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Determination of benzothiazoles and benzotriazoles by using ionic liquid stationary phases in gas chromatography mass spectrometry. Application to their characterization in wastewaters

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ABSTRACT

Benzothiazoles (BTHs) and benzotriazoles (BTRs) belong to a high production volume chemicals widely used in both industrial and household applications. Since they are recalcitrant to biodegradation, they are widespread in the environment. However, the BTHs and BTRs determination in environmental matrices is hindered by the coelution with coextracted organic matter and the poor selectivity in mass spectrometry due to the low mass of their diagnostic ions. Accordingly, this study examines the selectivity and suitability of new commercially available ionic liquid (IL) stationary phases for GC–MS and their application to the determination of BTHs and BTRs in wastewater samples. Five different IL columns were tested and the best results in terms of resolution, peak symmetry and analysis time were obtained with the SLB-IL59.

1. Introduction

Benzothiazoles (BTHs) and benzotriazoles (BTRs) belong to high production volume chemicals that find broad application in various industrial processes and household products [1]. BTHs are used as biocides in paper and leather manufacturing, as vulcanization accelerator in rubber production [2] and as corrosion inhibitors [3]. BTRs are commonly used as a corrosion inhibitor in dishwasher detergents and de-icing/anti-icing fluids, an ultraviolet light stabilizer in plastics, and an antifogging agent in photography [4].

BTHs and BTRs are water soluble, resistant to biodegradation and only partially removed in wastewater treatment [5,6]. In fact, BTHs and BTRs are typical examples of polar and poorly degradable trace organic pollutants. Owing to their widespread application, several BTHs and BTRs are widely distributed in wastewater both industrial and domestic [7–10] and surface water (i.e. river and lake) [8,11–13] at concentration levels from 0.1 to 6 μg/L. BTRs have been classified as emergent pollutants [14] because even at low concentrations, they elicit negative effects to aquatic organisms [13,15].

The BTHs and BTRs determination by gas chromatography coupled to mass spectrometry (GC–MS) in the electron impact (EI) from complex matrices suffers from interferences, even in the selective ion monitoring mode because of the low mass of the diagnostic ions. On the other hand, liquid chromatography coupled to mass spectrometry (LC–MS) [6,16] or mass spectrometry in tandem (LC–MS/MS) [7,11] is usually used for their determination in environmental matrices. However, these techniques suffer from some disadvantages like the ionic suppression or enhancement in the electrospray ion sources due to the charge competition with organic matter coextracted [17]. Recently, Jover et al. [8] described the application of comprehensive two-dimensional GC coupled to time-of-flight mass spectrometry (GC×GC-TOF MS) technique as an alternative method for the determination of these compounds in aqueous matrices taking advantage of the highest resolution of two dimensional GC separation.

Over the last decade, the interest of ionic liquids (ILs) increased enormously in different areas of analytical chemistry [18,19] because their unique physicochemical properties and are considered as environmental friendly solvents [20–22]. In this regard, IL as GC stationary phases have been introduced [23] and different IL stationary phases have been previously used to separate mixtures of a series of fatty acid methyl esters [24], polycyclic aromatic hydrocarbons and chlorinated pesticides [25], essential oils [26] and flavors and fragrances [27].

Room-temperature ionic liquids (RTILs), also known as organic liquids, molten or fused salts, are a class of nonmolecular ionic solvents with low melting points [28]. However, as ILs, it is often applied to any compound that has a melting point <100 °C [19]. Most common RTIL are composed of asymmetrically substituted

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organic cations containing nitrogen (e.g. imidazole, pyrrolidine, pyridine) with inorganic anions (e.g. Cl^-, PF^{6-}, BF_4^-).

