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Development of an ionic liquid-based ultrasonic-assisted liquid–liquid microextraction method for sensitive determination of biogenic amines: Application to the analysis of octopamine, tyramine and phenethylamine in beer samples

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ABSTRACT

A simple and efficient method, ionic liquid-based ultrasound-assisted liquid–liquid microextraction, has been developed for the determination of three biogenic amines including octopamine (OCT), tyramine (TYR) and phenethylamine (PHE). Fluorescence probe 2, 6-dimethyl-4-quinolinecarboxylic acid N-hydroxysuccinimide ester was applied for derivatization of biogenic amines and high-performance liquid chromatography coupled with fluorescence detection was used for the determination of the derivatives. The factors affecting the extraction efficiency, such as the type and volume of ionic liquid, ultrasonication time and centrifugation time have been investigated in detail. Under the optimum conditions, linearity of the method was observed in the range of 0.5–50 μg mL^{-1} for OCT and TYR, and 0.025–2.5 μg mL^{-1} for PHE, respectively, with correlation coefficients (γ) > 0.996. The limits of detection ranged from 0.25–50 ng mL^{-1} (S/N = 3). The spiked recoveries of three target compounds in beer samples were in the range of 90.2–114%. As a result, this method has been successfully applied for the sensitive determination of OCT, TYR and PHE in beer samples.

1. Introduction

Biogenic amines (BAs) are nitrogenous low molecular weight organic compounds, which have recognized biological activities [1]. High amounts of BAs are not only the markers of food spoilage but also can give rise to carcinogenic compounds [2,3]. The presence of BAs has been confirmed in many foods and beverages including cheese, sausage, fish, aged meat, soy sauces, wine and beer [4–8]. The types and levels of biogenic amines in beers are affected mainly by raw materials, brewing techniques and hygienic conditions. In view of the possible harmful effect of biogenic amines and the high amount of beer consumed, it is important to determine their concentration.

HPLC has been the most generally used technique in determination of BAs in different kinds of food due to its high selectivity and sensitivity [9–11]. However, BAs in food samples are not directly analyzed with HPLC due to their low concentrations and the complexity of the sample matrix. In general, trace determination of BAs in samples usually requires a sample preparation step prior to chromatographic analysis. Some sample preparation techniques developed for this propose include liquid–liquid extraction (LLE) [12], solid-phase extraction (SPE) [13], solid-phase microextraction (SPME) [14] and the single drop microextraction (SDME) [15]. However, LLE consumes relatively large amounts of hazardous organic solvents and a large volume of sample is often required for trace analysis. The extraction procedures listed above may be time-consuming and extraction equilibrium is not attained in a short time in most cases.

Ultrasonic radiation aids sample pre-treatment by facilitating and accelerating operations such as the extraction of organic and inorganic compounds from solid and liquid samples. Analytical applications of ultrasound, particularly sample preparation, have experienced a significant increase in the last decade [16,17]. Ultrasonic-assisted extraction methods proved to be a fast and efficient alternative to conventional extraction techniques [18,19]. Ionic liquids (ILs), which are liquids entirely composed of organic cations and inorganic or organic anions at or close to the room temperature, have the characteristics of high thermal stability, non-flammability and good solubility for inorganic and organic compounds. They have been investigated as replacements for conventional organic solvents in some extraction processes, such as LLE, liquid-phase microextraction, SPE and aqueous two-phase systems extraction [20–23]. Recently, ILs used in ultrasonic-assisted extraction methods has emerged as an attractive alternative for

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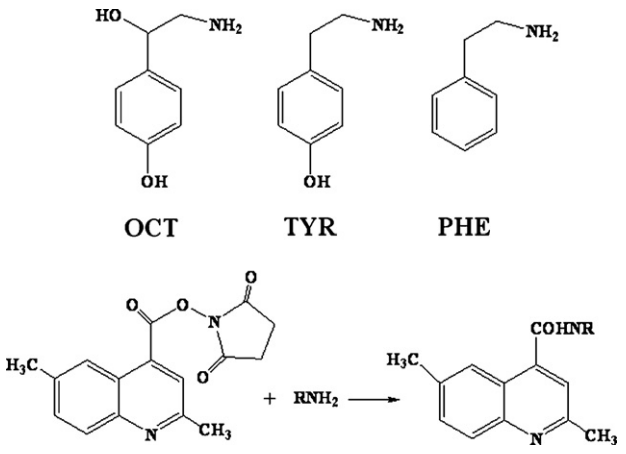

