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Continuous, on-line monitoring of haloacetic acids via membrane extraction

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Abstract

Haloacetic acids are an important class of disinfection byproducts that are being regulated. In this paper we report novel instrumentation for continuous monitoring of the nine haloacetic acids. Hollow fiber liquid–liquid membrane extraction (LLME) and supported liquid membrane extraction (SLME) followed by on-line HPLC-UV detection were studied. With continuous LLME, seven halo-acetic acids could be analyzed and enrichment factor (EF) was around 50. All the nine acids could be extracted and quantified by continuous SLME. Experiments with laboratory standards demonstrated that EF and extraction efficiency could be as high as 500 and 54%, respectively. Relative standard deviations based on seven replicates were between 3.3 and 10.3%, and the MDLs were at sub-ppb levels.

Keywords: Supported liquid membrane extraction; Membrane extraction; Haloacetic acids; Water monitoring; Disinfection byproducts

1. Introduction

Chlorination is one of the most common methods for disinfecting drinking water [1]. Chlorine reacts with naturally occurring acids to form halogenated disinfection byproducts (DBPs), some of which are known carcinogens. Trihalomethanes (THMs) and haloacetic acids (HAAs) are the major volatile and nonvolatile DBPs [2]. The names, abbreviations, pK_a values and octanol–water partition coefficients (log P) of the nine HAAS are included in Table 1. USEPA has classified DCAA as a probable human carcinogen and TCAA as a possible human carcinogen. Furthermore, decarboxylation of HAAs results in the formation of THMs, which are also carcinogens [3]. USEPA has regulated the total maximum contaminant level (MCL) in drinking water of the five HAAs: MCAA, MBAA, DCAA, BCAA, and DBAA to be less than 60 μg/L [4].

Currently there are several USEPA approved methods for HAAs analysis (EPA method 552.1, 552.2 and 6251) [5–6]. All these methods involve cumbersome liquid–liquid extraction or ion exchange and derivatization, followed by GC-ECD detection. They have several limitations, for example, EPA method 552.1 uses ion exchange and derivatization followed by GC-ECD detection. It consumes less solvent, however the interference from anions increases the detection limits [5], and it can only determine six of the HAAs. Typical analysis time for the above methods varies between three to four hours. Alternative methods that do not need the derivatization prior to analysis have been developed based on, liquid chromatography (LC) [7–8], ion chromatography (IC) [9–14], capillary electrophoresis (CE) [15], and electrospray ionization high-field asymmetric waveform ion mobility spectrometry and mass spectrometry (ESI-FAIMS–MS) [16]. ESI-FAIMS–MS provides low detection limit, has excellent sensitivity and selectivity, but the high cost limits its availability. The detection limits of the LC, IC and CE methods are higher than the GC methods. Many of the alternative methods have been used for five or six HAAs, and only a few are applicable for all the nine HAAs.

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Table 1
Analytical performance of continuous SLME-HPLC

Names	Abbreviation	log P[a]	pK_a[a]	RSD[b] (%)	EF	EE (%)	MDL[c] (ng/mL)
Monochloroacetic acid	MCAA	0.22	2.87	10.3	71.3	8.9	6.84
Dichloroacetic acid	DCAA	0.92	1.26	10.3	335.5	41.9	0.32
Monobromoacetic acid	MBAA	0.41	2.89	3.5	335.9	42.0	0.33
Bromochloroacetic acid	BCAA	1.14	1.39	4.2	273.6	34.2	0.13
Dibromoacetic acid	DBAA	1.69	1.47	4.8	412.1	51.5	0.15
Trichloroacetic acid	TCAA	1.33	0.51	5.7	383.4	48.0	0.18
Bromodichloroacetic acid	BDCAA	2.31	1.09	5.9	412.3	51.5	0.18
Chlorodibromoacetic acid	CDBAA	2.91	1.09	3.3	428.4	53.6	0.10
Tribromoacetic acid	TBAA	3.46	3.13	8.8	305.5	38.2	0.28

[a] log P and pK_a values are from Ref. [30].
[b] Relative standard deviations (RSD) based on seven replications were obtained with continuous SLME, the water containing 21 ppb MCAA, 3 ppb MBAA, and 1 ppb rest 7 HAAs flowed at 4 mL/min and the acceptor at 0.005 mL/min.
[c] The method detection limits (MDLs) were obtained following a standard EPA procedure [31].