The interest of using IL as GC stationary phases arise from its desirable properties. In fact, the unique properties of IL including wide liquid phase range, low volatility (negligible vapour pressure), high viscosity, good thermal stability and variable polarities, make them ideal for that purpose [29]. Moreover, ILs display unusual dual-nature retention behavior, separating both nonpolar and polar compounds [30]. IL-based column performed as a nonpolar column when retaining relatively nonpolar analytes. However, they behaved significantly different when retaining highly polar and proton-donor analytes [29]. One important feature of ILs is that varying the cation or anion might significantly affect their physical and chemical properties. Therefore, in order to obtain columns with unique selectivity, many modifications are possible (i.e. anion or cation or pendant groups to anion and cation) [19]. Moreover, IL stationary phase offer greater thermal stability compared to cyanopropyl substituted polysiloxanes or polyethylene glycols with lower column bleed. They exhibit a lower background in MS applications [31] and do not generate the typical ions obtained with polar conventional stationary phase degradation.

To the best of our knowledge, there are not many studies using IL as stationary phases in environmental applications. In this study, commercially available polar IL stationary phases with different polarity and chemical properties were evaluated as GC stationary phases for the GC–MS determination of BTHs and BTRs. Five IL columns were evaluated in order to obtain the best separation between the various target analytes as well as the highest peak symmetry. The optimum stationary phase was used for the detection and quantification of BTHs and BTRs by GC–MS. In this work, the application of IL stationary phases for the determination of polar analytes occurring in complex environmental matrices such wastewater is reported for the first time.

2. Materials and methods

2.1. Chemicals

GC grade (Suprasolv) hexane, methanol, acetone, and ethyl acetate were obtained from Merck (Darmstadt, Germany). Benzothiazole (BT), 2-hydroxybenzothiazole (OHBT), 2-(methylthio) benzothiazole (MTBT), 1*H*-benzotriazole (BTri), 5-methyl-1*H*-benzotriazole (5-TTri), 2-mercaptobenzothiazole (MBT), 5,6-dimethylbenzotriazole (XTri) (purity of ≥96% for all analytes) and triphenylamine (TPhA) analytical grade were purchased from Sigma–Aldrich (Steinheim, Germany). Strata-X polymeric solid-phase extraction (SPE) cartridges (100 mg/6 mL) were obtained from Phenomenex (Torrance, CA, USA) and the 0.7 μm glass fiber filters with a diameter of 47 mm were purchased from Millipore (Bedford, MA, USA).

2.2. Standard solutions

Individual stock solutions of all standards were prepared at a concentration of 3 mg/mL in ethyl acetate. A mix solution, which contained all standards at a concentration of 300 μg/mL was prepared in ethyl acetate by dilution from the individual stock solution. Working standard solutions were prepared by serial dilution with ethyl acetate to reach a concentration range of 0.031–3 μg/mL. All working standard solutions were stored at −20 °C and where stable at least for three months.

2.3. Optimization of the GC–MS conditions

The GC–MS method optimization was carried out with BTHs and BTRs mix solution prepared in ethyl acetate (Table 1). Their determination was performed in a Trace GC–MS (Thermo Scientific, Dreieich, Germany) in the electron impact mode (70 eV ionization energy). Identifier and qualifier ions of BTHs and BTRs are shown in Table 1. Five commercially available ionic liquid columns of 30 m × 0.25 mm ID coated with 0.20 μm film thickness of different IL stationary phases (i.e. SLB-IL59, SLB-IL61, SLB-IL76, SLB-IL82 and SLB-IL111) were obtained from Supelco (Bellefonte, PA, USA). According to the supplementary information provided by Supelco I+D department, these stationary phases show different composition and polarity. However, only the chemical composition for SLB-IL59 is disclosed (i.e. 1,12-diamide (tripropylphosphonium) dodecane bis (trifluoromethansulphonyl) amide) [32]. A SUPELCOWAX-10 (30 m × 0.25 mm ID, 0.25 μm film thickness) also from Supelco, was selected as a reference polar column. The final temperatures were selected according to the recommended upper temperature limit (UTL) at isothermal conditions and the temperature programming rates of different evaluated columns are shown in Table 2.

