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On-line coupling of in-tube boronate affinity solid phase microextraction with high performance liquid chromatography–electrospray ionization tandem mass spectrometry for the determination of cis-diol biomolecules

Jiangang He[a,b], Zhen Liu[a,*], Lianbing Ren[a], Yunchun Liu[a], Peng Dou[a], Kai Qian[b], Hong-Yuan Chen[a,**]

[a] Key Lab of Analytical Chemistry for Life Science, School of Chemistry and Chemical Engineering, Nanjing University, 22 Hankou Road, Nanjing 210093, Jiangsu, China
[b] Inspection Center of Industrial Products, Jiangsu Entry-Exit Inspection and Quarantine Bureau, Nanjing 210001, China

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ABSTRACT

Boronate affinity solid phase microextraction (BA-SPME) is a new format appeared recently with great potential for specific extraction of cis-diol-containing compounds. Unlike conventional SPME, BA-SPME relies on covalent interactions and thereby features with specific selectivity, eliminated matrix effect and manipulable capture/release. However, only on-fiber BA-SPME and its off-line combination with high performance liquid chromatography (HPLC) have been reported so far. In this study, we report on-line coupling of in-tube BA-SPME with HPLC–electrospray ionization tandem mass spectroscopy (in-tube BA-SPME-HPLC–ESI-MS/MS) for the specific and sensitive determination of cis-diol-containing biomolecules. A boronate affinity extraction phase was prepared onto the inner surface of the capillary by copolymerization of vinylphenylboronic acid (VPBA) and ethylene glycol dimethacrylate (EDMA). The extraction conditions were optimized by choosing appropriate extraction/desorption solutions and extraction time. The extraction capacity, linear range, reproducibility and life-time were investigated. The developed method was successfully applied for the determination of dopamine in urine samples. Since many cis-diol-containing compounds are of great biological importance, the in-tube BA-SPME-HPLC method can be a promising tool.

1. Introduction

Boronate affinity chromatography (BAC) [1–7], which has appeared for more than two decades, is a useful tool for the specific capture and isolation of cis-diol-containing molecules. The principle relies on the chemistry that boronic acids form covalent bonds with cis-diols to generate five- or six-membered cyclic esters in a basic aqueous media while the esters reversibly disassociate once upon the media pH is changed to an acidic condition. The boronic acids chemistry features with several significant advantages, which makes boronic acids ideal affinity ligands for molecular recognition and sample enrichment. First, the affinity is based on covalent reaction and therefore provides high specificity since compounds adsorbed by non-covalent interactions can be easily washed away. Second, the capture/release procedure can be facilely controlled through simply switching the media pH. Third, the acidic solution required for the elution is compatible with mass spectrometry (MS). Due to these merits, several new boronate affinity based techniques have emerged in the past years. Boronate affinity monolithic columns have been developed and applied for affinity separation and specific capture of cis-diol-containing molecules [8–12]. Boronate functionalized matrixes, including magnetic nanoparticles [13–16] and mesoporous silica [17], were synthesized and applied to the selective isolation of glycopeptides, glycoproteins and other cis-diol-containing biomolecules. Also, a borate complexation-assisted on-line sample concentration technique was proposed for pre-concentrating cis-diol-containing compounds in capillary electrophoresis (CE) [18]. Moreover, boronate affinity solid phase microextraction (BA-SPME) in on-fiber format was developed and off-line coupled with high performance liquid chromatography (HPLC) for the specific extraction and analysis of cis-diol-containing biomolecules [19].

Although a big variety of SPME have been reported, BA-SPME is quite different from its conventional counterparts as the latter are generally based on non-covalent interactions. Since on-fiber BA-SPME has already exhibited attractive features, including specific selectivity, eliminated matrix effect and manipulable on/off procedure [19], BA-SPME can be a promising tool. Therefore, it is necessary to further develop this methodology. In this paper, we report the development of in-tube BA-SPME and its on-

* Corresponding author. Tel.: +86 25 8368 5639; fax: +86 25 8368 5639.
** Corresponding author. Fax: +86 25 8359 4862.
E-mail addresses: zhenliu@nju.edu.cn (Z. Liu), hychen@nju.edu.cn (H.-Y. Chen).

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line combination with HPLC–electrospray ionization tandem mass spectroscopy (ESI-MS/MS) for the specific and sensitive determination of cis-diol-containing biomolecules. The in-tube BA-SPME was made through preparing a poly (vinylphenylboronic acid-co-ethylene glycol dimethacrylate) (poly(VPBA-co-EDMA)) extracting phase onto the inner surface of a capillary. On-line coupling of the prepared in-tube BA-SPME with HPLC was realized through using a six-port valve as an interface. Since BA-SPME is MS-compatible, MS was employed as a detector for high sensitivity. Dopamine, a typical cis-diol-containing biomolecule that plays important roles in nervous system, was used as target analyte. The extraction conditions were optimized by choosing appropriate extraction/desorption solutions and extraction time. The extraction capacity, linear range, reproducibility and life-time were investigated. The developed method was finally applied for the determination of dopamine in urine samples. The experimental results indicated that in-tube format together with the three inherent advantages of boronate affinity (specificity, easy-to-control capture/release procedure and MS-compatibility) make in-tube BA-SPME a useful sample preconcentration tool for on-line coupling with HPLC–ESI-MS/MS for specific and sensitive determination of cis-diol-containing biomolecules.

