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Isolation, purification and analysis of dissolved organic carbon from Gohagoda uncontrolled open dumpsite leachate, Sri Lanka

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ABSTRACT

Extract and analysis of the Dissolved Organic Carbon (DOC) fractions were analyzed from the leachate of an uncontrolled dumpsite at Gohagoda, Sri Lanka. DOC fractions, humic acid (HA), fulvic acid (FA) and the hydrophilic (Hyd) fractions were isolated and purified with the resin techniques. Spectroscopic techniques and elemental analysis were performed to characterize DOCs. Maximum TOC and DOC values recorded were 56,955 and 28,493 mg/L, respectively. Based on the total amount of DOC fractionation, Hyd dominated accounting for ~60%, and HA and FA constituted ~22% and ~17%, respectively, exhibiting the mature phase of the dumpsite. The elemental analysis of DOCs revealed carbon variation following HA > FA > Hyd, while hydrogen and nitrogen were similar in each fraction. The N/C ratio for HA was recorded as 0.18, following a similar trend in old dumpsite leachate elsewhere. The O/C ratios for HA and FA were recorded higher as much as 1.0 and 9.3, respectively, indicating high degree of carbon mineralization in the leachates. High content of carboxylic, phenolic and lactone groups in all DOCs was observed disclosing their potential for toxic substances transportation. The results strongly suggest the risk associated with DOCs in dumpsite leachate to the aquatic and terrestrial environment.

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KEYWORDS

DOC; humic acids; landfill leachate; leachate characterization; municipal solid waste



Introduction

Landfill leachate is the hazardous liquid formed primarily by the percolation of precipitation water through an open landfill. Leachate may contain four groups of pollutants: dissolved organic matter (DOM), inorganic macro-components, heavy metals and xenobiotic organic compounds [1]. In many cases, the leachates from landfills are highly contaminated and have higher concentrations of organic as well as inorganic pollutants and toxic substances, such as heavy metals [2,3]. The quality or composition of landfill leachates greatly vary with landfill age, seasonal weather variation, waste type and composition [4]. A combination of physical, chemical and microbial processes in the waste transfers pollutants from the waste material to the percolating water [5]. In any case, in most of the developing regions of the world, open dumpsites are the most dominant municipal solid waste (MSW) management practice where the landfill leachate directly flows to surface waterbodies or into groundwater [6,7]. There is extensive literature on the characterization of landfill leachate. However, most of these works have been reported on usual parameters (i.e. pH, electrical conductivity, chemical oxygen demand and nutrients), heavy metals and xenobiotics, such as phenols, halogenated hydrocarbons and aromatic hydrocarbons [7,8]. Only a few studies have paid attention to DOM and dissolved organic carbon (DOC) present in landfill leachate [2,9-11]. Therefore, there is a significant knowledge gap on the uncontrolled open dumpsites in developing countries to understand complex composition of landfill leachate in relation to

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identifying their fate, transport and optimum treatment methods. On the other hand, most of leachate characterization studies from developing countries have failed to stress the importance of DOCs, such as humic-like compounds [6,7]. These humic-like substances represent the intermediate degradation of organic waste in landfills. In the early phase or the acetogenic phase of leachate, a large quantity of volatile fatty acids is released, as much as 95% of the organic content [4]. As a landfill matures, methanogenic microorganisms develop in waste, and the volatile fatty acids are converted into biogas, like methane. Hence, the organic fraction in the leachate becomes dominant by non-biodegradable organic compounds, like humic substances [4].

Generally, DOM is defined as the organic matter fraction in solution that passes through a 0.45 μ m filtration membrane. The organic carbon remaining after filtering the sample with a 0.45 μ m filter membrane is known as DOC. Since amount of the carbon represents around 67% by mass in the bulk of the elemental composition of the organic matter, the DOM is often considered by its carbon content and referred to as DOC [12]. Low molecular weight substances, such as amino acids, carbohydrates and organic acids, and high molecular weight substances, like humic substances (i.e. HA, FA and humin), are referred as well-known DOC matrices [13].