A sample volume of 2 μL was injected in the splitless mode at an injector temperature of 270 °C and the purge valve activated 50 s after the injection. Helium was used as a carrier gas (99.9995% purity) at a flow rate of 1 mL/min. The transfer line and ion source were set at 280 °C and 200 °C respectively. Acquisition was performed in the full-scan mode ranging from m/z 50 to 500 amu at 2 scans/s with 6 min of solvent delay. Acquired data were processed by using the Themo Scientific X-calibur software.

The linearity range was from 0.031 to 3.0 μg/mL. The correlation coefficients (r^2) of the calibration curves were always higher than 0.993. The limit of detection (LOD) and limit of quantification (LOQ) were compound dependent in the range from 0.025 to 0.562 μg/L and from 0.060 to 0.815 μg/L respectively. These parameters were calculated as three times the standard deviation of a procedural blank, for the LOD and ten times for the standard deviation for the LOQ. Quantification and other quality control parameters have been reported elsewhere [8].

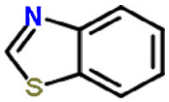
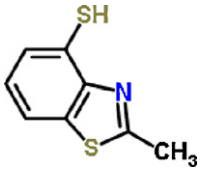
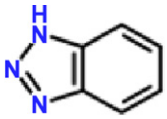
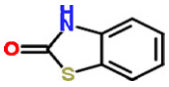
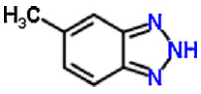
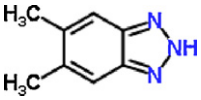
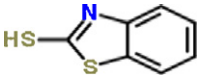
2.4. Sample extraction

The BTHs and BTRs in wastewater samples were analyzed following a filtration through a Whatman glass fiber filter with a pore size of 0.7 μm and processed as reported previously by Matamoros and et al. [33] (Fig. 1). This analytical methodology has been successfully developed for BTHs and BTRs determination in complex aqueous matrices [8] exhibiting recoveries in the range from 69 to 80% for BTHs and from 78 to 98% for BTRs in wastewater. Moreover, RSD obtained in water samples were lower than 10% for all analytes (n = 3). Briefly, a sample volume of 200 mL was acidified to pH = 3 with hydrochloric acid (HCl) and percolated through an activated polymeric solid-phase extraction cartridge (100 mg Strata X) from Phenomenex (Torrance, CA, USA). Loaded cartridges were eluted with 10 mL of hexane/ethyl acetate (1:1). The obtained extract was evaporated to ca. 20 μL under a gentle nitrogen stream and 186 ng of triphenylamine were added as internal standard. The vial was then reconstituted with ethyl acetate to a final volume of 300 μL. Final determination was performed on a TRACE GC–MS (Thermo Scientific, Dreieich, Germany).

2.5. Application to wastewater samples

The developed GC–MS methodology was applied to wastewater samples from a pilot wastewater treatment plant (Silvouta, NW, Spain). This treatment system consists of an upflow anaerobic sludge blanket (UASB) reactor and two constructed wetlands (CWs) operated in series. The first CW was a surface flow (SFCW) and the second a horizontal subsuperficial flow (SSFCW). Following sand and grease removal, the anaerobic reactor was fed with raw

Table 1
Chemical structures, acronyms, CAS number, physical properties and diagnostic ions used in GC–MS.