2. Experimental

2.1. Reagents and materials

4-Vinylphenylboronic acid (VPBA) was purchased from Alfa Aesar (Ward Hill, MA, USA). Ethylene glycol dimethacrylate (EDMA), γ-methacryloxypropyltrimethoxysilane (γ-MAPS) and sodium metabisulfite were obtained from Sigma (St. Louis, MO, USA). Ethylene glycol, diethylene glycol and azobisisobutyronitrile (AIBN) were from Sinopharm Chemical Reagent (Shanghai, China). HPLC-grade methanol and acetonitrile were from Merck (Darmstadt, Germany). 5-Hydroxytryptamine and dopamine were purchased from Sigma (St. Louis, MO, USA). All other chemicals were of analytical or HPLC grade. EDMA was extracted with 5% aqueous sodium hydroxide solution and dried over anhydrous magnesium sulfate. Azobisisobutyronitrile (AIBN) was recrystallized with methanol. Water was purified with a Milli-Q Advantage A10 System (Millipore, Milford, MA, USA), and was used to prepare all solutions. The other reagents and solvents were purchased as HPLC or AR grade reagents from normal commercial sources. Fused-silica capillaries with 75 μm ID and 375 μm OD were purchased from Yongnian Optic Fiber Plant (Hebei, China). A DB-1 gas chromatographic column (0.32 mm ID, 0.25 μm film thickness) coated with 100% polydimethylsiloxane (PDMS) was from J & W Scientific Inc. (Folsom, CA, USA).

2.2. Instruments

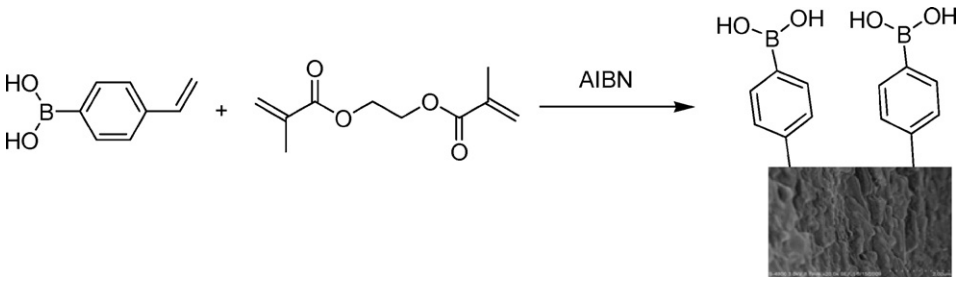
Scanning electron microscopy (SEM) analyses were performed on a field emission SEM S-4800 instrument (Hitachi, Tokyo, Japan). A Thermo ACCELA HPLC system equipped with TSQ Quantum Access mass spectrometer was employed for the analyses. The HPLC system was controlled by an Xcalibar 2.0 SR 2 chemstation. A ZORBAX Eclipse XDB-C8 analytical column (150 mm × 4.6 mm, 5 μm particle size) from Agilent Technologies (Walfbronn, Germany) was used for the separation of biomolecules. The column temperature was set at 20 °C. The mobile phase was 5 mM formic acid, at a flow rate of 0.2 mL/min.

2.3. Tandem mass spectrometric conditions

The analytes were detected in the multiple-reaction monitoring (MRM) mode of the tandem mass spectrometer. All results were generated in positive-ion mode. The general instrument settings applied as follows: spray voltage, 4.5 kV; capillary temperature, 350 °C; sheath gas pressure, nitrogen, 35 psi; Aux gas, nitrogen, 10 arbitrary pressure unit; collision gas, argon. MRM transitions monitored were m/z 154.0 → 137.0 and 137.0 → 91.0 for dopamine, and m/z 177.0 → 159.9 for 5-hydroxytryptamine. The quantitative product mass ion for dopamine was 137.0, while the qualitative product ions were 91.0 and 159.9 for dopamine and 5-hydroxytryptamine, respectively. The collision energy voltages applied for the analysis were 20 and 25 V for dopamine and 5-hydroxytryptamine, respectively.