The speciation and mobility of heavy metals in groundwater may be significantly influenced by the presence of DOCs due to the formation of dissolved complexes between DOCs and the heavy metals [14]. A variety of functional groups, including COOH, phenolic OH, alcoholic OH, lactone, guinone, hydroxyguinone and ether are also present in the complex structures of humic substances [15,16]. These functional groups significantly affect their high activity or functionality in soil and aquatic environments. The concentrations of DOC can be high up to 500 mg/L in leachate-polluted groundwater and significant complexation has been demonstrated in leachate-polluted groundwater with respect to Cd, Ni and Zn [14]. The complexing ability of DOC is primarily related to its content of functional groups (e.g. carboxylic and phenolic groups). Because these groups exhibit acid-base behavior, the complexation capacity of the DOC with respect to metals depends on the pH of the system [14]. On the other hand, it has been revealed that chlorine and its related species react with organic matter in water to produce chemical compounds known as the disinfection by-products. Of these, trihalomethanes and haloacetic acids can be found in the highest concentrations in treated drinking water [17]. It is widely recognized that the association between hydrophobic organic contaminants (HOC) and large molecules, especially DOM, significantly influences the apparent aqueous solubility of these HOCs and increases their mobility from solid matrix into aqueous environment [18]. Therefore, knowledge about the role and interactions of DOC as well as its characteristics is essential to evaluate the risk of pollution and to establish optimal remediation strategy [19].

Gohagoda open dumpsite, situated in Kandy, is one of the major dumpsites in Sri Lanka and is operated by the Kandy Municipal Council. At present, more than 130 tons of MSW per day collected within the city limit is being dumped at this site, which is 7 km away from the city. The leachate from Gohagoda dumpsite was recorded as highly polluted and directly flows to the Mahaweli river which is the main water source for the entire province due to the absence of a proper lining system or any treatment mechanism before disposal [7]. In fact this alone brings the focus to characterize DOC from Gohagoda uncontrolled dumpsite leachate, as they are precursors of carcinogenic chlorinated by-products that can be produced by chlorination of DOC-rich water. It is clear that the effluent discharge of Gohagoda dumpsite is exceeding the permissible standards, thus impacting the ecosystem, and during heavy rainfall the accumulated pollutants are washed into the river, which is a serious concern. Hence, the objectives of this study were isolation and purification of DOC from Gohagoda dumpsite leachate and their characterization using various analytical techniques.

Materials and methods

Landfill leachate collection and characterization

Leachate samples were collected from the top of the Gohagoda open dumpsite (located at a longitude and latitude of 7°18′47.85″N and 80°37′19.02″E, respectively) during March–October 2013 (Figure 1). Leachate samples were collected in sample bottles (washed with distilled water and leachate, respectively) and transported to the laboratory under 4°C conditions. Concentrations of total dissolved solids were measured by a Thermo Scientific conductivity meter (5 STAR, Environmental Instruments, USA). Measurements of TOC and DOC were made using a TOC analyzer (MULTI N/C 2100, Analytikjena, Germany).

Fractionation of DOC

The humic acid (HA), fulvic acid (FA) and hydrophilic (Hyd) fractions of DOC were separated and purified according to a previously described method with some modifications [2] (Figure 2). All the reagents were AR-grade chemicals.



Figure 1. Schematic of the Gohagoda MSW dumpsite, the leachate sampling point at the top of the dumpsite and leachate drainage pathway (in yellow dotted arrows).