Analyte	Acronym	Structure	CAS number	Polarizability[a] (cm^3/m)	Boiling point[a] (°C)	Diagnostic ions		
						Quantifier ion	Qualifier ion 1	Qualifier ion 2
Benzothiazole	BT		95-16-9	9.44	231	135	108	69
2-Methyl mercapto benzothiazole	MTBT		615-22-5	12.28	302	181	148	108
1-Benzotriazole	BTri		95-14-7	7.58	276	119	91	64
2-Hydroxy benzothiazole	OHBT		934-34-9	9.84	360	151	96	123
5-Methyl benzotriazole	5TTri		136-85-6	8.70	289	104	133	78
5,6-Dimethyl benzotriazole	XTri		4184-79-6	9.84	309	118	147	91
2-Mercapto benzothiazole	MBT		149-30-4	11.10	305	167	109	108

[a] Estimated by SPARC v.4.5.

municipal wastewater. A fraction of the UASB effluent (17–20 m^3/d) was diverted to an SFCW followed by a horizontal SSF CW. Both CWs (75 m^2 each) were planted with bulrushes (*Juncus effuses*) [34].

2.6. Statistical analysis

The correlation coefficients between the polarizability or boiling point and retention time for every analyte and column were calculated using parametric statistics (i.e. Pearson coefficient) with the SPSS v.15 package (Chicago, IL, USA).

3. Results and discussion

3.1. GC–MS optimization

The evaluated IL columns exhibit different polarities as indicated by the polarity index derived from McReynolds constants (normalized to SLB IL 100, Table 2). Despite the chemical composition of these IL stationary phases is not known, different selectivities are expected because their polarity range is quite broad (Polarity Index span 52 units). Moreover, a wax column with a polarity index of 52 was used as reference column.

The criteria parameters considered in the GC–MS methodology optimization were to obtain the highest resolution between the different target analytes, a suitable peak symmetry and the shortest analysis time. For this purpose, all columns were evaluated with a standard mix solution containing all the target analytes and the asymmetry factor (AF) [35] was determined with the following equation:

$$AF = \frac{b}{a} \quad (1)$$

where b is the distance from the center line of the peak to the back slope and a the distance from the center line of the peak to the front

Table 2
GC–MS analytical conditions.

Stationary phase and column polarity	UTL[c] (°C)	Initial (°C)	Hold time (min)	Ramp 1 (°C/min)	Temperature 1 (°C)	Hold time (min)	Ramp 2 (°C/min)	Temperature 2 (°C)	Hold time (min)	Ramp 3 (°C/min)	Temperature 3 (°C)	Hold time (min)
SLB – IL59[a]	300	65	2	10	150	–	5	290	10	–	–	–
SLB – IL61[a]	270	65	2	12	150	–	10	250	10	–	–	–
SLB – IL76[a]	270	65	2	14	150	–	5	250	5	–	–	–
SLB – IL 82[a]	270	65	2	10	150	–	5	250	10	–	–	–
SLB – IL111[a]	270	65	2	10	150	–	5	250	10	–	–	–
SUPELCOWAX-10[b]	280	65	2	12	150	–	10	220	–	5	270	10

[a] Column dimensions: 30 m × 0.25 mm × 0.20 μm. The stationary phase polarity index is given in the column code.
[b] Column dimensions: 30 m × 0.25 mm × 0.25 μm. The stationary phase polarity index is given in the column code.
[c] UTL: upper temperature limit.