Despite these recent developments, currently there is no method for continuous, on-line monitoring of all the nine HAAs. Automated on-line measurements are less expensive, provide real-time information and have better accuracy and precision [17]. Since there is less manual sample handling, these techniques tend to be less prone to contamination. The goal of this study is to develop automated, on-line methods for the continuous monitoring of all the nine HAAs in water.

Membrane extraction has recently emerged as a promising technique for sample enrichment. It has several advantages, such as simple instrumentation, requiring small solvent volumes and offering high enrichment factors. It allows continuous on-line extraction in a flow system, and can be coupled to a GC [18–22], HPLC [23–25], mass spectrometry (MS) [26] and CE [27] for continuous on-line monitoring.

There are two major approaches of membrane extraction, supported liquid membrane extraction (SLME) and liquid–liquid membrane extraction (LLME) [28]. SLME is a three-phase extraction system, where the analytes are extracted from an aqueous sample into an acceptor via an organic extractant held in the pores of the membrane. It works well for the extraction of highly polar and ionizable compounds [29]. Recently, we have reported supported liquid membrane micro-extraction (SLMME) for the extraction of HAAs from water [30]. This technique provides high enrichment and relatively short analysis time. LLME is a two-phase system, where the analytes are extracted from an aqueous sample into an organic acceptor. Here, the organic solvent contacts the water sample across the membrane without direct mixing. Another advantage of the membrane interface is that there is no emulsion formation, which is a common occurrence in conventional liquid–liquid extraction.

The objective of this study is to develop membrane extraction technique for continuous on-line monitoring of HAAs. Both continuous LLME and SLME followed by HPLC-UV detection were investigated in this research.

2. Experimental

The instrumentation used for SLME and LLME were quite similar and is shown in Fig. 1. It includes a hollow fiber membrane module, two pumps and a HPLC system. The first pump (a Hewlett-Packard 1050 HPLC pump) was used for the delivery of the acceptor, and the other (a Beckman 110B pump) for the donor. An automated six-port HPLC

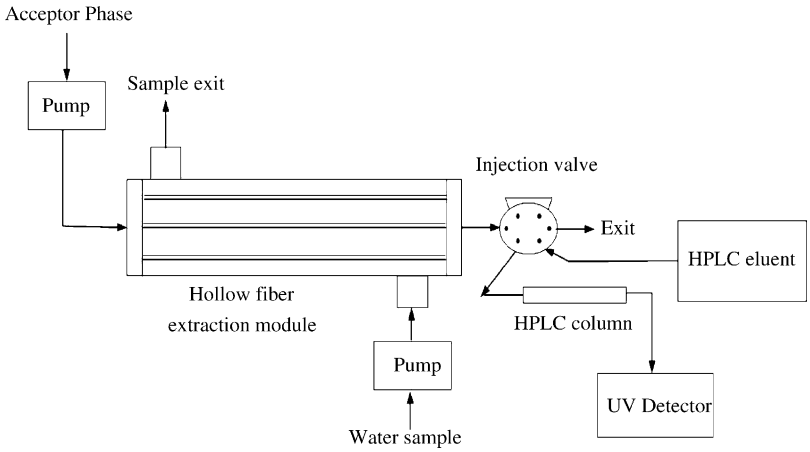

Fig. 1. Schematic diagram of continuous membrane extraction followed by HPLC-UV detection.

injection valve (Valco Instruments Co. Inc., Houston, TX, USA) was used to make repeat injections into the HPLC (Hewlett-Packard 1050) with a tunable absorbance UV detector (Waters 486). The wavelength was set at 210 nm. Donor flowed on the shell side of the membrane module, while the acceptor flowed inside the hollow fiber lumens. This approach has been described before [18–21]. The HAAs in the donor were extracted and enriched into the acceptor. The extract was injected (20 μL injection volume) into the HPLC for analysis. Minichrom V 1.62 software (VG Data System) was used for data acquisition.