HA fraction

Briefly, the HA fraction was separated by filtration (A-3S, EYELA, China) and ultra-centrifugation (261-234832-A, BECKMAN, UK), respectively. Prior to filtration, the leachate sample was acidified to pH 1.5 by the addition of few drops of concentrated HCI. The separated HA was redissolved in 0.05 N NaOH and then protonated by the dialysis process, for two days with each step for 12 hours, against 0.1 N HCl and deionized water (resistivity: $18.2 \text{ M}\Omega \text{ cm}^{-1}$). The dialyzed HA sample was separated from the solution by centrifugation (300807, BECKMAN, England), freeze dried (FD-1 EYELA, Tokiyo, Japan), labeled and finally stored below 4°C until use.

FA fraction

After separation of HA, the supernatant solution was passed through an Amberlite XAD-7 HP resin column. The residual which was sorbed onto XAD-7 resin is known as 'fulvic acids' (hydrophobic acids). The FA was desorbed with 0.1 N NaOH and acidified up to pH 2 using concentrated HCl. Then the solution was protonated by passing through a cation exchange resin column (Amberlite IR-120). The resulted solution was vacuum dried using a vacuum rotary evaporator (LABOR-OTA 4001, Heidolph, WB, Germany) below 50°C. Finally, the FA fraction was freeze dried, labeled and stored below 4°C until use.

Hyd fraction

The residual of solution which was not sorbed onto the Amberlite XAD-7 HP resin resulted from FA separation is referred to as the 'hydrophilic fraction'. The Hyd fraction was passed through an Amberlite IR-120 column and the resulted solution was vacuum dried. Then the isolated Hyd fraction was freeze dried, labeled and finally stored below 4°C until use. Percentages of isolated HA, FA and Hyd fractions were calculated based on Equations (1)–(3).

Percentage of HA

$$=\frac{[\text{DOC}]_{\text{leachate}} - [\text{DOC}]_{\text{supernatant of HA}}}{[\text{DOC}]_{\text{leachate}}} *100\%, \quad (1)$$

Percentage of FA

$$=\frac{[DOC]_{leachate} - [DOC]_{supernatant of FA}}{[DOC]_{leachate}} *100\%, \quad (2)$$

Percentage of Hyd

$$=\frac{[DOC]_{leachate} - [DOC]_{supernatant of Hyd}}{[DOC]_{leachate}} *100\%, \quad (3)$$

where [DOC] is the concentration of dissolved organic carbon.

Characterization of humic substances

The spectral properties of isolated fractions were examined by a Fourier Transform Infrared Spectrometer

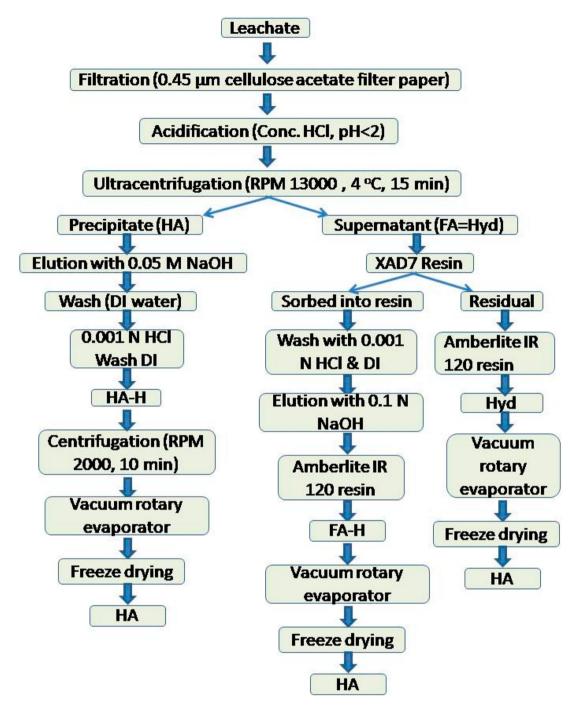


Figure 2. Isolation and the purification procedure for HA, FA and Hyd fractions in landfill leachate.

