



Hydrophobic organic chemicals (HOCs) removal from biologically treated landfill leachate by powder-activated carbon (PAC), granular-activated carbon (GAC) and biomimetic fat cell (BFC)

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ABSTRACT

Biological pretreatment efficiently remove organic matter from landfill leachate, but further removal of refractory hydrophobic organic chemicals (HOCs) is hard even with advanced treatment. In this work, three-stage-aged refuse bioreactor (ARB) efficiently removed chemical oxygen demand (COD) and biochemical oxygen demand (BOD) of fresh leachate produced in Shanghai laogang landfill, from 8603 to 451 mg L⁻¹ and 1368 to 30 mg L⁻¹, respectively. In downstream treatment, 3 g L⁻¹ powder-activated carbon (PAC), granular-activated carbon (GAC) and biomimetic fat cell (BFC) removed 89.2, 73.4 and 81.1% HOCs, but only 24.6, 19.1 and 8.9% COD, respectively. Through the specific HOCs accumulation characteristics of BFC, about 11.2% HOCs with low molecular weight (<1000 Da) in the biologically treated leachate were concluded. Since HOCs are competitively trapped by dissolved organic matters (DOM), the ultimate removal of HOCs from leachate is unreachable by activated carbon or BFC. It was also found that the biologically treated leachate effluent exhibited a wide molecular weight distribution (34–514,646 Da). These constitutes are derived from both autochthonous and allochthonous matters as well as biological activities.

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1. Introduction

Leachate, a highly contaminated and complex wastewater, is a by-product during the treatment or storage process of refuse. At the same time, leachate will become more complicated when landfill receives municipal, commercial and industrial wastes [1]. The main concern about landfill leachate is its impact of the surface and ground water [2–4].

Alternative methods such as coagulation, flocculation, upflow anaerobic sludge blanket (UASB) and sequence biological reactor (SBR) have been used for landfill leachate treatment, furthermore, advanced technologies such as membrane technology [5,6], activated carbon adsorption [7], advanced oxidation [8] and electrochemical oxidation [9,10] were also tried, but a efficient and economical method has not been found [11]. In brief, currently leachate treatment requires combined techniques, and a biological pretreatment is inevitable.

Chemical oxygen demand (COD) and biochemical oxygen demand (BOD) of fresh leachate could be biologically reduced to around 300–800 and 200–300 mg L⁻¹, but further removal of the rest called “refractory organic matters” which was considered as humic or humic-like material derived from the process of humic process [12–15] is difficult. Reverse osmosis (RO) technology shows its advantage in removing this refractory organic matters [5], whereas high cost and membrane fouling hinder its using, and the high concentration retained matter is also a problem.

Among this refractory organic matter, hydrophobic organic chemicals (HOCs) with chemical and biological stability [16,17], including some EPA organic priority pollutants, constitutes a potential risk to the quality of receiving water body such as surface water and groundwater, as well as accumulated in aquatic and soil food chains and further threaten ecology [18,19]. During the last decades, more and more HOCs have been detected at the all decomposition stages in landfill and leachate [2,3,20–22].

In this work, we employed powder-activated carbon (PAC), granular-activated fat carbon (GAC) and a novel agent biomimetic fat cell (BFC) to remove HOCs from a leachate which had been biologically treated by a three-stage-aged refuse bioreactor (ARB). On the other hand, primary evaluation of biologically treated landfill leachate and related HOCs were also conducted.

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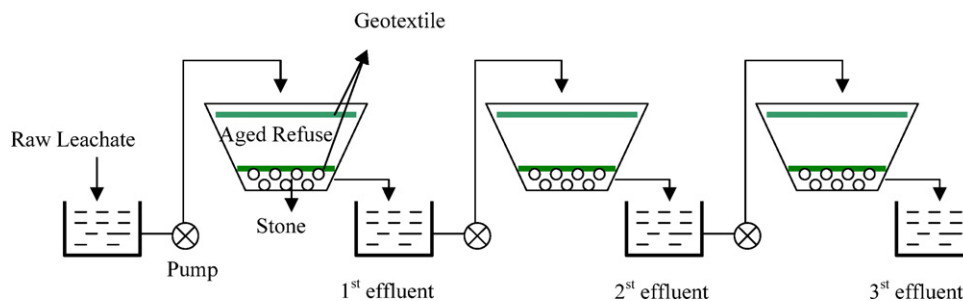


Fig. 1. Sketch of three-stage-aged refused bioreactor for raw leachate treatment.