Nine individual HAAs were purchased from Supelco (Supelco Park, PA, USA). All other chemicals used in this study were ACS reagent grade (Sigma Chemical Co., St. Louis, MO, USA). Deionized water was obtained from a Milli-Q® water purification system (Millipore Co., Bedford, MA, USA).

2.1. Continuous liquid–liquid membrane extraction

The membrane module for LLME was made by packing six pieces of 100 cm long composite hollow fiber membranes into a PTFE tube. Each end of the tube was connected to a tee union (Supelco Inc., PA, USA). Epoxy (Resin Technology Group, LLC, S. Easton, MA, USA) was used to seal the space between the membranes and the tee, preventing the mixing of water and the acceptor. The membrane used was Celgard X10 (Hoechst Celanese, Charlotte, NC, USA) with an I.D. of 0.240 mm and an O.D. of 0.290 mm, which was made of polypropylene. The acceptor used in LLME was 99.8% methyl *tert*-butyl ether (MTBE) (Fluka, Milwaukee, WI, USA). The HPLC column was a 3.9 mm I.D., 150 mm long Waters Resolve C_{18} with 5 μm spherical packing (Waters, Milford, MA, USA). The HPLC mobile phase was a 0.4 M ammonium sulfate and the flow programming was as follows: flow rate was held constant at 0.5 mL/min during the first 5 min, and was then increased gradually to 2.0 mL/min in the next 3 min, the flow rate was kept constant at that level between 8 and 13 min.

2.2. Continuous supported liquid membrane extraction

The membrane module for SLME was made by packing three pieces of 130 cm long hollow fiber membrane into a PTFE tube. The membrane was Celgard X20 (Hoechst Celanese, Charlotte, NC, USA). It had an I.D. of 400 μm and an O.D. of 460 μm, with an average pore size of 0.03 μm and porosity of 40%. The membranes were soaked in the membrane liquid, 5% trioctylphosphine oxide in dihexyl ether, which was optimized in a previous study [30] before the analysis. The HPLC column used here was a 150 mm × 4.6 mm YMC ODS-A C_{18} column with 3 μm packing. The HPLC mobile phase was 95:5 (v/v) 15 mM KH_2PO_4 (pH 2.2): acetonitrile at a flow rate of 1.0 mL/min.

The membrane module can be regenerated in this way: first clean the membrane by the flowing the acceptor, then dry it with nitrogen, and finally soak with the membrane liquid.

3. Results and discussion

Enrichment factor (EF) is defined as the ratio of analyte concentration in the extract (acceptor) to that in the water sample (donor) entering the membrane:

$$\mathrm{EF} = \frac{C_\mathrm{a}}{C_\mathrm{d}} \tag{1}$$

where C_a is the analyte concentration in the acceptor exiting the membrane module and C_d is the concentration in the donor. Sensitivity is proportional to the enrichment factor. Extraction efficiency (EE) is the fraction of analyte in the acceptor to that in the donor [28].

$$\mathrm{EE} = \frac{n_\mathrm{a}}{n_\mathrm{a}} = \frac{C_\mathrm{a}V_\mathrm{a}}{C_\mathrm{d}V_\mathrm{d}} = \mathrm{EF}\frac{V_\mathrm{a}}{V_\mathrm{d}} \tag{2}$$

where n_a and n_d are the total mass of analyte in the acceptor and the donor, V_a and V_d are the volumes of the acceptor exiting and that of the donor entering. In the continuous flowing system,

$$V_\mathrm{d} = F_\mathrm{d}t \tag{3}$$

where F_d is the flow rate of the water sample entering the membrane and t is the running time. In SLME system, the acceptor is an aqueous phase and its loss is negligible, thus, the volume of acceptor can be expressed as:

$$V_\mathrm{a} = F_\mathrm{a}t \tag{4}$$

where F_a is the flow rate of the acceptor. Consequently in the flowing SLME system, Eq. (2) reduces to:

$$\mathrm{EE} = \mathrm{EF}\frac{F_\mathrm{a}}{F_\mathrm{d}} \tag{5}$$

In LLME, the conditions are somewhat different. The acceptor is an organic solvent, which may be partially lost by dissolving in water [17], thus

$$V_\mathrm{a} = V_\mathrm{ai} - V_\mathrm{ls} = V_\mathrm{ai} - V_\mathrm{ai}L_\mathrm{s} = V_\mathrm{ai}(1 - L_\mathrm{s}) \tag{6}$$