Fig. 1. The chemical structures of octopamine, tyramine and phenylethylamine, and the reaction of DMQC-OSu with amines.

sample preparation because this technique showed high recovery and enrichment factor, simplicity, rapidness and low cost [24,25], and it has been successfully used for the extraction and sensitive determination of different pollutants including herbicides, pesticides and metal ions [26–28].

Since many BAs in food show neither satisfactory absorption in the visible or ultraviolet range, nor have fluorescence properties, chemical pre- or postcolumn derivatization has become widely accepted for their determination and usually shows great sensitivity and selectivity. For the fluorescence labeling of BAs, the fluorescence labeling reagent 2, 6-dimethyl-4-quinolinecarboxylic acid N-hydroxysuccinimide ester (DMQC-OSu) not only reacts readily with primary and secondary amines with good selectivity in aqueous solution, but also provides the advantages of having few by-products and mild reaction conditions [29].

The biogenic amines octopamine (OCT), tyramine (TYR), and phenethylamine (PHE) have the similar chemical structure (Fig. 1), and are usually present at low levels in beers. The objective of this work is to present a new method for the sensitive detection of these biogenic amines in beers based on DMQC-OSu derivatization followed by ionic liquid-based ultrasound-assisted liquid–liquid microextraction (IL-UALLME) and high-performance liquid chromatography–fluorescence detection (HPLC–FL). 1-butyl-3-methylimidazolium hexafluorophosphate (C_4MIMPF_6) was selected as extractant. The variables affecting the extraction procedure were studied and the analytical figures of merit were established. The method was applied to the determination of the target analytes in beer samples.

2. Experimental

2.1. Chemicals

DMQC-OSu was synthesized according to Ref. [29]. OCT, TYR and PHE were purchased from Sigma (St. Louis, MO, USA). A stock solution of these compounds was prepared in double-distilled water. Working solutions were prepared daily by proper dilution of the stock solution with double-distilled water. C_4MIMPF_6, 1-hexyl-3-methylimidazolium hexafluorophosphate (C_6MIMPF_6) and 1-octyl-3-methylimidazolium hexafluorophosphate (C_8MIMPF_6) were purchased from Jingchun Chemical Reagent Co., Ltd. (Shanghai, China). Unless otherwise specified, the purities of all reagents were ≥99.7% and used without further purification. All solutions were prepared with double-distilled water and were stored in the refrigerator at 4 °C and filtered through 0.45 μm nylon filters (Automaticscience, Instrument Co., Ltd., Tianjin, China) before use.

2.2. Instrumentation

An Agilent 1200 Series HPLC system, which consisted of a quaternary pump, a vacuum degasser and a fluorescence detector were used. A reversed-phase Eclipse XDB-C_{18} column (150 mm × 4.6 mm i.d., 5 μm, Agilent, USA) was used for separation at ambient temperature. A manual sample injector with a 20 μL loop was used. Agilent ChemStation for HPLC system was employed to acquire and process chromatographic data. The mobile phase was a mixture of methanol–water (60/40, v/v) delivered at a flow rate of 1.0 mL min^{-1}, and the detection wavelength was set at $\lambda_{ex}/\lambda_{em}$ = 326/412 nm. A KQ-500 ultrasonic water bath (Kunshan, Jiangsu, China; power: 100 W, frequency: 40 kHz) was used in the extraction step.