2.4. Preparation of poly(VPBA-co-EDMA) extracting phase

The in-tube SPME was prepared according to a method reported previously with slight modification [9]. The copolymerization reaction is schematically illustrated in Scheme 1. The capillary was first rinsed with 1 M NaOH solution for 30 min, followed with water until the pH value of the outlet solution reached 7.0. The capillary was then rinsed with 1 M HCl solution for 30 min, followed with water until the pH value of the outlet solution reached 7.0. The capillary was dried with passage of nitrogen in a GC-oven at 60 °C. After that, a solution of γ-MAPS/methanol (1:1, v/v) was injected into the capillary with a syringe. It was kept at 45 °C in a GC-oven overnight with both ends sealed with rubber. Finally, the capillary was rinsed with methanol to flush out the residual reagents and dried by passage of nitrogen. AIBN (0.1 mg) was dissolved in a 5 μL EDMA solution and 10 mg VPBA was added. The binary porogenic solution was prepared by mixing 195 μL diethylene glycol and 45 μL ethylene glycol, then the solution was mixed with the AIBN/EDMA/VPBA solution. The polymerization mixture was sonicated for 20 min to obtain a homogeneous solution and then purged with nitrogen for 10 min. Then the mixture was injected into a pretreated 45 cm capillary for a certain length by a syringe. Both ends of the capillary were sealed with rubber, and the capillary was submerged in a water bath at 75 °C for 2 h. The monolithic bed was pumped out of the capillary by a HPLC pump, leaving the residual poly(VPBA-co-EDMA) extracting phase on the inner surface of the capillary. The capillary was washed with 100 mM acetic acid for 30 min before use.



Scheme 1.

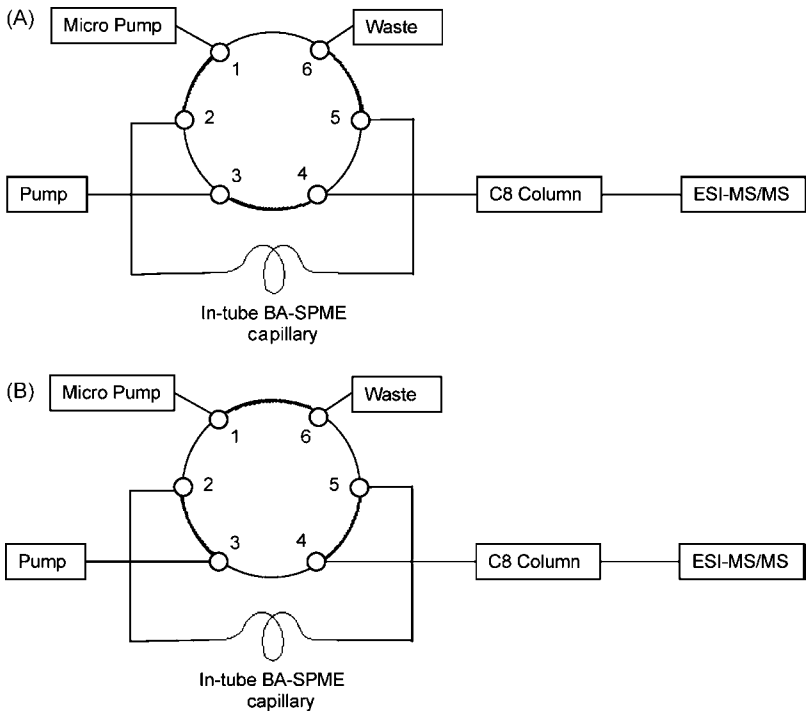


Fig. 1. Schematic diagram of the on-line in-tube BA-SPME-HPLC-ESI-MS/MS setup. (A) Extraction and rinse position (1–2 positions); (B) sample desorption and detection position (2–3 positions).

2.5. Preparation of sample solutions

A mixture of dopamine (5 μg/mL) and 5-hydroxytryptamine (5 μg/mL) dissolved in 100 mM sodium phosphate buffer (pH 9.0) was prepared. Fresh mixture solution was used to reduce possible oxidation of dopamine. A stock solution of dopamine (0.2 mg/mL) dissolved in 10 mM formic acid (pH 3.5) was prepared and stored in a freezer. Fresh solutions of dopamine (2 μg/mL) were prepared each day by diluting the stock solution with buffers, antioxidant sodium metabisulfite (6 mmol/L) was added in each prepared solution, to prevent the oxidation of dopamine.

2.6. Preparation of real sample solutions

To prepare the urine samples, 20 mL of fresh urine was added in a 50-mL beaker, and then the pH of sample was adjusted to 9.0 using 100 mM sodium hydroxide. Sodium metabisulfite (6 mmol/L) was added in prepared solution to avoid the oxidation of dopamine. In-tube SPME process was performed immediately after the preparation.