V_ai is the initial volume of the organic solvent entering the membrane lumen, while V_ls is the amount of solvent that is lost during the extraction. L_s is the percentage of the solvent lost.

$$V_\mathrm{ai} = F_\mathrm{a}t \tag{7}$$

where F_a is the flow rate of the organic solvent entering the membrane. According to Eqs. (2), (3), (6) and (7), extraction efficiency in flowing LLME system can be expressed as:

$$\mathrm{EE} = \mathrm{EF}(1 - L_\mathrm{s})\frac{F_\mathrm{a}}{F_\mathrm{d}} \tag{8}$$

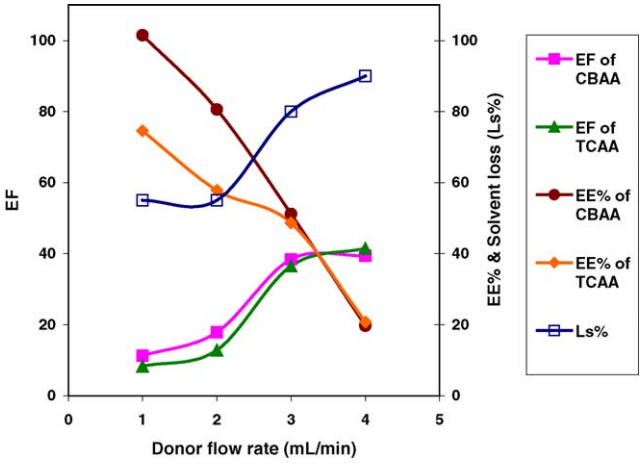

Fig. 2. EF, EE (%) and percentage solvent lost (Ls%) as a function of water sample (donor) flow rate, the flow rate of acceptor was kept constant at 0.2 mL/min. This was performed with LLME mode.

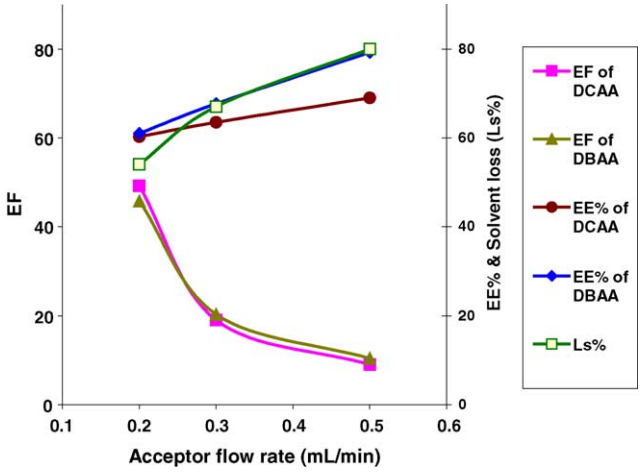

Fig. 3. EF, EE (%) and percentage solvent lost (Ls%) as a function of acceptor flow rate, the donor flow rate was kept constant at 3 mL/min. This was performed with LLME mode.

3.1. Continuous liquid–liquid membrane extraction

Continuous LLME was carried out across the hollow fiber. The donor consisted of 0.12 μg/mL (ppm) MCAA and DCAA, 0.08 ppm MBAA, BCAA and BDCAA, 0.04 ppm DBAA and TCAA, 0.2 ppm CDBAA and 0.4 ppm TBAA. The acceptor was 99.8% methyl *tert*-butyl ether (MTBE). During HPLC analysis MTBE co-eluted with CDBAA and TBAA. Consequently, only seven of the HAAs could be quantified.