Due to efficient and specific HOCs removal, BFC was selected. Earlier research indicated that BFC had 97.4% lindane (7 mg L^{-1}) removal close to 98.1% lindane removal by PAC in aqueous solution [23,24]. In addition, BFC can be regenerated easily using organic solvent (cyclohexane) dialysis, compared to high temperature/pressure used for activated carbon regeneration to overcome the stable bond between adsorbed chemicals and activated carbon.

2. Materials and methods

2.1. Materials

Raw leachate was produced at Shanghai Laogang Landfill, the largest landfill in China, which was put into operation at the end of 1989. After a short time storage at the hold tank and anaerobic lagoon, the leachate was transferred to a three-stage-aged refused bioreactor (ARB) (Fig. 1) with 8 year-aged refuse [4,25]. In one year's running and monitoring, the inlet COD concentration varied from 6000 to 11,000 mg L^{-1} and then reduced to 1300–3200 mg L^{-1} in 1st stage, to 680–2000 mg L^{-1} in 2nd stage and to 270–950 mg L^{-1} in 3rd stage. Influent and effluents were sampled per week and EPA standards methods were used to determine the concentrations of COD, BOD and $\text{NH}_3\text{-N}$. The quality of used leachate could be found at Table 1.

Based on the bioaccumulation characteristics of fat tissue [16,17,26,27], a novel agent, biomimetic fat cell (BFC) were synthesized employing with interfacial polymerization and described briefly here [24]: the white solid BFC (mean particle diameters: $4.968 \mu\text{m}$; median particle diameters: $3.41 \mu\text{m}$; specific area: $30.2755 \text{ m}^2 \text{ g}^{-1}$) was prepared at 0°C reacting temperature and 1500 rpm stirring rate. Organic phase includes 1,3,5-benzenetricarboxyl trichloride and terephthaloyldichloride, and water phase is piperazine. When the organic phase in a 250 mL separatory funnel was poured into the water phase in a 1-L beaker successively, BFC formed immediately and suspended in water. Decompress filtration was used to separate BFC. With three times distilled water washed, BFC dried at room temperature and stored in a desiccator for further use.

Fine PAC (300 mole sieve) and GAC (40 mole sieve) were obtained from Liyang Carbon Company, China. Analytical grade methylene chloride for HOCs extraction and potassium dichro-

mate for COD determination were purchased from Shanghai Yunjie Chemical Company (China).

2.2. Methods

2.2.1. Leachate-advanced treatment experiments

Typical jar-test procedure was employed for leachate-advanced treatment experiments [28], using 2.0 L beakers and magnetic stirring apparatus under room temperature. Accurately weighted PAC, GAC and BFC doses (0.5, 1.0, 2.0 and 3.0 g) were added into 1 L leachate with optimized 100 rpm stirring rate to keep agents in suspension. PAC and GAC used were boiled in 100°C hot water for 20 min to remove impurity, and cooled by adding distilled water prior to use [29]. To obtain better treatment, the reaction time was set for 48 h.

2.2.2. HOCs extraction and detection

HOCs Extraction was performed with SUPELCO C18 solid-phase extraction column operated on a SUPELCO SPE instrument (SUPELCO, USA). Before using, 5 mL methanol followed by 20 mL distilled water was used to activate column. 400 mL 0.45 μm filter filtrated biologically treated leachate was allowed to flow through a SPE column with optimized 4 mL min^{-1} flow rate, then washed with 20 mL distilled water, and finally 5 mL methylene chloride was used to elute SPE column. The extract was almost dried with gently blowing high purify nitrogen (>99.999%) and then quantified to 1 mL. $1 \mu\text{L}$ extracted was used for GC-MS determination.

HOCs determination was carried out by Trace DSQ GC-MS (Thermal, USA) with a DB 5 capillary column (30 m length, 0.25 mm diameter, and 0.25 μm film thicknesses). The oven temperature was programmed to hold at 50°C for 4 min, then increase to 280°C at $15^\circ\text{C min}^{-1}$, and finally hold constant at 280°C for 30 min. Helium was used as the carrier gas at the constant flow of 1 mL min^{-1} . Effluents from GC column were transferred to a 70-eV electron impact source held at 250°C . Full scan model was used with 500 amu s^{-1} speeds and 50–600 μm broad range.