2.7. Extraction and desorption

As shown in Fig. 1A, the prepared in-tube SPME capillary (30 cm in length) was connected between position 2 and position 5 of a six-port valve, replacing the conventional inject loop. To perform the extraction procedure, the injection valve was turned to the load-position, prepared samples were pumped through the capillary tube at the rate of 100 μL/min. After extraction, 10 mM ammonia (pH 9.5) passed through the tube at a flow rate of 200 μL/min for 5 minutes, to remove the un-extracted analytes. After extraction and rinse, the six-port valve was changed to the injection position, as shown in Fig. 1B, desorption solution was pass through the in-tube SPME, then the analytes were separated in the C8 column and detected by the mass spectrometer.

3. Results and discussion

3.1. Characterization of the extracting phase

The morphology of a poly(VPBA-co-EDMA)-coated capillary was investigated with SEM analysis using a capillary treated with γ-MAPS/methanol as a control. As shown in Fig. 2D, after the capillary was treated with γ-MAPS/methanol, no polymer was formed. The polymer was formed after the capillary was treated with AIBN/EDMA/VPBA, as shown in Fig. 2F, the thickness of the polymer was measured to be about 0.5 μm. The rough polymer extracting phase favored the extraction capacity of the in-tube SPME.

3.2. Desorption speed

Desorption kinetics is an important factor that greatly influences the on-line combination. Slow desorption speed of the analyte will cause severe peak tailing and carryover. Therefore, it is necessary to evaluate the desorption kinetics. Two consecutive runs were carried out. For the first run a dopamine sample was injected, while for the second run the desorption solution (formic acid) rather than the sample was injected. The desorption speed was evaluated in terms of the resulting peak shape and analyte carryover. As shown in Fig. 3, although the peak shape in the first run was tailing, there was no analyte carryover observed in the second run. This indicates that the desorption speed was acceptable, though it was not very fast.

3.3. Optimization of MRM transitions

Determination of the optimal MRM transitions for dopamine was performed using single-MS full scan mode followed by product ion scan mode. Dopamine standard solution at 1 μg/mL was directly injected into the MS spectrometer using a syringe pump. Different ionization interfaces and conditions were applied, giving ESI in positive-ion mode the best performance under the mobile phase conditions investigated. The predominant signal detected in

