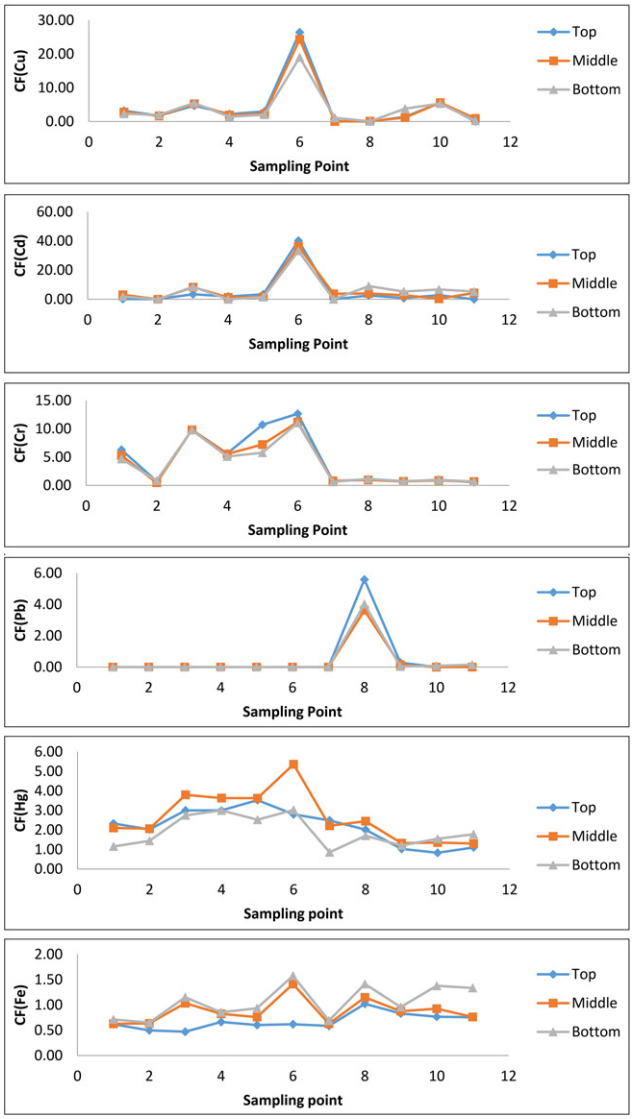

Figure 4. Spatial variation of CF along the Conolly Canal.

Table 8. Contamination factor (Hakanson 1980).

Value	Contamination level
CF < 1	Low
CF = 1–3	Moderate
CF = –6	High
CF > 6	Very High

(10.62,11.10, 11.26), Cd (−1.89, −2.60, −2.92), Cr (13.61, 13.47, 13.50), Pd (1.53, 1.38, 1.21), Hg (−2.23, −1.97, −2.41) and Fe (29.94, 30.22, 30.47). The metals Cu & Cr were in extreme pollution level. Further, the values did not show much variation across the different layers. Among the different sampling location S6 showed extreme pollution for all the metals. This showed the presence of a point source of contamination, which was similar to the conclusion made from the EF and the contamination factor (CF).