2.3. Ionic liquid-based ultrasound-assisted liquid–liquid microextraction

The derivatization process of DMQC-OSu and BAs has been studied in detail previously [30]. 100 μL of mixed amine in water, 400 μL of DMQC-OSu solution (2 mM), 200 μL of H_3BO_3–$Na_2B_4O_7$ buffer (pH 8.0) and 300 μL of double-distilled water were added to a 1.5-mL conical tube and then vigorously mixed. Then the solution was incubated at 20 °C for 40 min. The chemical structure of DMQC-OSu and its reaction with BAs are shown in Fig. 1.

After the derivatization process, a volume of 30 μL of IL was added and the mixture was sonicated for 1 min. The IL was dispersed into the aqueous solution, and nearly homogenous mixture was achieved. This was then centrifuged at 1677 × *g* for 3 min and the IL was collected in the bottom of the conical test tube. The upper aqueous phase was removed with a Pasteur pipette and the volume of sedimented phase was measured using a microsyringe. The ionic liquid extract was too viscous to be injected directly into the HPLC system, thus it was diluted to 0.5 mL with methanol. The mixture was filtered through 0.22 μm nylon filters and then 20 μL of resulting solution was injected into the HPLC system.

2.4. Method performance

Calibration graph using the optimized method was obtained by analyzing spiked beer samples (n = 4 for each concentration) over a wide range for all the analytes. At least nine calibration levels were included in each calibration line. The linearity was evaluated by calibration curves constructed using linear regression of the peak area (Y) versus biogenic amines concentration (X, μg mL^{-1}).

The limit of detection (LOD) and the limit of quantitation (LOQ) were determined at concentrations where the signal/noise ratios were equal to 3 and 10, respectively.

Intra-day, and inter-day precision values were determined by means of quadruple IL-UALLME/HPLC assays of the blank beer samples spiked with analytes. Accuracy values were calculated by comparison between the biogenic amines concentrations added to the beer samples with beer biogenic amines concentrations determined by the calibration curve. In this work, the recovery was defined as the method accuracy.

3. Results and discussion

3.1. Optimization of ionic liquid-based ultrasound-assisted liquid–liquid microextraction

The effects of ultrasound on the extraction efficiency have been attributed to cavitation, generating local high temperatures and

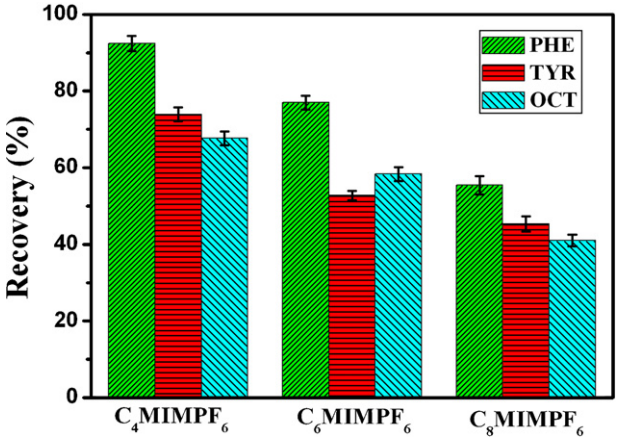

Fig. 2. Effect of type of ionic liquid on the recoveries. Analyte concentrations: C_{OCT} = 10 μg mL^{-1}, C_{TYR} = 10 μg mL^{-1}, C_{PHE} = 0.5 μg mL^{-1}. Volume of ionic liquid, 30 μL; ultrasonic agitation time, 1 min; centrifugation at 1677 × *g* for 3 min.

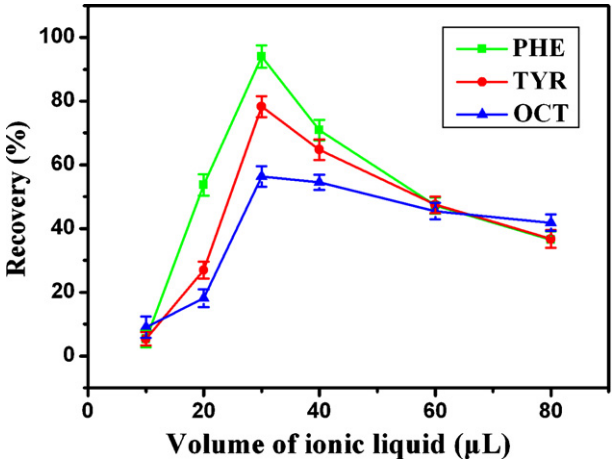

Fig. 3. Effect of volume of ionic liquid on the recoveries. Analyte concentrations: C_{OCT} = 10 μg mL^{-1}, C_{TYR} = 10 μg mL^{-1}, C_{PHE} = 0.5 μg mL^{-1}. Ultrasonic agitation time, 1 min; centrifugation at 1677 × *g* for 3 min.