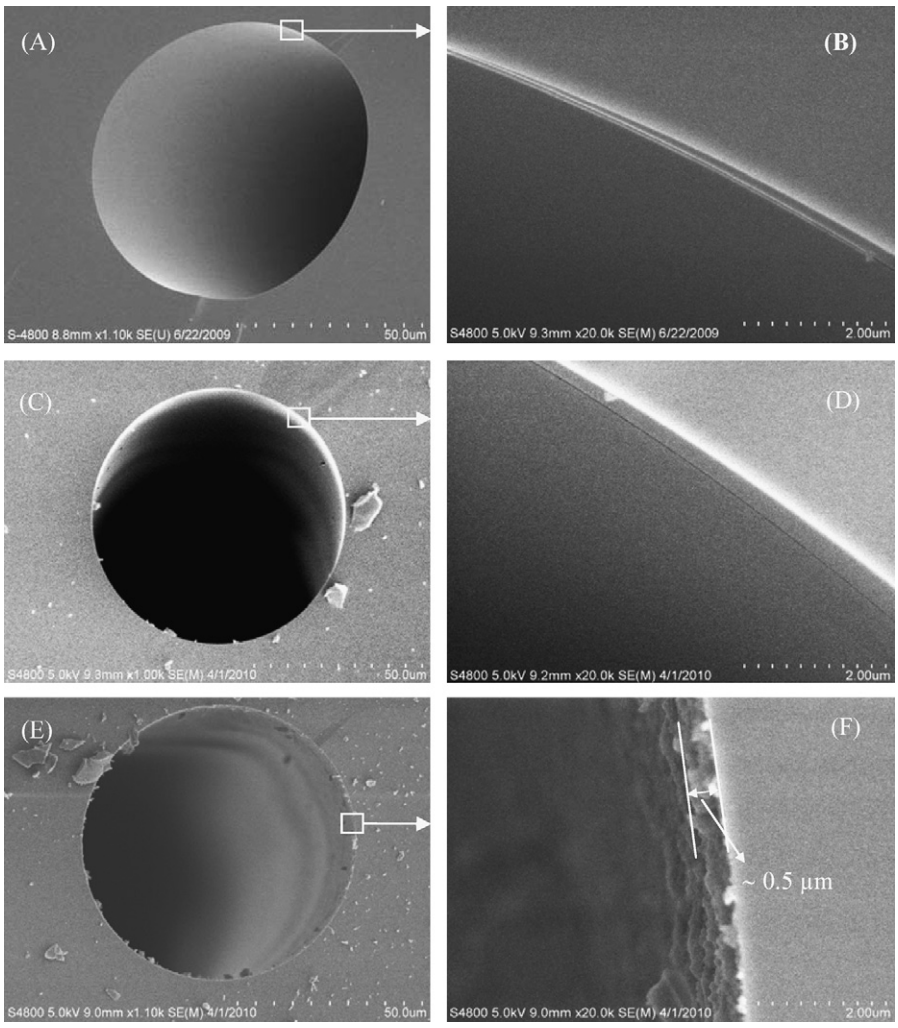


Fig. 2. SEM photographs of the poly(VPBA-co-EDMA)-coated capillary and the host capillaries: (A) cross-sectional view of the bare capillary; (B) enlarged image of the edge of the inner surface of the bare capillary; (C) cross-sectional view of the capillary treated with γ-MAPS/methanol; (D) enlarged image of the edge of the inner surface of the capillary treated with γ-MAPS/methanol; (E) cross-sectional view of the poly(VPBA-co-EDMA)-coated capillary; (F) enlarged image of the edge of the inner surface of the poly(VPBA-co-EDMA)-coated capillary.

the first quadrupole (Q1) was $[M+H]^+$ at m/z 154. Collision energies were optimized in the collision cell (Q2) from 5 to 50 V in order to generate diagnostic product ions from the investigated parent ion. The final quadrupole (Q3) was set to scan for the diagnostic product ions. The mass spectrum of dopamine showed the presence of product ions at m/z 137, m/z 119, m/z 91, and m/z 65. The most intense ion transition at m/z 137 was selected as a quantification ion and m/z 91 and m/z 65 were used as confirmation ions.

3.4. Extraction selectivity

The extraction selectivity of in-tube BA-SPME was investigated through extracting dopamine and 5-hydroxytryptamine from a mixture of these two compounds. Dopamine and 5-hydroxytryptamine are neurotransmitters which have similar structures; the main difference is that dopamine molecule contains cis-diol while 5-hydroxytryptamine molecule does not. As shown in Fig. 4A, dopamine and 5-hydroxytryptamine mixed in 5 mM formic acid were baseline separated in the C8 column. According to boronate affinity chromatography, under a basic loading pH, dopamine will bind to the stationary phase while 5-hydroxytryptamine not. When a mixture sample of dopamine and 5-hydroxytryptamine (5 μg/mL for each dissolved in 50 mM sodium phosphate solution, pH 9.0) was extracted by the in-tube BA-SPME, only dopamine was observed, as shown in Fig. 4B, confirming the boronate affinity selectivity of prepared in-tube BA-SPME. In comparison, when the PDMS-coated capillary was used for extraction, both analytes were extracted from the mixture, as shown in Fig. 4C. Clearly, the specific selectivity of the in-tube BA-SPME was due to the presence of poly(VPBA-co-EDMA) coating in the capillary.

3.5. Optimization of the extraction and desorption conditions

To optimize the extraction of dopamine by the in-tube BA-SPME, several parameters, such as the type and pH of the extraction solution, were investigated. Four different alkaline solutions (phosphate buffer, Tris–HCl, sodium carbonate–sodium bicarbonate, ammonium formate–ammonia) and three pH values (8.0, 8.5 and 9.0) were compared. The in-tube BA-SPME could extract dopamine in all four alkaline solutions, but best extraction efficiency was observed when phosphate solution was used. On the other hand, the extraction efficiency increased as increasing the pH, which is in good agreement with the general trend that higher pH favors the boronate affinity. Thus, phosphate solution at pH 9.0 was used for later investigations.

To choose an optimum desorption solution, three acids that are often used in LC–MS, including formic acid, acetic acid and triflu-

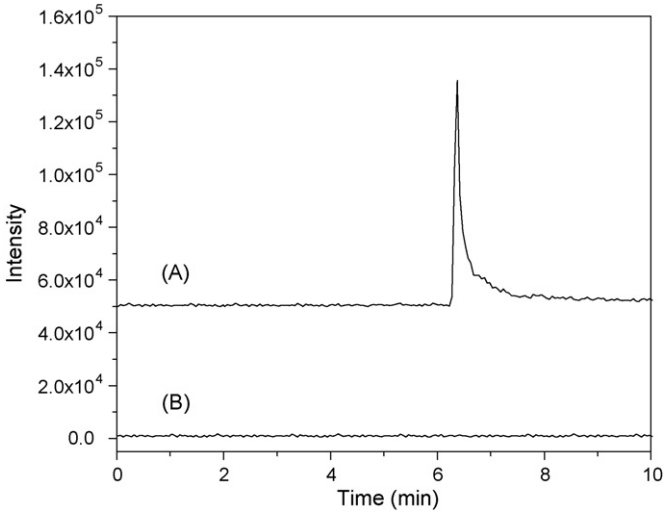


Fig. 3. Peak shape and carryover in two consecutive runs. (A) First run, 2 μg/mL dopamine in 50 mM sodium phosphate buffer (pH 9.0) was injected; (B) second run, 5 mM formic acid was injected. Desorption solution: 5 mM formic acid.

oroacetic acid, were tested. The experimental results showed that best ionization efficiency was reached when formic acid was used, which is consistent with the previous report [20]. Besides, methanol and acetonitrile were added into the formic acid (5 mM) to determine whether or not the ionization efficiency could be enhanced. The results showed that addition of methanol and acetonitrile did not influence the desorption and ionization of dopamine. For further investigations, formic acid (5 mM) was used as the desorption solution.