The effects of the donor flow rate on EF, EE and solvent loss were tested and the results are shown in Fig. 2. The flow rate of MTBE was kept constant at 0.2 mL/min, while the donor flow rate was increased from 1 to 4 mL/min. Solvent loss was measured by collecting the acceptor at the membrane outlets. Extraction efficiency was calculated according to Eq. (8). The EF of the seven HAAs increased as the donor flow rate increased from 1 to 3 mL/min. At higher donor flow rates, more analytes contacted the membrane, thus resulting in higher extracted amounts, which led to higher EF. EF as high as 50 was obtained by LLME. Solvent loss was an important consideration. It was found that as much as 55–90% of the solvent could be lost by permeation into the aqueous phase. As reported previously, solvent loss contributes to the high enrichment factors [21]. Solvent loss increased with the increase in donor flow rate, as the higher flow rate increase the pressure on the membrane. The EE decreased because although the increase of the donor flow rate brought more analytes into the system, a larger fraction went unextracted.

The effects of the acceptor flow rate on EF and EE and solvent loss in LLME were also tested. The donor flow rate was kept constant at 3 mL/min, while the acceptor flow rate was changed from 0.2 to 0.5 mL/min. The acceptor was collected offline and the solvent loss was calculated. The results are shown in Fig. 3. EF decreased significantly with the increase in the acceptor flow rate. This was to be expected because higher acceptor flow rate led to a more diluted extract, although the extraction efficiency increased. The solvent loss also increased at higher acceptor flow rates.

With continuous LLME, seven of the nine HAAs were extracted and quantified. Enrichment factor about 50 was achieved. However, the objective of this study was to develop a method for continuous on-line monitoring of all the nine HAAs at trace levels, thus continuous SLME was investigated.

3.2. Continuous supported liquid membrane extraction

SLME was carried out by two different modes: stop-and-flow SLME and continuous SLME. The former was a semi-static approach and is referred to as SF-SLME. In SF-SLME, the donor flowed continuously while the acceptor was held stagnant, and then collected after a certain period. The donor contained 83.2 μg/L (ppb) of the nine HAAs. Sulfuric acid (0.26%) was added to the donor to keep the acids in their uncharged molecular form. The donor was pumped at the rate of 1 mL/min for 60 min. The acceptor used here was 0.01 M sodium hydroxide (NaOH). The acceptor was stagnant in the membrane lumens. The membrane module was made by two pieces 128 cm Celgard X20 hollow fiber membranes. The internal volume of the membrane lumens was 0.322 mL. The membrane was soaked with 5% trioctylphosphine oxide (TOPO) in dihexyl ether (DHE) before extraction. After extraction, the acceptor was pumped at 0.1 mL/min and collected into HPLC vial inserts every minute. Results are shown in Fig. 4, and the EF was found to decrease with time. The concentration gradient in the acceptor along the length of the hollow fiber was evident. The concentration was higher in the static acceptor segment near the entrance point of the donor, which corresponded to the exit point of the acceptor. During counter-current contact across the membrane, the entering donor had high concentration and it first contacted the extract near the acceptor outlet and the extraction was initiated. As it flowed further, and toward the acceptor inlet,

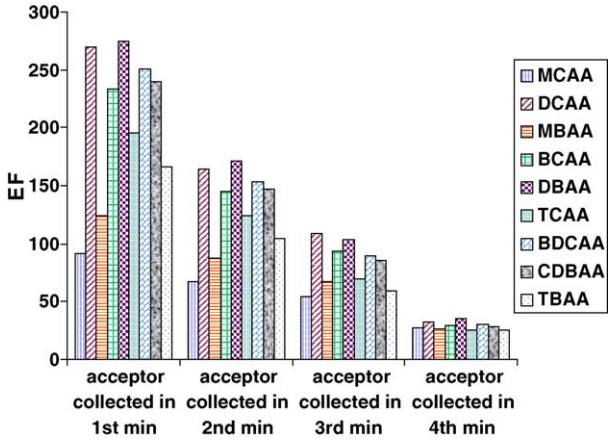

Fig. 4. EF as a function of time, the donor flowed at 1 mL/min for 60 min, while the acceptor was kept stagnant in the membrane lumens (SF-SLME mode). After the extraction, the acceptor was pumped at 0.1 mL/min. The acceptor exiting the membrane module was collected.