to mechanical action between different interfaces [31,32]. In this way, the efficiency of analyte extraction depends on the variables that influence the cavitation process. In order to obtain the maximal extraction efficiency, parameters that may influence the enrichment performance, such as the kind of IL, temperature, position of the vessels and sonication time were investigated. Other parameters (sample pH, centrifuging time) affected the extraction efficiency were also evaluated. Eqs. (1) and (2) were used to calculate the enrichment factor and recovery.

$$\mathrm{EF} = \frac{C_{sed}}{C_0} \quad (1)$$

where EF, C_{sed} and C_0 are the enrichment factor, the analyte concentration in the sediment and the initial analyte concentration in the aqueous samples, respectively. C_{sed} was calculated from the calibration graph.

$$R\% = \frac{C_{sed} \times V_{sed}}{C_0 \times V_{aq}} \times 100 = \mathrm{EF} \times \frac{V_{sed}}{V_{aq}} \times 100 \quad (2)$$

where *R*%, V_{sed}, V_{aq}, are the extraction recovery, the volume of the sediment phase and the volume of the aqueous sample, respectively.

3.1.1. Selection of ionic liquids

The alkyl chain length on the imidazolium ring of ILs has significant influence on its physical and chemical properties, such as density, viscosity and extraction performance [33,34]. Therefore, with the same anion PF_6^-, the effect of the changes of alkyl chain length on the imidazolium ring on the extraction efficiency was investigated. Three ionic liquids C_4MIMPF_6, C_6MIMPF_6 and C_8MIMPF_6 were studied and their extraction efficiencies were compared. As shown in Fig. 2, increasing alkyl chain length from butyl to octyl decreased the extraction efficiency. It has been reported that ultrasonic radiation could accelerate various steps of the analytical process in liquid samples [35]. In ultrasound-assisted liquid-phase microextraction, ultrasonic agitation makes the extractant completely disperse in aqueous phase and form vesicles to achieve efficient extraction. In this work, the increase of alkyl chain length of ILs may lead to the enhancement of viscosity, and this may hinder the ILs completely dispersing in the water phase in 1 min of ultrasonic agitation. Therefore, C_4MIMPF_6 was chosen as extraction solvent in the following studies.

3.1.2. Extraction vessel position

The effect of placement of the extraction vessel on the extraction recovery was investigated over a range of distances between vessel and ultrasound source from 5 to 50 mm. The extraction recoveries did not change with the increase of the distance from 5 to 20 mm, and decreased from 20 to 50 mm. A distance of 10 mm was chosen in this work.

3.1.3. Extraction temperature

Temperature has a significant effect on solubility and mass transfer [36]. The effect of different temperatures on the extraction recovery was evaluated from 20 to 60 °C. The extraction recoveries increased with the increase of temperature from 10 to 30 °C, and decreased above 30 °C. The extraction temperature of 30 °C was chosen in this study.

3.1.4. Effect of the volume of ionic liquid

The volume of extraction solvent is a parameter that has been found to significantly influence the extraction performance in liquid phase microextraction [36]. To examine the effect of IL volume on the extraction efficiency, different volumes of C_4MIMPF_6 ranging from 10 to 80 μL were subjected to the same IL-UALLME procedure. The results are shown in Fig. 3. The extraction recoveries increased with the increase of volume of IL from 10 to 30 μL, and decreased above 30 μL. Therefore, 30 μL of C_4MIMPF_6 was selected.