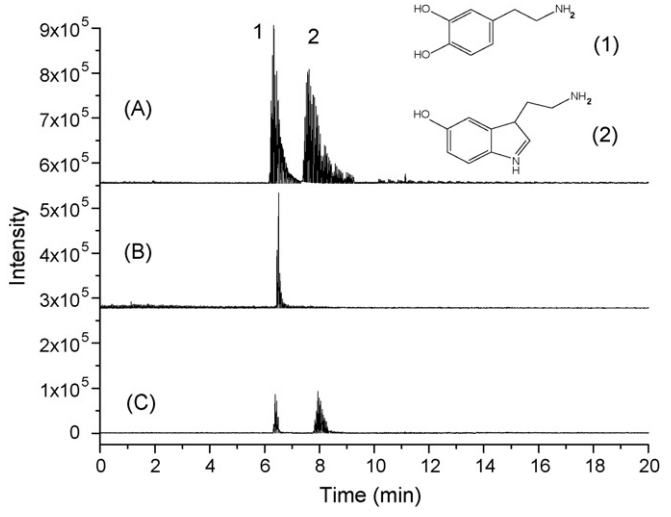


Fig. 4. Comparison of the chromatograms for a mixture of dopamine and 5-hydroxytryptamine (A), analytes extracted by an in-tube BA SPME from the mixture (B) and analytes extracted by the DB-1 column from the mixture (C). Sample: (A) 5 μg/mL dopamine and 5 μg/mL 5-hydroxytryptamine in 5 mM formic acid solution, volume injected: 10 μL; (B) analytes extracted by in-tube BA SPME from a mixture of 5 μg/mL dopamine and 5 μg/mL 5-hydroxytryptamine in 50 mM sodium phosphate buffer, pH 9.0; extraction time: 30 min, desorption solution: 5 mM formic acid; (C) analytes extracted by the DB-1 column, extraction and desorption conditions as same as (B). HPLC conditions: mobile phase, 5 mM formic acid; flow rate, 0.2 mL/min. MRM monitored were m/z 154.0 → 137.0 for dopamine; m/z 177.0 → 159.9 for 5-hydroxytryptamine. Peak identity: 1, dopamine; 2, 5-hydroxytryptamine.

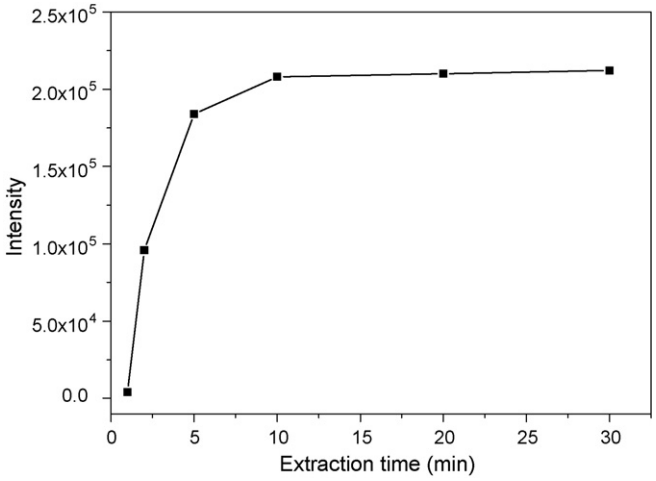


Fig. 5. Dependence of the peak area for dopamine on the extraction time. Sample: 5 μg/mL dopamine in 50 mM sodium phosphate buffer, pH 9.0.

3.6. Extraction and equilibrium

Extraction profile was investigated to evaluate the prepared in-tube BA-SPME. The sample was pumped through the in-tube-SPME at a flow rate of 100 μL/min for a predetermined duration. Fig. 5 shows the dependence of the peak intensity for the analyte extracted on the extraction time. The intensity increased rapidly with increasing the extraction time within initial 10 min, but kept nearly constant with further increasing the extraction time. This indicates that the equilibrium was reached in 10 min. The extraction equilibrium was much faster than that in previous study (30 min) [19]. Three aspects may have contributed to the faster equilibrium in this study. The first aspect is the structure of the extraction phase. In this study, the boronic acid ligands could protrude out of the polymeric extraction layer. As a comparison, boronic acid ligands in previous study [19] just stayed on the surface of the extraction phase. Clearly, the boronic acid ligands in this study are more accessible to analytes. The second favorable aspect is the flowing of the sample solution in the in-tube SPME. The third aspect is that a less thick coating favors fast extraction equilibrium. In the previous report the thickness of the coating was 7 μm, while in this study the coating had a thickness of only 0.5 μm. Since the equilibrium was reached in 10 min,