(FT-IR) (NICOLET 6700, USA) between 4000 and 400 cm⁻¹, with 1 cm⁻¹ resolution and 64 scans, using sample pellets prepared in fused KBr. The elemental composition (carbon, hydrogen and nitrogen) was determined using a CHN corder housed at the Saitama University, Japan and the presence of oxygen was calculated based on the difference to the total percentage. Organic functional groups (carboxyl, lactonic, phenolic and base groups) were determined by the Bohem titration method [20] using a fully automated titrator

(ORION EA 940, Orion Research Incorporated, MA, USA). In short, a known quantity (i.e. 0.15 g) of each isolated fraction was dissolved in 15 ml of pre-standardized solutions of 0.05 M NaOH, NaHCO₃, Na₂CO₃ and HCl in duplicate. The mixtures were shaken overnight at 100 rpm (EYELA, Shaker B 603), and samples were then filtered through a filter paper (Whatmann 42 filter paper). The filtered solution of HCl-containing sample was titrated with pre-standardized NaOH solution using methyl orange as the endpoint indicator. Other filtered

solutions were mixed with excess 0.05 M acid to ensure complete neutralization of bases and then back-titrated with NaOH. The amount of surface basic groups was calculated as moles neutralized by NaOH, in HCl-containing sample and amount of carboxylic, as the moles neutralized by NaHCO₃. The difference between molar NaOH–Na₂CO₃ and molar Na₂CO₃–NaHCO₃ was calculated as the phenolic and lactonic functional group contents, respectively [20].

Results and discussion

TOC and DOC in dumpsite leachate

The estimated TOC and DOC concentrations were quite high as average values remain at 56,955 and 28,493 mg/L, respectively. The TOC and DOC concentrations in leachate may vary with the age of landfill [19,21]. Apart from the age of landfill, it has been reported that a large variations or remarkably high fluctuations for TOC and DOC concentrations can be observed. During the acetogenic and methanogenic phases, TOC concentrations are reported in the range of 500-28,000 and 50-2200 mg/L, respectively [22]. Based on an international review, Robinson has predicted typical values for TOC as 8000 and <100 mg/L for acetogenic and methanogenic leachates respectively, from very large landfills [3]. However, the reported TOC and DOC values in the present study were high compared with previous studies [23,24].

Fractionation of DOC

The hydrophilic fraction has dominated in the analyzed leachate covering almost 60% of DOC in leachate (Figure 3). Considering the hydrophobic acids (HA and FA), HA was more abundant than FA. A landfill leachate-contaminated groundwater characterization

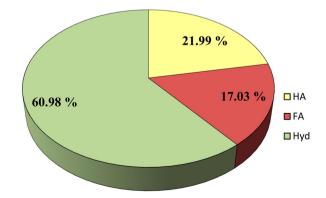


Figure 3. Percentage analysis of isolated HA, FA and Hyd fractions.

study revealed that the percentage of HA, FA and Hyd are 22, 17 and 60% of DOC, respectively [2]. Therefore, the observed percentages of HA and FA constituents in Gohagoda leachate have very much deviated from the literature. The concentrations of HA and FA depend on the age of the landfill leachate. When the leachate becomes older, the values of HA and FA tend to increase [2]. The molecular weight and aromaticity of DOCs, particularly for HA and FA have increased in old leachates [19]. On the other hand, higher HA and FA contents might increase the difficulty of leachate treatment [9]. Moreover, a higher content of hydrophilic fraction may be noticed, which is a sign of the better degradation of organic fraction of MSW [25].