3.1.5. Effect of sonication time

IL-UALLME is a type of equilibrium extraction, and the optimal extraction efficiency is obtained once the equilibrium is established. Hence, the effect of ultrasonic agitation time on extraction efficiency was investigated in the range of 1–8 min. The experimental results indicated that ultrasonic agitation time had no significant effects on the extraction efficiency (Fig. 4). After IL was dispersed by ultrasonic agitation to form vesicles, the surface area between the extraction solvent (C_4MIMPF_6) vesicle and the aqueous phase is large. Thus, the transfer of the analytes from aqueous phase to extraction phase was fast [38]. Therefore, IL-UALLME is a kind of fast equilibrium extraction procedure and the extraction time was very short. Herein, 1 min was enough time for the ultrasonic agitation extraction procedure.

3.1.6. Effect of pH

The sample pH might affect the extraction efficiency because it will affect the ionization of the analyte. Therefore, the effect of sample pH was optimized over the range of 3–11. The results showed that extraction recoveries of derivatives increased with the increase of pH value in the range of 3–8, and then decreased when the pH was further increased. Because pH 8 was equal to the pH of

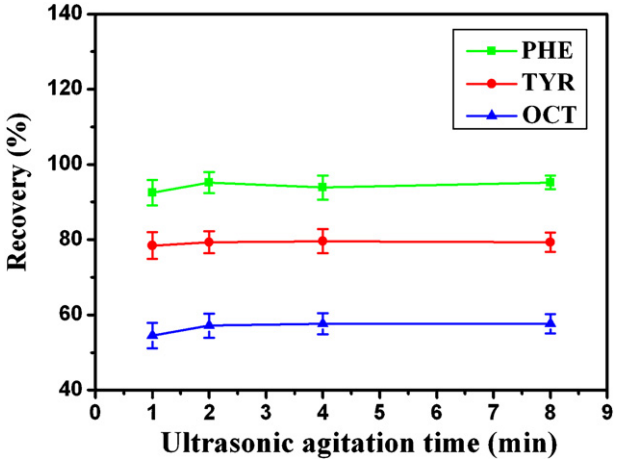

Fig. 4. Effect of ultrasonic agitation time on the recoveries. Analyte concentrations: C_{OCT} = 10 μg mL^{-1}, C_{TYR} = 10 μg mL^{-1}, C_{PHE} = 0.5 μg mL^{-1}. C_4MIMPF_6 volume, 30 μL; centrifugation at 1677 × *g* for 3 min.

H_3BO_3–$Na_2B_4O_7$ buffer that used in the derivatization experiment, the derivatization reaction solution was directly used in IL-UALLME without further pH adjustment.

3.1.7. Effect of centrifugation time

In the IL-UALLME process, centrifugation plays an important role in the separation procedure. The IL settles in the conical tube bottom during this process. Centrifugation time affects the size of the settled phase and the concentration of analyte in the extraction phase. The effect of the centrifugation time on the extraction efficiency was investigated in the range of 0.5–10 min. Similar results were achieved using centrifugation times between 3 and 10 min (Fig. 5). Thus, the lower value (3 min) was selected to speed up sample preparation.

Under selected conditions, a comparison of the chromatogram of BAs obtained from IL-UALLME–HPLC (B) and direct HPLC analysis (A) is shown in Fig. 6. No effects attributable to the ILs were observed on peak resolution, elution order and elution time; an unknown peak was obviously decreased and a dramatic peak enhancement was presented in the chromatogram obtained by IL-UALLME–HPLC. This exhibited the remarkable preconcentration ability of the IL-UALLME.