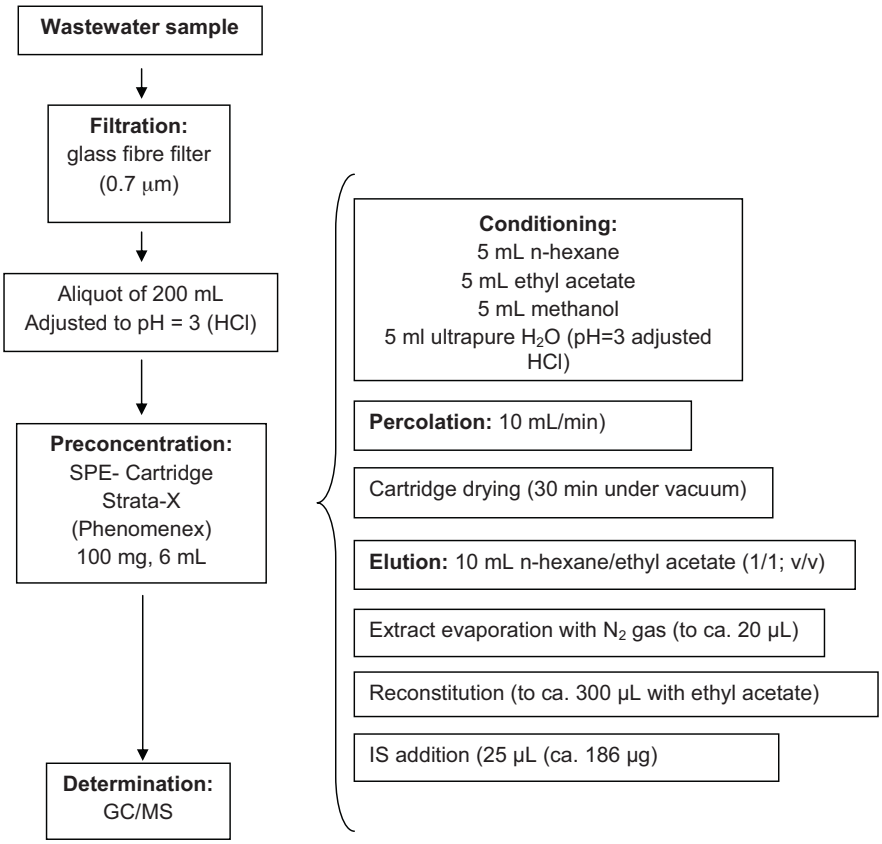


Fig. 1. Schematic diagram of the analytical procedure used for determination of BTHs and BTRs in wastewater.

slope with all measurements made at 10% of the maximum peak height.

The temperature programming was selected according with the UTL in isothermal conditions of each column according with the supplier (Table 2). From the TIC of the standard mix solution in the GC–MS determination, it was observed that all IL columns showed lower bleeding rates than the wax column selected, allowing an easier identification of the target analytes.

For the IL evaluated columns, the analyte retention behavior can be ascribed not only to the boiling point but also to the stationary phase interactions like dipolar (i.e. dipole–dipole or dipole-induced dipole), acid–base or hydrogen bonding. Accordingly, the linear correlation between analyte polarizability or boiling point versus analyte retention time was examined for the different evaluated columns (Table 2). However, no positive correlation was observed between these parameters for all evaluated analytes in the different columns ($p > 0.05$).

The elution profile normalized to BT retention time was determined (Table 3). This parameter was dependant on the evaluated column and slight variations were observed. However, only in the SLB-IL59 (UTL = 300 °C), all the target analytes eluted in the analytical conditions used and their retention times were lower than 26 min. Moreover, an appropriate resolution (>1.5) for all analytes to achieve a complete baseline separation was obtained [36]. Despite some tailing was obtained for several target analytes, particularly those eluting at higher retention time, this column provided the best peak symmetry (Table 3) with an AF value lower than 2.5, which is the maximum AF allowed for a precise quantitative analysis [37]. For the rest of IL columns, a partial elution of analytes was obtained. For SLB-IL76 (UTL = 270 °C; highly polar) only BT, MTBT and BTri eluted in the chromatographic conditions used. It could be attributed either to the lower UTL or a higher interaction between these analytes and the stationary phase. For SLB-IL61, SLB-IL 82 and SLB-IL111 (UTL = 270 °C; extremely polar) columns, although they exhibit the same UTL than SLB-IL76, a higher number of analytes were eluted indicating a lower interaction of these analytes with the stationary phases evaluated.

Table 3
Relative retention time to BT analyte and peak symmetry of different target compounds analyzed.