Characterization of humic substances

Elemental analysis

The elemental analysis is one of the most simple and important means of characterizing humic substances. The elemental compositions of HA, FA and Hyd fractions are given in Table 1 which are compared with the previous studies. The results obtained from the present study show that the carbon content varies according to HA > FA > Hyd. The hydrogen and nitrogen contents are moreover similar in each fractionated component. In all three fractions, the least element reported is hydrogen except in hydrophilic acids. The hydrogen and nitrogen contents are moreover similar in each fractionated component. Compared to the previous studies [2,19,27], the amount of carbon contents in FA and Hyd is much lower than the values reported while the oxygen content is higher. Also, the amounts of carbon and hydrogen were less than the reported values in HA. Among the three isolated fractions, FA contained the lowest amount of nitrogen, consistent with literature values. In each fraction, nitrogen concentration was reported to be higher than the literature values indicating the high ammonia nitrogen concentrations of leachate [19].

Table 1. The elemental composition of HA, FA and Hyd fractionsin Gohagoda dumpsite leachate.

	5									
	Gohagoda leachate				Literature values					
	HA	FA	Hyd	HAª	FA ^a	Hyd ^b	HAc	FAc		
С	44.17	8.80	2.96	56.1	50.9	47.40	55.2	52.6		
Н	2.53	2.19	2.97	7.2	6.4	4.70	53	4.5		
Ν	7.83	7.18	8.77	7.4	2.4	2.50	3.3	1.7		
0	45.48	81.83	85.30	29.3	35.2	42.20	35.8	40.9		
0/C	1.03	9.30	28.82	0.36	0.54	0.67				
H/C	0.06	0.25	1.00	1.88	2.53	1.19				
N/C	0.18	0.82	2.96	0.13	0.23	0.05				

^aKang et al. [19].

^bChristensen et al. [2]. ^cChristle [26].

The higher the H/C ratio, the higher is the aliphatic character [2,27]. Thus, hydrophilic acids fraction can be considered to be more aliphatic in nature than HA and FA. However, hydrophobic acids are more aromatic than hydrophilic acids. Since the calculated H/C ratio of FA is higher than that of HA, this resembles that HA is more aromatic than FA. When the H/C ratio is above 1.3, the chemistry of the substances tends to be more likely to be non-humic [28]. Hence, the Hyd fraction is more likely to be non-humic than HA and FA. In addition, the C/H ratio in HA is typically considered an indication of unsaturated level [29]. Accordingly, higher levels of C/H in HA than in other two fractions indicated the presence of more unsaturated structures [29]. Therefore, it is an indirect indication of the heterogeneity of HA as described previously. The O/C ratio for all the three fractions of DOCs shows relatively higher values than those found in the literature [19]. The N/C ratio of HA and FA fitted well with the previous results for leachate derived from old landfills [19]. High concentration of nitrogen in Hyd may be due to the high ammonia-N content in leachate, as reported in our previous study [7].

FT-IR spectroscopy

The FT-IR spectroscopy is a powerful tool for the determination of molecular structure of DOC fractions and this technique directly provides information about the presence of functional groups. The results obtained from FT-IR for HA, FA and Hyd fractions isolated from the leachate are shown in Figure 4. All spectra have typical absorption bands in the region of 3400–3200 cm⁻¹ (broad) due to intermolecular O-H stretching and 1654.8 cm⁻¹ due to C=C stretching of aromatic or asymmetric alkenes conjugated with C=O or carboxylates [30]. It has been reported that HA exhibits strong absorption bands at about 2900, 1720, 1520, 1200 and 1050 cm⁻¹ [25]. According to the results obtained from the isolated HA fraction, the peaks appear in 2923.1 and 2851.7, 1541.7, 1455.4 and 1383.9, 1233.1 and 1041.6 cm⁻¹ are similar to the literature.