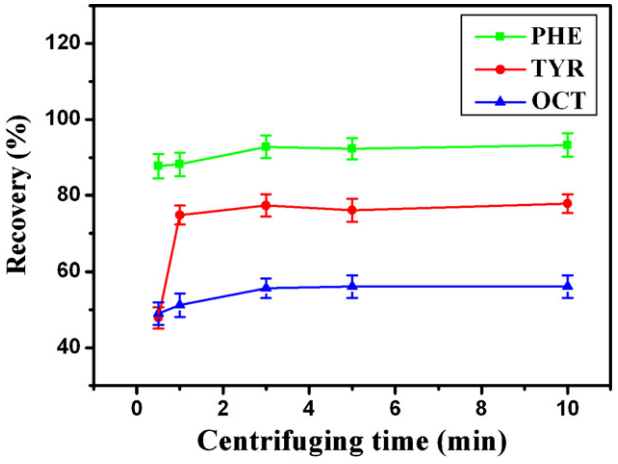

Fig. 5. Effect of time of centrifuging at 1677 × *g* on the recoveries. Analyte concentrations: C_{OCT} = 10 μg mL^{-1}, C_{TYR} = 10 μg mL^{-1}, C_{PHE} = 0.5 μg mL^{-1}. C_4MIMPF_6 volume, 30 μL; ultrasonic agitation time, 1 min.

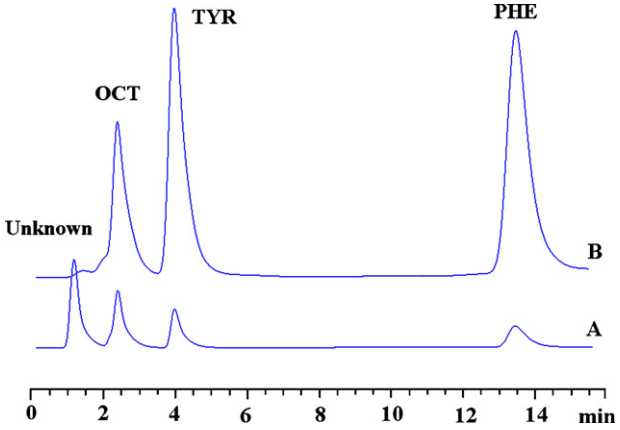

Fig. 6. Chromatograms for BA derivatives obtained by IL-UALLME–HPLC (B) and direct HPLC analysis (A). Extraction conditions: C_{OCT} = 10 μg mL^{-1}, C_{TYR} = 10 μg mL^{-1}, C_{PHE} = 0.5 μg mL^{-1}. The volume of C_4MIMPF_6 for 30 μL, ultrasonic agitation time for 1 min and 1677 × *g* centrifugation for 3 min. HPLC conditions: mobile phase, methanol/water (60/40, v/v); flow rate, 1 mL min^{-1}; detection wavelength fluorescence (326/412 nm).

3.2. Comparison of IL-UALLME with ionic liquid-based dispersive liquid–liquid microextraction (IL-DLLME)

Recently, a novel sample preparation technique called dispersive liquid phase microextraction (LPME) or dispersive liquid–liquid microextraction (DLLME) was developed by Assadi and co-workers. It is based on a ternary component solvent system like homogeneous liquid–liquid extraction (HLLE) and cloud point extraction (CPE) [39]. LPME is a simple, fast and easily controlled technique. Recently, ionic liquid-based dispersive liquid–liquid microextraction (IL-DLLME) has been explored for extraction purposes [40–42].

In the present work, for comparison, an IL-DLLME method was also developed for BAs determination according to the Ref. [30]. Acetonitrile and C_4MIMPF_6 were used as disperser solvent and extraction solvent, respectively. After the derivatization process, a solution of acetonitrile containing C_4MIMPF_6 was quickly introduced to the sample solution and then sonicated for 1 min. The cloudy solution was centrifuged for 3 min at 1677 × *g* and the dispersed fine droplets of C_4MIMPF_6 were settled to the bottom of centrifuge tube. The sedimented phase was collected and diluted with methanol to 0.5 mL. Subsequently, the extract was injected into the HPLC system for analysis. As shown in Fig. 7, the extraction efficiency decreased slightly with an increasing amount of acetonitrile. This indicated that acetonitrile was not beneficial in improving of extraction efficiency, and we conclude that IL-UALLME is preferred to IL-DLLME for BA determination in the present work.