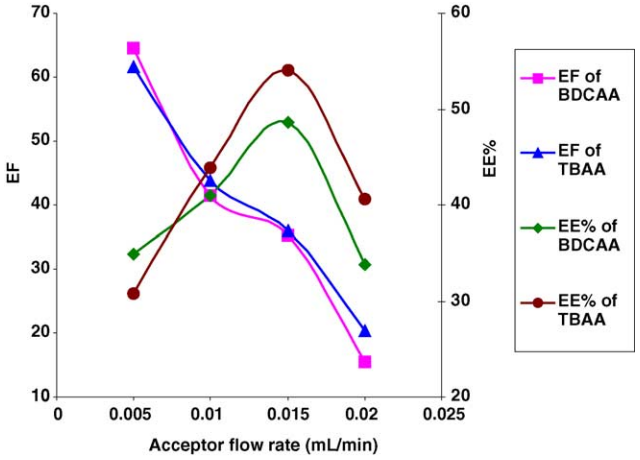

Fig. 5. EF and EE (%) as a function of acceptor flow rate, the donor flow rate was kept constant at 1 mL/min. This was performed with SLME mode.

its concentration decreased. Consequently, the extract near the donor inlet was more enriched than that near the outlet.

SF-SLME provided higher enrichment factor but lower extraction efficiency as the acceptor solution was kept stagnant in the hollow fiber lumens. There existed a concentration gradient in the acceptor for the SF-SLME. Shorter membrane fiber may decrease the concentration gradient across the hollow fiber.

To obtain real-time information, both the donor and acceptor need to flow continuously, which is referred as continuous SLME. In continuous SLME, the donor contained 80 ng/mL (ppb) of the nine HAAs in DI water, which was acidified with sulfuric acid to pH 1.9. The donor pH is acceptable even though it is somewhat higher than the pK_a values of the acids, as the protonation equilibrium is dynamic and quite fast. Lower pH may results in higher extraction efficiency, however very acidic solution is more corrosive, which may destroy the pump. The acceptor was 0.05 M tris buffer adjusted to pH 8.7, which was more than 3.3 units higher than the pK_a values that was required to prevent the extracted acids from re-entering the membrane. The membrane module was made of three pieces 130 cm Celgard X20 hollow fiber membranes. The membrane was soaked with 5% TOPO in DHE prior to use.

The effects of the acceptor flow rate on EF and EE in continuous SLME were studied. The donor flow rate was kept constant at 1 mL/min, while the acceptor flow rate was varied from 0.005 to 0.02 mL/min. It was found that the loss of acceptor during extraction was negligible. Thus the experiment was carried out on-line with HPLC-UV detection. EF and EE (%) as a function of the acceptor flow rate are shown in Fig. 5. EF decreased with the increase in the acceptor flow rate. At a lower flow rate, the contact time was longer and more analytes could be trapped into the acceptor. Moreover, higher acceptor flow rate diluted the extract, so the overall effect was the reduction in EF. According to Eq. (5), EE increases with the increase of the EF and the acceptor flow rate, and decreases with the donor flow rate. However, EF decreases with the increase of the acceptor flow rate. In Fig. 5 the EE increased with acceptor flow rate increase from 0.005 to 0.015 mL/min, then decreased as the acceptor flow rated increased to 0.02 mL/min due to the dramatic decrease of the EF.

The effects of the donor flow rate on EF and EE was also studied. The acceptor flow rate was kept constant at 0.005 mL/min, while the donor flow rate increased from 1 to 4 mL/min. EF and EE as a function of the donor flow rate is shown in Fig. 6. EF increased dramatically as the donor flow rate increased from 1 to 4 mL/min. With a higher donor flow rate, more analytes contacted the membrane, thus resulting in more analytes trapped in the acceptor, which led to higher EF. According to the Eq. (5) EE increases with the increase of EF but decreases with the increase of the donor flow rate and is also shown in Fig. 6.