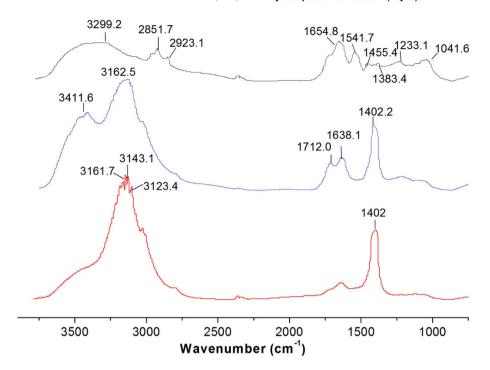
The absorbance peaks of HA around 2923.1 and 2851.7 cm⁻¹ are due to the asymmetrical or symmetrical aliphatic C–H bond stretching [2,18], which indicates the presence of methyl and methylene groups. The shoulder peak at 1718.4 cm⁻¹ for C=O stretching in carboxylic, aldehydes or ketones [25] with a peak at 1654.8 cm⁻¹ for C=C stretching of aromatic or asymmetric alkenes conjugated with C=O or carboxylates [30]. The existence of carboxylic and peptide groups in HA is explained by the IR band of 1541.7 cm⁻¹ (stretching vibrations of – CO–NH–) while the bands of lignin groups can be

observed in spectra at 1455.4 and 1383.9 cm^{-1} representing the aromatic structures [18].

The FT-IR spectra of isolated Hyd fraction resemble those of FA fraction except for a less dominating peak around 1640–1700 cm⁻¹ [2]. Both spectra have dominated by the peaks at 3180-3090 region and 1402.2 cm⁻¹ related to stretching vibrations of NH₂ (two bands) in primary amides [31] and stretching vibrations of C-O bond (sharp peak) in carboxylic groups or unsaturated alcohol compounds, respectively [9,18]. As reported by Christensen et al. [2], the FA and Hyd fractions are highly dominated by carboxylic groups. Moreover, the C=O bond stretching of carboxylic acids, aldehydes and ketones (peak at 1712.0 cm^{-1}) and C=C vibration of aromatic components conjugated with C=O (peak at 1654.8 cm⁻¹) observed in FA spectrum confirm the presence of carboxylic acids. Other than the sharp peak at 1402.2 cm^{-1} , the spectrum related to Hyd fraction possessing a small but not dominant peak around 1640–1700 cm⁻¹ indicates the presence of carboxylic groups. The enrichment of carboxylic groups in FA and Hyd fractions is also confirmed by the results of Boehm titration. In addition, the N-H bond vibration is more clearly observed as two adjoining peaks at 3162.5 and 3132.4 cm^{-1} in the FA spectrum than Hyd spectrum. The FT-IR spectrum of Hyd fraction presents some signals which are not well resolved; this fact is probably due to the complex structure of those molecules [9].

Boehm titration

The Boehm titration works on the principle that oxygen groups on carbon surfaces have different acidities and can be neutralized by bases of different strengths [32]. The dissociation properties of acidic functional groups are important parameters in order to understand the complexation capacity of humic substances [2]. Despite many researches of decades, the structures of complex DOCs such as HA and FA are ill defined yet. Nevertheless, the elemental composition and functional groups of some DOCs have been well understood [9]. According to a concept structure, HA is rich with carboxylic, phenols, lactones and basic groups [15]. The present study reveals that HA is more dominant in all carboxylic, phenols and lactones compared to FA and Hyd (Table 2). Based on an extensive characterization of DOCs, it has been concluded that the acidity of DOCs is primarily due to the presence of carboxylic and phenolic functional groups [28]. On the other hand, Ma et al. [28] has shown that the HA fractions had larger carboxyl content than other two fractions, while FA and Hyd were responsible for similar carboxyl content which is totally in accordance with the results from the present study. The presence of surface functional groups like



- Humic acid (HA) - Fulvic acid (FA) - Hydrophilic fraction (Hyd)

Figure 4. FT-IR bands of humic substances.

Table 2. Amount of surface functional groups in humic substances.