2.2.3. Molecular weight determinations

Twenty microliters 0.45 μm filter filtrated aliquots were used for molecular weight analysis by Lc-10ADVP Gel permeation chromatography (GPC) with RID-10A detector (SHIMADZU, Japan). The column was TSK G4000PWxl columns (SHIMADZU, Japan) and an optimal operation temperature was 40°C . The mobile phase was DDI water with 0.5 mL min^{-1} flow rate. Polystyrene Glycol/Oxide (Polymer Laboratories, England) with mean Mw 106, 1470, 25,820 and 460,000 Da were dissolved into DDI water as standard.

Number (M_n) and weight-average (M_w) molecular weight were defined using the following equations:

$$\bar{M}_n = \frac{\sum N_x M_x}{\sum N_x} \quad (1)$$

Table 1

COD, BOD and $\text{NH}_3\text{-N}$ concentrations of leachate

	BOD (mg L^{-1})	COD (mg L^{-1})	$\text{NH}_3\text{-N}$ (mg L^{-1})
Inlet leachate	1368	8603	861
1st ARB stage outlet	307	2016	391
2nd ARB stage outlet	97	983	131
3rd ARB stage outlet	30	451	27

Table 2
Removal of HOCs and COD in leachate with further treated by agents

Dosage (g L ⁻¹)	Samples	Relative mass abundance	HOC ₅ removal ratio (%)	COD removal ratio (%)
0.5	Raw leachate	7.52 E ⁷	0	0
	Treated by PAC	2.38 E ⁷	68.4	16.6
	Treated by BFC	2.53 E ⁷	66.4	6.2
	Treated by GAC	3.98 E ⁷	47.1	11.1
1.0	Raw leachate	7.01 E ⁷	0	0
	Treated by PAC	1.76 E ⁷	74.9	18.6
	Treated by BFC	2.13 E ⁷	69.6	7.1
	Treated by GAC	3.02 E ⁷	56.9	14.2
2.0	Raw leachate	6.58 E ⁷	0	0
	Treated by PAC	7.39 E ⁶	88.8	22.3
	Treated by BFC	1.19 E ⁷	81.9	9.2
	Treated by GAC	1.96 E ⁷	70.2	18.3
3.0	Raw leachate	6.72 E ⁷	0	0
	Treated by PAC	7.24 E ⁶	89.2	24.6
	Treated by BFC	1.27 E ⁷	81.1	8.9
	Treated by GAC	1.79 E ⁷	73.4	19.1

$$\bar{M}_w = \frac{\sum C_x M_x}{\sum C_x} = \frac{\sum N_x M_x^2}{\sum N_x M_x} \quad (2)$$

2.2.4. COD determination

COD was determined by a standard dichromate reflux method. Potassium dichromate was added into sample solutions after the samples had been digested with sulphuric acid, and then the samples were titrated with ferrous ammonium sulfate with ferroin as the indicator [4].

3. Results and discussion

3.1. Leachate biological treatment

COD, BOD and NH₃-N concentration are very high in raw leachate, variable with seasons, climate, operational modes used, and location of the landfill. In this research, the concentrations of COD, BOD and NH₃-N in raw leachate were 8603, 1368 and 861 mg L⁻¹, respectively.

After treatment by three-stage ARB, 94.8% COD, 97.8% BOD and 96.9% NH₃-N were removed, and the concentrations of COD, BOD and NH₃-N in effluent were 451, 30 and 27 mg L⁻¹, respectively. The mechanism of ARB mainly depends on the microbial degradation, ion exchange and sorption [4,25], and some humic substances in leachate interaction with the humic substances of aged refuse when the leachate flowed through the ARB was also proposed [25]. The low BOD value, 30 mg L⁻¹, indicates that the leachate barely contains the biodegradable organic matter.

3.2. Leachate-advanced treatment

After 48 h treatment, 0.5 g L⁻¹ PAC, GAC and BFC the removed 16.6, 11.1 and 6.2% COD, respectively (Table 2). With the increasing dosage from 0.5 to 3.0 g L⁻¹, the COD removal slowly increased, 8.0% for PAC, 18.0% for GAC and 2.7% for BFC.

Activated carbon removes not only HOCs, but also natural organic matter (NOM) and humic substances (HS) [30,31]. However, BFC only removes HOCs because of its specific hydrophobic chemicals bioaccumulation characteristic [23,24]. Hereby, the lower COD removal by BFC than that of PAC and GAC can be drawn.