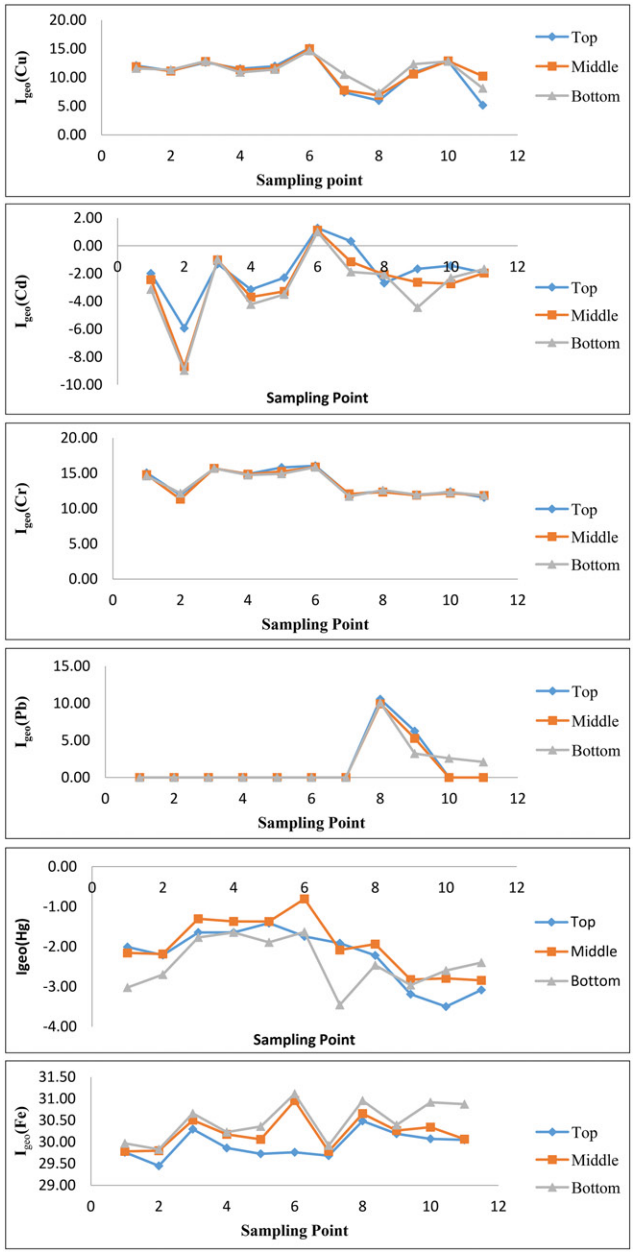

Figure 5. Spatial variation of Igeo along the Conolly Canal.

Comparison with human health based limits

World Health Organisation (WHO, 2006) has given the maximum tolerable soil concentrations of various toxic chemicals based on human health protection. The WHO recommendation for the metals Lead is 84 mg/kg and for Cadmium is 4 mg/kg. The test results of Pb and Cd are normalised with the WHO limiting value and represented in Figure 6. The normalised value above one represents the location having higher concentration than specified by the WHO. The concentrations at location S7 to S9 and S5 to S7, are above the limits set by WHO for Lead and Cadmium, respectively.

Table 9. Geo-accumulation index (Muller, 1969).

Igeo	Class	Contamination level
<0	0	unpolluted
0–1	1	Unpolluted to moderately polluted
1–2	2	Moderately polluted
2–3	3	Moderately to Strongly polluted
3–4	4	Strongly Polluted
4–5	5	Strongly to extremely polluted
>5	6	Extremely polluted

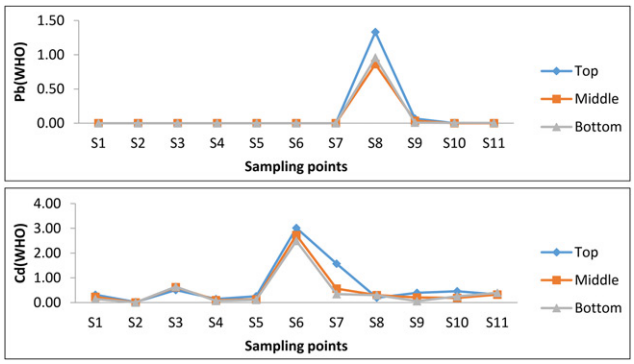

Figure 6. Spatial variations showing the metal concentration exceeding WHO limit.

Conclusion

For establishing the responsibility for pollution, the legal system requires reliable evidence. The acceptance of the evidence will depend on the procedures followed to collect, and interpret the particular evidence. In this regard, strict adherence to standard guidelines and procedures increases the acceptability of the evidence. In the present study for establishing the human influence of pollution in the urban water body, the sample collection procedure laid by the INTERPOL was followed. The testing of the samples were done in the calibrated instrument by following the standard procedure and the results were analysed and interpreted scientifically using the indices, EF, CF and I_{geo}.

All the three indices calculated showed severe pollution of the Conolly Canal, especially the location where the canal receives effluents from a major city drain. The levels of Cu, Cd and Cr in the sediments at this location were found to be of extreme concern. As expected, southern stretch that flows through highly urbanised areas are more polluted compared to the northern stretch. The spikes observed for the indices at some sampling, points to localised sources of pollution in its vicinity. The higher values of the indices for the top layer indicate recent or ongoing pollution and should be a matter concern for regulatory agencies. Understanding and assessing the human influence on the concentration of different contaminants will help regulators to enforce proper corrective measures. Also, it may be noted that the contamination levels at many locations exceeded the limits set for safeguarding human health. This, naturally, would call for extreme caution while disposing the sediments dredged from the Conolly Canal.

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