Analyte	Relative retention time						Asymmetry factor					
	IL-59	IL-61	IL-76	IL-82	IL-111	WAX	IL-59	IL-61	IL-76	IL-82	IL-111	WAX
BT	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.09	1.00	1.00
MTBT	1.33	1.33	1.48	1.30	1.36	1.33	1.00	1.25	1.14	1.11	1.00	1.06
BTri	2.13	2.38	3.18	2.17	2.68	2.01	1.34	6.00	1.29	3.00	1.42	2.20
OHBT	2.18	2.37	–	2.20	2.70	2.19	1.25	1.50	–	1.40	1.29	1.50
5TTri	2.25	2.49	–	2.30	2.78	2.11	1.33	6.36	–	3.25	1.42	2.60
XTri	2.47	2.77	–	2.64	3.02	2.38	1.60	6.60	–	2.80	2.00	2.00
MBT	2.73	–	–	–	–	–	1.33	–	–	–	–	–

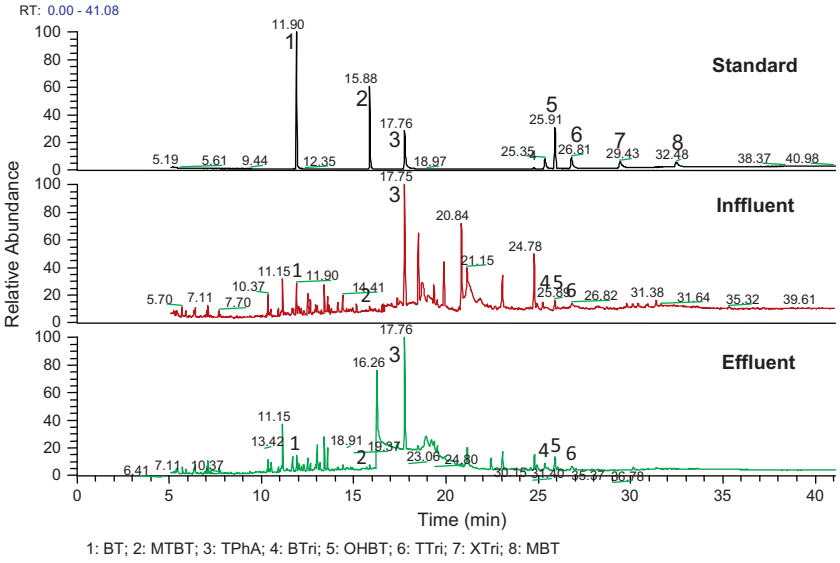


Fig. 2. Ion diagnostic GC–MS chromatogram (104 + 118 + 119 + 135 + 151 + 167 + 181 + 245) obtained for standard mix, influent and effluent.

Also, the resolution is used to express the degree, to which adjacent peaks (i.e. *A* and *B*) are separated. It was estimated according to the following expression:

$$R_s = \frac{2((t_R)_B - (t_R)_A)}{(w_b)_A + (w_b)_B} \quad (2)$$

where $(t_R)_x$ is the retention time and $(w_b)_x$ the peak width of the analyte *X*.

In these three evaluated columns, BTri and OHBT are poorly resolved but increasing R_s with the column polarity (i.e. $R_{SLB\text{-}IL61} = 0.78$, $R_{SLB\text{-}IL82} = 0.89$ and $R_{SLB\text{-}IL111} = 1.16$). Moreover, a variation in the elution order of these two analytes between these columns was observed showing a different selectivity.

On the other hand, the *AF* obtained for analytes, which eluted at higher retention time is greater, especially for the SLB-IL61. Indeed for this column, the *AF* exceed the reference value for 3 out 6 target analytes eluted, indicating that is not suitable for quantitative analysis [37]. These non-symmetrical peaks indicate that reversible adsorption takes place during the chromatographic process. Interestingly, the SLB-IL61 exhibits a similar polarity than SLB-IL 59 but its selectivity is clearly different. The SUPELCOWAX column (UTL = 280 °C; polar) was selected as reference of a conventional polar column. This column did not provide the complete elution of the target analytes in the run time. Moreover some differences in the elution profile were observed in front of IL evaluated columns and peak symmetry obtained for some analytes was poor (Table 3), with a worse *AF* that for the SLB-IL59 column.