The low COD removal by PAC, GAC and BFC interpreted that this part organic matter which mainly contributes the COD of biological

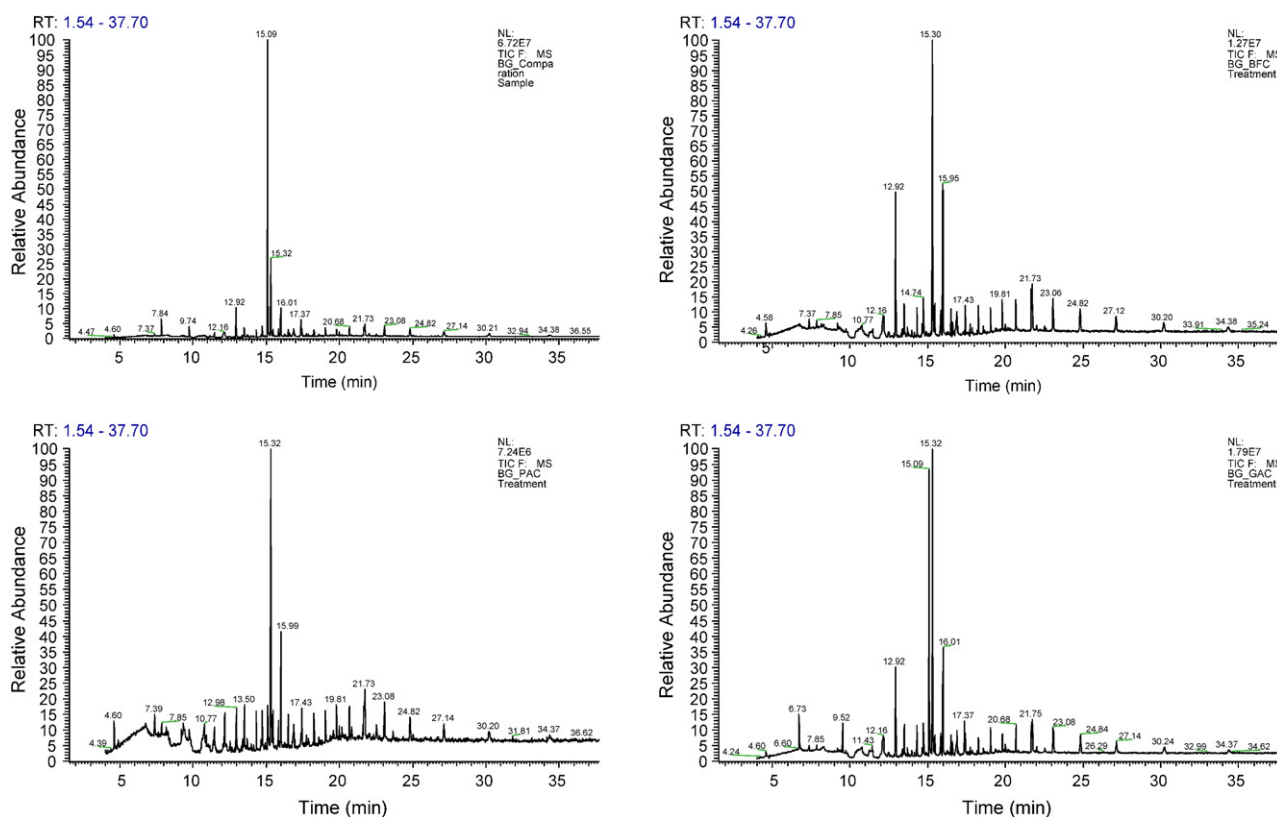


Fig. 2. Relative mass abundance of biologically treated and further treated leachate. Upper (left: biologically treated leachate; right: further treated by 3.0 g L⁻¹ BFC). Lower (left: further treated by 3.0 g L⁻¹ GAC; right: further treated by 3.0 g L⁻¹ PAC).

Table 3
Selected chemicals in leachate effluent

Name	Raw leachate	Leachate effluent treated by PAC	Leachate effluent treated by BFC	Leachate effluent treated by GAC
1,2-Benzenedicarboxylic acid dinonyl ester	+	–	–	+
Bis(2-ethylhexyl)phthalate	+	+	+	+
Phenol, <i>p-tert</i> -butyl	+	–	–	+
2,6-Bis(1,1-dimethylethyl) phenol	+	+	–	–
Diethylstilbestrol	+	+	–	–
Cholesterol	+	+	+	+
Hexadecane	+	+	+	+
1-Tetradecanol	+	+	+	+

+: positive; –: negative.

treated leachate is refractory, similar with the result of Rodríguez who used GAC to treat leachate which was recirculated at landfill units [32].