		Gohagoda leachate			Literature values	
	HA	FA	Hyd	HA ^a	FA ^a	Hyd ^a
Carboxylic groups	3271.73	2069.89	1760.92	3400.00	6100.00	6300.00
Phenolic groups	3113.16	1198.67	1364.26	1900.00	1300.00	2300.00
Lactone groups	2928.16	300.54	157.48	-	-	_
Basic groups	3909.57	4915.74	619.31	-	-	_

^aChristensen et al. [2].

carboxylic groups may result in total acidity below pH 8 in humic substances while weakly acidic functional groups (i.e. phenols) may result in total acidity, as above pH 8 [27].

Compared to the literature, the amounts of surface carboxylic groups in HA and surface phenol groups in FA have shown likeness (Table 2). Also, relatively low values have been reported in carboxylic groups in FA and Hyd in the present study than in the previous studies. However, the amount of carboxylic groups in HA could exceed in both FA and Hyd [28]. The lactone groups are significantly high in HA contrasted to both FA and Hyd. Lactones are a class of cyclic organic esters, usually formed by the reaction of a carboxylic acid group with a hydroxyl group or halogen atom present in the same molecule. The peak appears at the FT-IR spectrum of HA in relation to the lignin band might be due to high concentration of lactones. On the other hand, less quantity of lignin is an

indication of weak humification. Therefore compared to HA, FA and Hyd fractions are less humified substances. Further, basic groups have been observed in higher concentrations in HA and FA than Hyd. This observation is verified by the peak height of peptides in FT-IR spectroscopy. The dominant peak around 1540 cm^{-1} can be seen in HA spectrum and the strength of the peak is reduced as HA > FA > Hyd which is the same pattern of concentration variation of lactones in each. The presence of carboxylate and phenolate groups indicates DOCs' ability to form complexes with metal ions. The negatively charged polyelectrolytes behavior of humic substances is also caused to form associations between metal cations. Humic substances have two or more of these groups arranged so as to enable the formation of chelate complexes. The formation of (chelate) complexes is an important aspect of the biological role of HA in regulating bioavailability of metal ions [12].

Conclusions

In this study, the fractions of DOCs particularly HA, FA and Hyd were isolated and purified by resin techniques, and characterized for dumpsite leachate collected from an old uncontrolled dumpsite, Sri Lanka. Fractionation of DOCs and their chemical characteristics were investigated in elemental and spectroscopic levels. Hence, this study indicates a quantitative expression of HA, FA and Hyd in tropical dumpsite leachates. Apart from the specified DOCs, DOC (i.e. from TOC analyzer) and TOC were determined to understand dynamics of carbon contents in leachates. The observed maximum TOC and DOC values were 56,955 and 28,493 mg/L, respectively, which indicated high degree of organic discharge to the river Mahaweli. Therefore, the major source of drinking water to Kandy city is threatened by a high load of organic pollutants. Unfortunately, no limits can be found from Sri Lankan wastewater discharge guidelines for TOC and DOC. Of the total DOCs, Hyd, HA and FA were reported to be \sim 60, \sim 22 and \sim 17%, respectively. The elemental analysis of isolated DOCs revealed carbon variation following HA > FA > Hyd, whereas hydrogen and nitrogen are similar in each fraction. Moreover, the N/C ratio for HA was recorded as 0.18. The observed elemental composition of DOCs (i.e. N/C ratio for HA and FA) exhibits similar trends for old dumpsites investigated in previous studies. However, O/C ratios for HA and FA were recorded as high as 1.0 and 9.3, respectively, indicating higher degree of carbon mineralization in leachates perhaps due to the accelerated decomposition with tropical climatic conditions. In addition, high contents of carboxylic, phenolic and lactone groups in all DOCs were observed disclosing the ability in complexation of toxic substances; thereby enhancing transportation of pollutants in the aquatic and terrestrial environment. To decide an appropriate leachate treatment approach, future studies are needed to evaluate the temporal and spatial variations of DOCs. Further, new treatment or permissible units, specifically considering DOC and TOC are needed to be included to the Sri Lankan standards.

Disclosure statement

No potential conflict of interest was reported by the authors.

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