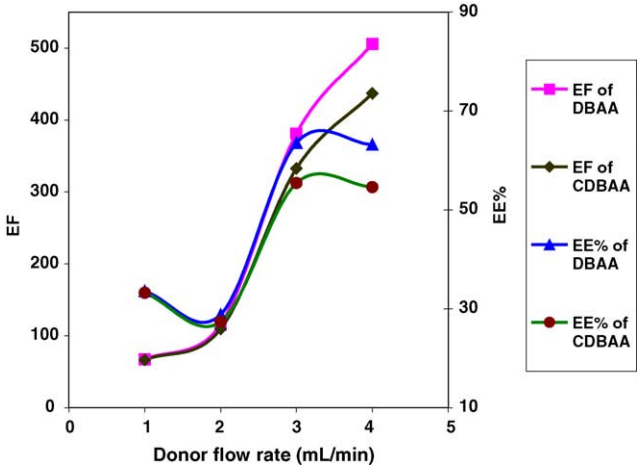

Fig. 6. EF and EE (%) as a function of water sample (donor) flow rate, the flow rate of acceptor was kept constant at 0.005 mL/min This was performed with SLME mode.

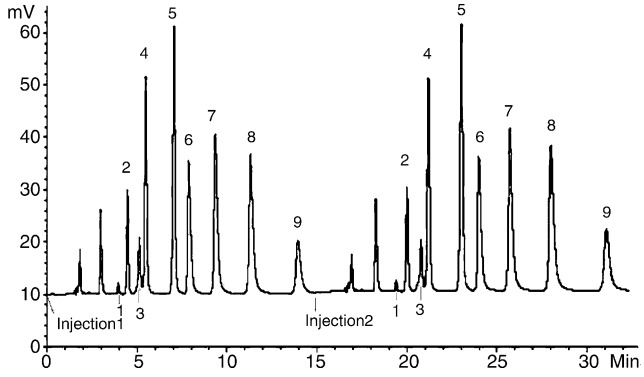

Fig. 7. Chromatogram of continuous SLME of reagent water spiked with 80 ng/mL (ppb) nine HAAs. The donor flow rate was 4 mL/min. The acceptor was 0.05 M tris buffer (pH 8.7) with a flow rate of 0.005 mL/min. Injections were made every 15 min. The numbered peaks in the chromatogram are: 1: MCAA; 2: DCAA; 3: MBAA; 4: BCAA; 5: DBAA; 6: TCAA; 7: BDCAA; 8: CDBAA; and 9: TBAA.

3.3. Analytical performance of continuous SLME

SLME showed higher EF compared to LLME, and its analytical performance was evaluated. The HAAs in acidified donor were extracted and trapped into the alkaline acceptor. The enriched acceptor was automatically injected into the HPLC-UV system every 15 min by automated injection valve. Sequential chromatograms were obtained and shown in Fig. 7. The donor used was reagent water (pH 1.9) spiked with 80 ppb nine HAAs at a flow rate of 4 mL/min. The acceptor was 0.05 M tris buffer (pH 8.7) at 0.005 mL/min. Good reproducibility in peak shape and retention time was observed.

Relative standard deviations, enrichment factors, extraction efficiencies and method detection limits (MDL) are listed in Table 1. The donor used was reagent water (pH 1.9) spiked with 21 ppb MCAA, 3 ppb MBAA and 1 ppb other seven HAAs. The acceptor and the flow rates were same as in Fig. 7. The RSDs were between 3.3 and 10.3% and the MDLs were at sub-ppb levels. The MDL of MCAA was higher than other HAAs. This is because MCAA is a very polar compound and its octanol–water partition coefficient (log *P*) is considerably lower than the other HAAs. This is consistent with previous observation [30,32].

The memory effect of the membrane was tested by flowing reagent water as the donor. The HAAs concentrations were about 2% of that obtained with a normal experiment. Thus the retention by the membrane itself was minor, and could be eliminated by washing the membranes with the acceptor for 1 min. The supported liquid membrane module worked well for about 4 weeks, after which it needed regeneration.

4. Conclusion

Both LLME and SLME were developed for the continuous analysis of the HAAs. Seven HAAs could be analyzed by LLME and the EF was about 50. Continuous SLME could be used to monitor all the nine HAAs and enrichment factors were as high as 500 with MDLs at sub-ppb levels. On the whole, the continuous SLME was found to be the more effective of the two approaches tested here, and is recommended for real-world applications.

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