3.3. HOCs removal

Since versatile heterogeneous chemicals mixed in landfill leachate and developed during landfill decomposition process, accurate determination of chemicals in landfill leachate by GC–MS could not be achieved, but the total mass abundance (TMA) of GC–MS which account for the mass ratio of detected chemicals could be used to evaluate the contents of total chemicals through a parallel comparison (Fig. 2).

As mentioned above that BFC specifically accumulates hydrophobic chemicals, here we proposed that the retained organic chemicals by BFC were HOCs. When the BFC doses increased from 0.5 to 3.0 g L⁻¹, HOCs removal increased to 81.1% from 64.4%, and the same trend was also observed at the using of PAC and GAC (Table 2). A close HOCs removal by 2.0 and 3.0 g L⁻¹ BFC showed that 2.0 g L⁻¹ BFC was efficient for HOCs removal.

Selected chemicals in ARB biologically treated leachate screened from GC–MS NISTDEMD chemical data bank were presented in Table 3, including some persistent organic pollutants (POPs) such as 1,2-benzenedicarboxylic acid dinonyl ester,

bis(2-ethylhexyl)phthalate and some normal chemicals such as hexadecane and 1-tetradecanol. Compared to the removal of selected chemicals (Table 3), BFC exhibited better removal than that of PAC and GAC, but some HOCs were still detected after treatment. The limited HOCs removal is possible because that a fraction of HOCs were trapped by DOM.

3.4. Molecular weight distribution of leachate

Molecular weight distribution of landfill leachate after ARB treatment demonstrated a wide distribution, ranging from 34 to 514,646 Da. Being an acceptor of the autochthonous and

Table 4
Average molecular weight, polydispersion and total molecular weight removal

	\bar{M}_n	\bar{M}_w	Total MW removal ratio (%)	<1000 Da MW removal ratio (%)
Leachate	376	2,937	0	0
PAC	476	8,317	12.8	11.2
BFC	484	10,723	7.7	7.2
GAC	557	12,998	10.5	9.4
Vejen landfill [36]	5,200	5,800		
Vejen landfill [37]	2,900	3,900		