3.3. Performance of the analytical procedure

To evaluate the proposed IL-UALLME method, the figures of merit of this method were investigated under the optimized conditions and the results are summarized in Table 1. Linearity was observed in the range of 0.5–50 μg mL^{-1} for OCT and TYR, and 0.025–2.5 μg mL^{-1} for PHE, respectively, with correlation coefficients (γ) ranging from 0.996 to 0.999. The intra-day precision ranged from 2.1 to 3.4% and the inter-day precision ranged from 3.5 to 4.2%. LODs and LOQs were in the range of 0.25–50 and 0.83–166.67 ng mL^{-1}, respectively. These results indicated that the present approach was an efficient and sensitive procedure to determine biogenic amines at trace level.

Table 1
The performance characteristics of the proposed method.

Compounds	Calibration range (μg mL^{-1})	Regression equation	γ	RSD (%, n = 4)		LOD (ng mL^{-1})	LOQ (ng mL^{-1})
				Intraday	Interday		
OCT	0.5–50	Y = 13.723X + 9.592	0.996	2.1	3.6	50	166.67
TYR	0.5–50	Y = 36.486X + 8.697	0.998	3.2	3.5	5	16.67
PHE	0.025–2.5	Y = 920.762X + 10.493	0.999	3.4	4.2	0.25	0.83

X: concentration of biogenic amines (μg mL^{-1}). Y: peak area of biogenic amines derivatives.

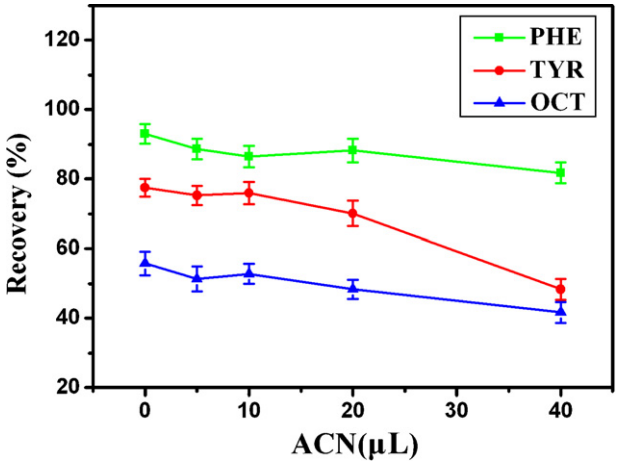

Fig. 7. Effect of volume of acetonitrile on the recoveries in IL-DLLME for the determination of biogenic amines. Analyte concentrations: C_{OCT} = 10 μg mL^{-1}, C_{TYR} = 10 μg mL^{-1}, C_{PHE} = 0.5 μg mL^{-1}. Ultrasonic agitation time, 1 min; centrifugation at 1677 × g for 3 min.

3.4. Analysis of beer samples

Under the optimized experimental conditions, the practical applicability of the recommended method was evaluated by extracting three BAs from different beer samples including Jigong hill beer, Weixue beer and Stout beer. Each sample was spiked with target species at three different concentration levels and analyzed in quadruplicate using the IL-UALLME–HPLC procedure to investigate the effect of sample matrices. The recoveries of analytes in three beer samples are listed in Table 2. The data display that the recoveries were in the range of 90.2–116%. These results indicate that the matrices of the real beer samples have little effect on the proposed IL-UALLME–HPLC method for the determination of BAs in beer samples. A typical chromatogram of a beer sample is shown in Fig. 8.

3.5. Comparison of IL-UALLME with other methods

Extraction and determination of BAs by the proposed method was compared with other methods [30,43–48] and the results are shown in Table 3. It can be seen that extraction time in the IL-

Table 2
Analytical results for the three biogenic amines in beer samples.