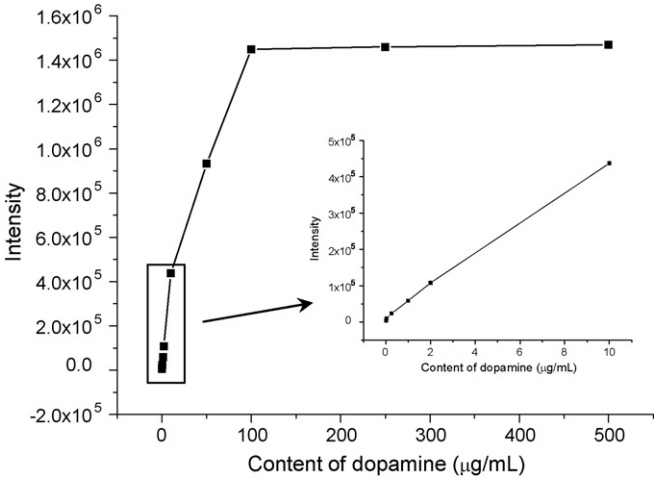


Fig. 6. Dependence of the peak area for the extracted dopamine on the concentration of dopamine in the samples. The intersection shows a close-up of the linear range.

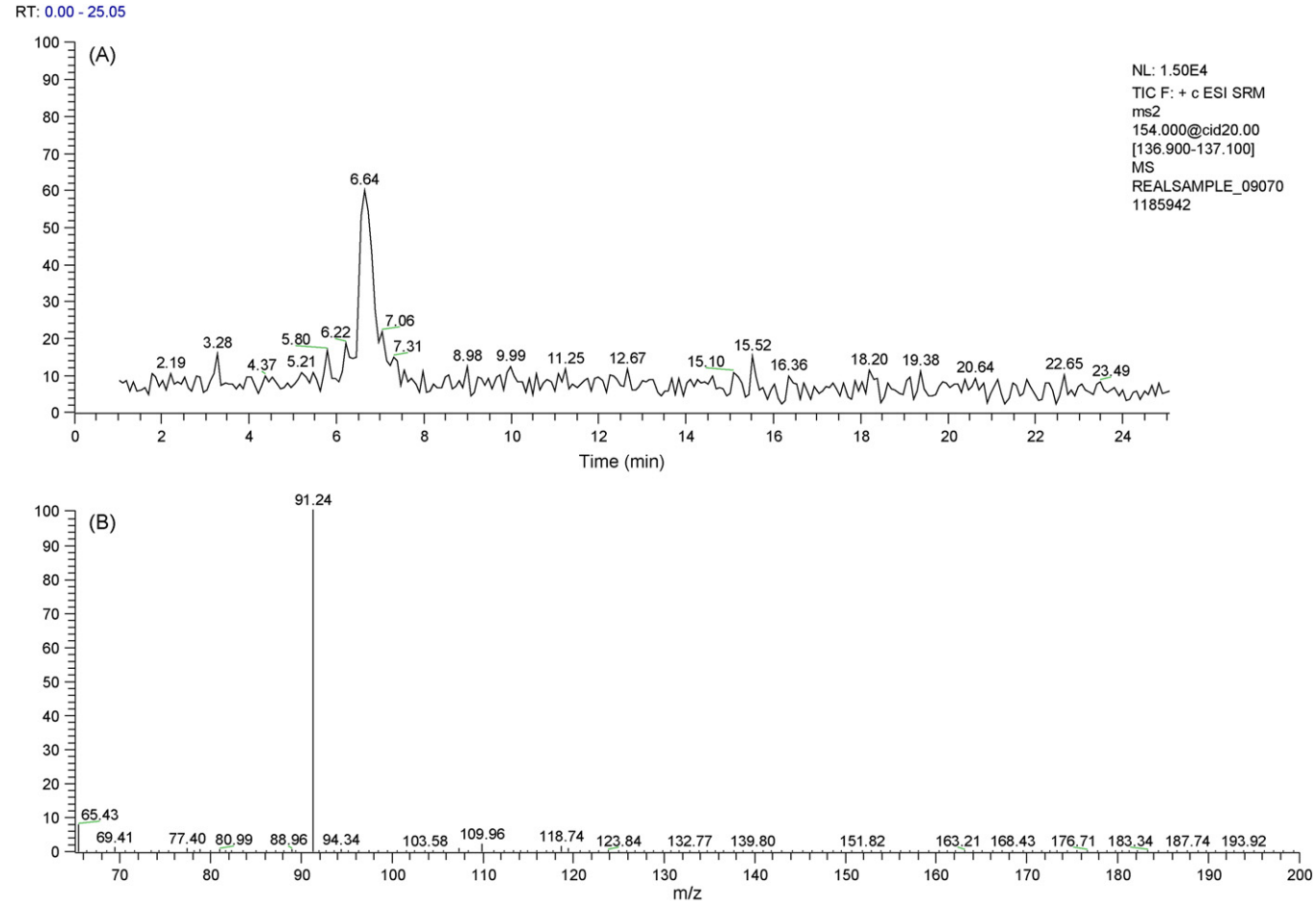


Fig. 7. A representative HPLC–MS/MS chromatogram of the extracted dopamine from urine sample. (A) Urine sample extracted by an in-tube BA-SPME capillary; extraction time: 10 min, desorption solution: 5 mM formic acid; quantitative products ion: *m*/*z* 137.0; (B) spectrum of qualitative product ion at *m*/*z* 91.0.