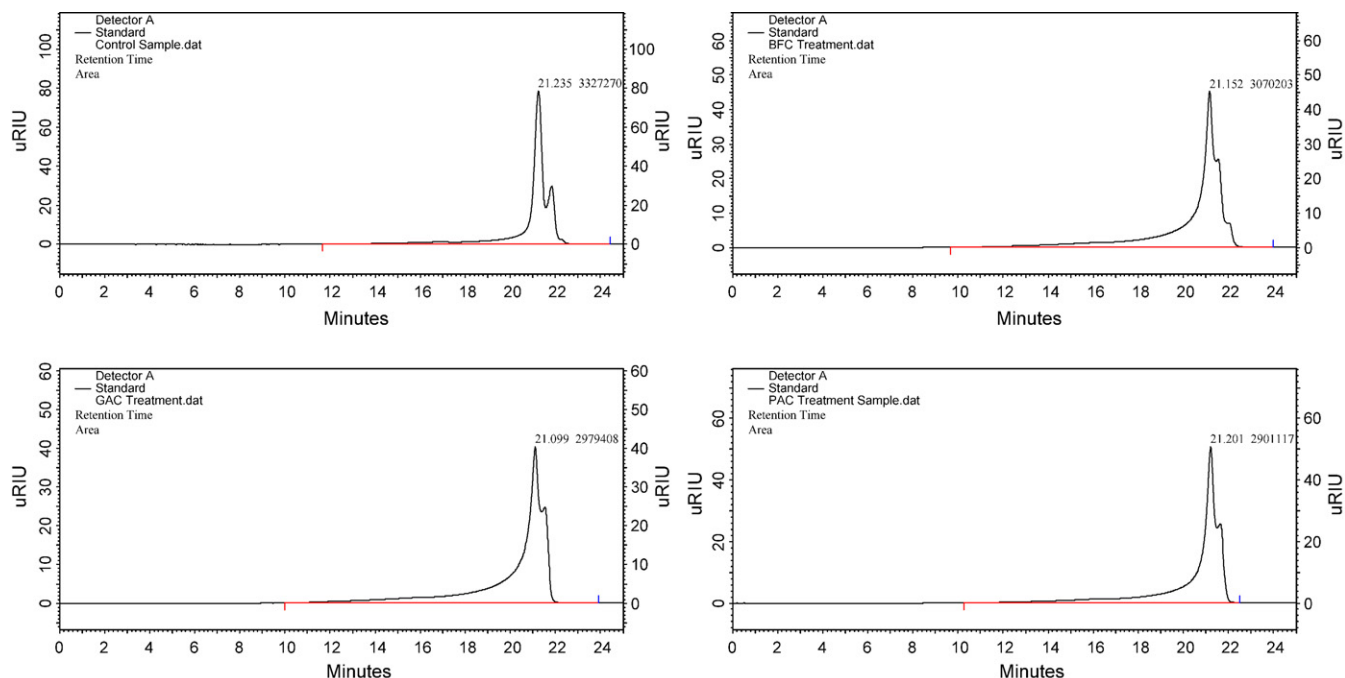


Fig. 3. GPC chromatography of biologically treated and further treated leachate. Upper (left: biologically treated leachate; right: further treated by 3.0 g L⁻¹ BFC). Lower (left: further treated by 3.0 g L⁻¹ GAC; right: further treated by 3.0 g L⁻¹ PAC).

allochthonous organic matters, the wide molecular weight distribution of landfill leachate could be accepted.

After further treatment by BFC, PAC and GAC, the \bar{M}_n and \bar{M}_w of leachate both increased, indicating that the percentage of macromolecular organic matter increased after much more low molecular weight organic matter was removed (Table 4).

During leachate decomposition process (an initial aerobic phase, an anaerobic acid phase, an initial methanogenic phase, a stable methanogenic phase and an additional aerobic or humic phase) [1,33,34], labile macromolecular decompose to refractory micro molecular and the molecular weight distribution will changed correspondingly. A molecular weight distribution of chemicals in a quite old age leachate plum (Vejen landfill run from 1961 to 1982, Denmark) was investigated [35], and DOM seemed to become more similar to fulvic acids present in uncontaminated groundwater [36] with respect to molecular weight along a 0–300 m gradient (Table 4). The reason is that the leachate naturally attenuates in a slow but steady manner and it can ultimately reach the regulated discharge standards and even surface water quality standards [4], whereas decades or even hundreds of years for such complete leachate stabilization is needed, depending on the refuse composition and moisture and on local climate and geological conditions [37]. As to leachate natural attenuation, 48 h activated carbon and BFC treatment could be neglected in this research. Then the change of the molecular weight distribution and polydispersion only depended on the removal of organic matters.

Biologically treated leachate and further treated leachate eluted from the GPC column showed as a broad, double-modal distribution with subtle shoulder and subpeaks.

Comparison of the peak between the biologically treated and further advanced treated leachate showed that the major removed chemicals had a longer retained time (RT: 21.2–22.2 min) which account for 100–400 Da fractions (Fig. 3). If GPC peak area stands for the distribution of total chemicals, the lost peak area could be calculated as the removed fractions. Hereby the total removed chemicals in further treated leachate by BFC, GAC and PAC were 7.7, 10.5 and 12.8%, close to the 7.2, 9.4 and 11.2% removal of the chemicals with <1000 Da molecular weights, respectively (Table 4). This result interpreted that the mainly removed fractions by activated carbon and BFC are the chemicals with low molecular weight (<1000 Da).

3.5. Primary evaluation of HOCs in biologically treated leachate

With a specific HOCs removal by BFC, combined the lost of COD and mass abundance of GC–MS, the content of HOCs in biologically treated leachate could be conducted roughly as:

$$\text{HOCs} = \frac{\text{COD}_{\text{lost}}}{\text{Mass}_{\text{lost}}} \times 100\% \quad (3)$$

$$\text{HOCs content} = \frac{9.2}{0.819} \times 100\% = 11.2\%$$

4. Conclusions

Landfill leachate treatment needs integrated technology, usually with a biological pretreatment which efficiently reduce COD and BOD to a relative low concentration, whereas the further refractory HOCs removal is hard. In this study, PAC, GAC and BFC were employed to remove HOCs from a leachate which had been biologically treated by a three-stages ARB.

In summary (1) 94.8% COD, 97.8% BOD and 96.9% NH₃-N in raw leachate were removed by three-stages ARB at 8603, 1368 and

861 mg L⁻¹ initial concentration, respectively. (2) In further treatment, 3 g L⁻¹ PAC, GAC and BFC removed 89.2, 73.4 and 81.1% HOCs, but only 24.6% 19.1 and 8.9% COD at 451 mg L⁻¹ initial COD concentration, respectively. (3) Biologically treated leachate mainly contains refractory organic matter which includes 11.2% HOCs with low molecular weight (< 1000 Da). (4) HOCs could not be ultimately removed by PAC, GAC and BFC because of the competitive sorption by DOM.

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