3.2. Application to BTH and BTR determination in wastewater

Since the SLB-IL59 column provided the complete elution of the target BTHs and BTRs with an appropriate response and peak symmetry, this column was selected to analyze these pollutants in real wastewater samples. Accordingly, samples from the influent and effluent of a wastewater treatment (i.e. UASB reactor and of two CWs) were analyzed. The preconcentration technique selected has been proved to be suitable for the determination of these compounds in aqueous matrices [8].

Extracted ion chromatograms of a standard mix solution, influent and effluent samples are displayed in Fig. 2. Clearly, the response of the different analytes depends on the retention time and BTri and MBT are not detected in the samples analyzed. It is reported [38] that the thiol functional group is prone to oxidation to disulphide namely, dis-(2-benzothiazolyl) disulphide (MTBST). On the other hand, BTHs and BTRs were detected in all the samples analyzed, thus the method proved to be appropriate. Moreover, the EI MS spectra obtained for the target analytes present in real samples enabled their positive identification by comparison with the NIST spectra library. The identification and confirmation ions exhibited a low background noise, except for MTBT which concentration is near to the LOQ value.

Fig. 3 shows the BTHs and BTRs concentrations detected in wastewater samples through the different treatment steps from a pilot treatment plant. The concentrations obtained are similar to those reported in wastewaters from diverse origin [7–10]. The 5TTri showed the highest concentration in every treatment step and MTBT showed the lowest concentration, nearly LOQ. From the BTHs and BTRs analyzed, only 5TTri was moderately removed in the pilot wastewater treatment system studied showing that these contaminants are recalcitrant in the passive treatment systems evaluated.

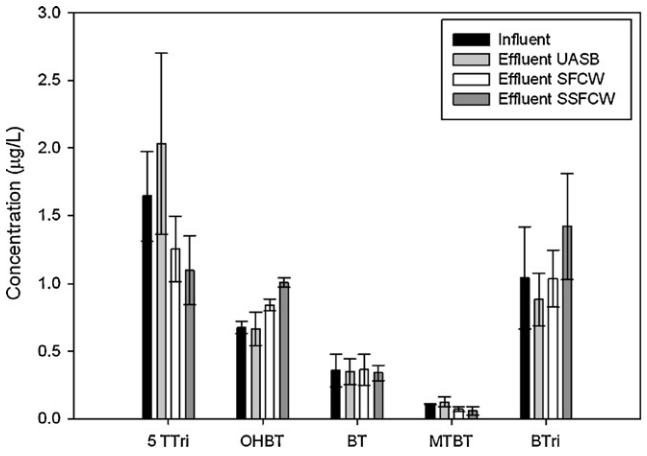


Fig. 3. Concentrations of 5TTri, OHBT, BT MTBM and BTri in the influent, effluent UASB, effluent SFCW and effluent SSFCW of the upper wet Mean concentration of BTri and BT in the wastewater treatment. The error bars indicate standard deviation of three sampling at independent days.

4. Conclusions

The BTHs and BTRs determination by GC–MS using conventional polar columns is a difficult task due to the high column bleeding and the low mass of the diagnostic ions, which coelute with coextracted organic matter. On the other hand, nonpolar stationary phases such as 5% diphenyl 95% dimethylpolysiloxanes lack of selectivity and poor chromatographic retention. Ionic liquid stationary phases offer interesting possibilities in the GC–MS determination of BTHs and BTRs in aqueous matrices because their unique selectivity. Among the IL columns evaluated, the SLB-IL59 provided the total elution of all the target analytes with the highest peak symmetry and the lowest analysis time. Moreover, the lower stationary phase bleeding obtained enabled their positive identification and quantification. The proposed methodology has been successfully applied to the quantification of BTHs and BTRs by GC–MS in real samples from a wastewater treatment plant.

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