the extraction time was set at 10 min for remaining investigations.

3.7. Linear range and reproducibility

To investigate the extracting capability and linear range of the prepared in-tube BA-SPME, solutions containing 5 ng/mL to 500 μg/mL dopamine were prepared for extraction. Fig. 6 shows the dependence of the peak intensity found for the extracted dopamine on the concentration of dopamine present in the sample solution. Within the range of 5–10,000 ng/mL, the dependence was linear. Since the content of dopamine in urine is below 1000 ng/mL, it is better to evaluate the linear regression equation within this range. The linear regression equation within the range of 5–1000 ng/mL was $y = 50{,}700x + 8320$, with a squared correlation coefficient of 0.9989 ($n = 5$). As the dopamine concentration in the sample increased to 100 μg/mL, the in-tube BA capillary began to be saturated. Reproducibility was examined with an aqueous solution of 200 ng/mL dopamine. For intra-day reproducibility, the relative standard deviation (RSD) value for peak intensity was found to be 6.5% ($n = 6$), while the inter-day RSD was 7.6% ($n = 6$). The prepared capillary tubes were found to be able to endure for 3 months with at least 350 uses while keeping run-to-run RSD less than 10%. The limit of detection (LOD) and limit of quantification (LOQ) of the on-line in-tube BA-SPME-HPLC–ESI-MS/MS method was examined with an aqueous solution of 5 ng/mL dopamine. The LOD was determined to be 1.2 ng/mL or 7.8×10^{-9} M (3× s.d.), while the LOQ was found to be 4.0 ng/mL or 2.6×10^{-8} M (10× s.d.).

3.8. Application to real sample

Catecholamines, such as dopamine and 5-hydroxytryptamine, play a significant role in the nervous system for numerous organisms. Excessive secretion of catecholamines in human body may cause symptoms such as sustained or intermittent hypertension, sweating, tachycardia, and palpitations. Catecholamine levels in plasma and urine are independent of dietary influences and may more accurately reflect endogenous production, plasma or urinary catecholamines have been shown to be reliable biochemical indices to detect majority of catecholamine-secreting tumors, such as phaeochromocytoma [21]. In this study, urine was used as a real sample to verify the feasibility of in-tube BA-SPME established in this study.

A certain volume of urine of a healthy volunteer was added into the bottle, along with the addition of the antioxidant, and then the pH was adjusted to 9.0. The prepared solution was pumped through the in-tube SPME capillary tube. After extraction, ammonia (pH 9.5) was passed through the tube at a flow rate of 200 μL/min to remove the un-extracted dopamine, followed with on-line SPME-HPLC–MS/MS analysis. A representative chromatogram for the extracted dopamine from urine sample is shown in Fig. 7A, the product ion at *m*/*z* 137 was monitored and the peak intensity was recorded. The qualitative product ion at *m*/*z* 91.0 was monitored and the spectrum is shown in Fig. 7B. From the calibration curve of the dopamine, the concentration of dopamine in the urine sample was determined to be 131 ± 9 ($n = 3$) ng/mL. The recovery of the on-line in-tube BA-SPME-HPLC–ESI-MS/MS method was assessed

by replicate analysis ($n = 3$) of urine sample spiked with standard solution (100 ng/mL), the result is 85.6%, which is in the acceptable range.

4. Conclusions

In this study, in-tube BA-SPME was prepared and on-line coupled with HPLC–ESI-MS/MS for the specific extraction and analysis of cis-diol-containing biomolecules. On-line combination and sensitive detection were accomplished through the employment of in-tube format of BA-SPME and MS detection. The on-line hyphenation benefited from one of the inherent merits of boronate affinity, easy-to-control on/off procedure while the sensitive MS detection benefited from other two of the inherent merits of boronate affinity, specificity and MS-compatibility. The specificity excluded interfering molecules and whereas the MS-compatibility facilitated the ionization efficiency. Since a lot of important biomolecules contain 1,2- or 1,3-cis-diols, the in-tube boronate affinity SPME-HPLC coupling can be developed into a promising tool for bioanalysis. We foresee the potential applications of the on-line hyphenated method in the fields of metabolomics and glycoproteomics.

Acknowledgements

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