Samples	Biogenic amines	Added (μg mL^{-1})	Found (μg mL^{-1})	Recovery (%)	RSD (%)(n = 4)
Jigongshan beer	Octopamine	0	0		
		1	1.12	112	4.6
		10	10.02	100.2	3.4
		40	40.8	102	4.1
	Tyramine	0	2.21		4.9
		1	3.34	113	5.8
		10	11.33	91.2	5.4
		40	40.04	100.1	3.9
	Phenethylamine	0	0		
		0.005	0.0057	114	3.6
		0.05	0.0451	90.2	4.1
		0.2	0.2018	100.9	5.3
Weixue beer	Octopamine	0	0		
		1	1.12	112	6.3
		10	10.01	100.1	4.4
		40	40.08	100.2	3.9
	Tyramine	0	1.36		6.3
		1	2.52	116	6.4
		10	9.58	95.8	5.8
		40	39.56	98.9	4.1
	Phenethylamine	0	0.00		
		0.005	0.0053	106	5.5
		0.05	0.0459	91.8	3.4
		0.2	0.2002	100.1	4.6
Stout beer	Octopamine	0	0		
		1	1.06	106	6.5
		10	11.28	112.8	4.2
		40	41.84	104.6	5.6
	Tyramine	0	3.47		7.1
		1	4.52	105	5.4
		10	12.96	94.9	5.5
		40	41.76	104.4	2.9
	Phenethylamine	0	0.0095		6.8
		0.005	0.0152	114	5.2
		0.05	0.0594	99.8	4.7
		0.2	0.2062	103.1	8.2

Table 3
Comparison of IL-UALLME with other methods for the determination of biogenic amine in sample matrix.

Method	Sample volume (mL)	Extraction time (min)	Analysis time (min)[a]	LOD (ng mL^{-1})	RSD (%)
Micellar extraction-FL [43]	5	5	10	0.56–166.02	2.67–9.04
SPE-FL [44]	0.6	–	–	15–50	3.54–12.50
Cloud point extraction-UV [45]	5	5	10	15–100	–
SPME-UV [46]	5	60	75	4.43–7.34	≤3.09
Liquid-phase microextraction-UV [47]	10	30	–	10–30	3.1–6.9
SPE-electrochemical detection [48]	4	30	35	0.1	–
This work	1	1	4	0. 25–50	2.1–4.2

[a] The time of total extraction procedure including extraction time, centrifugal time, desorption time and so on.

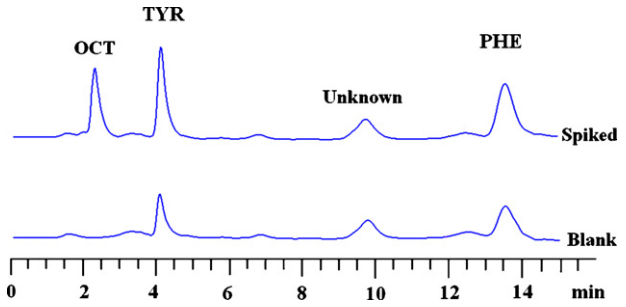

Fig. 8. The typical chromatograms of biogenic amines in spiked beer samples of stout beer. Spiked concentration: C_{OCT} = 2 μg mL^{-1}, C_{TYR} = 2 μg mL^{-1}, C_{PHE} = 0.001 μg mL^{-1}. Found concentration: C_{OCT} = 2.1 μg mL^{-1}, C_{TYR} = 1.89 μg mL^{-1}, C_{PHE} = 0.00097 μg mL^{-1}.

UALLME procedure was very short (1 min) and only 4 min were needed before HPLC analysis. The present technique provides lower limit of detection in comparison with other techniques. These results indicate that IL-UALLME–HPLC was a fast, reproducible and simple technique that could be used for the determination of BAs from the beer samples.

4. Conclusions

In this study, a green and effective method based on the ionic liquid-based ultrasound-assisted liquid–liquid microextraction was developed combined with HPLC–FL for the determination of the three BAs in beer samples. The room-temperature ionic liquid C_4MIMPF_6 was used as extraction solvent. Good repeatability and spiked recoveries were obtained for the BAs analysis. The proposed approach provided higher extraction efficiency and reduced extraction time compared to other techniques. Additionally, the use of an ionic liquid provided some advantages such as the reduction of exposure to toxic solvent, possibility of obtaining more reproducible results since evaporation of extractant was not required and obtaining a directly analyzable extract in a short single-step procedure.

Acknowledgments

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